Bayer CropScience

Document Title

Summary of the residues in or on treated products, food and feed for Prothioconazole

Data Requirements

EU Regulation 1107/2009 & EU Regulation 283/2013
Document MCA
Section 6: Residues in or on treated products, food and feed

According to the guidance document SANCO 10181/2013 for preparing dossiers for the approval of a chemical active substance

Date

2015-10-31

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[redacted]

Bayer CropScience

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Version history

Date	Data points containing amendments or additions[1] and brief description	Document identifier and version number

[1] It is suggested that applicants adopt a similar approach to showing revisions and version history as outlined in SANCO/10180/2013 Chapter 4 How to revise an Assessment Report

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CA 6 RESIDUES IN OR ON TREATED PRODUCTS, FOOD AND FEED

A dossier on prothioconazole (CAS No. 178928-70-6) was submitted February 2002 by Bayer CropScience to the EU RMS United Kingdom for agricultural use as a fungicide. Prothioconazole was included into Annex I of the Council Directive 91/414/EEC by the Commission Directive 2008/44/EC published 4 April 2008, with an entry into force by 1 August 2008.

This Supplemental Dossier contains only detailed study summaries of additional studies which were not part of the dossier during the first Annex I inclusion of prothioconazole and were, therefore, not evaluated during the first EU review of this compound. The summaries on the relevant endpoints were taken from the DAR from the first Annex I inclusion under EC 91/414 and its Addenda and supplemented with additional information (additional studies, additional references, further comments). In order to facilitate discrimination between additional and original information, the additional information is written in black letters whereas grey letters describe the original information.
All studies which have been already submitted by Bayer CropScience for the first Annex I inclusion are content of the DAR from the first Annex I inclusion under EC 91/414 and its Addenda and are included in the Baseline Dossier provided by Bayer CropScience.

Synonymous names for prothioconazole used at several locations in this Supplementary Dossier are JAU 6476 and PTZ.

CA 6.1 Storage stability of residues

The storage stability of prothioconazole-desthio has been evaluated for the EU review of prothioconazole in the Draft Assessment Report, Annex B.7.7 prepared by the RMS UK CRD. Studies demonstrate that the residue of interest prothioconazole-desthio is stable in deep frozen wheat matrices (grain, forage and straw) for greater than 540 days (report MR-282/00). A brief summary of the results is presented in Table 6.1.1.

This storage stability study was further extended until 36 months (report MR-354/01) without any indication for degradation of the compound. This supplementary data was not peer-reviewed during the evaluation of the Annex II dossier for prothioconazole, and is summarised below under KCA 6.1/01.

The storage stability for prothioconazole-desthio was also examined in canola (seed, pod and straw), spinach (leaves), sugar beet (body, leaf with root collar), tomato (fruit) and pea (pea dried) for 24 months (report MR-07/282). These data were not peer-reviewed during the evaluation of the Annex II dossier for prothioconazole, therefore they are summarised thereafter under KCA 6.1/02.

For all prothioconazole-desthio-hydroxy metabolites storage stability up to 24 months was tested in 5 matrices (oilseed rape, orange fruit, potato tuber, soybean and tomato fruit). This data was not presented in the Annex II dossier for prothioconazole and is therefore summarised under KCA 6.1/03.

Table 6.1- 1: Summary of storage stability of prothioconazole-desthio in plant

Compound	Plant matrix	Stability	Storage conditions	Reference
Prothioconazole-desthio (JAU6476-desthio)	Wheat forage Wheat grain Wheat straw	At least for 18 months (540 days)	≤ -18°C	Already evaluated KCA 6.0/01 [redacted], 2001d) Report No. MR-282/00
Prothioconazole-desthio (JAU6476-desthio)	Wheat forage Wheat grain Wheat straw	At least for 36 months (1088 to 1128 days)	-18°C	KCA 6.1/02 M-081351-02-1
Prothioconazole-desthio (JAU6476-desthio)	Canola seed Canola pod Canola straw Spinach (leaves) Sugar beet (body) Sugar beet (leaf with root collar) Tomato Field pea, dried	At least for 24 months	-18°C	KCA 6.1/02 M-258955-02-1
Prothioconazole-α-hydroxy-desthio, Prothioconazole-3-hydroxy-desthio, Prothioconazole-4-hydroxy-desthio, Prothioconazole-5-hydroxy-desthio, and Prothioconazole-6-hydroxy-desthio	Oilseed rape Orange fruit Potato tuber Soybean Tomato fruit	At least for 24 months	-18°C	KCA 6.1/03 M-405410-01-1

The new studies that were not evaluated for the EU review of prothioconazole are summarised below.

Report: KCA 6.1/01 [redacted]; 2003; M-081351-02-1
Title: 36 Months storage stability of residues of JAU 6476 and JAU 6476-desthio during frozen storage in/on wheat matrices
Report No.: MR-354/01
Document No.: M-081351-02-1
Guideline(s): EU Council Directive 91/414/EEC amended by the Commission Directive 96/68/EC
US EPA Residue Chemistry Test Guideline OPPTS 860.1380: Storage Stability Data
Guideline deviation(s): not specified
GLP/GEP: yes

As parent prothioconazole is not to be regarded as the residue of concern (relevant to MRLs and risk assessment), only storage stability results conducted with prothioconazole-desthio are presented in the following summaries.

Material and methods

A deep-freezer storage stability study was conducted with prothioconazole-desthio in/on wheat (forage, grain and straw) for 36 months. Individual aliquots of homogenised sample materials were fortified with prothioconazole-desthio at 0.50 to 1.5 mg/kg for forage, 0.10 to 0.15 mg/kg for grain, and 0.50 to 0.75 mg/kg for straw samples. The fortified samples were stored in a freezer at minus 18 °C or below for up to ca. 36 months. Control samples that had not been fortified were stored under the same conditions to allow procedural recovery determination from freshly fortified samples. Samples were analysed after nominal storage periods of 0, 30, 60, 90, 120, 180, 360, 540, 720 and 1080 days.

Three stored fortified samples (except for the day 0 samples where five samples have been spiked with prothioconazole-desthio), one stored control sample, and two stored control sample freshly fortified with prothioconazole-desthio at the same level as the stored fortified samples, were analysed for each matrix at each of the investigated storage periods.

Residues of prothioconazole-desthio were analysed according to method 00598 by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution (please refer to Section 4.2.1).

The analytical method was validated prior to analysis by running a set of two recoveries with prothioconazole-desthio at the limit of quantitation (0.01 mg/kg for grain and 0.05 mg/kg for green material and straw, respectively).

Findings

The analytical method was validated prior to analysis by running a set of recoveries at the limit of quantitation (0.01 mg/kg for grain, 0.05 mg/kg for forage and straw). In addition, during analysis of the stored samples concurrent recoveries were determined at 0.50 to 1.5 mg/kg for forage, 0.10 to 0.15 mg/kg for grain, and 0.50 to 0.75 mg/kg for straw samples. Both validation and concurrent recovery experiments shown in Table 6.1-2 were performed by spiking two control samples with prothioconazole-desthio at each storage period.

For method validation, recoveries were performed at the respective LOQ of 0.01 mg/kg for grain, 0.05 mg/kg for forage and straw. In all matrices, the recovery rates for prothioconazole-desthio were in the range of 67 to 108% (single values) leading to mean values ranging from 72% to 99%.
For concurrent recoveries, control samples were fortified at 0.50 to 1.5 mg/kg for forage, 0.10 to 0.15 mg/kg for grain, and 0.50 to 0.75 mg/kg for straw. The recovery rates for prothioconazole-desthio were in the range of 67 to 107% (single values) leading to mean values ranging from 76% to 100%. This demonstrates the accuracy of the analytical determination.
Table 6.1-3 summarises the amount of prothioconazole-desthio recovered in the stored samples after the various storage intervals. The values presented in these tables were neither corrected for the concurrent recoveries at the respective intervals nor for the recoveries at day 0, since the mean concurrent recoveries are within a range of 70% and 110%.

This storage stability study revealed that residues of prothioconazole-desthio were stable in a deep-freezer for a period of 36 months, in/on wheat (forage, grain and straw). Mean recovery rates for JAU 6476-desthio were between 86 and 117% (normalised to day 0).

Table 6.1-2: Method Validation Data

Sample material	Storage interval (days)	Mean of recovery rates from two freshly fortified samples (%)		Mean of recovery rates from two freshly fortified samples (%)	
		Levels (mg/kg)	Method Validation	Levels (mg/kg)	Concurrent Recoveries
Wheat Forage	0	0.05	87	0.50	93
	34	0.05	92	0.50	92
	57	0.05	80	0.50	93
	97	0.05	93	0.50	91
	100	0.05	93	0.50	96
	121	0.05	89	0.50	100
	169	0.05	94	0.50	100
	393	0.05	92	1.5	87
	576	0.05	89	1.5	87
	763	0.05	92	1.5	88
	1022	0.05	92	1.5	86
	1126	0.05	90	1.5	89
Wheat Grain	0	0.01	88	0.10	85
	23	0.01	88	0.10	81
	53	0.01	76	0.10	90
	92	0.01	93	0.10	92
	122	0.01	81	0.10	98
	197	0.01	99	0.10	84
	352	0.01	94	0.15	87
	533	0.01	79	0.15	86
	731	0.01	76	0.15	76
	975	0.01	80	0.15	83
	987	0.01	-	0.15	92
	1088	0.01	75	0.15	90
Wheat Straw	0	0.05	93	0.50	90
	34	0.05	89	0.50	89
	57	0.05	84	0.50	89
	96	0.05	91	0.50	91
	120	0.05	93	0.50	93
	167	0.05	72	0.50	93
	392	0.05	85	0.75	91*
	575	0.05	85	0.75	81
	762	0.05	81	0.75	96
	1016	0.05	86	0.75	89
	1128	0.05	88	0.75	90

*only one concurrent recovery

Table 6.1-3: Storage stability of prothioconazole-desthio in/on various wheat matrices for up to 36 months

Sample material	Storage interval (days)	Residue level in stored samples		
		Mean* (mg/kg)	RSD (%)	% of day 0
Wheat Forage	0	1.6	8.4	100
	34	1.7	9.8	106
	57	1.7	2.2	106
	97	1.6	2.7	96
	100	1.7	3.8	108
	121	1.7	10.4	103
	169	1.6	3.6	102
	393	1.5	3.2	93
	576	1.6	4.2	98
	763	1.5	2.3	93
	1022	1.8	4.1	113
	1126	1.9	7.5	117
Wheat Grain	0	0.23	3.5	100
	23	0.22	4.3	94
	53	0.23	3.1	101
	92	0.23**	0.6	100
	122	0.26	4.6	114
	197	0.23	13.7	98
	352	0.24	10.4	105
	535	0.22	3.2	97
	731	0.20**	19.9	86
	975	0.22	0.0	97
	987	0.21	12.9	93
	1088	0.22	6.7	94
Wheat Straw	0	1.9	2.1	100
	34	2.0	1.6	101
	57	2.0	4.4	101
	96	1.9	2.3	100
	120	2.2	5.2	114
	167	2.0	0.7	103
	392	1.9	2.2	98
	575	1.8	2.8	93
	762	2.0	2.5	101
	1016	2.0	4.6	101
	1128	1.9	3.3	99

* Mean from three analysed samples (except at day 0 where five samples have been spiked)
** Mean from two analysed samples

Conclusion

This storage stability study demonstrates that prothioconazole-desthio is stable under deep frozen storage for up to 36 months in/on wheat matrices (forage, grain and straw).

Report:	KCA 6.1/02[redacted]E; 2007; M-258955-02-1
Title:	Storage stability of prothioconazole-desthio in/on canola, spinach, sugar beet, tomato, and pea during freezer storage for 24 months
Report No.:	MR-07/282
Document No.:	M-258955-02-1
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Material and methods

A deep-freezer storage stability study was conducted with prothioconazole-desthio in/on canola (seed, pod, straw), spinach (leaves), sugar beet (body, leaf with root collar), tomato (fruit), and field pea (field pea dried). Individual aliquots of the homogenised sample materials were fortified with 0.50 mg/kg of prothioconazole-desthio which corresponds to the 50-fold limit of quantitation of the residue analytical method. The fortified samples were stored in a freezer at about minus 18 °C or below for up to ca. 24 months. Control samples that had not been fortified with prothioconazole-desthio were stored under the same conditions to allow procedural recovery determination from freshly fortified samples.
Samples were analysed after nominal storage periods of 0, 2, 4, 6, 12, and 24 months.

Three stored fortified samples (except for the day 0 samples where five samples have been spiked with prothioconazole-desthio), one stored control sample, and two stored control sample freshly fortified with prothioconazole-desthio at the same level as the stored fortified samples, were analysed for each matrix at each of the investigated storage periods. In addition, the analytical method was validated prior to analysis by running a set of two recoveries at the limit of quantitation (0.01 mg/kg).
Residues of prothioconazole-desthio were determined according to method 00647/E001 by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration, and dilution. (For details on method 00647/E001, see Section 2 Point 5.3.1).

Results and discussion

The analytical method was validated prior to analysis by running a set of recoveries at the limit of quantitation (0.01 mg/kg). In addition, during analysis of the stored samples concurrent recoveries were determined at 0.50 mg/kg. Both validation and concurrent recovery experiments shown in Table 6.1-4 were performed by spiking two control samples with prothioconazole-desthio at each storage period.

In the case of recovery experiments for method validation, recoveries at the respective LOQ were performed at 0.01 mg/kg. The recovery rates for prothioconazole-desthio were in the range of 84 to 111%. For concurrent recoveries, control samples were fortified at 0.50 mg/kg. The recovery rates for prothioconazole-desthio were in the range of 84 to 107%. This demonstrates the accuracy of the analytical determination.

Table 6.1-5 summarises the amount of prothioconazole-desthio recovered in the stored samples after the various storage intervals. The values presented in these tables were neither corrected for the concurrent recoveries at the respective intervals nor for the recoveries at day 0, since the mean concurrent recoveries are in general within a range of 70% and 110%.

This storage stability study revealed that residues of prothioconazole-desthio were stable for a deep-freezer period of 24 months, in/on canola (seed, pod, straw), spinach (leaves), sugar beet (body leaf with root collar), tomato (fruit), and field pea (field pea dried). Mean recovery rates for prothioconazole-desthio were between 94 and 107% (normalised to day 0).

Table 6.1-4: Method Validation Data (Fortification levels: 0.01 mg/kg for method validation and 0.50 mg/kg for concurrent recoveries)

Sample material	Storage interval (days)	Mean of recovery rates from two freshly fortified samples (%)	
		Method Validation	Concurrent Recoveries
Canola (Seed)	0	98	90
	64	--	88
	128	--	88
	174	88	86
	336	92	89
	735	90	85
Canola (Pod)	0	107	100
	64	--	102
	128	--	100
	174	109	99
	336	98	102
	735	102	99
Canola (Straw)	0	96	98
	64	--	100
	128	--	100
	174	99	98
	336	102	98
	735	101	97
Spinach (Leaves)	0	100	105
	64	--	102
	128	--	98
	174	101	98
	336	104	104
	735	100	98
Sugar Beet (body)	0	101	95
	63	--	96
	127	--	98
	173	98	97
	336	96	98
	734	102	96
Sugar Beet (leaf with root collar)	0	95	93
	63	--	97
	127	--	95
	173	98	101
	336	104	101
	734	102	96
Tomato (fruit)	0	99	101
	63	--	102
	127	--	99
	173	103	106
	336	101	100
	734	105	99
Field pea (dried)	0	106	95
	63	--	90
	127	--	92
	173	94	94
	336	102	100
	734	97	90

Table 6.1-5: Storage stability of prothioconazole-desthio in/on various crops for up to 24 months (Fortification levels: 0.50 mg/kg)

Sample material	Storage interval (days)	Residue level in stored samples		
		Mean* (mg/kg)	RSD (%)	% of day 0
Canola Seed	0	0.433	5.4	100
	64	0.447	2.7	103
	128	0.454	1.7	105
	174	0.425	1.7	98
	336	0.455	2.9	105
	735	0.424	0.9	98
Canola Pod	0	0.489	2.4	100
	64	0.504	0.3	103
	128	0.522	4.0	107
	174	0.493	1.1	101
	336	0.498	0.3	102
	735	0.486	0.6	99
Canola Straw	0	0.507	2.2	100
	64	0.482	2.3	95
	128	0.512	2.4	101
	174	0.480	1.1	95
	336	0.493	0.8	97
	735	0.488	1.3	96
Spinach Leaves	0	0.515	2.9	100
	64	0.498	1.7	97
	128	0.497	1.6	97
	174	0.499	1.7	97
	336	0.513	2.1	100
	735	0.485	0.5	94
Sugar Beet (body)	0	0.477	2.4	100
	63	0.487	0.5	102
	127	0.488	0.6	102
	173	0.489	1.7	103
	336	0.500	0.9	105
	734	0.477	0.8	100
Sugar Beet (with root collar)	0	0.474	3.0	100
	63	0.482	2.8	102
	127	0.478	5.1	101
	173	0.469	2.7	99
	336	0.488	1.6	103
	734	0.463	1.9	98
Tomato Fruit	0	0.506	1.0	100
	63	0.496	1.4	98
	127	0.521	1.2	103
	173	0.505	2.8	100
	336	0.513	1.6	101
	734	0.487	1.9	96
Field Pea, Dried	0	0.472	1.8	100
	63	0.447	3.3	95
	127	0.457	3.5	97
	173	0.451	2.1	96
	336	0.505	1.6	107
	734	0.465	1.8	98

* Mean from three analysed samples (except at day 0 where five samples have been spiked)

Conclusion

The results of the study demonstrate the stability of prothioconazole-desthio residues upon deep frozen storage for up to 24 months in/on canola (seed, pod, straw), spinach (leaves), sugar beet (body, leaf with root collar), tomato (fruit), and field pea (field pea dried).

Report:	KCA 6.1/03 [redacted].; 2011; M-405410-01-1
Title:	Storage stability of prothioconazole-á-hydroxy-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, and prothioconazole-6-hydroxy-desthio in/on tomato fruit, potato tuber, soybean, orange fruit and oil seed rape for 24 months
Report No.:	MR-08/024
Document No.:	M-405410-01-1
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Material and methods

Report MR-08/024 describes the stability of residues of the metabolites prothioconazole-α-hydroxy-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, and prothioconazole-6-hydroxy-desthio in fortified control samples of plant origin (tomato fruit, potato tuber, soybean, orange fruit and oil seed rape) during freezer storage for 24 months. The samples were fortified with a mixture containing all the analytes at a level of 0.10 mg/kg each (each expressed as parent equivalent).
The samples were stored in amber glass bottles at – 18 °C or below and were analysed at nominal intervals of 0, 30, 60, 90, 180, 360, 540 and 720 days.

5 g aliquots of the homogenised control materials were weighed into the bottles. These samples were fortified, resulting in levels of 0.10 mg/kg in all matrices. After fortification, the solvent was allowed to evaporate for about 15 - 30 min. In addition, untreated samples of each sample material were prepared for control and recovery experiments. Subsequently, the bottles were closed and deep-frozen until analysis. Samples were analysed in the 24 hours following their extraction.

Residues of the hydroxy metabolites of prothioconazole-desthio were determined by LC/MS/MS according to method 00979/M001 (please refer to Section 4.2.1).

Findings

The analytical method was validated prior to analysis by running a set of recoveries for method validation. In addition, during analysis of the stored samples concurrent recoveries were determined.
At each storage interval, the samples for the determination of the method performance (concurrent recoveries and method validation) were extracted and analysed concurrently with the control sample and the spiked stored samples.
In the control samples (at each sampling event at least one control sample per matrix was analysed), the residues of each analyte were always below 30% of the LOQ.

Overall recoveries for method validation as well as overall concurrent recoveries were within the acceptable range of 70-110%. Overall RSDs were below 20%.

Recoveries for Method Validation:
To demonstrate the accuracy of prothioconazole-α-hydroxy-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, and prothioconazole-6-hydroxy-desthio determination during this study, a set of two recovery experiments were performed for each plant matrix at the nominal storage intervals of 0, 30 (except tomato and soybean), 180, 360, 540 and 720 days. For this purpose, control samples were freshly fortified with a mixture containing all analytes at 0.01 mg/kg each (expressed as prothioconazole-desthio equivalents) and then analysed. The obtained recovery data for method validation are presented in Table 6.1-6 to Table 6.1-10.

Procedural (or "Concurrent") Recoveries:
During analysis of the samples concurrent recovery experiments were performed by spiking control samples with a mixture containing all analytes. Concurrent recoveries were conducted in each plant matrix at the nominal storage intervals of 30, 60, 90 (except orange and potato), 180, 360, 540 and 720 days. On day 0 (zero time analyses) samples were analysed during the analysis of storage samples. Since these samples are recovery samples, it was not necessary to include concurrent recoveries. For this purpose, stored control samples were freshly fortified with a mixture containing all analytes at 0.10 mg/kg each (expressed as prothioconazole-desthio equivalents). The freshly fortified samples were then extracted and analysed concurrently with the control and spiked samples of these actual storage intervals. The obtained concurrent recovery data are presented in Table 6.1-6 to Table 6.1-10.

Table 6.1-11 to Table 6.1-15 summarises the amount of each prothioconazole-hydroxy-desthio metabolite recovered in the stored samples after the various storage intervals. The values presented in these tables were not corrected for the concurrent recoveries at the respective intervals.
After a deep-freezer storage period of 24 months, mean recovery rates for all compounds analysed and in all plant matrices ranged between 71 and 103%. No degradation during the deep-freezer storage could be observed.
It is concluded that residues of prothioconazole-α-hydroxy-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, and prothioconazole-6-hydroxy-desthio are stable for at least 24 months under deep-freezer storage conditions in samples of plant origin (rape oil seed, orange fruit, potato tuber, tomato fruit and soybean).

Conclusion

The results of the study demonstrate the stability of residues of prothioconazole-α-hydroxy-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, and prothioconazole-6-hydroxy-desthio upon deep frozen storage at – 18 °C for up to 24 months in all tested matrices of plant origin (rape oil seed, orange fruit, potato tuber, tomato fruit and soybean).

Table 6.1- 6: Method performance for prothioconazole-α-hydroxy-desthio

Date of Extraction	Sample Material	Storage Interval [days]		Method Validation [%] FL 0.01 mg/kg*			Concurrent Recoveries [%] FL 0.10 mg/kg*		
		nominal	actual	Single Values		Mean	Single Values		Mean
2009-01-16	Rape Oil seed	0	0	96	97	97	---	---	---
2009-01-06		30	35	91	96	94	89	---	---
2009-02-02		60	62	---	---	---	86	86	86
2009-03-09		90	97	---	---	---	100	99	100
2009-06-09		180	189	106	111	109	94	97	96
2009-11-30		360	363	89	95	92	87	81	84
2010-09-06		540	643	105	102	104	104	103	104
2011-01-05		720	764	91	---	91	91	90	91
Overall and RSD [%]				**98**	**7.2**		**93**	**7.8**	
2008-12-03	Orange Fruit	0	0	92	83	88	---	---	---
2009-01-07		30	35	---	---	---	89	---	89
2009-02-03		60	61	---	---	---	89	93	91
2009-03-11		90	98	98	89	94	---	---	---
2009-06-10		180	189	101	92	97	104	95	100
2009-12-01		360	363	97	101	99	99	100	100
2010-09-08		540	644	98	100	99	106	108	107
2011-01-06		720	764	83	---	83	96	96	96
Overall and RSD [%]				**94**	**7.1**		**98**	**6.5**	
2008-12-08	Potato Tuber	0	0	95	97	96	---	---	---
2009-01-08		30	31	---	---	---	96	95	96
2009-02-06		60	60	---	---	---	93	96	95
2009-03-11		90	95	98	89	94	---	---	---
2009-06-23		180	197	90	96	93	97	93	95
2009-12-02		360	359	99	101	100	97	93	95
2010-10-07		540	668	105	97	101	97	101	99
2011-01-06		720	759	108	110	109	105	103	104
Overall and RSD [%]				**99**	**6.5**		**97**	**4.0**	
2008-12-09	Tomato Fruit	0	0	117	114	116	---	---	---
2009-01-13		30	35	---	---	---	91	94	93
2009-02-09		60	62	---	---	---	110	95	103
2009-03-13		90	94	---	---	---	106	104	105
2009-06-24		180	197	94	99	97	99	98	99
2009-12-03		360	359	83	98	91	92	100	96
2010-10-07		540	667	96	98	97	92	103	98
2010-01-10		720	762	108	106	107	104	104	104
Overall and RSD [%]				**101**	**10.0**		**99**	**6.0**	
2008-12-10	Soybean	0	0	105	98	102	---	---	---
2009-01-12		30	33	---	---	---	99	98	99
2009-02-09		60	61	---	---	---	94	96	95
2009-03-13		90	93	---	---	---	97	98	98
2009-06-25		180	197	101	96	99	101	101	101
2009-12-09		360	364	105	119	112	120	117	119
2010-10-07		540	666	87	99	93	95	100	98
2011-01-10		720	761	92	101	97	92	91	92
Overall and RSD [%]				**100**	**8.6**		**100**	**8.5**	

***** FL: Fortification level expressed as prothioconazole-desthio equivalents

Table 6.1- 7: Method performance for prothioconazole-3-hydroxy-desthio

Date of Extraction	Sample Material	Storage Interval [days]		Method Validation [%] FL 0.01 mg/kg*			Concurrent Recoveries [%] FL 0.10 mg/kg*		
		nominal	actual	Single Values		Mean	Single Values		Mean
2009-01-16	Rape Oil seed	0	0	99	102	101	---	---	---
2009-01-06		30	35	93	96	94	87	---	87
2009-02-02		60	62	---	---	---	88	88	88
2009-03-09		90	97	---	---	---	102	100	101
2009-06-09		180	189	91	98	95	92	98	95
2009-11-30		360	363	95	92	94	85	80	83
2010-09-06		540	643	101	97	99	105	105	105
2011-01-05		720	764	75		75	85	82	84
Overall and RSD [%]				**94**	**7.8**		**92**	**9.6**	
2008-12-03	Orange Fruit	0	0	78	79	79	---	---	---
2009-01-07		30	35	---	---	---	90	---	90
2009-02-03		60	61	---	---	---	90	95	93
2009-03-11		90	98	95	87	91	---	---	---
2009-06-10		180	189	99	92	96	100	98	99
2009-12-01		360	363	89	99	94	95	93	94
2010-09-08		540	644	101	100	101	109	111	110
2011-01-06		720	764	72	---	72	90	91	91
Overall and RSD [%]				**90**	**11.2**		**97**	**7.7**	
2008-12-08	Potato Tuber	0	0	101	97	99	---	---	---
2009-01-08		30	31	---	---	---	99	100	100
2009-02-06		60	60	---	---	---	97	99	98
2009-03-11		90	95	98	90	94	---	---	---
2009-06-23		180	197	96	96	96	97	93	95
2009-12-02		360	359	98	99	99	96	95	96
2010-10-07		540	668	115	104	110	112	116	114
2011-01-06		720	759	94	95	95	103	99	101
Overall and RSD [%]				**99**	**6.3**		**101**	**6.8**	
2008-12-09	Tomato Fruit	0	0	96	96	96	---	---	---
2009-01-13		30	35	---	---	---	92	98	95
2009-02-09		60	62	---	---	---	111	98	105
2009-03-13		90	94	---	---	---	108	105	107
2009-06-24		180	197	95	99	97	99	98	99
2009-12-03		360	359	84	92	88	103	102	103
2010-10-07		540	667	100	105	103	106	115	111
2011-01-10		720	762	95	93	94	101	100	101
Overall and RSD [%]				**96**	**5.8**		**103**	**5.9**	
2008-12-10	Soybean	0	0	86	75	81	---	---	---
2009-01-12		30	33	---	---	---	102	103	103
2009-02-09		60	61	---	---	---	98	97	98
2009-03-13		90	93	---	---	---	96	97	97
2009-06-25		180	197	102	100	101	98	101	100
2009-12-09		360	364	106	115	111	110	110	110
2010-10-07		540	666	96	108	102	108	113	111
2011-01-10		720	761	81	84	83	90	90	90
Overall and RSD [%]				**95**	**13.8**		**101**	**7.1**	

* FL: Fortification level expressed as prothioconazole-desthio equivalents

Table 6.1- 8: Method performance for prothioconazole-4-hydroxy-desthio

Date of Extraction	Sample Material	Storage Interval [days]		Method Validation [%] FL 0.01 mg/kg*			Concurrent Recoveries [%] FL 0.10 mg/kg*		
		nominal	actual	Single Values		Mean	Single Values		Mean
2009-01-16	Rape Oil seed	0	0	94	95	95	---	---	---
2009-01-06		30	35	89	93	91	83	---	83
2009-02-02		60	62	---	---	---	80	82	81
2009-03-09		90	97	---	---	---	100	100	100
2009-06-09		180	189	94	100	97	92	98	95
2009-11-30		360	362	88	84	86	80	76	78
2010-09-06		540	643	90	102	96	102	98	100
2011-01-05		720	764	82	---	82	85	82	84
Overall and RSD [%]				**92**	**6.7**		**89**	**10.6**	
2008-12-03	Orange Fruit	0	0	73	74	74	---	---	---
2009-01-07		30	35	---	---	---	87	---	---
2009-02-03		60	61	---	---	---	85	89	87
2009-03-11		90	98	93	84	89	---	---	---
2009-06-10		180	189	99	92	96	99	96	98
2009-12-01		360	363	96	98	97	90	93	92
2010-09-08		540	644	101	98	100	103	106	105
2011-01-06		720	764	73	---	73	89	93	91
Overall and RSD [%]				**89**	**12.5**		**94**	**7.2**	
2008-12-08	Potato Tuber	0	0	94	91	93	---	---	---
2009-01-08		30	31	---	---	---	95	97	96
2009-02-06		60	60	---	---	---	94	93	94
2009-03-11		90	95	97	91	94	---	---	---
2009-06-23		180	197	93	98	96	97	93	95
2009-12-02		360	359	93	99	96	91	93	92
2010-10-07		540	668	99	98	99	96	100	98
2011-01-06		720	759	106	105	106	102	101	102
Overall and RSD [%]				**97**	**5.1**		**96**	**3.7**	
2008-12-09	Tomato Fruit	0	0	95	97	96	---	---	---
2009-01-13		30	35	---	---	---	90	93	92
2009-02-09		60	62	---	---	---	104	93	99
2009-03-13		90	94	---	---	---	106	102	104
2009-06-24		180	197	92	97	95	99	98	99
2009-12-03		360	359	82	92	87	90	96	93
2010-10-07		540	667	88	100	94	89	98	94
2011-01-10		720	762	106	102	104	99	97	98
Overall and RSD [%]				**95**	**7.3**		**97**	**5.4**	
2008-12-10	Soybean	0	0	89	78	84	---	---	---
2009-01-12		30	33	---	---	---	98	97	98
2009-02-09		60	61	---	---	---	91	94	93
2009-03-13		90	93	---	---	---	94	94	94
2009-06-25		180	197	102	95	99	95	97	96
2009-12-09		360	364	106	107	107	113	115	114
2010-10-07		540	666	91	96	94	96	99	98
2011-01-10		720	761	89	94	92	89	88	89
Overall and RSD [%]				**95**	**9.3**		**97**	**8.1**	

* FL: Fortification level expressed as prothioconazole-desthio equivalents

Table 6.1- 9: Method performance for prothioconazole-5-hydroxy-desthio

Date of Extraction	Sample Material	Storage Interval [days]		Method Validation [%] FL 0.01 mg/kg*			Concurrent Recoveries [%] FL 0.10 mg/kg*		
		nominal	actual	Single Values		Mean	Single Values		Mean
2009-01-16	Rape Oil seed	0	0	99	98	99	---	---	---
2009-01-06		30	35	87	92	90	80	---	80
2009-02-02		60	62	---	---	---	82	84	83
2009-03-09		90	97	---	---	---	100	100	100
2009-06-09		180	189	89	102	96	92	98	95
2009-11-30		360	363	89	90	90	76	75	76
2010-09-06		540	643	91	99	95	102	102	102
2011-01-05		720	764	83	---	83	84	83	84
Overall and RSD [%]				**93**	**6.5**		**89**	**11.5**	
2008-12-03	Orange Fruit	0	0	76	71	74	---	---	---
2009-01-07		30	35	---	---	---	87	---	---
2009-02-03		60	61	---	---	---	86	92	89
2009-03-11		90	98	96	86	91	---	---	---
2009-06-10		180	189	89	87	88	99	97	98
2009-12-01		360	363	92	98	95	97	90	94
2010-09-08		540	644	100	98	99	104	107	106
2011-01-06		720	764	73	---	73	88	89	89
Overall and RSD [%]				**88**	**11.9**		**94**	**7.6**	
2008-12-08	Potato Tuber	0	0	95	91	93	---	---	---
2009-01-08		30	31	---	---	---	97	97	97
2009-02-06		60	60	---	---	---	93	95	94
2009-03-11		90	95	97	90	94	---	---	---
2009-06-23		180	197	95	104	100	97	93	95
2009-12-02		360	359	92	98	95	94	93	94
2010-10-07		540	668	106	98	102	96	101	99
2011-01-10		720	759	98	99	99	99	97	98
Overall and RSD [%]				**97**	**5.0**		**96**	**2.6**	
2008-12-09	Tomato Fruit	0	0	94	99	97	---	---	---
2009-01-13		30	33	---	---	---	89	92	91
2009-02-09		60	62	---	---	---	106	95	101
2009-03-13		90	94	---	---	---	104	103	104
2009-06-24		180	197	94	97	96	99	98	99
2009-12-03		360	359	84	97	91	100	99	100
2010-10-07		540	667	91	98	95	90	100	95
2011-01-10		720	762	100	100	100	97	93	95
Overall and RSD [%]				**95**	**5.2**		**98**	**5.3**	
2008-12-10	Soybean	0	0	92	82	87	---	---	---
2009-01-12		30	33	---	---	---	97	98	98
2009-02-09		60	61	---	---	---	93	96	95
2009-03-13		90	93	---	---	---	94	94	94
2009-06-25		180	197	101	94	98	100	100	100
2009-12-09		360	364	108	107	108	106	110	108
2010-10-07		540	666	86	93	90	96	99	98
2011-01-10		720	761	89	90	90	85	87	86
Overall and RSD [%]				**94**	**9.1**		**97**	**6.7**	

* FL: Fortification level expressed as prothioconazole-desthio equivalents

Table 6.1- 10: Method performance for prothioconazole-6-hydroxy-desthio

Date of Extraction	Sample Material	Storage Interval [days]		Method Validation [%] FL 0.01 mg/kg*			Concurrent Recoveries [%] FL 0.10 mg/kg*		
		nominal	actual	Single Values		Mean	Single Values		Mean
2009-01-16	Rape Oil seed	0	0	84	88	86	---	---	---
2009-01-06		30	35	77	84	81	71	---	71
2009-02-02		60	62	---	---	---	73	80	77
2009-03-09		90	97	---	---	---	102	101	102
2009-06-09		180	189	86	87	87	92	98	95
2009-11-30		360	363	83	80	82	81	68	75
2010-09-06		540	643	87	85	86	92	88	90
2011-01-05		720	764	70	---	70	74	75	75
Overall and RSD [%]				**83**	**6.5**		**84**	**14.1**	
2008-12-03	Orange Fruit	0	0	88	85	87	---	---	---
2009-01-07		30	35	---	---	---	77	---	77
2009-02-03		60	61	---	---	---	84	87	86
2009-03-11		90	98	89	82	86	---	---	---
2009-06-10		180	189	93	97	95	90	92	91
2009-12-01		360	363	82	93	88	86	85	86
2010-09-08		540	644	93	92	93	98	101	100
2011-01-06		720	764	62	---	62	75	76	76
Overall and RSD [%]				**87**	**11.0**		**86**	**9.9**	
2008-12-08	Potato Tuber	0	0	108	108	108	---	---	---
2009-01-08		30	31	---	---	---	98	98	98
2009-02-06		60	60	---	---	---	97	96	97
2009-03-11		90	95	92	86	89	---	---	---
2009-06-23		180	197	92	98	95	97	93	95
2009-12-02		360	359	100	100	100	95	99	97
2010-10-07		540	668	99	89	94	87	99	93
2011-01-06		720	759	93	94	94	92	91	92
Overall and RSD [%]				**97**	**7.1**		**95**	**3.9**	
2008-12-09	Tomato Fruit	0	0	96	102	99	---	---	---
2009-01-13		30	35	---	---	---	84	92	88
2009-02-09		60	62	---	---	---	105	93	99
2009-03-13		90	94	---	---	---	103	102	103
2009-06-24		180	197	93	98	96	99	98	99
2009-12-03		360	359	76	88	82	96	101	99
2010-10-07		540	667	94	89	92	86	93	90
2011-01-10		720	762	89	87	88	87	85	86
Overall and RSD [%]				**91**	**7.9**		**95**	**7.5**	
2008-12-10	Soybean	0	0	91	81	86	---	---	---
2009-01-12		30	33	---	---	---	98	97	98
2009-02-09		60	61	---	---	---	89	92	91
2009-03-13		90	93	---	---	---	95	95	95
2009-06-25		180	197	100	94	97	103	103	103
2009-12-09		360	364	100	107	104	105	106	106
2010-10-07		540	666	80	87	84	89	91	90
2011-01-10		720	761	80	74	77	82	84	83
Overall and RSD [%]				**89**	**12.1**		**95**	**8.0**	

* FL: Fortification level expressed as prothioconazole-desthio equivalents

Table 6.1- 11: Storage stability of prothioconazole-α-hydroxy-desthio in/on various matrices for up to 24 months

Date of Extraction	Sample Material	Storage Interval [days]		Stored Samples FL 0.10 mg/kg*					Mean [%]	RSD [%]
		nominal	actual	Single Values [%]						
2009-01-16	Rape Oil seed	0	0	91	96	98	97	98	96	3.0
2009-01-06		30	35	88	91	95	---	---	91	3.8
2009-02-02		60	62	87	88	99	---	---	91	7.3
2009-03-09		90	97	102	100	104	---	---	102	2.0
2009-06-09		180	189	97	97	97	---	---	97	1.0
2009-11-30		360	363	89	82	78	---	---	83	6.7
2010-09-06		540	643	102	98	98	---	---	99	2.1
2011-01-05		720	764	92	95	90	---	---	92	2.7
Overall									**94**	**6.6**
2008-12-03	Orange Fruit	0	0	80	84	78	83	83	82	3.1
2009-01-07		30	35	93	98	92	---	---	94	3.4
2009-02-03		60	61	91	92	97	---	---	93	3.4
2009-03-11		90	98	102	102	101	---	---	102	0.6
2009-06-10		180	189	104	101	99	---	---	101	2.5
2009-12-01		360	363	94	93	98	---	---	95	2.8
2010-09-08		540	644	99	98	101	---	---	99	1.5
2011-01-06		720	764	108	93	98	---	---	100	7.7
Overall									**95**	**8.2**
2008-12-08	Potato Tuber	0	0	92	90	88	91	89	90	1.8
2009-01-08		30	31	94	97	---	---	---	96	2.2
2009-02-06		60	60	95	93	93	---	---	94	1.2
2009-03-11		90	95	99	102	99	---	---	100	1.7
2009-06-23		180	197	99	89	93	---	---	94	5.4
2009-12-02		360	359	79	78	80	---	---	79	1.3
2010-10-07		540	668	92	94	98	---	---	95	3.2
2011-01-06		720	759	99	98	95	---	---	97	2.1
Overall									**93**	**6.8**
2008-12-09	Tomato Fruit	0	0	97	96	92	86	89	92	5.0
2009-01-13		30	35	92	91	100	---	---	94	5.2
2009-02-09		60	62	96	97	96	---	---	96	0.6
2009-03-13		90	94	103	103	101	---	---	102	1.1
2009-06-24		180	197	101	101	98	---	---	100	1.7
2009-12-03		360	359	94	92	89	---	---	92	2.7
2010-10-07		540	667	102	101	99	---	---	101	1.5
2011-01-10		720	762	93	95	100	---	---	96	3.8
Overall									**96**	**4.9**
2008-12-10	Soybean	0	0	91	93	74	67	74	80	14.4
2009-01-12		30	33	101	95	99	---	---	98	3.1
2009-02-09		60	61	90	96	95	---	---	94	3.4
2009-03-13		90	93	98	102	103	---	---	101	2.6
2009-06-25		180	197	99	101	100	---	---	100	1.0
2009-12-09		360	364	114	109	99	---	---	107	7.1
2010-10-07		540	666	98	99	95	---	---	97	2.1
2011-01-10		720	761	74	83	88	---	---	82	8.7
Overall									**94**	**12.0**

* FL: Fortification level expressed as prothioconazole-desthio equivalents; RSD: relative standard deviation

Table 6.1- 12: Storage stability of prothioconazole-3-hydroxy-desthio in/on various matrices for up to 24 months

Date of Extraction	Sample Material	Storage Interval [days]		Stored Samples FL 0.10 mg/kg*					Mean [%]	RSD [%]
		nominal	actual	Single Values [%]						
2009-01-16	Rape Oil seed	0	0	96	98	100	99	99	98	1.5
2009-01-06		30	35	90	92	93	---	---	92	1.7
2009-02-02		60	62	87	90	102	---	---	93	8.5
2009-03-09		90	97	101	102	104	---	---	102	1.5
2009-06-09		180	189	97	94	98	---	---	96	2.2
2009-11-30		360	363	82	73	76	---	---	77	6.0
2010-09-06		540	643	104	103	100	---	---	102	2.0
2011-01-05		720	764	83	79	79	---	---	80	2.9
Overall									**93**	**10.0**
2008-12-03	Orange Fruit	0	0	71	73	69	75	73	72	3.2
2009-01-07		30	35	91	100	91	---	---	94	5.8
2009-02-03		60	61	92	95	100	---	---	96	4.2
2009-03-11		90	98	100	101	101	---	---	101	0.6
2009-06-10		180	189	102	102	96	---	---	100	3.5
2009-12-01		360	363	81	86	85	---	---	84	3.1
2010-09-08		540	644	103	102	105	---	---	103	1.5
2011-01-06		720	764	107	92	96	---	---	98	7.9
Overall									**92**	**12.7**
2008-12-08	Potato Tuber	0	0	93	92	89	92	90	91	1.8
2009-01-08		30	31	---	96	101	---	---	99	3.6
2009-02-06		60	60	96	97	95	---	---	96	1.0
2009-03-11		90	95	99	102	100	---	---	100	1.5
2009-06-23		180	197	101	94	96	---	---	97	3.7
2009-12-02		360	359	79	79	80	---	---	79	0.7
2010-10-07		540	668	103	104	110	---	---	106	3.6
2011-01-06		720	759	92	92	88	---	---	91	2.5
Overall									**94**	**8.1**
2008-12-09	Tomato Fruit	0	0	85	82	79	74	75	79	5.9
2009-01-13		30	35	95	93	103	---	---	97	5.5
2009-02-09		60	62	100	100	101	---	---	100	0.6
2009-03-13		90	94	102	101	101	---	---	101	0.6
2009-06-24		180	197	101	100	99	---	---	100	1.0
2009-12-03		360	359	86	84	80	---	---	83	3.7
2010-10-07		540	662	116	116	112	---	---	115	2.0
2011-01-10		720	762	93	91	100	---	---	95	5.0
Overall									**95**	**12.2**
2008-12-10	Soybean	0	0	77	77	64	59	65	68	11.9
2009-01-12		30	33	103	97	101	---	---	100	3.0
2009-02-09		60	61	96	105	100	---	---	100	4.5
2009-03-13		90	93	96	108	101	---	---	102	5.9
2009-06-25		180	197	102	101	101	---	---	101	0.6
2009-12-09		360	364	109	108	115	---	---	111	3.4
2010-10-07		540	666	109	112	106	---	---	109	2.8
2011-01-10		720	761	73	81	87	---	---	80	8.7
Overall									**94**	**17.0**

* FL: Fortification level expressed as prothioconazole-desthio equivalents; RSD: relative standard deviation

Table 6.1-13: Storage stability of prothioconazole-4-hydroxy-desthio in/on various matrices for up to 24 months

Date of Extraction	Sample Material	Storage Interval [days]		Stored Samples FL 0.10 mg/kg*					Mean [%]	RSD [%]
		nominal	actual	Single Values [%]						
2009-01-16	Rape Oil seed	0	0	94	95	95	95	95	95	0.5
2009-01-06		30	35	84	86	86	---	---	85	1.4
2009-02-02		60	62	79	84	90	---	---	84	6.5
2009-03-09		90	97	93	93	96	---	---	94	1.8
2009-06-09		180	189	84	86	89	---	---	86	2.9
2009-11-30		360	363	85	81	80	---	---	82	3.2
2010-09-06		540	643	97	96	92	---	---	95	2.8
2011-01-05		720	764	85	90	77	---	---	84	7.8
Overall									**89**	**6.7**
2008-12-03	Orange Fruit	0	0	68	70	64	71	71	69	4.3
2009-01-07		30	35	88	96	89	---	---	91	4.8
2009-02-03		60	61	89	91	94	---	---	91	2.8
2009-03-11		90	98	94	96	95	---	---	95	1.1
2009-06-10		180	189	93	92	85	---	---	90	4.8
2009-12-01		360	363	92	93	90	---	---	92	1.7
2010-09-08		540	644	96	96	99	---	---	97	1.8
2011-01-06		720	764	105	88	117	---	---	103	14.1
Overall									**89**	**13.4**
2008-12-08	Potato Tuber	0	0	86	85	81	86	83	84	2.6
2009-01-08		30	31	91	92	95	---	---	94	2.3
2009-02-06		60	60	91	91	89	---	---	90	1.3
2009-03-11		90	95	93	95	94	---	---	94	1.1
2009-06-23		180	197	94	84	87	---	---	88	5.8
2009-12-02		360	359	81	82	76	---	---	80	4.0
2010-10-07		540	668	91	91	96	---	---	93	3.1
2011-01-06		720	759	90	91	86	---	---	89	3.0
Overall									**88**	**5.9**
2008-12-09	Tomato Fruit	0	0	84	83	79	76	76	80	4.8
2009-01-13		30	35	90	89	98	---	---	92	5.3
2009-02-09		60	62	96	97	96	---	---	96	0.6
2009-03-13		90	94	97	95	96	---	---	96	1.0
2009-06-24		180	197	93	90	91	---	---	91	1.7
2009-12-03		360	359	94	96	92	---	---	94	2.1
2010-10-07		540	667	100	101	98	---	---	100	1.5
2011-01-10		720	762	92	88	98	---	---	93	5.4
Overall									**92**	**7.7**
2008-12-10	Soybean	0	0	78	78	65	59	63	69	12.9
2009-01-12		30	33	100	95	99	---	---	98	2.7
2009-02-09		60	61	91	97	95	---	---	94	3.2
2009-03-13		90	93	90	100	96	---	---	95	5.3
2009-06-25		180	197	93	96	93	---	---	94	1.8
2009-12-09		360	364	117	114	111	---	---	114	2.6
2010-10-07		540	666	85	91	82	---	---	86	5.3
2011-01-10		720	761	71	82	85	---	---	79	9.3
Overall									**89**	**16.4**

* FL: Fortification level expressed as prothioconazole-desthio equivalents; RSD: relative standard deviation

Table 6.1-14: Storage stability of prothioconazole-5-hydroxy-desthio in/on various matrices for up to 24 months

Date of Extraction	Sample Material	Storage Interval [days]		Stored Samples FL 0.10 mg/kg*					Mean [%]	RSD [%]
		nominal	actual	Single Values [%]						
2009-01-16	Rape Oil seed	0	0	93	95	98	97	95	96	2.0
2009-01-06		30	35	83	87	84	---	---	85	2.5
2009-02-02		60	62	85	85	98	---	---	89	8.4
2009-03-09		90	97	100	101	104	---	---	102	2.0
2009-06-09		180	189	97	95	99	---	---	97	2.1
2009-11-30		360	363	86	80	76	---	---	81	6.2
2010-09-06		540	643	101	98	96	---	---	98	2.6
2011-01-05		720	764	86	88	76	---	---	84	8.1
Overall									**92**	**8.8**
2008-12-03	Orange Fruit	0	0	68	68	65	72	70	69	3.8
2009-01-07		30	35	89	96	87	---	---	91	5.2
2009-02-03		60	61	90	92	95	---	---	92	2.7
2009-03-11		90	98	102	101	103	---	---	102	1.0
2009-06-10		180	189	104	102	99	---	---	102	2.5
2009-12-01		360	363	90	94	93	---	---	92	2.3
2010-09-08		540	644	99	97	101	---	---	99	2.0
2011-01-06		720	764	101	84	89	---	---	91	9.6
Overall									**90**	**13.4**
2008-12-08	Potato Tuber	0	0	89	86	84	86	85	86	2.2
2009-01-08		30	31	---	93	97	---	---	95	3.0
2009-02-06		60	60	94	93	92	---	---	93	1.1
2009-03-11		90	95	102	104	102	---	---	103	1.1
2009-06-23		180	197	103	98	101	---	---	101	2.5
2009-12-02		360	359	86	85	87	---	---	86	1.2
2010-10-07		540	668	92	94	98	---	---	95	3.2
2011-01-06		720	759	92	96	91	---	---	93	2.8
Overall									**93**	**6.7**
2008-12-09	Tomato Fruit	0	0	85	83	82	78	79	81	3.5
2009-01-13		30	35	92	90	99	---	---	94	5.0
2009-02-09		60	62	96	97	97	---	---	97	0.6
2009-03-13		90	94	104	103	103	---	---	103	0.6
2009-06-24		180	197	104	101	100	---	---	102	2.0
2009-12-03		360	359	92	89	91	---	---	91	1.7
2010-10-07		540	667	100	102	97	---	---	100	2.5
2011-01-10		720	762	90	83	95	---	---	89	6.7
Overall									**94**	**8.6**
2008-12-10	Soybean	0	0	81	79	64	58	65	69	14.5
2009-01-12		30	33	100	95	99	---	---	98	2.7
2009-02-09		60	61	91	95	96	---	---	94	2.8
2009-03-13		90	93	98	104	102	---	---	101	3.0
2009-06-25		180	197	100	101	101	---	---	101	0.6
2009-12-09		360	364	111	105	108	---	---	108	2.8
2010-10-07		540	666	97	99	93	---	---	96	3.2
2011-01-10		720	761	70	81	86	---	---	79	10.4
Overall									**92**	**15.6**

* FL: Fortification level expressed as prothioconazole-desthio equivalents; RSD: relative standard deviation

Table 6.1-15: Storage stability of prothioconazole-6-hydroxy-desthio for up to 24 months

Date of Extraction	Sample Material	Interval [days]		Stored Samples FL 0.10 mg/kg*					Mean	RSD
		nominal	actual	Single Values [%]					[%]	[%]
2009-01-16	Rape Oil seed	0	0	82	86	88	89	87	86	3.1
2009-01-06		30	35	73	77	70	---	---	73	4.8
2009-02-02		60	62	73	72	92	---	---	79	14.3
2009-03-09		90	97	88	91	91	---	---	90	1.9
2009-06-09		180	189	87	86	93	---	---	89	4.0
2009-11-30		360	363	79	68	74	---	---	74	7.5
2010-09-06		540	643	94	92	85	---	---	90	5.2
2011-01-05		720	764	70	80	64	---	---	71	11.3
Overall									**82**	**10.9**
2008-12-03	Orange Fruit	0	0	86	91	86	95	93	90	4.5
2009-01-07		30	35	83	89	79	---	---	84	6.0
2009-02-03		60	61	82	89	92	---	---	88	5.9
2009-03-11		90	98	98	98	96	---	---	97	1.2
2009-06-10		180	189	98	99	94	---	---	97	2.7
2009-12-01		360	363	81	84	87	---	---	84	3.6
2010-09-08		540	644	95	95	99	---	---	96	2.4
2011-01-06		720	764	94	78	81	---	---	84	10.1
Overall									**90**	**7.5**
2008-12-08	Potato Tuber	0	0	109	109	105	106	105	107	1.9
2009-01-08		30	31	97	96	97	---	---	97	0.7
2009-02-06		60	60	93	96	93	---	---	94	1.8
2009-03-11		90	95	98	101	100	---	---	100	1.5
2009-06-23		180	197	103	94	100	---	---	99	4.6
2009-12-02		360	359	86	84	95	---	---	88	6.6
2010-10-07		540	668	87	89	93	---	---	90	3.4
2011-01-06		720	759	89	92	86	---	---	89	3.4
Overall									**96**	**7.6**
2008-12-09	Tomato Fruit	0	0	94	92	91	85	88	90	3.9
2009-01-13		30	35	89	88	96	---	---	91	4.8
2009-02-09		60	62	94	96	96	---	---	95	1.2
2009-03-13		90	94	100	100	99	---	---	100	0.6
2009-06-24		180	197	104	99	95	---	---	99	4.5
2009-12-03		360	359	97	90	92	---	---	93	3.9
2010-10-07		540	667	96	95	89	---	---	93	4.1
2011-01-10		720	762	83	75	84	---	---	81	6.1
Overall									**93**	**6.9**
2008-12-10	Soybean	0	0	88	82	70	59	70	74	15.4
2009-01-12		30	33	99	94	96	---	---	96	2.6
2009-02-09		60	61	89	92	93	---	---	91	2.3
2009-03-13		90	93	97	102	100	---	---	100	2.5
2009-06-25		180	197	98	101	101	---	---	100	1.7
2009-12-09		360	364	111	108	108	---	---	109	1.6
2010-10-07		540	666	90	91	86	---	---	89	3.0
2011-01-10		720	761	67	77	81	---	---	75	9.6
Overall									**90**	**14.8**

* FL: Fortification level expressed as prothioconazole-desthio equivalents; RSD: relative standard deviation

CA 6.2 Metabolism, distribution and expression of residues

CA 6.2.1 Plants

Data to address this point were presented in the dossier submitted for first inclusion in Annex I (March 2002) and were deemed acceptable following evaluation and peer review at EU level (2007).

Additionally metabolism studies using [phenyl-UL-^{14}C]- and [3,5-triazole-^{14}C]-labelled prothioconazole were conducted after the dossier submission on request of US EPA. The metabolism studies and an amended Annex II Tier 2 summary of the Section 4, Points IIA 6.2 and 6.6.2 (reference M-301848-01-1) were submitted at EU level to the RMS (UK PSD) in May 2008 as supportive data.

For details of data submitted previously please refer also to the Baseline dossier CA 6.2. For completeness, a summary of these previously submitted studies are included below.

The metabolism of [^{14}C]prothioconazole was investigated in wheat, peanuts and sugar beets after spray treatment employing the phenyl- as well as the triazole-labelled compound. A study in wheat following seed treatment using the phenyl-label was also conducted. Additionally, the triazole-labelled desthio derivative of prothioconazole was used as a test compound in a wheat metabolism study after spray treatment. A summary of the main parameters of these studies are compiled in the table below.

Report	Crop	Formulation	Treatment	Application rate	Harvest	Label
KCA 6.2.1/01 [redacted] & [redacted] (2000) M-041657-01-1	wheat	EC 250	spray treatment	2x200 g a.s./ha	48 days*	phenyl
KCA 6.2.1/02 [redacted] & [redacted] (2004) M-001524-01-1	wheat	SC 480	spray treatment	178 + 292 g a.s./ha	49 days*	triazole
KCA 6.2.1/03 [redacted] (2001a) M-030412-01-3	wheat	N/A	seed treatment	200 g a.s./100 kg seed	153 days	phenyl
KCA 6.2.1/04 [redacted] et al. (1993) M-008633-01-1	wheat desthio-prothio-conazole	WP 25	spray treatment	2 x 250 g a.s./ha	48 days*	triazole
KCA 6.2.1/05 [redacted] (2001b) M-033059-01-2	peanut	EC 250	spray treatment	3 x 297 g a.s./ha	157 days*	phenyl
KCA 6.2.1/06 [redacted] (2003) M-103268-01-2	peanut	EC 250	spray treatment	3 x 297 g a.s./ha	148 days*	triazole
KCA 6.2.1/07 [redacted] & [redacted] (2004a) M-001059-01-1	sugar beet	N/A	spray treatment	4 x 288 g a.s./ha	7 days*	phenyl
KCA 6.2.1/08 [redacted] & [redacted] (2004b) M-001049-01-1	sugar beet	N/A	spray treatment	4 x 289 g a.s./ha	7 days*	triazole

*After last treatment.

For convenience, the executive summaries of these plant metabolism studies are again provided in the following chapters.

Wheat

Report:	KCA 6.2.1/01 [redacted],; [redacted]; 2000; M-041657-01-1
Title:	Metabolism of JAU6476 in spring wheat (after foliar application)
Report No.:	MR-198/99
Document No.:	M-041657-01-1
Guideline(s):	US EPA OPPTS 860.1300; EU 91/414/EEC (Final 11/97)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Executive Summary

The metabolic fate of phenyl-labelled prothioconazole was investigated after two spray applications in spring wheat. Wheat was treated at growth stages tillering (BBCH[1] code 32-37) and full flowering (BBCH code 65) allowing a 17 days time period between the two applications and a 48 days interval until harvest of straw and seeds. Each treatment was performed with phenyl-labelled prothioconazole formulated as EC 250 at a rate of 200 g active substance (a.s.)/ha.

Total radioactive residue (TRR) levels in forage, hay, straw and grain were 10.45 mg/kg, 8.90 mg/kg, 26.74 mg/kg and 0.08 mg/kg (a.s. equivalents) respectively.

Identified metabolites in the raw agricultural commodities (RACs) accounted for 73.1% of the TRR in forage, 64.7% in hay, 66.2% in straw and 33.7% in grain.

Major metabolite identified in the different RACs included JAU 6476-desthio *(M04)*, (as the main metabolite) and its derivatives JAU 6476-desthio-4-hydroxy *(M15)*, isomers (3- and 4-) of JAU 6476-desthio-hydroxy *(M08, M13)* and the corresponding glucosides *(M21-23)*. Two other metabolites of prothioconazole were identified as JAU 6476-sulfonic acid *(M02)* and JAU 6476-triazolinone *(M03)* (percentages of TRR and mg a.s. equivalents/kg of these metabolites are provided in Table 6.2.1-1 below).

[1] BBCH Code (1994): Compendium of growth stage identification: Keys for mono- and dicotyledoneus plants
BBA, IVA and other members of the BBCH

Table 6.2.1-1: Parent compound and major metabolites after spray application of phenyl-labelled prothioconazole to wheat (in % of TRR and mg a.s. equivalents/kg)

Parent compound / metabolite	Forage		Hay		Straw		Grain	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Prothioconazole	3.3	0.35	2.6	0.24	3.7	0.98	1.3	< 0.01
JAU 6476-sulfonic acid *(M02)*	7.1	0.75	3.3	0.29	8.5	2.24	n.d.	n.d.
JAU 6476-triazolinone *(M03)*	6.9	0.71	5.1	0.46	6.1	1.64	1.3	< 0.01
JAU 6476-desthio *(M04)*	35.4	3.70	18.5	1.64	22.3	5.95	15.5	0.74
JAU 6476-desthio-3-hydroxy *(M14)*	2.4	0.25	8.5	0.75	2.9	0.75	1.1	< 0.01
JAU 6476-desthio-4-hydroxy *(M15)*	1.2	0.13	6.7	0.60	2.7	0.72		
JAU 6476-desthio-6-hydroxy *(M17)*	1.1	0.12	1.2	0.11	1.2	0.32	n.d.	< 0.01
3 isomers of JAU 6476-desthio-hydroxy-glucoside *(M21-23)*	8.6	0.91	2.6	0.24	7.3	1.96	8.4	< 0.01
JAU 6476-desthio-α-hydroxy *(M18)*	4.5	0.47	9.4	0.85	5.8	1.54	2.8	0.04
Sum of 8 comp. at maximum	2.4	0.25	6.7	0.59	5.8	1.55	3.2	0.0[illegible]

n.d. = not detected

Prothioconazole was extensively metabolised in wheat. The proposed metabolic reactions involved:

- oxidation and loss of sulphur of the triazolinethione ring resulting in the main metabolite JAU 6476-desthio *(M04)*
- hydroxylation of the chlorobenzyl methylene C-atom and hydroxylation of the chlorobenzyl ring at position 3, 4 and 6 of JAU 6476-desthio
- conjugation of all these hydroxylated metabolites and to a minimal extent of JAU 6476-desthio

Minor degradation reactions were:

- exchange of sulfur against oxygen
- elimination of the triazole moiety and conjugation of the benzylpropyldiol rest

Report: [redacted]; 2004; M-001524-01-1
Title: The metabolism of [triazole-3,5-14 C] JAU 6476 in wheat
Report No.: 200733
Document No.: M-001524-01-1
Guideline(s): OPPTS 860, 1300; DACO 6.3
Guideline deviation(s): --
GLP/GEP: **yes**

Executive Summary

The metabolic fate of triazole-labelled prothioconazole was investigated after two spray applications in wheat. The wheat was treated at BBCH crop growth stages 32 (node 2 at least 2 cm above node 1) and 65 (full flowering). Each treatment was performed with triazole-labelled prothioconazole formulated as SC 480. The treatment rates for the first and the second application were 178 and 292 g a.s./ha, respectively.

The total radioactive residue (TRR) in wheat forage, hay, straw, and grain were 7.96 mg/kg, 11.18 mg/kg, 7.94 mg/kg, and 4.97 mg/kg, respectively.

Identified metabolites in the raw agricultural commodities (RACs) accounted for 66% of the TRR in forage, 75% in hay, 61% in straw and 94% in grain.

The major residues found in all matrices were JAU 6476-desthio *(M04)*, triazolylalanine (TA, *M31*), triazolylacetic acid (TAA, *M29*), triazolylhydroxypropionic acid (THP, *M30*), JAU 6476-desthio-α-hydroxy *(M18)* and prothioconazole (percentages of TRR and mg/kg equivalents a.s. of these major metabolites are provided in Table 6.2.1-2 below).

Table 6.2.1-2: Parent compound and major metabolites after spray application of triazole-labelled prothioconazole to wheat (in % of TRR and mg a.s. equivalents/kg)

Parent compound / metabolite	Forage		Hay		Straw		Grain	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Prothioconazole	5	0.394	3	0.326	6	0.481		
JAU 6476-desthio *(M04)*	19	1.498	11	1.279	9	0.700		
JAU 6476-desthio-hydroxy *(M14-M17)*	2[a)]	0.162[a)]	n.d.	n.d.	7	0.485		
JAU 6476-desthio-α-hydroxy *(M18)*	9	0.673	7	0.764	5	0.395		
TA *(M31)*	12	0.951	25	2.869	4	0.321	71	3.537
THP *(M30)*	3	0.220	8	0.849	8	0.609	< 1	0.028
TAA *(M29)*	1	0.108	5	0.490	5	0.365	19	0.946
Sum of 17 comp. in maximum	15	1.139		1.295		1.285		

n.d. = not detected
a) = JAU 6476-desthio-3-hydroxy *(M14)* and JAU 6476-desthio-4-hydroxy *(M15)*

Following the initial metabolism of prothioconazole to JAU 6476-desthio *(M04)* two major metabolic processes were observed:

- one major pathway involved hydroxylation of the phenyl ring and/or benzylic carbon followed by conjugation with malonic acid and/or glucose;
- the other major pathway involved the release of the triazole moiety and subsequent formation of triazole conjugates.
  The fact that no free 1,2,4-triazole *(M13)* was found in any wheat matrix suggests an immediate conjugation of the released triazole.
- minor metabolic processes led to the formation of JAU 6476-triazolinone *(M03)* and JAU 6476-desthio-phenylalanine *(M44)*, conjugation of JAU 6476-desthio *(M04)* with glucose and malonic acid, oxidation of the sulfur atom of the triazolinethione ring of prothioconazole to form JAU 6476-sulfonic acid *(M02)*, and cleavage of the benzylic group to form JAU 6476-triazolyl-ethanol *(M45)* and its glucoside *(M46)*.

Report: [redacted]; 2001; M-030412-01-3
Title: Metabolism of JAU 6476 in spring wheat after seed dressing
Report No.: 1d0881
Document No.: M-030412-01-3
Guideline(s): US EPA OPPTS 860.1300; EU 91/414/EEC (Final 11/97)
Guideline deviation(s): not specified
GLP/GEP: yes

Executive Summary

The metabolic fate of phenyl-labelled prothioconazole was investigated in spring wheat after seed dressing. The results were compared to the results of the study were phenyl-labelled prothioconazole had been applied by spraying (MR-198/99, summarised in KCA 6.2.1/01.

Wheat seeds were treated at a rate of 20 g a.s./100 kg seeds, corresponding to 7.97 µg a.s./seed (so called 1X experiment). In a 5X overdose experiment 40.0 µg a.s./seed were applied.

Total radioactive residue (TRR) levels of the 1X experiment were very low and amounted to 0.07 mg/kg in forage, 0.02 mg/kg in hay, 0.03 mg/kg in straw and 0.008 mg/kg (a.s. equivalents) in grain, respectively. None of the extraction phases obtained after partitioning of the extracts, reached the identification threshold values.

Due to these low concentrations, further analysis for metabolite identification was conducted for the RACs forage, hay and straw of the 5X overdose experiment, where TRRs were determined to be 0.07 mg/kg for forage, 0.09 mg/kg for hay, 0.28 mg/kg for straw and 0.01 mg/kg for grain.

Major metabolites, identified in the different RACs, included JAU 6476-desthio *(M04)*, (as main metabolite), its hydroxylated isomers *(M14/M15/M17)* and the corresponding glucosides. In addition to the parent compound the following metabolites were identified: JAU 6476-glutonic acid *(M02)*, JAU 6476-triazolinone *(M03)*, JAU 6476-benzylpropyldiol-glucosides *(M43)*, JAU 6476-desthio-α-hydroxy *(M18)* and JAU 6476-desthio-α-acetoxy *(M19)*. One metabolite JAU 6476-disulfide *(M11)* was tentatively identified.

In wheat grain the radioactivity concentration was below 0.01 mg/kg rendering a metabolite identification impossible.

The percentages of TRR and mg/kg equivalents a.s. of the main metabolites are provided in Table 6.2.1-3 below.

Table 6.2.1-3: Parent compound and major metabolites after application of phenyl-labelled prothioconazole as seed dressing on wheat (5X experiment) in % of TRR and mg a.s. equivalents/kg

Parent compound / metabolite	Forage		Hay		Straw		Grain	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Prothioconazole	0.4	< 0.001	0.8	0.001	0.6	0.001	TRR < 0.01 mg/kg	
JAU 6476-desthio *(M04)*	10.9	0.008	6.5	0.005	6.6	0.019		
JAU 6476-desthio-3-hydroxy *(M14)*	14.0	0.010	3.8	0.004	3.8	0.011		
JAU 6476-desthio-4-hydroxy *(M15)*					2.4	0.006		
JAU 6476-desthio-3/4/6-hydroxy-glucosides *(M21-23)*					10.6	0.030		
Sum of 6 comp. in maximum	25	0.018	11	0.009	24	0.067		

The following metabolic reactions were involved:

- oxidation and loss of sulphur of the triazolinethione ring, resulting in the main metabolite JAU 6476-desthio *(M04)*
- hydroxylation of the chlorobenzyl methylene C-atom and hydroxylation of the chlorobenzyl ring at positions 3, 4 and 6 of JAU 6476-desthio

Minor degradation reactions were:

- exchange of sulfur against oxygen
- elimination of the triazole moiety and conjugation of the benzylpropyldiol rest

Metabolism of prothioconazole-desthio in wheat

The objective of this study was to determine the overall metabolic fate of JAU 6476-desthio *(M04)* and the total radioactive residues in summer wheat following spray application.

Report:	KCA 6.2.1/04 [redacted] N; [redacted]; [redacted]; 1993; M-008633-01-1
Title:	Metabolism of SXX 0665 in summer wheat
Report No.:	PF3906
Document No.:	M-008633-01-1
Guideline(s):	US EPA 171-4
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Executive Summary

The metabolic fate of triazole-labelled prothioconazole-desthio *(M04)* was investigated after two spray applications in wheat. Each treatment was performed with the labelled test compound formulated as WP 25 and at a rate of approximately 250 g /ha. The wheat was treated at BBCH crop growth stages 31 (one-node stage) and 59 (end of heading) allowing a 17 days time period between the two applications. Forage samples were collected immediately (day 0) and 14 days after the second application. Mature wheat samples (straw, glumes and grain) were harvested 48 days after the second application.

The measurement of total radioactive residues (TRR) in forage resulted in 10.1 mg/kg (day 0) and 10.9 mg/kg (day 14). The TRRs in straw, glumes and grain were 28.7 mg/kg, 11.6 mg/kg and 2.85 mg/kg, respectively.

Identified metabolites in the raw agricultural commodities (RACs) accounted for 89.9 – 93.7% of the TRR in forage, 92.6% in straw and 94.5% in grain.

Metabolites identified in the different RACs included JAU 6476-desthio (M04), and its hydroxylated derivatives JAU 6476-desthio-4-hydroxy *(M14)* and JAU 6476-desthio-hydroxy isomers *(M14, M15, M16, M17)*. In addition triazolylacetic acid (TAA, *M29*), a main metabolite in grain, triazolylhydroxy-propionic acid (THP, *M30*) and triazolylalanine (TA, *M31*) were identified.

An overview on percentages of TRR and mg/kg equivalents JAU 6476-desthio of the identified metabolites is provided in Table 6.2.1-4 below.

Table 6.2.1-4: Identified metabolites after spray application of triazole-labelled prothioconazole-desthio to wheat (in % of TRR and mg a.s. equivalents/kg)

Compound	Forage (day 0)		Forage (day 14)		Straw		Grain	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
JAU 6476-desthio *(M04)*	86.8	8.94	76.9	8.36	71.9	20.61	2.3	0.07
JAU 6476-desthio-3-hydroxy *(M14)*	1.2	0.12	1.5	0.16	1.3	0.37		
JAU 6476-desthio-4-hydroxy *(M15)*	0.8	0.08	1.8	0.20	0.8	0.23		
JAU 6476-desthio-5-hydroxy *(M16)*	1.5	0.15	1.8	0.20	0.8	0.23		
JAU 6476-desthio-6-hydroxy *(M17)*	0.3	0.03	0.5	0.06	0.2	0.06		
JAU 6476-desthio-dihydroxy *(M18)*	1.6	0.16	3.5	0.37	4.3	1.23	0.1	< 0.01
TA *(M31)*	0.3	0.03	1.3	0.14	0.2	0.06	60.2	1.72
THP *(M30)*	0.6	0.06	1.4	0.16	3.2	0.92		
TAA *(M29)*	0.6	0.06	1.2	0.13	0.9	0.26	31.9	0.91

n.d. = not detected

Prothioconazole-desthio underwent the following metabolic conversions:

- hydroxylation of the chlorobenzyl methylene group
- mono-hydroxylation of the chlorobenzyl ring at all unsubstituted positions
- conjugation of all hydroxylated compounds
- cleavage of the H_2C-N bond to release the triazole moiety leading to the formation of triazolylalanine (TA, *M31*) and triazolylhydroxypropionic acid (THP, *M30*) and further metabolism of the triazole conjugates to triazolylacetic acid (TAA, *M29*). The fact that no free 1,2,4-triazole *(M13)* was found in any of the wheat matrices suggests an immediate conjugation of the released triazole.

Peanut

Report:	KCA 6.2.1/05 [redacted]; 2001; M-033059-01-2
Title:	Metabolism of [phenyl-UL-14C]JAU6476 in peanuts
Report No.:	MR-193/01
Document No.:	M-033059-01-2
Guideline(s):	US EPA OPPTS 860.1300; Canada PMRA Ref.: DACO 6.3; EU 91/414/EEC amended by 96/68/EC
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Executive Summary

The metabolic fate of prothioconazole was investigated after spray application to peanuts. Peanuts were treated 3 times allowing a 3 week time period between the three applications and a 14 days interval until harvest (PHI). Each treatment was performed with phenyl-labelled prothioconazole formulated as an EC 250 at a rate of 297 g a.s./ha.

Total radioactive residue (TRR) levels in peanut hay were 107.5 mg/kg, whereas TRR levels in nutmeat were quite low at 0.29 mg/kg (as a equivalents).
Identified metabolites in the raw agricultural commodities (RACs) accounted for 74.1% of the TRR in peanut hay and 65.4% in nutmeat.

Major metabolites in peanut hay included JAU 6476-desthio *(M04)* (as the main metabolite, 28.2% of TRR, 30.4 mg/kg) and its derivatives, the two isomers (3- and 4-) of JAU 6476-hydroxy-desthio *(M14/M15)*. In addition to the parent compound, two other metabolites of prothioconazole were identified as JAU 6476-sulfonic acid *(M02)* and JAU 6476-triazolinone *(M03)*. None of these compounds were detected in nutmeat.

The presence of metabolites derived from JAU 6476-desthio and JAU 6476-sulfonic acid but lacking the aromaticity of the phenyl ring was confirmed in hay and in the case of JAU 6476-desthio derivatives also in nutmeat. But the main portion of radioactivity (47.8% of TRR in the extracts) of nutmeat was characterized as natural constituents and identified as fatty acids.

Prothioconazole was intensively metabolised in peanuts. The proposed metabolic reactions in peanut hay involved:

- oxidation and loss of sulphur of the triazolinethione ring, resulting in the main metabolite JAU 6476-desthio *(M04)*
- hydroxylation of the chlorophenyl ring
- conjugation of these hydroxylated metabolites
- exchange of sulfur against oxygen
- oxidative hydroxylation of the phenyl moiety leading to de-aromatisation

Report:	[redacted]9; [redacted]; 2003; M-103268-01-2
Title:	Metabolism of [triazole-UL-14C]JAU6476 in peanuts
Report No.:	MR-194/02
Document No.:	M-103268-01-2
Guideline(s):	US EPA OPPTS 860.1300; Canadian PMRA Ref.: DACO 6.3; EU 91/414/EEC amended by 96/68/EC
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Executive Summary

The metabolism of prothioconazole was investigated in peanuts following spray application. The peanuts were treated 4 times allowing a 3 week period between the applications and a 14 day pre-harvest interval (PHI). Each treatment was performed with triazole-labelled prothioconazole formulated as an EC 250 at a rate of 297 g a.s./ha.

The TRR in hay was 47.38 mg/kg, the TRR in nutmeat was much lower (1.4 mg a.s. equivalents/kg). Identified metabolites in the RAC accounted for 77.0% of the TRR in peanut hay and 82.7% in nutmeat.

Major metabolites in peanut hay included JAU 6476-desthio *(M04)* (identified as main metabolite in peanut hay, 23.6% of TRR, 11.2 mg/kg) and its derivatives, the two isomers (3- and 4-) of JAU 6476-hydroxy-desthio *(M14/M15)*. Two other metabolites of prothioconazole were identified as JAU 6476-sulfonic acid *(M02)* and JAU 6476-triazolinone *(M03)*. Furthermore, metabolites derived from JAU 6476-desthio *(M04)* and JAU 6476-sulfonic acid *(M02)* but lacking the aromaticity of the phenyl ring were detected in the hay. With the exception of JAU 6476-desthio *(M04)*, none of these metabolites were detected in nutmeat. The major metabolites in nutmeat, i.e. triazolylalanine (TA, *M31*) and triazolylhydroxypropionic acid (THP, *M30*), are conjugates of 1,2,4-triazole *(M13)*. However, free 1,2,4-triazole *(M13)* was not detected. A small portion of the radioactivity (3.0% of the TRR in the extracts) in nutmeat was characterised as natural constituents, and identified as fatty acids.

Prothioconazole was extensively metabolised. The proposed metabolic reactions in peanuts involved:

- oxidation and loss of sulfur of the triazolinethione ring, resulting in the main metabolite JAU 6476-desthio *(M04)* (peanut hay)
- hydroxylation of the chlorophenyl ring of JAU 6476-desthio *(M04)* at positions 3 and 4
- (higher) conjugation of these hydroxylated metabolites
- exchange of sulfur against oxygen
- release of the triazole moiety, but no detection of free triazole. However, the triazole conjugates triazolylalanine (TA, *M31*) and triazolylhydroxypropionic acid (THP, *M30*) were detected mainly in nutmeat

A quantitative overview on percentages of TRR and mg/kg equivalents prothioconazole of the identified metabolites is provided in Tables 6.2.1-6 and 6.2.1-7 below.

Table 6.2.1-6: Prothioconazole and metabolites after spray application of phenyl- and triazole-labelled prothioconazole to peanuts (in mg a.s. equivalents/kg)

Label Parent compound / metabolite	Hay *phenyl*	*triazole*	Nutmeat *phenyl* extraction 2[a]	extraction 1[b]	*triazole*
a.s.	1.98	3.11			
Metabolites common to both labels:					
M02	2.28	1.24		< 0.01	
M03	1.72	1.16			
M11	3.48	2.55			
Σ: M25, M26	7.89	0.73			
M04	30.37	11.13			0.09
M14	7.81	3.13			
M15	2.19	1.40			
M14 or M15[c)]	0.91				
Σ: M21, M22	0.97	0.52	< 0.01	< 0.01	
Σ: M54, M55		2.82			
M24	5.59		0.03	0.02	
Σ: M47, M64		2.99			
M64	15.09		0.02	0.04	
M44		0.78			
Metabolites specific to the triazole-label:					
M31		0.66			0.67
M30		0.30			0.34
M29		0.33			0.02
M45		0.21			
M46		0.71			
Σ: fatty acids			0.14	0.13	0.04
Total identified	79.98	36.36	0.20	0.20	1.16
Total radioactive residue (TRR)	107.51	40.38	0.29	0.30	1.40

a.s. = prothioconazole
M02 = JAU 6476-sulfonic acid
M03 = JAU 6476-triazolinone
M04 = JAU 6476-desthio
M11 = JAU 6476-disulfide
M14 = JAU 6476-desthio-3-hydroxy
M15 = JAU 6476-desthio-4-hydroxy
M21 = JAU 6476-desthio-3-hydroxy-glucoside
M22 = JAU 6476-desthio-4-hydroxy-glucoside
M24 = JAU 6476-desthio-dihydroxy-dienyl-cysteine
M25 = JAU 6476-dihydroxy-diene-sulfonic acid
M26 = JAU 6476-dihydroxy-olefin-sulfonic acid
M29 = triazolylacetic acid (TAA)
M30 = triazolylhydroxypropionic acid (THP)
M31 = triazolylalanine (TA)
M44 = JAU 6476-desthio-phenyl-cysteine
M45 = JAU 6476-triazolyl-ethanol
M46 = JAU 6476-triazolyl-ethanol-glucoside
M47 = JAU 6476-desthio-dihydroxy-diene-glucoside
M54 = JAU 6476-desthio-3-hydroxy-glucoside-malonic acid
M55 = JAU 6476-desthio-4-hydroxy-glucoside-malonic acid
M64 = JAU 6476-desthio-dihydroxy-olefin-glucoside
a) = extraction procedure 2 (MSP)
b) = extraction procedure 2 (n-hexane reflux)
c) = trace amounts, no discrimination between isomers possible

Table 6.2.1-7: Prothioconazole and metabolites after spray application of phenyl- and triazole-labelled prothioconazole to peanuts (in % of TRR)

Label Parent compound / metabolite	Hay *phenyl*	*triazole*	Nutmeat *phenyl* extraction 2[a)]	extraction 1[b)]	*triazole*
TRR in mg a.s. equivalents/kg	107.51	47.38	0.29	0.30	1.40
a.s.	1.8	6.6			
Metabolites common to both labels:					
M02	2.1	2.7		1.5	
M03	1.6	3.7			
M11	3.2	1.4			
Σ: M25, M26	7.4	1.5			
M04	28.2	23.6			6.2
M14	7.3	6.2			
M15	2.0	3.0			
M14 or M15[c)]	0.8				
Σ: M21, M22	9.9	7.6	1.0	3.4	
Σ: M54, M55		1.0			
M24	5.2	2.0	2.0	3.4	
Σ: M47, M64		4.2			
M64	4.1		7.1	12.3	
M44		1.7			
Metabolites specific to the triazole-label:					
M31		1.2			47.8
M30		0.6			24.5
M29		0.7			1.2
M45		0.5			
M46		1.3			
Σ: fatty acids			77.8	42.6	3.0
Total identified	74.1	77.0	65.4	65.1	82.7
Total radioactive residues (TRR)	100.0	100.0	100.0	100.0	100.0

a.s. = prothioconazole
M02 = JAU 6476-sulfonic acid
M03 = JAU 6476-triazolinone
M04 = JAU 6476-desthio
M11 = JAU 6476-dimethide
M14 = JAU 6476-desthio-3-hydroxy
M15 = JAU 6476-desthio-4-hydroxy
M21 = JAU 6476-desthio-3-hydroxy-glucoside
M22 = JAU 6476-desthio-4-hydroxy-glucoside
M24 = JAU 6476-desthio-hydroxy-diene-cysteine
M25 = JAU 6476-dihydroxy-diene-sulfonic acid
M28 = JAU 6476-dihydroxy-olefin-sulfonic acid
M29 = triazolylacetic acid (TAA)
M30 = triazolylhydroxypropionic acid (THP)
M31 = triazolylalanine (TA)
M44 = JAU 6476-desthio-phenyl-cysteine
M45 = JAU 6476-triazolyl-ethanol
M46 = JAU 6476-triazolyl-ethanol-glucoside
M47 = JAU 6476-desthio-dihydroxy-diene-glucoside
M54 = JAU 6476-desthio-3-hydroxy-glucoside-malonic acid
M55 = JAU 6476-desthio-4-hydroxy-glucoside-malonic acid
M64 = JAU 6476-desthio-dihydroxy-olefin-glucoside
a) = extraction procedure 2 (MTPD)
b) = extraction procedure 2 (n-hexane reflux)
c) = trace amounts, no discrimination between isomers possible

Sugar beet

Report:	[redacted]1; [redacted]; [redacted]; 2004; M-001059-01-1
Title:	The metabolism of [phenyl-UL-14C]JAU6476 in sugar beets
Report No.:	200466
Document No.:	M-001059-01-1
Guideline(s):	EPA Ref.: OPPTS 860.1300, Nature of the Residue - Plants
Guideline deviation(s):	--
GLP/GEP:	**yes**

Executive Summary

Four foliar spray applications of phenyl-labelled prothioconazole were made to sugar beet plants at an average rate of 288 g a.s./ha/application for a total rate of 1152 g a.s./ha. Applications were made at 14-day intervals with the last application 7 days prior to harvest of sugar beet tops and roots.

The total radioactive residue (TRR) in sugar beet tops was 4.33 mg/kg, and in roots 0.2 mg/kg.

In sugar beet tops, 65% of the TRR (2.84 mg/kg) was identified, and an additional 33% (1.44 mg/kg) was characterised. All radioactive residues greater than 5% of the TRR (0.2 mg/kg) were identified. In roots, 60% of the TRR (0.001 mg/kg) was identified and an additional 32% (0.138 mg/kg) was characterised. All radioactive residues greater than 9% (0.011 mg/kg) were identified.

Major metabolites identified in the different RACs (esp. in sugar beet tops) were JAU 6476-desthio *(M04)* and isomers of its hydroxy-glucosides *(M21/M22/M23)*, JAU 6476-desthio-hydroxy-dienyl-cysteine isomers *(M24)* and JAU 6476-hydroxy-sulfonic acid glucoside *(M59)*. The percentages of TRR and mg/kg equivalents a.s. of these 5 main metabolites are provided in Table 6.2.1-8 below.

Table 6.2.1-8: Prothioconazole and major metabolites after spray application of phenyl- labelled prothioconazole to sugar beets (in % of TRR and mg/kg equivalents/kg).

Parent compound / metabolite	Sugar beet tops		Sugar beet roots	
	% of TRR	mg/kg	% of TRR	mg/kg
Prothioconazole		0.323		
JAU 6476-hydroxy-sulfonic acid glucoside *(M59)*	8	0.351		
JAU 6476-desthio *(M04)*	28	1.249	58	0.068
JAU 6476-desthio-hydroxy-glucosides, isomers *(M21/M22/M23)*	5	0.222		
JAU 6476-desthio-hydroxy-dienyl-cysteine isomers *(M24)*	10	0.454		
Sum of components at maximum	7	0.24	2	0.003

Prothioconazole was extensively metabolised in sugar beets. The proposed reactions involved are:

- oxidation and loss of sulfur of the triazolinethione ring, resulting in the main metabolite JAU 6476-desthio *(M04)*
- hydroxylation of the phenyl ring or the benzyl carbon to form multiple isomers
- followed by subsequent conjugation with glucose or further reaction to yield isomers of JAU 6476-desthio-hydroxy-glucosides *(M21/M22/M23)* or JAU 6476-desthio-hydroxy-dienyl-cysteine *(M24)*, respectively.

Report:	[redacted]8;[redacted];[redacted]; 2004; M-001049-01-1
Title:	The metabolism of [triazole-UL-14C]JAU6476 in sugar beets
Report No.:	200467
Document No.:	M-001049-01-1
Guideline(s):	EPA Ref.: OPPTS 860.1300, Nature of the Residue - Plants
Guideline deviation(s):	--
GLP/GEP:	**yes**

Executive Summary

Four foliar spray applications of triazole-labelled prothioconazole were made to sugar beets at an average rate of 289 g a.s./ha/application for a total rate of 1157 g a.s./ha. Applications were made at 14-day intervals with the last application 7 days prior to harvest of sugar beet tops and roots.

The total radioactive residue (TRR) in sugar beet tops was 5.19 mg/kg, and in roots 0.13 mg/kg.

In sugar beet tops, 69% (3.54 mg/kg) of the TRR was identified and an additional 26% (1.52 mg/kg) was characterised. All individual radioactive components containing more than 6% (0.33 mg/kg) of the TRR were identified. In sugar beet roots, 67% (0.090 mg/kg) of the TRR was identified, and an additional 33% (0.042 mg/kg) was characterised. All radioactive residues greater than 12% (0.016 mg/kg) of the TRR were identified. The percentages of TRR and mg/kg equivalents a.s. of these major metabolites are provided in Table 6.2.1-9 below.

Table 6.2.1-9: Prothioconazole and major metabolites after spray application of triazole- labelled prothioconazole to sugar beets (in % of TRR and mg/kg equivalents/kg).

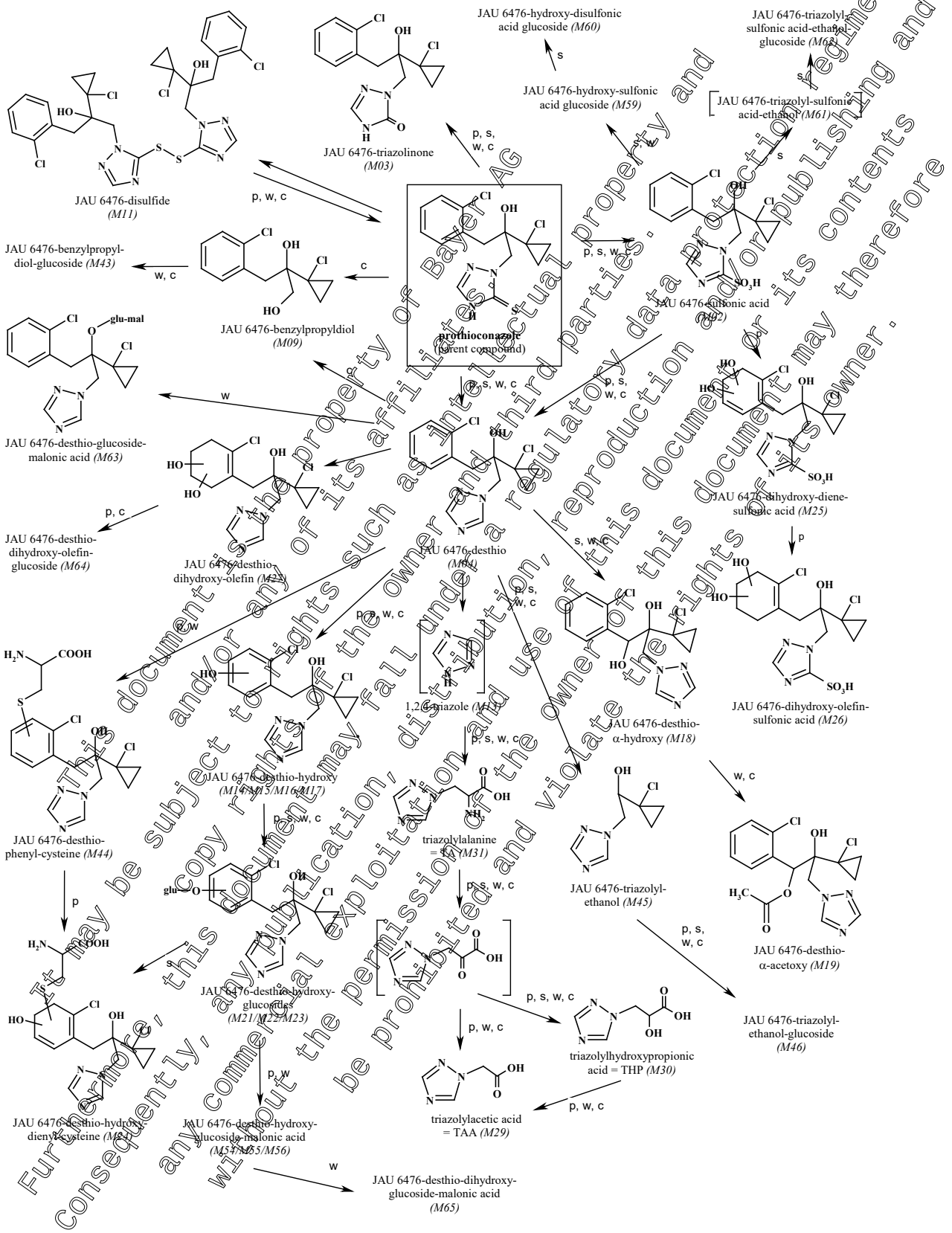
Parent compound /metabolite	Sugar beet tops		Sugar beet roots	
	% of TRR	mg/kg	% of TRR	mg/kg
Prothioconazole	5	0.265		
JAU 6476-hydroxy-sulfonic acid glucoside isomer *(M59)*	6	0.316		
JAU 6476-desthio *(M04)*	19	0.988	25	0.033
JAU 6476-desthio-monohydroxy isomers *(M14/M15/M16/M17)*	1	0.063		
JAU 6476-desthio-monohydroxy-glucoside isomers *(M21/M22/M23)*	6	0.334		
JAU 6476-desthio-hydroxy-dienyl-cysteine isomers *(M24)*	10	0.512	5	0.007
Triazolylalanine = TA *(M31)*	2	0.084	29	0.038
Sum of 6 components at maximum	19	0.974	2	0.002

Prothioconazole was extensively metabolised in sugar beets to numerous components; only a small quantity of unchanged parent compound was detected. The major metabolite was JAU 6476-desthio *(M04)* arising from oxidation of the sulfur of the triazolinethione ring to the corresponding sulfonic acid *(M02)* with subsequent elimination of the sulfonic acid group. Hydroxylation of the phenyl ring or benzylic carbon yielding several monhydroxylated isomers *(M14.M17)* was observed with subsequent conjugation with glucose *(M21-23)* or further reaction to produce JAU 6476-desthio-hydroxy-dienyl-cysteine *(M24)*.

The triazole moiety was released leading to triazolylalanine (TA, *M31*) and triazolylhydroxypropionic acid (THP, *M30*). Free 1,2,4-triazole *(M13)* was not identified. Additional triazole-label specific metabolites were formed by elimination of the chlorophenyl moiety and subsequent conjugation of the resulting metabolite with glucose.

The proposed metabolic pathway of prothioconazole in wheat, peanuts, sugar beets and rotational crops (see CA 6.6.1) is shown in Figure 6.6.2- 1.

Figure 6.6.2- 1: Proposed metabolic pathway of prothioconazole in plants



p = peanuts, s = sugar beets, w = wheat, c = confined rotational crop

CA 6.2.2 Poultry

Two studies to address this point were presented in the dossier submitted for first inclusion in Annex I (March 2002) and were deemed acceptable following evaluation and peer review at EU level (2007).

Report:	KCA 6.2.2/01 [redacted]; [redacted]; 2001; M-032686-01-2
Title:	[Phenyl-UL-14C]JAU6476 - Absorption, distribution, excretion and metabolism in laying hens
Report No.:	MR-309/01
Document No.:	M-032686-01-2
Guideline(s):	US EPA OPPTS860.1300; Canada PMRA Ref.: DACO 6.2; EU 91/414/EEC amended by 96/68/EC
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.2.2/02 [redacted]カ; [redacted]; 2003; M-109936-02-1
Title:	[Triazole-UL-14C]JAU6476: Absorption, distribution, excretion and metabolism in laying hens
Report No.:	MEF-005/03
Document No.:	M-109936-02-1
Guideline(s):	US EPA OPPTS 860.1300; Canadian PMRA Ref.: DACO 6.2; EU 91/414/EEC amended by 96/68/EC
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

For details of data submitted previously please refer also to the Baseline dossier CA 6.2. For completeness, a summary of the key parameters of these previously submitted studies and an overall summary of the two metabolism studies of prothioconazole in poultry is provided below.

Report	Administered compound	Label	Dosing	Time of sacrifice	Number of animals
CA 6.2.2/01 [redacted] & [redacted] (2001) M-032686-01-2	Prothioconazole	Phenyl	3 x 10 mg/kg bw/day	5 h after last dose	6
CA 6.2.2/02 [redacted] & [redacted] (2003) M-109936-02-1	Prothioconazole	Triazole	3 x 10 mg/kg bw/day	5 h after last dose	6

Overall summary of the metabolism of prothioconazole in poultry

The kinetic behaviour and metabolism of prothioconazole in laying hens was investigated in two separate studies employing the [phenyl-UL-^{14}C]- and the [triazole-UL-^{14}C] labelling positions following an identical design: Six laying hens were orally dosed (3x) with radiolabelled prothioconazole at a dose level of 10 mg/kg body weight. The hens received the doses on three consecutive days in intervals of 24 hours. The animals were sacrificed 5 hours after the last administration.

The kinetics and the metabolism of prothioconazole were comparable in both studies. The kinetics were characterised by a fast and almost complete absorption process of the administered radioactivity, followed by fast excretion. The predominant portion of the totally administered dose was found in the excreta whereas only trace amounts were detected in eggs (0.01%) and edible organs and tissues (0.8% to 0.9%).

The total radioactive residues were also well comparable, for liver and fat slightly higher concentrations were detected in the study with phenyl-labelled prothioconazole (see Table 6.2.2-1).

Table 6.2.2- 1 Total radioactive residues (TRR) determined in eggs and edible tissues of laying hen after administration of prothioconazole

	Liver		Eggs		Muscle		Fat	
Label	phenyl	triazole	phenyl	triazole	phenyl	triazole	phenyl	triazole
TRR [mg/kg]	4.017	1.531	0.036	0.050	0.089	0.122	0.450	0.290

Major metabolites were JAU 6476-S-methyl *(M01)*, JAU 6476-desthio *(M02)* and JAU 6476-S-glucuronide *(M06)*. Also, a number of sulfate conjugates of hydroxylated JAU 6476-desthio (*M82, M83* and *M84*) was detected as a significant portion in liver. Label-specific metabolites were only detected in the study employing the triazole-labelled prothioconazole.

The percentages of the TRR and mg/kg as equivalents/kg values of prothioconazole and its metabolites are shown in Tables 6.2.2-2 and 6.2.2-3.

Major compounds in both studies were the parent compound and JAU 6476-S-glucuronide *(M06)*. The metabolites JAU 6476-S-methyl *(M01)* and JAU 6476-desthio *(M02)* were also detected in all matrices, but were only of major significance in fat. The label specific metabolites 1,2,4-triazole *(M13)* and JAU 6476-triazolylethanol *(M15)* were detected in all matrices in the study using triazole-labelled prothioconazole. They represented major fractions of the total radioactive residues in eggs and muscle, but the absolute concentrations did not exceed 0.04 mg/kg. However, it has to be considered that the hens were administered with highly exaggerated doses with respect to the field residues in cereal grains and straw (more than a factor of 100 for straw, which shows generally the highest residues).

Table 6.2.2- 2 Parent compound and metabolites in eggs, tissues and organs of laying hens after application of phenyl- and triazole-labelled prothioconazole (in % of TRR)

Compound	Liver		Eggs		Muscle		Fat	
Label	% TRR phenyl	triazole	% TRR phenyl	triazole	% TRR phenyl	triazole	% TRR phenyl	triazole
TRR (mg a.s. equiv./kg)	4.017	3.531	0.036	0.050	0.089	0.122	0.450	0.299
Prothioconazole (JAU 6476)	24.8	30.7	3.6	3.4	11.3	2.5	30.7	3.9
Metabolites common to both labels								
JAU 6476-S-methyl *(M01)*	2.2	1.7	1.9	1.2	6.6	2.2	19.6	28.8
JAU 6476-N-glucuronide *(M05)*	1.1[d]	0.2			1.1[d]			
JAU 6476-S-glucuronide *(M06)*[e]	11.9[a]	14.9[a]	17.0	23.7[b]	15.5	9.8	5.3	7.2
JAU 6476-4-hydroxy *(M08)*	0.7[d]	0.3			1.6[d]			
JAU 6476-hydroxy-glucuronide *(M10)*	2.6							
JAU 6476-dihydroxy-diene *(M40)*	0.9[d]				1.3[d]			
JAU 6476-desthio *(M04)*	4.2	4.1	28.1	4.2	7.2	2.1	2.0	26.8
JAU 6476-desthio-4-hydroxy *(M15)*	2.7	0.1	3.3[d]					
JAU 6476-desthio-hydroxy-sulfate *(M84)*	3.3	1.6						
JAU 6476-desthio-3,4-dihydroxy-dienyl-glucuronide *(M52)*	2.2				2.5[d]			
JAU 6476-desthio-hydroxy-methoxy-sulfate *(M82)*, JAU 6476-desthio-dihydroxy-sulfate *(M83)* and JAU 6476-desthio-hydroxy-sulfate *(M84)*	7.8	11.9			1.5			0.7
Metabolites specific to the triazole label								
1,2,4-triazole *(M13)*		1.0		11.4		18.7		1.5[c]
JAU 6476-triazolyl-ethanol *(M45)*		3.6		13.6		28.3		1.6
Thiocyanate *(M80)*		0.7		9.1		4.0		1.5[c]
Total identified	61.7	72.3	42.8	71.4	41.9	67.6	84.1	82.3
Total characterised	10.6	12.0	10.4	10.4	11.7	11.3	-	5.9

a) may contain a trace amount of JAU 6476-desthio-3-hydroxy *(M14)*
b) may contain a minor amount of JAU 6476-desthio-4-hydroxy *(M15)* and a trace amount of JAU 6476-desthio-3-hydroxy *(M14)*
c) for the fat, the values of 1,2,4-triazole *(M13)* and thiocyanate *(M80)* could only be determined as the sum of both metabolites
d) identification by HPLC only, no other unambiguous confirmation by HPTLC (= characterised peak)
e) position of conjugation was unassigned in the study with the phenyl-label, but a retrospective identification followed in the goat study by spectroscopic evidence ([redacted], E.; [redacted]; Hund [redacted], K.; 2003, amended 2005; KCA, 6.2.2/02.)

Table 6.2.2- 3 Parent compound and metabolites in eggs, tissues and organs of laying hens after application of phenyl- and triazole-labelled prothioconazole (in mg a.s. equiv./kg)

Label	Liver		Eggs		Muscle		Fat	
	phenyl	triazole	phenyl	triazole	phenyl	triazole	phenyl	triazole
prothioconazole (JAU 6476)	0.995	1.085	0.001	0.002	0.010	0.003	0.137	0.042
Metabolites common to both labels								
JAU 6476-S-methyl *(M01)*	0.090	0.059	0.001	0.001	0.006	0.003	0.008	0.003
JAU 6476-N-glucuronide *(M05)*	0.043[d]	0.006			0.001[d]			
JAU 6476-S-glucuronide *(M06)*[e]	0.479[a]	0.526[a]	0.006	0.012[b]	0.014	0.012	0.021	0.011
JAU 6476-4-hydroxy *(M08)*	0.029[d]	0.011			0.001[d]			
JAU 6476-hydroxy-glucuronide *(M10)*	0.103							
JAU 6476-dihydroxy-diene *(M40)*[d]	0.035[d]				0.001[d]			
JAU 6476-desthio *(M04)*	0.167	0.172	0.001	0.003	0.006	0.003	0.136	0.008
JAU 6476-desthio-4-hydroxy *(M15)*	0.109	0.031	0.001					
JAU 6476-desthio-hydroxy-sulfate *(M84)*	0.120	0.055						
JAU 6476-desthio-3,4-dihydroxy-dienyl-glucuronide *(M52)*	0.092				0.001[d]			
JAU 6476-desthio-hydroxy-methoxy-sulfate *(M82)*, JAU 6476-desthio-dihydroxy-sulfate *(M83)* and JAU 6476-desthio-hydroxy-sulfate *(M84)*	0.014	0.419			0.001			0.002
Metabolites specific to the triazole label								
1,2,4-triazole *(M13)*		0.037		0.006		0.023		0.004[c]
JAU 6476-triazolyl-ethanol *(M45)*		0.129		0.006		0.035		0.005
Thiocyanate *(M80)*		0.023		0.005		0.005		0.004[c]
Total identified	2.480	2.554	0.015	0.036	0.037	0.083	0.379	0.239
Total characterised	0.471	0.425	0.001	0.005	0.010	0.014	-	0.017

a) may contain a trace amount of JAU 6476-desthio-3-hydroxy *(M14)*
b) may contain a minor amount of JAU 6476-desthio-4-hydroxy *(M15)* and trace amount of JAU 6476-desthio-3-hydroxy *(M14)*
c) for the fat the values of 1,2,4-triazole *(M13)* and thiocyanate *(M80)* could only be determined as the sum of both metabolites
d) identification by HPLC only, or no unambiguous confirmation by HPTLC (= characterised peak)
e) position of conjugation was not assigned in the study with the phenyl label, but a retrospective identification followed in the goat study by spectroscopic evidence ([redacted]; [redacted] H. and [redacted] E.; 2003, amended 2005; KCA, 6.2.2/02)

The consolidated metabolic routes of prothioconzole combining the results of both studies are characterised by the following reactions:

- Conjugation of parent compound with glucuronic acid either at the sulfur (high significance) or at the nitrogen atom (low significance) of the triazolinethione moiety
- Methylation of the triazolinethione moiety
- Elimination of sulfur from the triazolinethione moiety
- Mono- and dihydroxylation of the chlorophenyl moiety of parent compound and of metabolite JAU 6476-desthio;
  conjugation with sulfate in case of the hydroxylated metabolites of JAU 6476-desthio; conjugation with glucuronic acid in case of the hydroxylated parent compound
- Methylation of JAU 6476-desthio-dihydroxy *(M34)* to JAU 6476-desthio-hydroxy-methoxy *(M28)* (of low significance and was only postulated since a sulfate conjugate of JAU 6476-desthio-hydroxy-methoxy *(M82)* was detected)
- De-aromatisation of the chlorophenyl moiety of the parent compound by oxidative hydroxylation was also detected to a minor extent; followed by glucuronidation
- Cleavage of the aliphatic carbon bond yielding the later specific metabolites 1,2,4-triazole *(M13)* and JAU 6476-triazolyl-ethanol *(M45)*
- Cleavage of the triazolinethione moiety resulting in the formation of thiocyanate (M80)

In conclusion, the metabolic pathway of prothioconazole in laying hens follows two main branches:

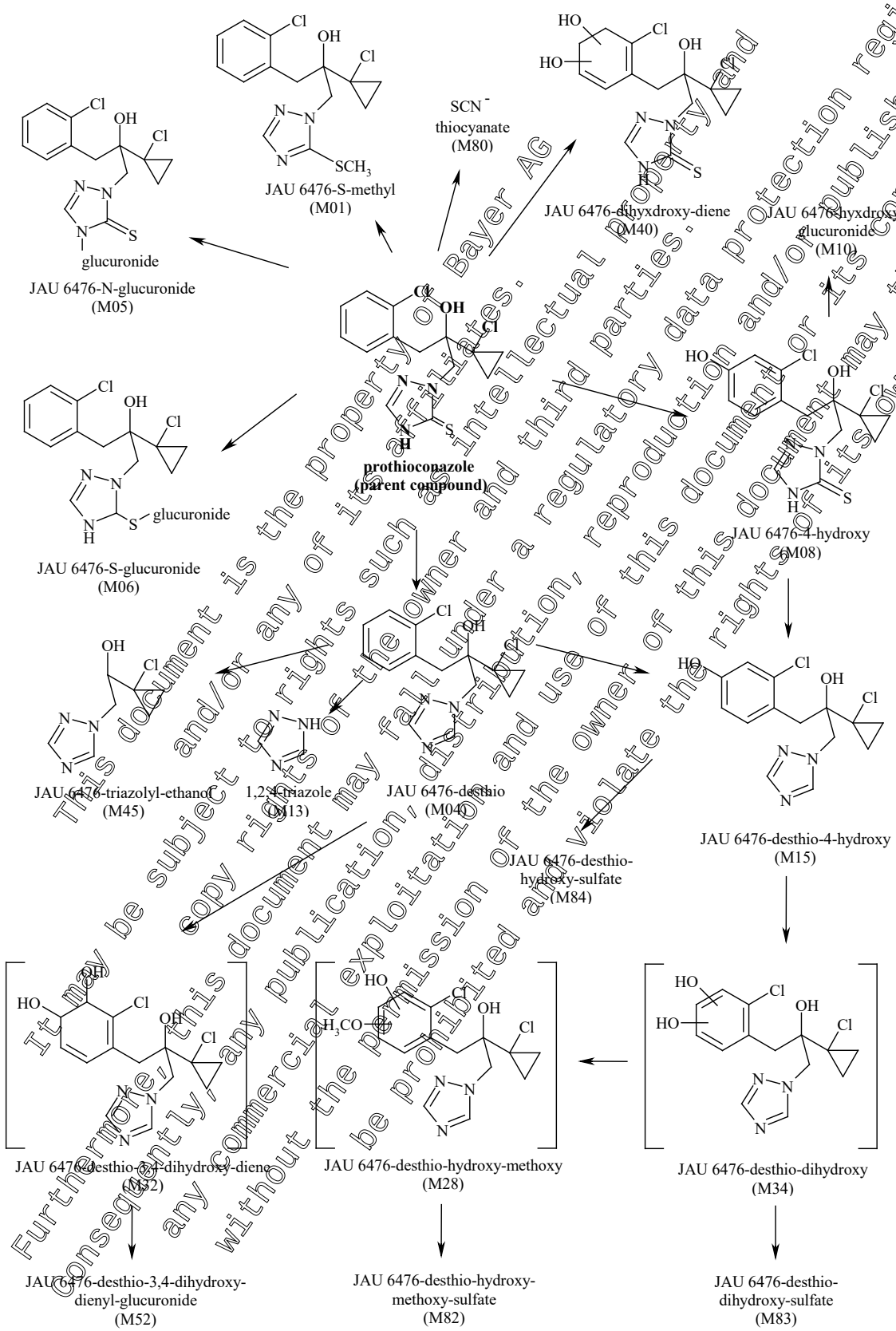
- metabolic transformations of the parent compound under conservation of the triazolinethione moiety
- metabolic transformations after elimination of the sulfur from the triazolinethione moiety

Metabolic routes starting from the parent compound under conservation of the triazolinethione moiety are mainly focused on this triazolinethione moiety. Conjugation with glucuronic acid, methylation or cleavage of this moiety are observed to a significant extent. Hydroxylation and oxidation of the chlorophenyl moiety is of minor significance.

Elimination of the sulfur from the triazolinethione moiety and formation of metabolite JAU 6476-desthio *(M04)* is the starting point of the second branch of the metabolic pathway. Metabolic routes via JAU 6476-desthio *(M04)* are mainly based on the hydroxylation of the chlorophenyl moiety, followed by methylation, oxidation and to a major extent by subsequent conjugation with glucuronic acid or sulfate. Additionally, elimination of the chlorophenyl moiety is observed resulting in the metabolites JAU 6476-triazolyl ethanol *(M45)* and 1,2,4-triazole *(M13)*.

According to the detected metabolites and considering their concentrations in the various matrices, it can be concluded that the metabolic routes deriving directly from the intact parent compound are the dominating ones. The results of these studies provide a comprehensive understanding of the metabolism of prothioconazole in laying hens. The metabolic degradation of prothioconazole is summarised in Figure 6.2.1-1.

Figure 6.2.2- 1 Proposed metabolic pathway of phenyl- and triazole-labelled prothioconazole in laying hens



compounds in brackets [ ] = postulated intermediates

CA 6.2.3 Lactating ruminants

Four studies to address this point were presented in the dossier submitted for first inclusion in Annex I (March 2002) and were deemed acceptable following evaluation and peer review at EU level (2007).

Report:	KCA 6.2.3/01 [redacted],; [redacted]; 2001; M-034900-01-2
Title:	[Phenyl-UL-14C]JAU6476 - Absorption, distribution, excretion and metabolism in the lactating goat
Report No.:	MR-092/01
Document No.:	M-034900-01-2
Guideline(s):	US EPA OPPTS 860.1300; Canadian PMRA Ref.: DACO 6.2; EU 91/414/EEC amended by 96/68/EC
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	[redacted]8; [redacted]; [redacted]; [redacted]; 2005; M-116219-02-1
Title:	[Triazole-UL-14C]JAU 6476: Absorption, distribution, excretion, and metabolism in the lactating goat
Report No.:	MR-448/02
Document No.:	M-116219-02-1
Guideline(s):	US EPA OPPTS 860.1300; Canadian PMRA Ref.: DACO 6.2; EU 91/414/EEC amended by 96/68/EC; Japanese MAFF, 12 Nousan 8147
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	[redacted]n; [redacted]; [redacted]; [redacted]; 2002; M-041101-01-2
Title:	[Phenyl-UL-14C]JAU6476-desthio - Absorption, distribution, excretion, and metabolism in the lactating goat
Report No.:	MR-091/01
Document No.:	M-041101-01-2
Guideline(s):	US EPA OPPTS 860.1300; Canadian PMRA Ref.: DACO 6.2; EU 91/414/EEC amended by 96/68/EC
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	[redacted]d; [redacted]; 2006; M-279178-01-1
Title:	[Phenyl-UL-14C]JAU 6476-desthio: Absorption, distribution, excretion and metabolism in the lactating goat - Subsequent identification of metabolite hydrolysis products
Report No.:	MEF-06/469
Document No.:	M-279178-01-1
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

For details of data submitted previously please refer also to the Baseline dossier CA 6.2. For completeness, a summary of the key parameters of these previously submitted studies and an overall summary of the four metabolism studies on prothioconazole and prothioconazole-desthio in the lactating goat is provided below.

A recently conducted goat metabolism study employing [triazole-UL-^{14}C]prothioconazole-desthio, which was not previously submitted, is summarised in detail after the overall summary of the four studies described before.

Report	Administered compound	Label	Dosing	Time of sacrifice	Number of animals
CA 6.2.3/01 [redacted], E.; [redacted] H., [redacted], K.; 2001 M-034900-01-2	Prothioconazole	Phenyl	3 x 10 mg/kg bw/day	5 h after last dose	1
CA 6.2.3/02 [redacted], E.; [redacted] H., [redacted], K.; 2003, amended 2005 M-116219-02-1	Prothioconazole	Triazole	3 x 10 mg/kg bw/day	5 h after last dose	1
CA 6.2.3/03 [redacted], H.; [redacted] E., [redacted], K.; 2002 M-041101-01-2	Prothioconazole-desthio	Phenyl	3 x 10 mg/kg bw/day	5 h after last dose	1
CA 6.2.3/04 [redacted] E.; 2006 M-279178-01-1	Prothioconazole-desthio	Phenyl	3 x 10 mg/kg bw/day	5 h after last dose	1

Overall summary of the metabolism of prothioconazole and its metabolite prothioconazole-desthio *(M04)* in lactating ruminants

Livestock metabolism studies using phenyl- and triazole-labelled prothioconazole and phenyl-labelled prothioconazole-desthio (SXX 0665; *M04*) were conducted in the lactating goat as a model for ruminants.

The metabolism study with phenyl-labelled JAU 6476-desthio was conducted since the dominating residue in livestock feed items is the desthio-derivative of prothioconazole.

All studies followed the same design. A lactating goat was three times orally dosed with the radiolabelled test substance (prothioconazole or JAU 6476-desthio) at a dose level of 10 mg/kg body weight. The goats received the dose on three consecutive days in intervals of 24 hours. The animals were sacrificed 5 hours after the last administration.

The kinetics and the metabolism of prothioconazole were very well comparable in the lactating goat studies performed with the two different labels. The kinetics were characterised by a fast and almost complete absorption process of the administered radioactivity, followed by fast excretion. The predominant portion of the totally administered dose was found in the excreta. Renal excretion was preferred. Only trace amounts of the totally administered radioactivity were secreted with the milk (≤ 0.03% of the dose) and detected in edible tissues and organs (0.7% to 1.0%).

Also for the study performed with JAU 6476-desthio a fast and almost complete absorption process of the administered radioactivity was observed, followed by rapid distribution and a fast elimination phase, with a major amount of radioactivity in the urine. Only trace amounts of the totally administered radioactivity were secreted with the milk (0.05% of the dose) and detected in edible tissues and organs (1.9%).

The total radioactive residues were also well comparable for the studies performed with prothioconazole. Slightly higher concentrations were detected in milk, liver, muscle and fat in the study with the triazole-label.

Table 6.2.3- 1 Total radioactive residues (TRR) determined in milk and tissues of lactating goats after administration of prothioconazole

JAU 6476	Milk		Liver		Muscle		Kidney		Fat	
Label	phenyl	triazole	phenyl	triazole	phenyl	triazole	phenyl	triazole	phenyl	triazole
TRR [mg/kg]	0.037	0.150	6.092	6.248	0.088	0.117	6.762	4.507	0.[illegible]	0.174

The total radioactive residues in the study performed with JAU 6476-desthio were higher than in the studies performed with prothioconazole. Significantly higher concentrations were detected in the metabolising organs liver and kidney.

Table 6.2.3- 2 Total radioactive residues (TRR) determined in milk and tissues of lactating goats after administration of metabolite JAU 6476-desthio (*M04*)

M04	Milk	Liver	Muscle	Kidney	Fat
Label	phenyl	phenyl	phenyl	phenyl	phenyl
TRR [mg/kg]	0.286	18.021	0.266	18.975	0.234

Since cleavage of the molecule was not very pronounced in the studies with prothioconazole, the detected metabolites and their concentrations in the different matrices corresponded very well in the studies with the phenyl and the triazole label. Major compounds (> 10% of TRR) detected in the majority of the matrices were the parent compound and the metabolite JAU 6476-glucuronide (*M06*). JAU 6476-desthio (*M04*) was only prominent in fat and JAU 6476-4-hydroxy (*M08*) in liver.

The key metabolite in all matrices, except for fat, was JAU 6476-S-glucuronide (*M06*). Due to the conjugation with glucuronic acid, the sulfur was obviously protected against cleavage. Thus, the metabolic route via JAU 6476-desthio (*M04*) was impeded. JAU 6476-desthio (*M04*) and all its derivatives accounted in each sample matrix for less than 20% of the TRR, indicating that the major metabolic routes are based on the intact parent compound.

Thiocyanate (*M80*) was the only label specific metabolite detected and was also formed directly from the intact parent compound. It was a major metabolite in milk, muscle and fat in the study performed with triazole-labelled prothioconazole. A corresponding counterpart was not detected in the study using the phenyl-label.

Thiocyanate is not of toxicological concern. It is well known as the main detoxification product after cyanide exposure[2]. It is also a physiologic component of the lactoperoxidase-thiocyanate-H_2O_2-system and is therefore a natural component in raw milk[3, 4]. The natural content in milk can be much higher than the amounts found in the presented studies. Moreover, FAO and WHO recommended to add and adjust the naturally occurring substances thiocyanate and hydrogen peroxidase of the lactoperoxidase (LPS) system in raw milk for conservation purposes. The method of activating LPS in raw milk is based

2 [redacted]. (1980) in: Spencer, P.S. and Schaumburg, H.H. Experimental and Clinical Neurotoxicology, Williams & Wilkins, Baltimore/London; pp 617 - 618

3 [redacted] (1976) Nonspecific bactericidialactivity of the lactoperoxidases-thiocyanate-hydrogen peroxide system of milk against Escherichia coli and some gram-negative pathogens; Infection and Immunity, 13 (3); pp.800 - 807

4 [redacted] (2000) Thiocyanate mediatedantifungal and antibacterial property of goat milk lactoperoxidase; Life Science 66 (25), pp. 2433 - 2439

5 [redacted]. (1997) Natural thiocyanate content and optimum conditions for activation of lactoperoxidase system in raw buffalo milk; [redacted] Sci. 25 (2); pp.241 - 252

on increasingly insufficient levels of thiocyanate present in milk up to about 15 ppm and subsequently adding an equimolar amount of hydrogen peroxide[6].

A comparison of metabolites detected in the milk, tissue and organs of the lactating goat after administration of prothioconazole (phenyl- and triazole-label) is provided in Table 6.2.3- 3 and Table 6.2.3- 4.

Table 6.2.3- 3 Parent compound and metabolites in milk and tissues of the lactating goat administered with [phenyl-UL-^{14}C]- and [triazole-UL^{14}C]prothioconazole (in % of TRR)

Compound	Milk		Liver		Muscle		Kidney		Fat	
	% TRR		% TRR		% TRR		% TRR		% TRR	
Label	phen.	triaz.	phen.	triaz.	phen.	triaz.	phen.	triaz.	phen.	triaz.
TRR (mg/kg)	0.037	0.150	6.092	6.248	0.088	0.113	6.767	4.567	0.169	0.174
prothioconazole *(a.s.)*	0.9	3.2	12.9	16.8	13.1	7.2	18.0	19.5	13.3	16.1
Metabolites common to both labels										
-S-methyl (*M01*)				0.6						
-N-glucuronide *(M05)*	1.3		3.8	4.6	1.1		2.6	3.4	0.7	
-S-glucuronide *(M06)*	12.0	4.4	10.0	6.1	14.7	15.6	35.3	32.9	10.1	11.9
-4-hydroxy (*M08*)		3.3	1.5	1.0	1.3		3.6			8.3
-hydroxy-glucuronide (*M10*)		3.6	3.1	1.0	3.4	4.7	7.4	6.1	3.2	11.2
-4-hydroxy-glucuronide (*M69*)			2.4		2.0	2.5	4.0	3.6	2.5	
-hydroxy-sulfate (*M79*)				6.5						
-lactoside *(M78)*			4.4							
-desthio *(M04)*	2.8	1.4	1.2	4.4	3.0	0.9	1.3	3.0	19.0	15.1
-desthio-4-hydroxy (*M15*)			0.9							
group of M38, M40, M49, M74, M91 (dienes, glucuronides)	10.1									
group of M38, M52 (dienes, glucuronides)			16.4							
Metabolites specific to the triazole label										
thiocyanate *(M80)*		44.1		12.0	29.6			9.0		12.4
Total identified	29.2	61.3	53.6		44.5		70.8		52.4	
Total characterised	38.9	11.0	13.0		12.0		7.1		8.5	

M38 JAU 6476-desthio-dihydroxy-diene
M40 JAU 6476-dihydroxy-diene
M49 JAU 6476-desthio-hydroxy-methoxy-glucuronide
M52 JAU 6476-desthio-3,4-dihydroxy-phenyl-glucuronide
M74 JAU 6476-desthio-4-hydroxy-glucuronide
M91 JAU 6476-desthio-3,4-dihydroxy-glucuronide

6 FAO/WHO (1991) Codex Alimentarius Commission; 19th Session of the Codex Alimentarius Commission, Rome, 1-10 July 1991. Alinorm 91/13

Table 6.2.3- 4 Parent compound and metabolites in milk and tissues of the lactating goats administered with [phenyl-UL-^{14}C]- and [triazole-UL^{14}C]prothioconazole (in mg a.s. equivalents/kg)

Compound	Milk		Liver		Muscle		Kidney		Fat	
Label	mg/kg phen.	triaz.	mg/kg phen.	triaz.	mg/kg phen.	triaz.	mg/kg phen.	triaz.	mg/kg phen.	triaz.
TRR (mg/kg)	0.037	0.150	6.092	6.248	0.088	0.117	6.702	4.507	0.169	0.174
prothioconazole	<0.001	0.005	0.788	1.047	0.012	0.008	0.215	0.879	0.022	0.028
Metabolites common to both labels										
-S-methyl *(M01)*				0.038						
-N-glucuronide *(M05)*	<0.001		0.170	0.284	0.001		0.179	0.155	0.001	
-S-glucuronide *(M06)*	0.004	0.007	0.610	0.879	0.013	0.016	2.321	1.526	0.017	0.025
-4-hydroxy *(M08)*		0.005	0.092	0.686		0.006		0.164		0.014
-hydroxy-glucuronide *(M10)*		0.005	0.307	0.315	0.001	0.005	0.503	0.275	0.005	0.019
-4-hydroxy-glucuronide (*M69*)			0.046		0.002	0.004	0.271	0.250	0.004	
-hydroxy-sulfate *(M79)*				0.408						
-lactoside *(M78)*		0.007								
-desthio *(M04)*	0.001	0.002	0.076	0.309	0.003	0.001	0.087	0.132	0.032	0.026
-desthio-4-hydroxy *(M15)*			0.092	0.179						
group of M38, M40, M49, M74, M91 (dienes, glucuronides)	0.004									
group of M38, M52 (dienes, glucuronides)			0.392							
Metabolites specific to the triazole label										
thiocyanate *(M80)*		0.061		0.125		0.007		0.406		0.022
Total identified	0.041	0.092	3.265	3.953	0.034	0.075	4.786	3.790	0.089	0.131
Total characterised	0.015	0.016	0.839	1.793	0.011	0.013	0.482	0.363	0.014	

M38 JAU 6476-desthio-dihydroxy-diene
M40 JAU 6476-dihydroxy-diene
M49 JAU 6476-desthio-hydroxy-methoxy-glucuronide
M52 JAU 6476-desthio-3,4-dihydroxy-dienyl-glucuronide
M74 JAU 6476-desthio-4-hydroxy-glucuronide
M91 JAU 6476-desthio-3,4-dihydroxy-glucuronide (the dihydroxylation positions were determined in an additional study [redacted] E.; 2006; KCA 6.2.2.4)

The main metabolites derived directly from the parent compound. The main metabolic routes can be summarized as follows:

- Conjugation of the parent compound with glucuronic acid at the sulfur (high importance) or the nitrogen atom (low importance) of the triazolinethione moiety
- Cleavage of the triazolinethione moiety of the parent compound resulting in metabolite thiocyanate (*M80*)
- Mono-hydroxylation of the chlorophenyl moiety of the intact parent compound, followed by conjugation with glucuronic acid or sulfate
- Elimination of sulfur from the triazolinethione moiety

As described above, conjugation of prothioconazole with glucuronic acid at the sulfur atom of the triazolinethione moiety was the dominating metabolic reaction. Due to this conjugation, the metabolic routes via JAU 6476-desthio (*M04*) became less important. Nevertheless, JAU 6476-desthio was formed and several metabolic routes can be derived from this metabolite.

The most important metabolic reactions with JAU 6476-desthio *(M04)* as precursor were:

- Mono- and dihydroxylation of the chlorophenyl moiety of JAU 6476-desthio; conjugation with glucuronic acid was observed to a high extent.

A further minor metabolic route which left the triazolinethione moiety intact was the methylation of this moiety. The resulting metabolite JAU 6476-S-methyl (*M01*) was detected only in very low amounts in liver. Conjugation of the parent compound with lactose was detected only in milk. Both metabolites were only detected in the study performed with triazole-labelled prothioconazole.

Additional minor metabolic routes with metabolite JAU 6476-desthio as precursor were the formation of JAU 6476-desthio-hydroxy-methoxy (*M28*) and JAU 6476-desthio-glucuronide (*M71*). JAU 6476-desthio-hydroxy-methoxy was detected only in faeces as glucuronic acid conjugate. Oxidation of the chlorophenyl moiety of the parent compound or of JAU 6476-desthio *(M04)* was also of minor importance and led partly to de-aromatisation. As a consequence, several diene compounds were detected. To some extent, these dienes were conjugated with glucuronic acid.

In the study performed with JAU 6476-desthio *(M04)*, no single main metabolite or key metabolite was detected covering all matrices. Besides the test substance, mainly conjugates of hydroxylated JAU 6476-desthio were identified. Since the metabolic patterns in the various matrices were quite complex, an additional way for the characterisation of metabolites or metabolic groups was established by treating the extracts with boiling hydrochloric acid. The aim of the hydrolysis was to cleave conjugates and to convert non-aromatic compounds back into aromatic compounds with known structures. As a result, up to five relevant marker compounds were formed during acidic treatment. The majority of the metabolites could be traced back to the following compounds: JAU 6476-desthio (*M04*), JAU 6476-desthio-3-hydroxy (*M14*), JAU 6476-desthio-4-hydroxy (*M15*), JAU 6476-desthio-3,4-dihydroxy (*M33*), JAU 6476-desthio-3,5-dihydroxy (*M35*) and JAU 6476-desthio-glucuronide (*M71*). JAU 6476-desthio-glucuronide is an O-glucuronide which is not completely cleaved under the conditions applied.

The hydroxylation positions of JAU 6476-desthio-3,4-dihydroxy and of its corresponding glucuronic acid conjugate were assigned unambiguously in an additional study ([redacted] E., 2006; KCA, 6.2.2/04). The metabolites described as JAU 6476-desthio-dihydroxy *(M33)* and JAU 6476-desthio-dihydroxy-glucuronide (*M72*) in all livestock studies were assigned retrospectively to JAU 6476-desthio-3,4-dihydroxy *(M33)* and JAU 6476-desthio-3,4-dihydroxy-glucuronide *(M91)*.

A comparison of metabolites detected in the milk, tissue and organs of the lactating goat after administration of phenyl-labelled prothioconazole and phenyl-labelled JAU 6476-desthio is provided in Table 6.2.2/5 and Table 6.2.3- 6.

Table 6.2.3- 5 Parent compound and metabolites in milk and tissues of the lactating goat administered with phenyl-labelled prothioconazole and phenyl-labelled JAU 6476-desthio *(M04)* (in % of TRR)

Compound	Milk		Liver		Muscle		Kidney		Fat	
-	a.s.	M04	a.s.	M04	a.s.	M04	a.s.	M04	a.s.	M04
TRR (mg/kg)	0.037	0.286	6.092	8.421	0.088	0.266	6.762	18.975	0.169	0.231
	% TRR		% TRR		% TRR		% TRR		% TRR	
Prothioconazole	0.9		12.9		13.4		18.0		13.3	
-N-glucuronide *(M05)*	1.3		2.8		1.1		2.6		0.8	
-S-glucuronide *(M06)*	12.0		10.0		14.8		34.3		10.1	
-4-hydroxy *(M08)*			1.5							
-hydroxy-glucuronide *(M10)*			2.4		21.0		4.0		2.5	
-4-hydroxy-glucuronide *(M69)*			2.4		21.0		4.0		2.5	
-desthio *(M04)*	2.8		1.2	3.2	3.0	1.8	1.3	7.7	19.0	13.9
-desthio-glucuronide *(M71)*		6.2				3.6[3]		24.1		4.2[4]
-desthio-3-hydroxy *(M14)*				1.0		4.8		1.1		
-desthio-4-hydroxy *(M15)*				8.4		3.1		3.1		14.6
-desthio-4-hydroxy-glucuronide *(M74)*		5.1		2.8		1.8		7.3		4.7
-desthio-3,4-dihydroxy *(M33)*[1]		1.6		2.15		1.72				5.36
-desthio-4,5-dihydroxy *(M35)*		1.4		4.8[2]		2.8				
-desthio-3,4-dihydroxy-diene *(M32)*		2.4		1.2[5]		3.6		1.6		4.3
-desthio-3,4-dihydroxy-dienyl-glucuronide *(M52)*		4.4		1.8		20.9		21.0		22.9
-desthio-hydroxy-methoxy-glucuronide *(M49)*		0.0				5.2				
group of M86, M87, M91 (glucuronides)		3.6		4.7				4.9		5.3
group of M82, M83, M84 (sulfate conjugates)		44.0								
group of M38, M40, M49, M74, M91 (dienes, glucuronides)		4.4		2.4						
group of M38, M42 (dienes, glucuronides)		10.1								
Total identified	29.2	58.1	53.6	53.2	44.7	49.4	70.8	58.0	52.4	60.8
Total characterised	38.4	30.4	11.9	17.7	12.0	27.6	7.1	28.4	8.5	23.3

1) assigned as JAU 6476-desthio-dihydroxy in this report, identified as JAU 6476-desthio-3,4-dihydroxy in a separate study ([redacted] E.; 2006; KCA 6.2.2/04)
2) co-elution with JAU 6476-desthio-glucuronide possible
3) co-elution with JAU 6476-desthio-dihydroxy-glucuronide and JAU 6476-desthio-hydroxy-glucuronide possible
4) co-elution with JAU 6476-desthio-hydroxy-methoxy-glucuronide possible
5) co-elution with JAU 6476-desthio-4,5-dihydroxy-dienyl-glucuronide possible

M38 JAU 6476-desthio-dihydroxy-diene
M42 JAU 6476-dihydroxy-diene
M52 JAU 6476-desthio-3,4-dihydroxy-dienyl-glucuronide
M82 JAU 6476-desthio-hydroxy-methoxy-sulfate
M83 JAU 6476-desthio-dihydroxy-sulfate
M84 JAU 6476-desthio-hydroxy-sulfate
M86 JAU 6476-desthio-4,5-dihydroxy-gluc.
M87 JAU 6476-desthio-3-hydroxy-gluc.
M91 JAU 6476-desthio-3,4-dihydroxy-gluc.

Table 6.2.3- 6 Parent compound and metabolites in milk and tissues of the lactating goat administered with phenyl-labelled prothioconazole and phenyl-labelled JAU 6476-desthio *(M04)* (in mg test item equivalents/kg)

Compound	Milk		Liver		Muscle		Kidney		Fat	
-	a.s.	M04	a.s.	M04	a.s.	M04	a.s.	M04	a.s.	M04
TRR (mg/kg)	0.037	0.286	6.092	8.421	0.088	0.266	6.762	18.975	0.169	0.231
	mg/kg		mg/kg		mg/kg		mg/kg		mg/kg	
Prothioconazole	<0.001		0.788		0.012		1.215		0.022	
-N-glucuronide *(M05)*	<0.001		0.170		0.001		0.179		0.001	
-S-glucuronide *(M06)*	0.004		0.610		0.013		2.321		0.017	
-4-hydroxy *(M08)*			0.092							
-hydroxy-glucuronide *(M10)*			0.3[illegible]		0.005		0.503		0.005	
-4-hydroxy-glucuronide *(M69)*			0.146		0.002		0.271		0.004	
-desthio *(M04)*	0.001		0.074	5.721	0.003	0.185	0.087	5.454	0.032	0.032
-desthio-glucuronide *(M71)*		0.015				0.009[3)]		4.567		0.010[4)]
-desthio-3-hydroxy *(M14)*				0.178		0.013		0.231		
-desthio-4-hydroxy *(M15)*			0.093	1.542		0.056		0.970		0.034
-desthio-4-hydroxy-glucuronide *(M74)*		0.01[illegible]		0.[illegible]		0.016		0.388		0.011
-desthio-3,4-dihydroxy *(M33)*[1)]		0.004		0.396		0.00[illegible]				0.012
-desthio-4,5-dihydroxy *(M35)*		0.00[illegible]		0.878[2)]		0.007				
-desthio-3,4-dihydroxy-diene *(M32)*		0.007		0.213[5)]		0.00[illegible]		0.307		0.010
-desthio-3,4-dihydroxy-dienyl-glucuronide *(M52)*		0.016		1.089		0.056		3.992		0.053
-desthio-hydroxy-methoxy-glucuronide *(M49)*						0.00[illegible]				
group of M06, M10, M69 (glucuronides)		0.008		0.004				0.933		0.012
group of M82, M83, M84 (sulfate conjugates)		0.126		0.006						
group of M38, M40, M47, M74, M91 (dienes, glucuronides)	0.004									
group of *M38*, *M52* (dienes, glucuronides)				0.3[illegible]						
Total identified	0.011	0.166	2.265	9.805	0.040	0.131	4.786	1.013	0.089	0.141
Total characterised	0.015	0.087	0.832	3.251	0.011	0.074	0.482	5.378	0.014	0.054

1) assigned as JAU 6476-desthio-dihydroxy in the report, identified as JAU 6476-desthio-3,4-dihydroxy in a separate study ([redacted] 2006, KCA, 6.2.2/04)
2) co-elution with JAU 6476-desthio-glucuronide possible
3) co-elution with JAU 6476-desthio-dihydroxy-glucuronide and JAU 6476-desthio-hydroxy-glucuronide possible
4) co-elution with JAU 6476-desthio-hydroxy-methoxy-glucuronide possible
5) co-elution with JAU 6476-desthio-4,5-dihydroxy-dienyl-glucuronide possible

M38 JAU 6476-desthio-dihydroxy-diene
M40 JAU 6476-dihydroxy-diene
M47 JAU 6476-desthio-tri-dihydroxy-dienyl-glucuronide
M82 JAU 6476-desthio-hydroxy-methoxy-sulfate
M83 JAU 6476-desthio-dihydroxy-sulfate
M84 JAU 6476-desthio-hydroxy-sulfate
M86 JAU 6476-desthio-4,5-dihydroxy-gluc.
M87 JAU 6476-desthio-3-hydroxy-gluc.
M91 JAU 6476-desthio-3,4-dihydroxy-gluc.

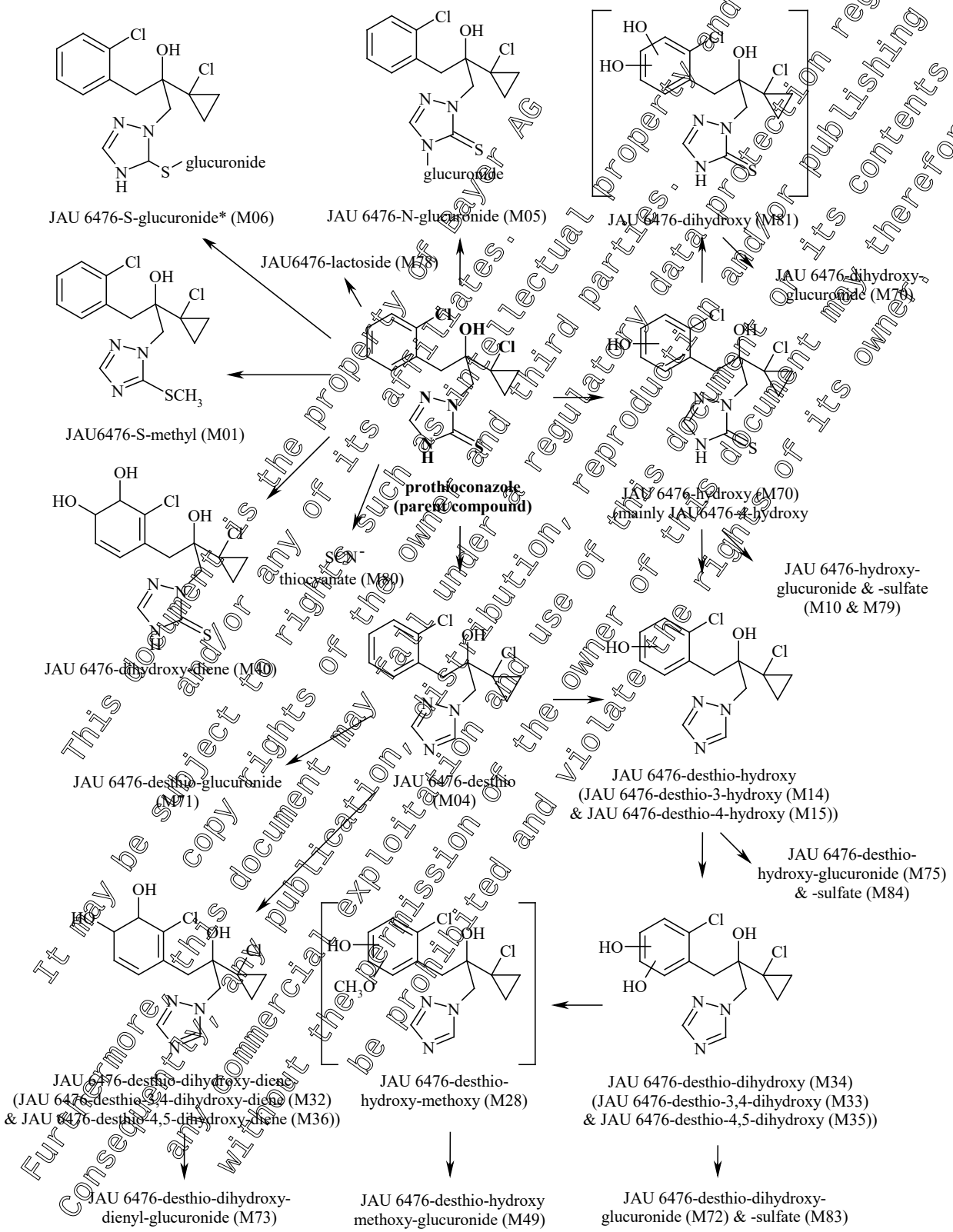
The metabolic pathway of JAU 6476-desthio *(M04)* in the lactating goat was characterised by the following main reactions, which were already observed as minor metabolic routes in the studies performed with prothioconazole as test compound.

- Conjugation with glucuronic acid forming the O-conjugate of JAU 6476-desthio.
- Hydroxylation resulting in the formation of the isomers JAU 6476-desthio-3-hydroxy (*M14*) and JAU 6476-desthio-4-hydroxy (*M15*) followed by conjugation with glucuronic acid and sulfate
- Further hydroxylation of the chlorophenyl moiety leading to two isomers of JAU 6476-desthio-dihydroxy followed by conjugation with glucuronic acid and sulfate.
- Oxidation of the chlorophenyl moiety of the JAU 6476-desthio-hydroxy metabolites leading to de-aromatisation. As a consequence, JAU 6476-desthio-3,4- (*M32)* and JAU 6476-desthio-4,5-dihydroxy-diene (*M36*) were formed. The oxidation can be followed by conjugation with glucuronic acid.

There was also some evidence for the occurrence of JAU 6476-desthio-hydroxy-methoxy (*M28*) as an intermediate prior to conjugation with glucuronic acid.

A proposed, consolidated pathway of prothioconazole, including also all metabolic routes with metabolite JAU 6476-desthio *(M04)* as precursor is shown in Figure 6.2.3-1.

Figure 6.2.3- 1 Proposed metabolic pathway of prothioconazole in the lactating goat (consolidated results of the studies using phenyl and triazole-labelled prothioconazole as well as phenyl-labelled JAU 6476-deshio (*M04*)).



In addition to the goat metabolism studies summerised above (KCA, 6.2.2 /01-04) a metabolism study with prothioconazole-desthio labelled in the [triazole-UL-^{14}C]-position (JAU 6476-desthio, *M04*) was conducted. This study is described in detail below.

Report:	KCA 6.2.3/06 [redacted],; [redacted]; [redacted]; 2011; M-404996-02-1
Title:	[Triazole-UL-14C]JAU 6476-desthio: Metabolism in the lactating goat
Report No.:	MEF-11/011
Document No.:	M-404996-02-1
Guideline(s):	OECD 503; US EPA OPPTS 860.1300; Health Canada PMRA Ref.: DACO 6.2; EU 91/414/EEC amended by 96/68/EC, Appendix F
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Executive Summary

Prothioconazole-desthio is the main crop metabolite of the triazolinethione fungicide prothioconazole. Since the metabolite was detected in significant amounts in livestock feed items, the kinetic behaviour and metabolism of JAU 6476-desthio was investigated in the lactating goat as a model for ruminants. The goat was orally dosed five times in 24 h intervals with 10 mg [triazole-UL-^{14}C]JAU 6476-desthio per kg body weight per day (corresponding to 224.28 mg a.s./kg dry feed/day) and sacrificed about five hours after the last dose. Total radioactivity was measured time-dependently in milk and excreta, and in liver, kidney, muscle and fat at sacrifice. Milk, edible organs and tissues and urine were analysed for prothioconazole-desthio and metabolites.

Recovery

The overall recovery was 70.56 % of the total dose administered. The missing amount of approx. 30% at sacrifice may have been still present in the gastro-intestinal tract. Up to the time of sacrifice, the excretion accounted for 69.71 % of the total dose. A high portion of 44.40 % was found in the urine and 25.31 % in the faeces. The cumulative urinary and faecal excretion was characterised by a more or less linear increase during the whole testing period.

The time course of the radioactivity concentrations from the evening and morning milk pool samples after each dosing showed a clear diurnal pattern. The residues increased significantly during the eight hour period after each dosing followed by a decrease to approx. the original level measured prior to the delivery of the next dose. A plateau was reached at about 24 to 32 hours after the first dosing.

Total radioactive residues in milk and in organs and tissues

The TRR-values in milk samples ranged from 0.109 to 0.224 mg/kg. The highest value was detected 56 hours after the first administration. In terms of amounts, 0.04% of the total dose was found in the milk during the whole testing period. Pooled samples of morning milk (TRR: 0.126 mg/kg) and evening milk (TRR: 0.208 mg/kg) were extracted for metabolism investigations. At sacrifice, the highest concentrations were measured in liver (14.850 mg/kg) and kidney (6.618 mg/kg), which shows the significance of these organs for metabolic conversion and excretion. In relation to the totally administered dose, these values corresponded to 0.544% and 0.039%, respectively. The concentrations in the different types of muscle and fat were significantly lower and ranged from 0.247 mg/kg to 0.331 mg/kg. The radioactivity concentrations in the total body muscle and total body fat were calculated assuming that 30% or 12% of the body weight were represented by muscle and fat, respectively. The

residue concentrations accounted for 0.267 mg/kg (muscle) and 0.306 mg/kg (fat), corresponding to 0.153% and 0.070% of the totally administered dose.

Metabolism

For identification and quantification of parent compound and metabolites, milk, organs and tissues were extracted consecutively with acetonitrile/water mixtures and in the case of fat additionally with heptane. The post-extraction solids of muscle, liver and kidney were additionally submitted to exhaustive extraction steps. In total, approximately 74% to 96% of the TRR was extracted, the residues remaining in milk and the edible tissues muscle and fat were below 0.05 mg/kg.

All extracts were degreased and the resulting phases were analysed by HPLC or TLC using radio-detection. Label specific metabolites were isolated and identified by HPLC and/or TLC co-chromatography with authentic reference compounds or by direct HPLC comparison. Non-label specific metabolites were identified based on the metabolite assignments conducted in the goat metabolism study using [phenyl-UL-^{14}C]JAU 6476-desthio. Corresponding metabolite profiles of each matrix were compared before and after treatment of the final aqueous extracts with hydrochloric acid. The hydrolysis step was performed to reduce the huge number of the detected non-label specific compounds to five common moiety products covering the major part of the TRR.

All findings in the present study corresponded very well with those of the study performed with [phenyl-UL-^{14}C]JAU 6476-desthio. The following amounts of JAU 6476-desthio and metabolites - expressed as % of the total radioactive residue and as equivalent concentration (mg/kg) - were found in milk and in edible tissues of the lactating goat following conventional extraction:

	Morning milk		Evening milk	
	% TRR	mg/kg	% TRR	mg/kg
JAU 6476-desthio	7.2	0.009	0.7	0.002
1,2,4-triazole	32.6	0.041	21.6	0.045
-triazolyl-ethanol	5.0	0.006	10.6	0.022
-3,4-dihydroxy-dienyl-glucuronide (D1)	2.3	0.003	4.6	0.010
-3,4-dihydroxy-dienyl-glucuronide (D2)	2.8	0.004	3.5	0.007
-3,4-dihydroxy-diene (D1)	2.6	0.003	3.1	0.006
-3,4-dihydroxy-diene (D2)	---	---	6.7	0.014
-3-hydroxy-glucuronide/ -3,4-dihydroxy-glucuronide/ -4,5-dihydroxy-glucuronide	7.2	0.009	5.2	0.011
-glucuronide	2.9	0.004	7.6	0.016
-4-hydroxy-glucuronide	2.3	0.004	7.1	0.015
-3,4-dihydroxy	2.2	0.003	2.4	0.005
-4,5-dihydroxy	8.5	0.011	1.2	0.003
-4-hydroxy	2.4	0.003	1.5	0.003
Total identified	71.8	0.090	75.8	0.158
Total characterised	20.5	0.026	17.0	0.035
Total extracted	92.3	0.116	93.4	0.194

TRR (mg/kg)	Muscle		Fat		Liver		Kidney	
	0.267		0.306		14.850		6.618	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
JAU 6476-desthio	1.7	0.005	12.9	0.040	25.9	3.849	7.6	0.504
1,2,4-triazole	17.0	0.045	6.2[1)]	0.019[1)]	1.1[2)]	0.162[2)]	9.3[1)]	0.616[1)]
-triazolyl-ethanol	8.3	0.022	3.8	0.011	3.9	0.583	3.5	0.232
-3,4-dihydroxy-dienyl-glucuronide (D1)	3.3	0.009	5.8	0.018	1.2	0.172	5.7	0.377
-3,4-dihydroxy-dienyl-glucuronide (D2)	5.0	0.013	9.4	0.029	3.1	0.463	9.0	0.595
-3,4-dihydroxy-diene (D1)	2.9	0.008	1.3	0.004	1.5	0.223	1.3	0.084
-3,4-dihydroxy-diene (D2)	5.5	0.015	6.6	0.020	1.0	0.148	1.1	0.071
-3-hydroxy-glucuronide/ -3,4-dihydroxy-glucuronide/ -4,5-dihydroxy-glucuronide	2.0	0.005	1.9	0.006	4.9	0.728	5.4	0.355
-glucuronide	1.4	0.004	3.7	0.011	3.5	0.520	2.8	0.186
-4-hydroxy-glucuronide	0.7	0.002	1.9	0.006	6.9	1.024	18.0	1.193
-hydroxy-methoxy-glucuronide	0.6	0.001	1.3	0.004	0.5	0.067	1.0	0.067
-3,4-dihydroxy	0.4	0.001	---	---	---	---	---	---
-4,5-dihydroxy	1.4	0.004	2.4	0.007	0.7	0.107	0.7	0.048
-3-hydroxy	9.3	0.025	18.4	0.056	5.6	0.825	9.7	0.648
-4-hydroxy	59.3	0.158	75.7	0.232	58.7	8.710	75.2	4.977
Total identified	20.6	0.055	12.9	0.039	13.4	1.989	21.2	1.404
Total characterised	81.8	0.218	96.0	0.294	73.6	10.928	96.4	6.454
Total extracted								

[1]) = coeluting with triazolyl alanine
2) = no unamiguous identification, fraction was related to the characterized compounds

All main metabolic degradation pathways identified in the goat metabolism study with [phenyl-UL-^{14}C]JAU 6476-desthio were confirmed in the present study with [triazole-UL-^{14}C]JAU 6476-desthio. Additionally, cleavage of the molecule was detected resulting in the label specific metabolites 1,2,4-triazole, triazolyl alanine and JAU 6476-desthio-triazolylethanol.

Thus, the main metabolic reactions of [triazole-UL-^{14}C]JAU 6476-desthio identified in the lactating goat were:

- mono- and dihydroxylation of the chlorophenyl moiety
- dearomatisation of the chlorophenyl moiety by oxidative hydroxylation
- subsequent conjugation of the hydroxylated metabolites with glucuronic acid
- conjugation of the test compound with glucuronic acid
- cleavage of the test compound resulting in 1,2,4-triazole and JAU 6476-desthiotriazolyl-ethanol
- conjugation of 1,2,4-triazole with serine

I. Material and Methods

A. Materials

1. Test Material: [Triazole-UL-^{14}C]JAU 6476-desthio

Specific radioactivity	4.24 MBq/mg (114.6 µCi/mg)
Synthesis code	KATH 6179
Radiochemical purity	> 99% by radio-HPLC
Chemical purity	> 99% by HPLC (UV detection at 210 nm)
CAS No.	120983-64-4

For administration, one part of the test substance was radiodiluted with 14 parts of non-radiolabelled test substance. The resulting specific radioactivity in the administration suspension was 0.397 MBq/mg (10.7 µCi/mg). The radiolabelled test substance proved to be stable in the 0.5% aqueous tragacanth suspensions for at least 4 h at room temperature, as shown by radio-HPLC analysis. The evaluation of the chromatograms resulted in a radiochemical purity of >99% in all cases.

2. Test Animals

Species	Lactating goat (*capra hircus*)
Strain	"Bunte Deutsche Edelziege"
Breeding facility	[redacted]
Sex and numbers involved	Female, one animal
Age	19 months
Body weight	42 kg at first administration, 40.13 kg at sacrifice
Acclimatisation	9 days
Identification	Skin markings, individual cage card
Housing	during the period of acclimation, the animal was kept in a raised stall with a metal grid as base and straw and hay as bedding. - one day prior to the start of the study and during the whole duration of the test, the animal was kept in an electro-polished stainless steel metabolism cage for farm animals allowing for an almost separate and quantitative collection of urine and faeces. The cage was equipped with a variable restraining device and was supplied by [redacted]
Environmental conditions	Air-conditioned rooms Temperature: 22 - 28 °C Relative humidity: 47 - 75% Light: 12 / 12 hours light / dark cycle Air change: 10 - 15 times per hour.
Feed and water	During the whole residence time, the goat was fed *ad libitum* with hay, apples and supplementary ruminant feed. The feed consumption was recorded by back-weighing during the experiment. Tap water was offered ad libitum; water specification were in accordance to the local drinking water regulations.

B. Study Design and Methods

1. Dosing regime

Administration	oral
Dose rate	10 mg/kg bw/day
Feed consumption	ca. 4.46% of body weight, corresponding to 224.28 mg a.s./kg dry feed/day in the diet
Vehicle	0.5% aqueous tragacanth suspension
Timing	once daily
Duration	5 days

2. Sample collection

Milk collection	twice daily
Urine and faeces collection	once daily
Interval from last dose to sacrifice	ca. 5 hours
Tissues collected at sacrifice and analysed	liver (without gall bladder), kidney (whole organ), three different types of muscle (round, flank, loin), three different types of fat (perirenal, omental, subcutaneous), gall bladder for optional analysis

3. Storage stability of samples

All conventional and exhaustive extraction experiments and subsequent analyses of the resulting extracts from milk, muscle, fat, liver and kidney were performed within approximately 3 months after sacrifice. An exception was the exhaustive extraction of the solids of muscle remaining after conventional extraction. These solids were extracted ca. 11 months after sacrifice. Analysis of the extracts was not possible since only minor radioactivity amounts were released and the concentrated sample was too cloudy for HPLC-injection.

Direct analyses of the conventional extracts by HPLC or TLC were performed within 1 to 6 days after sample preparation. Hydrolysis of the final aqueous extracts with hydrochloric acid to simplify the metabolite pattern was performed 2 to 11 weeks after sample preparation. HPLC analysis of the purified hydrolysed samples followed ca. 4 to 5 days later. Thus, quantification of test compound and metabolites was performed latest ca. 3 months after sacrifice (corresponding to approx. 2.5 months after sample preparation). All investigations on metabolite identification were also performed within three months after sacrifice.

Hence, investigations on storage stability of the residues in the samples were not necessary. It can be concluded that the metabolic profiles represent the residues in the matrices and analysed samples at sacrifice.

4. Extraction of residues

All sample materials were extracted with acetonitrile/water mixtures and in the case of fat additionally with heptane (conventional solvent extraction). Exhaustive extraction steps (microwave extraction at slightly increased temperature) followed in case of muscle, liver and kidney due to an insufficient extraction efficiency after conventional solvent extraction (< 90% of the TRR was extracted). Degreasing of the conventional solvent extracts was performed by solid phase extraction (SPE) using a C18-cartridge in the case of milk or by liquid/liquid partitioning with n-hexane in the case of organs and tissues. The aqueous phases of the SPE and the partitioning steps were concentrated and analysed by radio-HPLC for metabolite profiling. The hexane phases after partitioning were analysed by TLC for metabolite profiling, if the radioactivity in the phase exceeded 10% of the TRR.

5. Identification of metabolites

Metabolites common to both radiolabels were identified based on the metabolite assignments provided in the goat metabolism study with [phenyl-UL-^{14}C]JAU 6476-desthio (KCA, 6.2.2 /03 and KCA, 6.2.2 /04). In this study, identification of metabolites was achieved by structure elucidation of isolated and purified urine metabolites using HPLC-MS/MS and NMR spectroscopy. The isolated and identified metabolites were used as reference compounds for HPLC and HPTLC co-chromatography or comparison when analysing the extracts of milk and tissues in the study described here. In all extracts, the presence of major metabolites was confirmed by two independent chromatographic methods. However, the metabolic patterns in all matrices were quite complex and several minor metabolites (conjugates) were present. Therefore, an additional experiment for characterisation and identification of metabolites or metabolic groups was performed: The final aqueous extracts of all matrices were treated with boiling hydrochloric acid. The aim of the hydrolysis was to cleave conjugates and to re-convert non-aromatic metabolites back into aromatic compounds with known structures. In fact, besides some minor components, five relevant compounds (two isomers of JAU6476-desthiodihydroxy, two isomers of JAU6476-desthio-hydroxy and the test compound JAU6476-desthio) were formed due to the hydrolytic treatment. Based on this additional information, a large number of minor metabolites (partly combined in metabolic groups) could be traced back to a few basic structures. This approach was also utilised in the residue analytical method for the determination of JAU 6476-desthio residues in animal matrices

II. Results and Discussion

A. Recovery and distribution of residues

The overall recovery including excreta, milk, organs and tissues amounted to 70.56% of the totally administered dose. Considering the short survival period after the last dose and the low quantities of radioactivity determined or estimated in body muscle and fat, a significant portion of radioactivity (about 30% of the total dose) was assumed to be still present in the gastro-intestinal tract of the animal after sacrifice.

The total excretion amounted to 69.71% of the total dose. 44.40% thereof was excreted with the urine and 25.31% with the faeces. An extremely low amount (0.04% of the total dose) was secreted with the milk. Only trace amounts (0.81%) were found in the edible organs and tissues (Table 6.2.3- 7).

Table 6.2.3- 7 Recovery of radioactivity after oral administration of 10 mg/kg b.w. triazole-labelled JAU 6476-desthio (M04) to a lactating goat on five consecutive days.

	Time after the first dose [h]	Administration no.	% of the totally administered radioactivity
Urine (plus urine funnel rinsing)	0 24 48 72 96 101	1 2 3 4 5 sacrifice	------- 9.48 10.51 10.65 11.07 2.69
subtotal			44.40
Faeces	0 24 48 72 96 101	1 2 3 4 5 sacrifice	----- 4.31 6.26 6.88 6.54 1.31
subtotal			25.31
Milk	0 8 24 32 48 56 72 80 96 101	1 2 3 4 5 sacrifice	------ 0.003 0.005 0.005 0.005 0.005 0.006 0.006 0.005 0.004
subtotal			0.044
Totally excreted			69.75
Estimated residue in edible tissues			0.81
Recovery			70.56

The radioactivity levels measured in the milk samples are recorded inTable 6.2.3- 8. Concentrations of 0.192 µg/mL and 0.222 µg/mL were measured in the milk 8 hours after the first and second dosage, respectively. These concentrations declined from 8 to 24 hours after the first and second administration to values of 0.109 µg/mL and 0.125 µg/mL, respectively. This finding indicates that there is no risk of a significant bioaccumulation of residues in the milk after repeated dosage. The highest concentration of 0.224 µg/mL was measured at 56 h after administration. In terms of amounts, a very low fraction of 0.044% of the total dose was found in the milk during the whole test period.

Table 6.2.3- 8 Radioactivity in the milk of the lactating goat after oral administration of 10 mg/kg b.w. triazole-labelled JAU 6476-desthio to a lactating goat on five consecutive days.

Time after the first dosage [h]	Dosage no.	Concentration [µg a.s. equiv./mL]	% of the RA secreted per fraction	% of the RA (cumulative)
0	1	-------	--------	-------
8		0.192	0.003	0.003
24[1)]		0.109	0.005	0.008
32	2	0.222	0.005	0.013
48[1)]		0.125	0.005	0.018
56	3	0.224	0.005	0.023
72[1)]		0.130	0.006	0.029
80	4	0.202	0.006	0.035
96[1)]		0.142	0.005	0.040
101	5	0.197	0.004	0.044

1) immediately prior to administration

The mean recoveries of the totally administered radioactivity in edible organs and tissues along with the corresponding concentrations are summarised in Table 6.2.3- 9. The levels of residues were all clearly below 1.0% of the dose for each organ and tissue. The highest concentration was measured in the liver (14.850 mg/kg), followed by that obtained for the kidney (6.618 mg/kg). This result reflects the significance of these organs for excretion and metabolism of the compound. These concentrations were followed in decreasing order in omental fat (0.331 mg/kg), subcutaneous fat (0.323 mg/kg), round muscle (0.270 mg/kg), perirenal fat (0.266 mg/kg), flank muscle (0.258 mg/kg) and loin muscle (0.247 mg/kg).

Table 6.2.3- 9 Residual radioactivity in the edible organs and tissues of a lactating goat after oral administration of 10 mg/kg b.w. triazole-labelled JAU 6476-desthio on five consecutive days.

Tissue/organ	Concentration [mg a.s. equiv./kg]	% of the RA totally administered
Liver	14.850	0.544
Kidney	6.618	0.039
Round muscle (sample)	0.270	--------
Flank muscle (sample)	0.258	--------
Loin muscle (sample)	0.247	--------
Total body muscle[1)]	0.267[2)]	0.153
Perirenal fat (sample)	0.266	--------
Subcutaneous fat (sample)	0.323	--------
Omental fat (sample)	0.331	--------
Total body fat[1)]	0.306[2)]	0.070
Calculated/estimated residue in the edible tissues/organs		0.806

1) calculated from the body weight, assuming 30% and 12% of the body weight for total body muscle and total body fat, respectively
2) weighted mean equivalent concentrations of the three different types of muscle or fat

B. Extraction efficiency, quantification and identification of metabolites

For the identification of the test compound and metabolites, milk, organs and tissues were extracted with acetonitrile/water mixtures and in the case of fat additionally with heptane. These conventional solvent extraction steps at ambient temperature released between 74% to 96% of the total radioactive residue (TRR). To increase the extraction efficiencies in muscle, liver and kidney, the remaining solids were additionally submitted to exhaustive extraction steps (two microwave extraction steps at 60°C). Exhaustive extraction of muscle and liver released only small additional amounts of radioactivity (1.8%), whereas 10.6% of the TRR was exhaustively extracted for kidney. As a result more than 80%

of the TRR was extracted from all matrices, except liver. For liver, the total extraction efficiency accounted to ca. 74% of the TRR.

The conventional extracts of milk, organs and tissues were degreased by solid phase extraction (SPE) or liquid-liquid extraction (LLE). The resulting final aqueous extracts were analysed by radio-HPLC. Hexane phases resulting from LLE were analysed by TLC, if the radioactivity in the phase exceeded 10% of the TRR.

The label specific metabolites 1,2,4-triazole and JAU 6476-desthio-triazolyl-ethanol were identified in each matrix, triazolyl alanine was only identified in fat and kidney. JAU 6476-desthio-triazolyl-ethanol was identified by HPLC comparison.

Identification of the test compound and non-label specific metabolites was performed by comparing the metabolite patterns with those obtained in the corresponding goat metabolism study with [phenyl-UL-^{14}C]JAU 6476-desthio (KCA, 6.2.2 /03 and KCA, 6.2.2 /04). Generally, the metabolite patterns corresponded quite well for each matrix, although the concentrations of the metabolites differed in some cases significantly. However, it has to be considered that the goat received two additional doses of the test substance in the present study and that the vast majority of the identified metabolites were conjugates which could have been cleaved to some extent during sample preparation and analysis.

A successful approach to overcome this difference was to convert the multitude of compounds in the extracts into a few aglycons by an acidic hydrolysis step as described in B.5. HPLC analysis of the hydrolysed and purified extracts showed simplified patterns of only a few major compounds. These major compounds were identified by HPLC comparison with the corresponding hydrolysed profiles obtained in the goat metabolism study with [phenyl-UL-^{14}C]JAU 6476-desthio (KCA, 6.2.2 /03 and KCA, 6.2.2 /04) and additionally by HPLC comparison with authentic reference compounds. In doing so, the assignments made in the non-hydrolysed aqueous extracts were additionally supported by the aglycons identified in the hydrolysed aqueous extracts.

1. Urine

The urine sample collected within 24 hours after the first administration was the basis for the identification of metabolites in the goat metabolism study with [phenyl-UL-^{14}C]JAU 6476-desthio (KCA, 6.2.2 /03 and KCA, 6.2.2 /04). Therefore, urine samples were also analysed in the present study. Comparison of the urine samples collected within 24 hours after the first administration showed a very good consistence of the HPLC fingerprints. Thus it was demonstrated that the less polar – and therefore non-label specific part of the JAU 6476-desthio residues corresponded very well. The label specific metabolites represented only a minor portion of the urine residues. Two polar regions were isolated from the urine sample collected from 96 to 101 hours after the first administration and were analysed with a zwitterionic stationary phase. Comparison with authentic reference compounds revealed the presence of 1,2,4-triazole (eluting in the void volume) and other metabolites, which could not be identified unambiguously on the basis of the available reference compounds. Probably, triazolyl acetic acid was one component of the polar region. Another potential component is metabolite JAU 6476-desthio-triazolyl-ethanol, a label-specific metabolite identified in the laying hen metabolism study performed with [triazolyl-UL-^{14}C]JAU 6476 (KCA, 6.2.2 /02).

2. Evening milk

A total of 93.4% of the TRR was extracted from the evening milk in three subsequent steps. The extracts from the first two extraction steps were combined and purified by SPE for HPLC analysis. The quantification of the metabolites is presented in Table 6.2.3- 10.

All metabolites identified accounted for about 76% of the TRR (0.158 mg/kg) and all characterised compounds for about 17% (0.035 mg/kg). The main components were 1,2,4-triazole (22% of the TRR, 0.045 mg/kg) and JAU 6476-desthio-triazolyl-ethanol (11%, 0.022 mg/kg). All other metabolites identified were non-specific to the triazole label and corresponded very well with the metabolites detected in the goat metabolism study with [phenyl-UL-^{14}C]JAU6476-desthio (KCA, 6.2.2/03 and 6.6.2/04). Each of them ranged from approx. 1% to 8% of the TRR. Seven minor compounds were characterised by their chromatographic behaviour. None of them exceeded 6% of the TRR (approx. 0.01 mg/kg).

3. Morning milk

A total of 92.3% of the TRR was extracted from the morning milk in three subsequent steps. All extracts were combined and purified by SPE for HPLC analysis. The the quantification of the metabolites is presented in Table 6.2.3- 10.

All metabolites identified accounted for about 72% of the TRR (0.090 mg/kg) and all characterised compounds for about 21% (0.026 mg/kg) of the morning milk pool. By far the main compound detected was 1,2,4-triazole (approx. 33% of the TRR, 0.041 mg/kg) followed by JAU 6476-desthio-4,5-dihydroxy which accounted for about 9% of the TRR. All other components ranged from 2% and 7% of the TRR. None of the characterised compounds exceeded 6% of the TRR (approx. 0.01 mg/kg).

Table 6.2.3- 10 Summary of characterisation and identification of metabolites in the milk of the lactating goat after oral administration of 10 mg/kg b.w. [triazole-UL-^{14}C]JAU 6476-desthio on five consecutive days.

	Morning milk		Evening milk	
	% TRR	mg/kg	% TRR	mg/kg
JAU 6476-desthio	7.2	0.009	0.7	0.002
1,2,4-triazole	32.6	0.041	21.6	0.045
-triazolyl-ethanol	5.0	0.006	10.6	0.022
-3,4-dihydroxy-dienyl-glucuronide (D1)	2.3	0.003	4.6	0.010
-3,4-dihydroxy-dienyl-glucuronide (D2)	2.8	0.004	3.5	0.007
-3,4-dihydroxy-diene (D1)	2.6	0.003	3.1	0.006
-3,4-dihydroxy-diene (D2)	---	---	6.7	0.014
-3-hydroxy-glucuronide/ -3,4-dihydroxy-glucuronide/ -4,5-dihydroxy-glucuronide	7.2	0.009	5.2	0.011
-glucuronide	2.9	0.004	7.6	0.016
-4-hydroxy-glucuronide	3.3	0.004	7.1	0.015
-3,4-dihydroxy	2.2	0.003	2.4	0.005
-4,5-dihydroxy	8.5	0.011	1.2	0.003
-4-hydroxy	2.4	0.003	1.5	0.003
Total identified	71.8	0.090	75.8	0.158
Total characterised	20.5	0.026	17.0	0.035
Total extracted	92.3	0.116	93.4	0.194

4. Muscle

From the muscle pool, 79.9% of the TRR was released by conventional solvent extraction. The combined extracts were degreased by liquid/liquid partitioning, resulting in an aqueous and a hexane phase. An aliquot of the aqueous extract was analysed by HPLC, whereas the hexane phase was subjected to TLC.

Extraction efficiency was moderate after conventional solvent extraction (79.9%) and was not improved significantly by exhaustive extraction under microwave assistance which released only in the first of two extraction steps an additional small portion of 1.8% of the TRR (0.005 mg/kg). An analysis of the microwave extract was impossible since the resulting extract contained extremely low residues but a high matrix burden. The residue level in the post-extraction solids was below 0.050 mg/kg.

As for the milk samples, a first assignment of compounds and an indication of label specific compounds was possible after comparing the fingerprint of the metabolite profile of the aqueous extract of muscle with that obtained in the study performed with [phenyl-UL-^{14}C]JAU 6476-desthio (KCA, 6.2.2 /03 and KCA, 6.2.2 /04). In total, 81.8% of the TRR was extractable from muscle, 59.3% was identified and 20.6% was characterised. The main compounds identified were 1,2,4-triazole (approx. 17% of the TRR, 0.045 mg/kg) and JAU 6476-desthio-triazolyl-ethanol (approx. 8%, 0.022 mg/kg). All other components ranged from < 1% to 7% of the TRR. Approximately two thirds of the characterised TRR was detected in the aqueous extract and ca. one third in the final hexane extract after partitioning. Both extracts were analysed by HPLC or TLC. Thus, the unknown metabolites or fractions were well characterised by their partitioning and chromatographic retention behaviour. The quantification of the identified metabolites is compiled in Table 6.2.3- 11.

5. Fat

The fat pool was extracted consecutively with an acetonitrile/water mixture after addition of heptane to dissolve the fatty matrix. In total, 96.0% of the TRR was extracted that way. Approximately 7% (< 0.03 mg/kg) was partitioned into the heptane phase, leaving ca. 87% in the combined acetonitrile/water extracts. Due to an additional degreasing step of the combined and concentrated acetonitrile/water extracts with hexane, two phases for analysis resulted. An aliquot of the degreased aqueous extract was analysed by HPLC, whereas the hexane phase was analysed by TLC.

As for the muscle sample, a first assignment of compounds and an indication of label-specific compounds was possible after comparing the fingerprint of the metabolite profile of the final aqueous extract of fat with that obtained in the study performed with [phenyl-UL-^{14}-C]JAU 6476-desthio (KCA, 6.2.2 /03 and KCA, 6.2.2 /04). Direct comparison of the HPLC profiles showed a very good correspondence. In contrast to the metabolite profiles of milk and the other tissues and organs, the label-specific compounds were not predominant in fat.

Additionally, the assignment of metabolites was confirmed after acidic treatment of the aqueous extract. The majority of the detected compounds was converted to the common moiety products JAU 6476-desthio-3-hydroxy, JAU 6476-desthio-4-hydroxy, JAU 6476-desthio-3,4-dihydroxy and parent compound prothioconazole. HPLC comparison with authentic reference compounds provided the basis for the identification of the common moiety products in the present study besides the comparison of the metabolite profiles of the hydrolysed extracts.

In total, 96.0% of the TRR was extractable from fat. A portion of 75.7% was identified and 12.9% was characterised by the partitioning and/or chromatographic retention behaviour of the unknown compounds or fractions. Approximately 12% of the TRR was assigned to five unknown compounds, the largest constituent accounting for ca. 0.01 mg/kg. Approximately 7% of the TRR was recovered in the heptane phase obtained during conventional solvent extraction. This phase was not analysed but it can be assumed that it contained the test compound and JAU 6476-desthio-4-hydroxy as the hexane phase which was obtained after partitioning of the concentrated aqueous extract in the degreasing step. The residue level in the unextractable solids was low and accounted for 0.012 mg/kg. The quantification of the identified metabolites is compiled in Table 6.2.3- 11.

6. Liver

In total, 73.6% of the TRR was extracted from the liver sample, conventional extraction released approx. 72% and subsequent extraction under microwave assistance only ca. 2%. The combined conventional extracts were degreased by liquid/liquid partitioning, resulting in an aqueous and a hexane phase. An aliquot of the aqueous extract was analysed directly by HPLC. The hexane phase was not analysed since only < 2% of the TRR partitioned into that phase. It was assumed that – in analogy to the hexane phases analysed for muscle, fat and kidney – JAU 6476-desthio and at least one of the monohydroxylated metabolites (most probably JAU 6476-desthio-4-hydroxy) was present. The microwave extracts were combined and analysed by HPLC as well.

As for the other matrices, a first assignment of compounds and an indication of label specific compounds was possible after comparing the fingerprint of the metabolite profile of the aqueous extract of liver with that obtained in the study performed with [phenyl-UL-^{14}C]JAU 6476-desthio (KCA, 6.2.2 /03 and KCA, 6.2.2 /04). Comparison of the metabolite profiles showed a good correspondence - the non-label specific metabolites were dominant while the label specific fraction was insignificant (< 5% of the TRR).

A confirmation of the assignments for these non-label specific metabolites was possible after acidic treatment of the final aqueous extract. The majority of the compounds was converted to the common moiety products JAU 6476-desthio-3,4-dihydroxy, JAU 6476-desthio-4,5-dihydroxy, JAU 6476-desthio-3-hydroxy, JAU 6476-desthio-4-hydroxy and JAU 6476-desthio, as also shown for the hydrolysed liver extract in the goat metabolism study performed with [phenyl-UL-^{14}C]JAU 6476-desthio (KCA, 6.2.2 /03 and KCA, 6.2.2 /04). The metabolite JAU 6476-desthio-glucuronide was assigned based on its HPLC retention time in HPLC chromatograms before and after acidic treatment.

In total, a portion of 58.7% of the TRR was identified in liver and 13.4% was characterised by the partitioning and chromatographic retention behaviour of the unknown compounds or fractions. A significant portion of the TRR (ca. 26%) was bound in the post-extraction solids even after exhaustive extraction steps, as often observed for liver samples. The quantitative data of the analysis of the conventional and the microwave extracts of liver are compiled in Table 6.2.3- 11.

7. Kidney

In total, 96.4% of the TRR was extracted from the kidney sample, conventional extraction released ca. 86% and subsequent extraction under microwave assistance an additional 11%. The combined conventional extracts were degreased by liquid/liquid partitioning, resulting in an aqueous and a hexane phase. An aliquot of the aqueous extract was analysed directly by HPLC, whereas the hexane phase was analysed by TLC.

A first assignment of compounds and an indication of label specific compounds was performed by comparing metabolite profiles. Direct comparison of the HPLC profile of the aqueous extract of the kidney sample of the present study with that of the study performed with [phenyl-UL-^{14}C]JAU 6476-desthio (KCA, 6.2.2 /03 and KCA, 6.2.2 /04) revealed a very good correspondence of the fingerprints. The metabolite patterns were almost identical. The label-specific portion was of minor importance.

Assignment of the non-label specific compounds in the aqueous extract of kidney was performed based on the metabolite profiles obtained in the goat metabolism with [phenyl-UL-^{14}C]JAU 6476-desthio as mentioned above. One isomer of the glucuronic acid conjugate of the hydroxylated test substance was the main metabolite detected, followed by the isomeric glucuronic acid conjugates of JAU 6476-desthio-3,4-diene and JAU 6476-desthio-4-hydroxy. The unchanged test compound, which was a major compound in the metabolite profile of the goat study performed with [phenyl-UL-^{14}C]JAU 6476-desthio, was detected as main compound in the hexane phase of the present study. Thus, adding the

metabolite profiles obtained after conventional extraction, an excellent correspondence was observed between the studies with both radiolabels.

The assignments for the non-label specific metabolites were additionally confirmed after acidic treatment of the aqueous extract. The hydrolytic fingerprints showed very good consistence with the fingerprint detected in the study with [phenyl-UL-^{14}C]JAU 6476-desthio (KCA, 6.2.2 /03 and KCA, 6.2.2 /04). The majority of the compounds was converted to the common moiety products JAU 6476-desthio-3,4-dihydroxy, JAU 6476-desthio-4,5-dihydroxy, JAU 6476-desthio-3-hydroxy, JAU 6476-desthio-4-hydroxy and JAU 6476-desthio, which accounted in total for nearly 50% of the TRR. Additionally, minor amounts of JAU 6476-desthio-glucuronide were detected. This metabolite was a major compound detected in the study with [phenyl-UL-^{14}C]JAU 6476-desthio, even after acidic treatment.

Extraction efficiency was quite good after conventional solvent extraction (85.9% of the TRR), but was additionally improved by exhaustive extraction under microwave assistance. Two exhaustive extraction steps at increased temperature (60°C) released an additional portion of ca. 11% (0.704 mg/kg). The combined microwave extracts were concentrated and analysed by HPLC. One predominant polar fraction was detected, which was eluting in the void volume. This fraction was isolated and analysed by TLC. Co-chromatography with 1,2,4-triazole, triazolyl acetic acid and triazolyl alanine confirmed the presence of triazolyl alanine (main compound) and traces of 1,2,4-triazole.

In total, 75.2% of the TRR was identified in kidney and 21.2% was characterised by the partitioning and the chromatographic retention behaviour of the unknown compounds or fractions. The two biggest unknown compounds accounted for approx. 4% and 6% of the TRR, all other unknowns were below 2.5%. The quantitative data for kidney are summarised in Table 6.2.3-11.

Table 6.2.3- 11 Summary of characterisation and identification of metabolites in the edible tissues and organs of the lactating goat after oral administration of 10 mg/kg b.w. [triazole-UL-^{14}C]JAU 6476-desthio on five consecutive days.

TRR (mg/kg)	Muscle		Fat		Liver		Kidney	
	0.267		0.306		14.850		6.618	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
JAU 6476-desthio	1.7	0.005	12.9	0.040	25.9	3.849	7.6	0.504
1,2,4-triazole	17.0	0.045	6.2[1)]	0.019[1)]	1.1[2)]	0.162[2)]	9.3[1)]	0.616
-triazolyl-ethanol	8.3	0.022	3.8	0.011	3.9	0.583	3.5	0.232
-3,4-dihydroxy-dienyl-glucuronide (D1)	3.3	0.009	5.8	0.018	1.2	0.172	5.7	0.377
-3,4-dihydroxy-dienyl-glucuronide (D2)	5.0	0.013	9.4	0.029	3.1	0.463	9.0	0.595
-3,4-dihydroxy-diene (D1)	2.9	0.008	1.3	0.004	1.5	0.223	1.3	0.084
-3,4-dihydroxy-diene (D2)	5.5	0.015	6.6	0.020	1.0	0.148	1.1	0.071
-3-hydroxy-glucuronide/ -3,4-dihydroxy-glucuronide/ -4,5-dihydroxy-glucuronide	2.0	0.005	1.9	0.006	4.9	0.728	5.4	0.355
-glucuronide -4-hydroxy-glucuronide	1.4	0.004	3.7	0.011	3.5	0.520	2.8	0.186
-hydroxy-methoxy-glucuronide	0.7	0.002	1.9	0.006	6.9	1.024	18.0	1.193
-3,4-dihydroxy	0.6	0.001	1.3	0.004	0.8	0.067	1.0	0.067
-4,5-dihydroxy	0.4	0.001	---	---	---	---	---	---
-3-hydroxy	1.4	0.004	2.4	0.007	0.7	0.107	0.7	0.048
-4-hydroxy	9.3	0.025	18.4	0.056	5.6	0.825	9.7	0.648
Total identified	59.3	0.158	75.7	0.232	58.7	8.710	75.2	4.977
Total characterised	20.6	0.055	12.9	0.039	13.4	1.989	21.2	1.404
Total extracted	81.8	0.218	96.0	0.294	73.6	10.928	96.4	6.454

[1]) = coeluting with triazolyl alanine
2) = no unamiguous identification, fraction was related to the characterized compounds

C. Biotransformation Pathway

The present goat metabolism study performed with [triazole-UL-^{14}C]JAU 6476-desthio confirmed the metabolic reactions identified in the goat metabolism study with [phenyl-UL-^{14}C]JAU 6476-desthio. Additionally, cleavage of the test substance was detected yielding the metabolites 1,2,4-triazole, triazolyl alanine and JAU 6476-desthio-triazolyl-ethanol.

The main metabolic reactions of [triazole-UL-^{14}C]JAU 6476-desthio identified in the lactating goat were:

- mono- and dihydroxylation of the chlorophenyl moiety
- dearomatisation of the chlorophenyl moiety by oxidative hydroxylation
- subsequent conjugation of the hydroxylated metabolites with glucuronic acid
- conjugation of the test compound with glucuronic acid
- cleavage of the test compound resulting in JAU 6476-desthio-triazolyl-ethanol
- cleavage of the test compound and loss of 1,2,4-triazole
- subsequent conjugation of 1,2,4-triazole with serine to triazolyl alanine.

The proposed biotransformation pathway is shown in Figure 6.2.3- 2.

D. Conclusion

A lactating goat was orally dosed on five consecutive days in time intervals of 24 hours with [triazole-UL-^{14}C]JAU6476-desthio at a daily dose level of 10 mg/kg body weight, corresponding to 224 mg/kg in the feed. The goat was sacrificed 5 hours after the final administration, and the edible tissues and organs as well as the milk collected during the experimental phase were analysed for residues. Identification of test compound and metabolites was performed in each matrix. The kinetic behaviour was derived from the excretion parameters and the secretion of the residues in milk.

The kinetic and metabolic behaviour of [triazole-UL-^{14}C]JAU 6476-desthio in the lactating goat can be characterised by the following observations:

- A significant amount of the administered radioactivity (ca. 70%) was excreted via urine and faeces. The cumulative urinary and faecal excretion was characterised by a linear increase during investigation.
- Each dose was almost completely absorbed from the gastrointestinal tract.
- A very low amount of 0.04% of the administered dose was secreted with the milk.
- The total radioactive residues in milk showed a diurnal pattern. A plateau level was reached between 24 and 32 hours after the first dosing, which was indicated by the decline of TRR-values prior to the delivery of the next dose to about the same level measured on the previous day. These findings showed that the radioactivity is not accumulating in the milk after repeated administration.
- At sacrifice, the residue levels in milk, as well as in tissues were significantly lower in relation to the daily dose level with exception of the residue levels in the metabolising organs kidney and liver. Kidney showed a concentration of 6.618 mg/kg while in the liver a distinctly higher concentration of 14.850 mg/kg was observed. The residue concentrations in the different types of muscle and fat as well as in milk were in the narrow range of 0.1 - 0.3 mg/kg.

It is important to note that

- an exaggerated dose level of 224 mg/kg feed/day was administered,
- the animal was dosed five times in time intervals of 24 hours,
- the sacrifice was 5 hours after the last dose, i.e. at a time of relatively high concentrations in the tissues and organs

in order to facilitate metabolite characterisation and identification. Hence, the determined residue concentrations in the organs, tissues and milk do not reflect a realistic exposure situation.

- Radioactive residues were efficiently extracted from milk, edible organs and tissues (approx. 74% to 96% of the TRR). Exhaustive extraction steps with the assistance of microwave at a slightly increased temperature of 60°C were only efficient for extraction of the label specific metabolites triazolyl alanine and 1,2,4-triazole as shown for kidney. Non label specific metabolites were released only to minor amounts, although it can be assumed that they formed the major part of the non-extractable residues in muscle and liver.
- [Triazole-UL-^{14}C]JAU 6476-desthio was metabolised extensively in the lactating goat –more than twenty components were detected.

- Metabolic reactions detected in the lactating goat were:

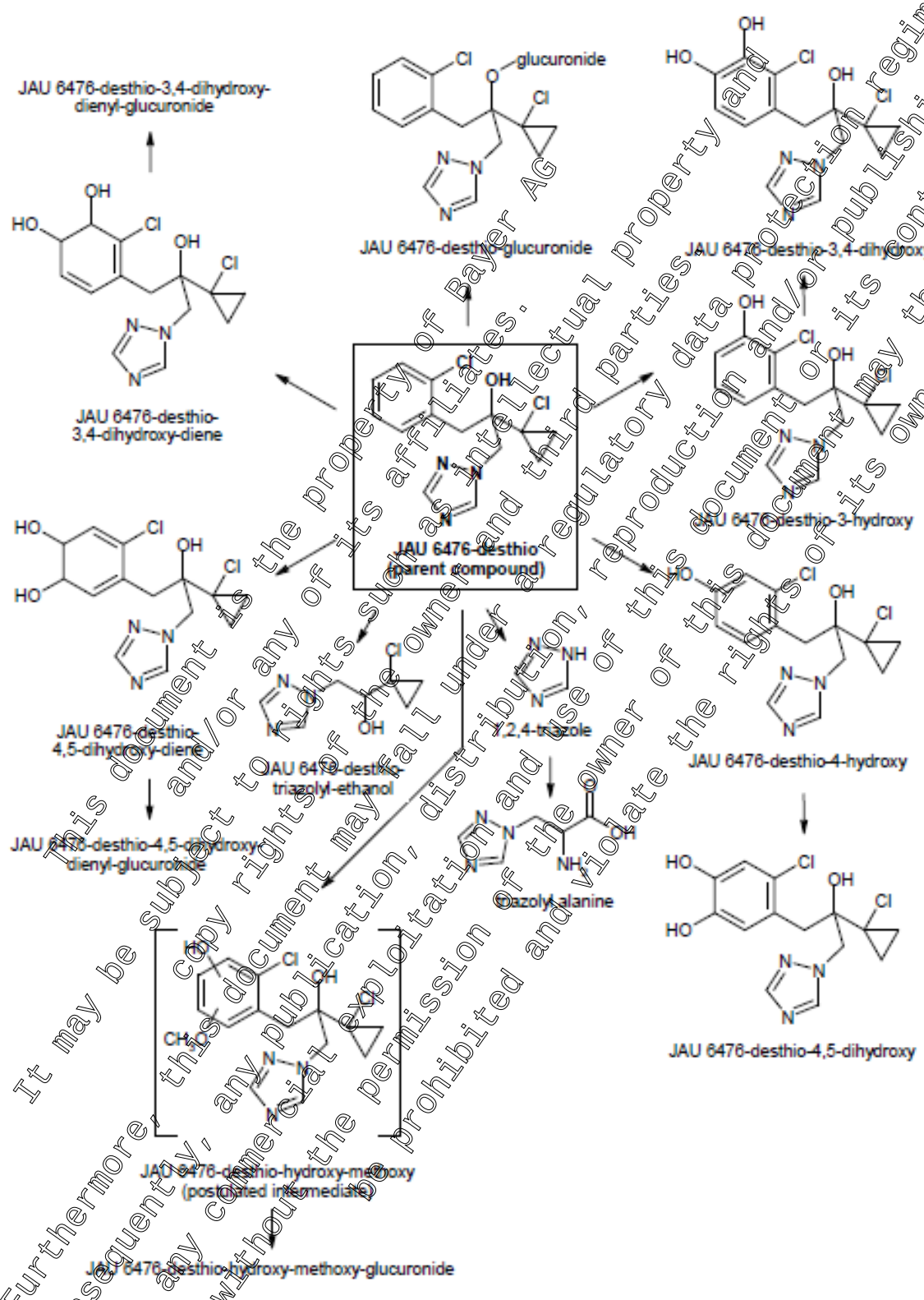
- mono- and dihydroxylation of the chlorophenyl moiety
- dearomatisation of the chlorophenyl moiety by oxidative hydroxylation
- subsequent conjugation of the hydroxylated metabolites with glucuronic acid
- conjugation of the test compound with glucuronic acid
- cleavage of the molecule yielding JAU 6476-triazolyl-ethanol and 1,2,4-triazole
- conjugation of 1,2,4-triazole with serine to triazolyl alanine

- 1,2,4-triazole was the main metabolite in milk and muscle ranging from ca. 17% in muscle to ca. 33% in morning milk. Although the percentage proportions are high, the corresponding residue levels did not exceed 0.045 mg/kg. Considering the fact, that all radioactive residues are calculated as equivalents of the active substance, the effective concentrations of 1,2,4-triazole are by a factor of approx 4.5 lower and account for maximally ca. 0.01 mg/kg. Thus, in spite of the apparent selective transfer of the metabolite into milk, low effective residues resulted. 1,2,4-triazole was also detected in fat, liver, and kidney accounting for ca. 4%, 1% and 1%, respectively. Co-elution with triazolyl alanine was proven for all three matrices. The exhaustive extract of kidney revealed major amounts of triazolyl alanine (ca. 8%) indicating that 1,2,4-triazole was transformed into the even more water-soluble triazolyl alanine to facilitate fast excretion via urine.

  JAU 6476-desthio-triazolyl ethanol was also a major metabolite in milk and muscle. It represented ca. 8% of the TRR in muscle and approx. 11% in evening milk. However the absolute residue levels were low (0.02 mg a.s. equiv./kg in both matrices, or ca. 0.01 mg/kg considering the molecular weight of the metabolite).
  Additionally, it has to be considered that the residue concentrations of free 1,2,4-triazole and JAU 6476-desthio-triazolyl ethanol in the organs, tissues and milk do not reflect a realistic exposure situation as mentioned above. Most probably none of the metabolites would have been detectable in the edible tissues under realistic conditions.
- The unchanged test compound JAU 6476-desthio was prominent (> 10% of the TRR) in fat and liver, JAU 6476-desthio-4-hydroxy in fat and JAU 6476-desthio-4-hydroxy-glucuronide in kidney. All other metabolites were present at lower percentages, the majority of them as conjugates or in the case of the dihydroxylated metabolites as dienes after de-aromatisation. Since the conjugates and metabolites with diene structure can be easily hydrolysed, an acidic treatment of the extracts was additionally performed. Under the conditions chosen, glucuronides and other conjugates were cleaved and the compounds with diene structure were converted into metabolites with known aromatic structure. The same simplified metabolic pattern resulted in both studies. The resulting five common moiety products JAU 6476-desthio-3,4-dihydroxy, JAU 6476-desthio-4,5-dihydroxy, JAU 6476-desthio-3-hydroxy, JAU 6476-desthio-4-hydroxy and JAU 6476-desthio covered the main residues originating from all non-label specific metabolites in each matrix.

The results of the metabolism study with [triazole-UL^{14}C]JAU 6476-desthio in the lactating goat is in very good agreement with the results of the corresponding metabolism study with [phenyl-UL^{14}C]JAU 6476-desthio.

Figure 6.2.3- 2 Proposed metabolic pathway of [triazole-UL^{14}C]JAU 6476-desthio in the lactating goat.



CA 6.2.4 Pigs

No studies to address the data requirement metabolism in pigs were included in the submission in support of the inclusion of prothioconazole in Annex I of Directive 91/414/EEC.

Studies are only required when the metabolism in ruminants differs from that in the rat. The metabolism of prothioconazole in ruminants and rats is considered comparable. No new studies have therefore been performed.

CA 6.2.5 Fish

No studies on metabolism in fish were included in the submission in support of the inclusion of prothioconazole in Annex I of Directive 91/414/EEC since this was not a data requirement at the time.

Commission Regulation (EU) No 283/2013 of 1 March 2013 setting out the data requirements for active substances, in accordance with Regulation (EC) No 1107/2009 of the European Parliament and of the Council concerning the placing of plant protection products on the market stipulates under point 6.2.5: "*Metabolism studies on fish may be required where the plant protection product is used in crops whose parts or products, also after processing, are fed to fish and where residues in feed may occur from the intended applications*."

However, for this data requirement no test guideline is available yet. As stated in SANCO document 10181/2013-rev. 3 (12 December 2014), in cases where "*test methods or guidance documents are not yet available for particular data requirements [...] waiving of these particular data requirement points is considered acceptable as long as no test methods or guidance documents are published in form of an update of the Commission Communications 2013/C 95/01 and 2013/C 95/02.*"

Therefore, no fish metabolism study was conducted.

CA 6.3 Magnitude of residue trials in plants

For the renewal of prothioconazole in Europe, the representative formulations are Aviator, a formulation containing 150 g/L prothioconazole and 75 g/L bixafen and Redigo FS100, a seed treatment formuation containing 100 g/L prothioconazole. The representative use patterns (or Good Agricultural Practices - GAPs) using this formulation are summarised in Table 6.3-1 and Table 6.3-2.

Table 6.3-1: Summary of the critical GAP for the representative foliar uses for prothioconazole*

Crop	Region	FT**	F, G, I***	Timing of application	Max. n° of applic.	Minimum Application interval [days]	Maximum application rate [g a.s./ha]	PHI [days]
Wheat, rye, tritiale, spelt	EU-N EU-S	EC 225	F	Up to BBCH 69	2	14	187.5	†
Barley, Oat	EU-N EU-S	EC 225	F	Up to BBCH 61	2	14	150	†

EU-S = Southern Europe EU-N = northern Europe
* justification of the critical GAP for each crop (all zones) applied for is provided in documents D1 and D2.
** FormulationType: EC 225 = EC formulation containing 75 g/L bixafen and 150 g/L prothioconazole.
*** F Field; G Greenhouse; I Indoor † As the application in cereals is driven purely by growth stage, no PHI is necessary.

Table 6.3-2: Summary of the critical GAP for the representative seed treatment uses for prothioconazole*.

Crop	Region	FT	F, G, I*	Mode of application	n° of applic.	Maximum application rate		PHI
						(g a.s./dt seeds)	[g a.s./ha]	
Wheat, rye, tritiale, spelt	EU-N and EU-S	FS100	F	Seed treatement	1	PTZ: 10.0	PTZ: 18.0[a]	†
Barley, Oat	EU-N EU-S	FS100	F	Seed treatement	1	PTZ: 10.0	PTZ: 18.0[a]	†

EU-S = Southern Europe EU-N = northern Europe
* justification of the critical GAP for each crop (all zones) applied for is provided in documents D1 and D2.
** FormulationType FS100 Seed treatment formulation containing 100 g/L prothioconazole
*** F Field; G Greenhouse; I Indoor †As the application in cereals is driven purely by growth stage, no PHI is necessary.
[a] Based on a seeding rate of 180 kg seed/ha

General remarks:
• In this section of the dossier, only the residues relevant to prothioconazole will be described in detail. As the products applied also contained other active substances, residues of those compounds were also determined, but these results are not considered relevant to this dossier. For details on the results for the other compounds, see the study reports.
• Tier 1 summary forms of residue trials are provided in Appendix 1 of this section.

CA 6.3.1 Wheat

The wheat uses and GAPs supported for this AIR dossier are summarised in Table 6.3.1- 1 and Table 6.3.1- 2.

Foliar use (Bixafen & Prothioconazole EC 225 (75 + 150 g/L)) Aviator

Table 6.3.1-1: Summary of the critical GAP for the representative foliar uses for prothioconazole*

Crop	Region	FT**	F, G, I***	Timing of application	Max. n° of applic.	Minimum Application interval [days]	Maximum application rate [g a.s./ha]	PHI [days]
Wheat, rye, tritiale, spelt	EU-N EU-S	EC 225	F	Up to BBCH 89	2	14	187.5	†

EU-N = northern Europe EU-S = southern Europe
* justification of the critical GAP for each crop (all zones) applied for is provided in documents D1 and D2.
** Formulation Type EC 225 = EC formulation containing 75 g/L bixafen and 150 g/L prothioconazole.
*** F Field; G Greenhouse; I Indoor † As the application in cereals is driven purely by growth stage, no PHI is necessary.

Seed treatment use (Prothioconazole FS 100 (100 g/L)) Redigo

Table 6.3.1- 2: Summary of the critical GAP for the representative seed treatment uses for prothioconazole*

Crop	Region	FT	F, G, I*	Mode of application	n° of applic.	Maximum application rate		PHI
						[g a.s./dt seeds]	[g a.s./ha]	
Wheat, rye, tritiale, spelt	EU-N and EU-S	FS100	F	Seed treatement	1	PTZ: 10.0	PTZ: 18.0[a]	†

EU-S = Southern Europe EU-N = northern Europe
* justification of the critical GAP for each crop (all zones) applied for is provided in documents D1 and D2.
** FormulationType FS100 Seed treatment formulation containing 100 g/L prothioconazole
*** F Field; G Greenhouse; I Indoor †As the application in cereals is driven purely by growth stage, no PHI is necessary.
[a] Based on a seeding rate of 180 kg seed/ha

According to EU guidance document SANCO 7525/VI/95-rev.9 of March 2011 ("Guidelines on comparability, extrapolation, group tolerances and data requirements for setting MRLs") the data obtained from trials conducted on wheat can be extrapolated to rye, triticale and spelt.

The current EU MRL for rye (0.1 mg/kg) will be replaced by an MRL of 0.05 mg/kg in spring 2016 following the review of the existing MRLs for prothioconazole according to Article 12 of Regulation (EC) N° 396/2005. Please refer to Regulation (EU) 843/2013 as well as draft Regulation SANCO 11481/2014. The current EU MRL for wheat is 0.05 mg/kg.

Summary of the trials from the Annex 1:

The residue trials on wheat that have already been evaluated during the EU review of prothioconazole are summarised in Table 6.3.1-3. Since these residue reports have been previously evaluated by the RMS UK (refer to Draft Assessment Report, Annex B7.6.1, Table B7.18) They are only presented for reference purposes. The critical supported GAP consisted in one seed treatment application with 15 g a.s./dt seed followed by 3 sprayings at rate of 200 g a.s./ha with last treatment performed at BBCH growth stage 69 at the latest.

Table 6.3.1- 3: Number of residue trials conducted per geographical region and vegetation period

Crop	GAP	Region	Formu-lation*	Number of Trials				Report No.	Dossier Reference
				Vegetation period			Total		
				1998	1999	2000			
Annex II data – seed treatment plus foliar									
Wheat, spring	ST 15 g a.s./dt and SPI 3 x 200 g a.s./ha	N-EU	FS 200 and EC 250	3	1	-	10	RA-2095/99	KCA 6.3.1.1.2
Wheat, spring	SPI 3 x 200 g a.s./ha		EC 250	-	1	5		RA-2003/99 RA-2104/00	
Wheat, spring	ST 15 g a.s./dt and SPI 3 x 200 g a.s./ha	S-EU	FS 200 and EC 250	-	5	-	9	RA-2149/98	
Wheat, Spring, Triticale	SPI 3 x 200 g a.s./ha		EC 250	-	-	4		RA-2105/00	
Annex II data – seed treatment use									
Cereal (wheat)	ST 19.5-22 g/ha (15 g a.s./dt)	N-EU	FS 200	-	2	2	4	RA-2010/99 RA-2091/00	KCA 6.3.1.1.1
Cereal (wheat)		S-EU	FS 200	-	2	2	4	RA-2110/99 RA-2090/00	

N-EU: northern Europe S-EU: southern Europe ST: seed treatment SPI: spray
* FS 200: flowable concentrate formulations containing 200 g/L of prothioconazole
250 EC: emulsifiable concentrate containing 250 g/L of prothioconazole

Seed treatment + foliar use:

With the Annex II dossier, a total of 19 residue trials were conducted on wheat in both European regions with the 'prothioconazole FS 200' formulation, a flowable concentrate for seed treatment containing 200 g/L prothioconazole and/or with the 'prothioconazole EC 250' formulation, an emulsifiable concentrate for spraying containing 250 g/L prothioconazole. A short summary of the data is given below.

In 9 trials, 'prothioconazole FS 200' was used for seed dressing at a rate of 15 g a.s./dt followed by 3 spray applications of 'prothioconazole EC 250' at a rate of 200 g a.s./ha. This use pattern is considered to represent the EU critical GAP and can be used as a basis for setting the EU MRLs.
10 separate trials were conducted with 3 spray applications of 'prothioconazole EC 250' at a rate of 200 g a.s./ha. The sprayings were performed at BBCH growth stages 32/33, 39-41 and 69, respectively, with the last treatment performed 34 to 36 days before harvest.

Residues of prothioconazole-desthio were determined at various development stages of the treated plants: treated seeds, green material, rest of plant, ears, straw and grain depending on the growth stage at sampling. Straw and grain samples were taken in most trials on day 34-37 as well as 42-71 days after the final treatment. The latter sampling interval was needed to ensure that samples of mature plants were available independent of the growth stage that was reached following the fixed pre-harvest interval of 35 days.

For the Annex II data, the desired PHI was 35 days. If 35 days after application, the grain could not be separated, sample materials available at that time (ear and "rest of plant") were taken, and grain and straw were then sampled later at harvest maturity (GS 89). Also, if grain samples could be taken on day 35, but the commodity had not yet reached final harvest maturity, a second set of grain and straw samples was taken at harvest.

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.01 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

No residues above the LOQ could be detected in any of the control samples.
An overall summary of the obtained residues in feed and food items is presented in Table 6.3.1- 4.

Table 6.3.1- 4: Summary of residue data from wheat trials with prothioconazole 250 EC (foliar treatment or seed treatment followed by foliar)

Application Rate	Region	Commodity	No. of trials	Residue level of prothioconazole desthio (mg/kg)			Reference
				Minimum	Maximum (HR)	STMR	
3 x 200 g a.s./ha or seed treatment (15 g a.s./dt seed) followed by 3 x 200 g a.s./ha (PHI 35 days)	N-EU	Wheat, grain	10	< 0.01	0.01	< 0.01	EFSA Scientific Report (2007) 106
	S-EU		9	< 0.01	< 0.01	< 0.01	
	N-EU	Wheat, straw	10	0.08	0.72	0.19	
	S-EU		9	0.2	0.81	0.53	

N-EU = Northern Europe, S-EU = Southern Europe

Seed treatment:

For seed treatment in cereals, 8 trials in wheat (4 in northern Europe and 4 in Southern Europe) were carried out and evaluated during Annex I inclusion. The product was a flowable concentrate for seed treatment, containing 200 g/L JAU 6476. The seed was dressed at a rate of 75 mL product per 100 kg of seed, corresponding to 15 g JAU 6476 per 100 kg of seed. The actual dressing rate was determined with method 00598/M001 by taking seed samples after seed dressing prior to drilling.

For data gathering purposes the samples taken from these trials were analysed for prothioconazole-desthio using methods 00598/M001 or 00647 with a limit of quantitation of 0.01 mg/kg for grain and 0.05 mg/kg for cereal green material and straw.

No residues above the LOQ could be detected in any of the control samples.
An overall summary of the obtained residues in feed and food items is presented in Table 6.3.1- 5.

Table 6.3.1- 5: Overall summary of residue of prothioconazole-desthio in wheat with seed treatment

Application Rate	Region	Com-modity	No. of trials	Residue level of prothioconazole-desthio (mg/kg)			Reference
				Minimum	Maximum (HR)	STMR	
19.5-22 g/ha (15 g a.s./dt seed)	N-EU and S-EU	Wheat , grain	8	< 0.01	< 0.01	0.01	EFSA Scientific Report (2007) 106
		Wheat, straw	8	< 0.05	< 0.05	0.05	

N-EU = northern Europe; S-EU = southern Europe

EU - northern region

Table 6.3.1- 6: Number of residue trials conducted per geographical region and vegetation period

Year	GAP	Formulation	N° of trials	Study number Report No.	Dossier Reference
Wheat EU foliar spray residue trials – northern EU					
2000	2 x 150 g a.s./ha BBCH69	EC200 (100 g/L fluoxastrobin, 100 g/L prothioconazole)	4	RA-2014/00 M-091521-01-1	KCA 6.3.1/08
2000	2 x 200 g a.s./ha BBCH69	EC460 (160 g/L prothioconazole, 300 g/L spiroxamine)	4	RA-2092/00 M-087669-01-1	KCA 6.3.1/09
2003	2 x 150 g a.s./ha BBCH69	EC300 (175 g/L fluoxastrobin, 150 g/L prothioconazole, 75 g/L trifloxystrobin)	2	RA-2018/03 M-001696-01-1	KCA 6.3.1/10
2003	2 x 175 g a.s./ha BBCH69	SC325 (175 g/L prothioconazole, 150 g/L trifloxystrobin)	2	RA-2108/03 M-067473-01-1	KCA 6.3.1/11
2005	2 x 200 g a.s./ha BBCH69	SC263 (175 g/L prothioconazole, 88 g/L trifloxystrobin)	4	RA-2575/05 M-268270-01-1	KCA 6.3.1/12
2007	2 x 187 g a.s./ha BBCH69	EC225 (75 g/L bixafen, 150 g/L prothioconazole)	4	RA-2037/07 M-298112-01-1	KCA 6.3.1/13
2007	2 x 150 g a.s./ha BBCH69	EC400 (50 g/L bixafen, 100 g/L prothioconazole, 250 g/L spiroxamine)	4	RA-2040/07 M-298182-02-1	KCA 6.3.1/14
2009	2 x 200 g a.s./ha BBCH69	EC260 (60 g/L bixafen, 100 g/L prothioconazole)	2	09-2057 M-393820-01-1	KCA 6.3.1/15
2010	2 x 175 g a.s./ha BBCH69	EC190 (40 g/L bixafen, 50 g/L fluoxastrobin and 100 g/L prothioconazole)	2	10-2205 M-414652-01-1	KCA 6.3.1/16
2013	2 x 150 g a.s./ha BBCH69	EC200 (100 g/L fluoxastrobin, 100 g/L prothioconazole)	3	13-2138 M-501083-02-1	KCA 6.3.1/17
2013	2 x 150 g a.s./ha BBCH69	EC200 (100 g/L fluoxastrobin, 100 g/L prothioconazole)	2	13-2159 M-501715-01-1	KCA 6.3.1/18
TOTAL northern EU region			**33**		

Report:	KCA 6.3.1/08 [redacted],; [redacted]; 2002; M-091521-01-1
Title:	Determination of residues of HEC 5725 & JAU 6476-desthio on winter wheat following spray application of HEC 5725 & JAU 6476 200 EC in Sweden, France, Great Britain and Germany
Report No.:	RA-2011/00
Document No.:	M-091521-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of 15 July, 1991, Annex II, part A, point 6 and Annex III, part A, point 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 1999/2000, a set of 4 residue trials was conducted in northern Europe. The trials were located in Sweden, northern France, Great Britain and Germany.

In each trial, wheat was treated twice at a product rate of 1.5 L/ha fluoxastrobin & prothioconazole EC 200 corresponding to 0.15 kg a.s./ha for each mixing partner. The water rate was 300 L/ha in all trials. The spray interval was 19 days in 3 trials and 37 days in one trial. The time of application was:

1. when the flag leaf sheath was opening (BBCH 47) in 3 trials and beginning of heading (BBCH 51) in one trial,
2. at the end of flowering (BBCH 69) in 3 trials and early dough stage (BBCH 81) in one trial.

Samples were taken at the following intervals:

- prior to and immediately after the final application,
- at a pre-harvest interval of 35 days as well as 42-61 days after the final treatment.

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 7.

Table 6.3.1- 7: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	267	8.9	RA-2011/00 M-091521-01-1
		grain	224	7.5	
		Rest of plant	267	8.9	
		straw	224	7.5	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 8. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 8: RA-2011/00: Concurrent recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Wheat grain / Ear	0.01	101; 101; 97	100	2.3	0.01
		0.10	105; 103; 105; 104; 104; 102; 106; 104; 108; 102; 102; 105; 102; 103; 100; 97; 103; 103; 102; 104; 104; 102; 102; 109; 100; 102; 99	103	2.4	
		Overall (n = 30)		**103**	**2.6**	
Prothio-conazole-desthio	Rest of plant	0.05	107; 98; 107	104	5.0	0.05
		0.50	105; 102; 102; 105; 106; 105; 102; 101; 104; 103; 100; 99; 120; 98; 102; 101; 99; 103	103	4.6	
		Overall (n = 21)		**103**	**4.6**	
Prothio-conazole-desthio	Straw	0.05	98; 97; 96	97	1.0	0.05
		0.50	106; 102; 102; 102; 103; 97; 100; 103; 103; 109; 103; 99; 105; 101; 101	102	2.8	
		Overall (n = 18)		**102**	**3.3**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:

The residue results are summarised in Table 6.3.1- 9.

Table 6.3.1- 9: Residues of prothioconazole-desthio in/on winter wheat applied with fluoxastrobin & prothioconazole EC 200

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2011/00 R 2000 0146/0 0146-00 GLP: yes 2000	Wheat, winter Abbot	United Kingdom GB-[redacted] Europe, North	200 EC	2	0.1500	0.05000	69	ear	0(1) 0 35	0.01 0.60 0.02
								rest of plant	0(1) 0 35	<0.05 0.93 0.07
								straw	58	0.11
								grain	58	<0.01
RA-2011/00 R 2000 0145/2 0145-00 GLP: yes 2000	Wheat, winter Shango	France F-[redacted] Europe, North	200 EC	2	0.1500	0.05000	69	ear	0(1) 0 35	0.04 0.54 0.02
								rest of plant	0(1) 0 35	<0.05 0.67 0.10
								straw	48	0.09
								grain	48	<0.01
RA-2011/00 R 2000 0147/9 0147-00 GLP: yes 2000	Wheat, winter Flair	Germany D-[redacted] Europe, North	200 EC	2	0.1500	0.05000	81	ear	0(1) 0	<0.01 0.70
								rest of plant	0(1) 0	<0.05 1.1
								straw	35 42	<0.05 0.05
								grain	35 42	<0.01 <0.01
RA-2011/00 R 2000 0144/4 0144-00 GLP: yes 2000	Wheat, winter Tarso	Sweden S-[redacted] Europe, North	200 EC	2	0.1500	0.05000	65	ear	-1 0 35	0.09 1.3 0.04
								rest of plant	-1 0 35	0.10 0.74 0.08
								straw	61	0.07
								grain	61	<0.01

FL = Formulation, DALT = Days after last treatment
* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) : before last application
** : the 2nd treatment was carried out at a later stage as originally intended (BBCH 81 instead of 69).

In the 4 trials conducted in 2000 (report RA-2011/00), residues of prothioconazole-desthio in wheat grain at harvest were always below the limit of quantification (LOQ) of 0.01 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.11 mg/kg.

Conclusion

Four wheat residue trials were conducted with fluoxastrobin & prothioconazole EC 200 in northern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha.
Residues of prothioconazole-desthio in wheat grain at harvest were always below the limit of quantification (LOQ) of 0.01 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.11 mg/kg.

Report:	KCA 6.3.1/09[redacted].;[redacted]; 2001; M-087669-01-1
Title:	Determination of residues of JAU 6476-desthio & KWG 4168 on spring wheat following spray application of JAU 6476 & KWG 4168 460 EC in Great Britain, France, Germany and Italy
Report No.:	RA-2092/00
Document No.:	M-087669-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of 15 July, 1991, Annex II, part A, point 6 and Annex III, part A, point 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2000, a set of 4 residue trials was conducted on winter wheat in northern Europe. The studies were located in northern France, Germany and the United Kingdom.

In each trial, wheat was treated twice at a product rate of 1.25 L/ha prothioconazole & spiroxamine EC 460 corresponding to 200 g a.s./ha of prothioconazole. The water rate was 300 L/ha. The spray interval ranged from 30 to 51 days. The time of application was:
1. during stem elongation (BBCH 32), and
2. at the end of flowering (BBCH 69)
with the last treatment performed 42 (41) days prior to harvest.

Samples were taken at the following intervals:
- immediately after the final application,
- at a pre-harvest interval of 28 days, 35 days, 42 and at grain maturity, *i.e.* up to 58 days after the final treatment.

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 10.

Table 6.3.1- 10: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	322	10.7	RA-2092/00 M-087669-01-1
		grain	275	9.2	
		Rest of plant	316	10.5	
		straw	275	9.2	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).

Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 11. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 11: RA-2092/00: Concurrent recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Wheat grain / Ear	0.01	101; 101; 97	100	2.3	0.01
		0.10	105; 103; 105; 104; 104; 102; 106; 104; 108; 102; 102; 105; 102; 103; 100; 97; 103; 103; 102; 104; 104; 102; 102; 109; 100; 102; 99	103	2.4	
		Overall (n = 30)		**103**	**2.6**	
Prothio-conazole-desthio	Rest of plant	0.05	107; 98; 107	104	5.0	0.05
		0.50	105; 102; 102; 105; 106; 105; 102; 101; 104; 103; 100; 99; 120; 98; 102; 101; 99; 103	103	4.6	
		Overall (n = 21)		**103**	**4.6**	
Prothio-conazole-desthio	Straw	0.05	98; 97; 96	97	1.0	0.05
		0.50	106; 102; 102; 102; 103; 97; 100; 103; 103; 109; 103; 99; 105; 101; 101	102	2.8	
		Overall (n = 18)		**102**	**3.3**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:

The residue results are summarised in Table 6.3.1- 12.

Table 6.3.1- 12: Residues of prothioconazole-desthio in/on spring wheat applied with prothioconazole & spiroxamine EC 460

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2092/00 R 2000 0081/2 0081-00 GLP: yes 2000	Wheat, spring Chablis	United Kingdom GB-[redacted] Europe, North	460 EC	2	0.2000	0.06672	69	ear	0 28 35	1.1 <0.03 <0.02
								rest of plant	0 28 35	2.2 0.08 0.06
								grain	42 56	<0.01 <0.01
								straw	42 56	0.06 0.06
RA-2092/00 R 2000 0431/1 0431-00 GLP: yes 2000	Wheat, spring Lavett	Germany D-[redacted] Europe, North	460 EC	2	0.2000	0.06672	69	ear	0 28 35	1.5 0.10 0.07
								rest of plant	0 28 35	2.9 0.39 0.36
								grain	41 49	<0.01 <0.01
								straw	41 49	0.28 0.14
RA-2092/00 R 2000 0433/8 0433-00 GLP: yes 2000	Wheat, spring Furio	France F-[redacted] Europe, North	460 EC	2	0.2000	0.06672	69	ear	0 28	0.92 0.04
								rest of plant	0 28	1.9 0.06
								grain	35 42	<0.01 <0.01
								straw	35 42	0.06 0.06
RA-2092/00 R 2000 0430/3 0430-00 GLP: yes 2000	Wheat, spring Lavett	Germany D-[redacted] Europe, North	460 EC	2	0.2000	0.06672	69	ear	0 28 35 42	1.7 0.06 0.04 0.04
								rest of plant	0 28 35 42	2.4 0.05 0.06 <0.05
								grain	58	<0.01
								straw	58	0.07

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio

In the 4 trials conducted in 2000 (report RA-2092/00), residues of prothioconazole-desthio in wheat grain at harvest were always <0.01 mg/kg. The residues in straw at harvest ranged between 0.06 mg/kg and 0.14 mg/kg.

Conclusion

Four wheat residue trials were conducted with prothioconazole & spiroxamine EC 460 in northern Europe. The product application corresponded to a prothioconazole rate of 2x200 g a.s./ha.

Residues of prothioconazole-desthio in wheat grain at harvest were always <0.01 mg/kg. The residues in straw at harvest ranged between 0.06 mg/kg and 0.14 mg/kg.

Report:	KCA 6.3.1/10[redacted].;[redacted]; 2004; M-001696-01-1
Title:	Determination of residues of fluoxastrobin (HEC 5725), prothioconazole (JAU 6476) and trifloxystrobin in/on wheat following spray application of HEC 5725 & JAU 6476 & CGA 279202 300 EC in the field in Germany
Report No.:	RA-2018/03
Document No.:	M-001696-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2002/2003, a set of 2 residue trials was conducted in northern Europe. The trials were located in Germany (2).

In each trial, wheat was treated twice at a product rate of 1.0 L/ha fluoxastrobin & prothioconazole & trifloxystrobin EC 300 corresponding to 0.150 kg a.s./ha of prothioconazole. The employed water rate was 300 L/ha in all trials. The applications were scheduled for growth stages BBCH 47 and 69. The applications were carried out at growth stages 47-51 (1st application) and 69 (2nd application; corresponding to intervals of 12 and 17 days).

Samples were taken at the following intervals:
- prior to and immediately after the final application;
- at a pre-harvest interval of 35 days
- at harvest (BBCH 89), 35-46 days after the final treatment.

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 13.

Table 6.3.1- 13: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	167	5.6	RA-2018/03 M-001696-01-1
		grain	132	4.4	
		Rest of plant	167	5.6	
		straw	132	4.4	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 14. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 14: RA-2018/03: Concurrent recoveries and validation recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Wheat grain	0.01	94; 94; 92	93	1.2	0.01
		0.1	*100; 95; 88; 95*	95	5.2	
		Overall (n = 7)		**94**	**3.8**	
Prothio-conazole-desthio	Straw	0.05	98; 94; 108	100	7.2	0.05
		0.50	*89; 98; 97*	95	5.2	
		Overall (n = 6)		**97**	**6.4**	
Prothio-conazole-desthio	Rest of plant	0.05	101; 92; 98	97	4.7	0.05
		0.50	*101; 94*	98	-	
		Overall (n = 5)		**97**	**4.2**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation. Concurrent recoveries in italics.

- Residue results:
The residue results are summarised in Table 6.3.1- 15.

Table 6.3.1- 15: Residues of prothioconazole-desthio in/on wheat applied with fluoxastrobin & prothioconazole & trifloxystrobin EC 300

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2018/03 R 2003 0133/2 0133-03 GLP: yes 2003	Wheat Magnus	Germany D-[redacted] [redacted]Europe, North	300 EC	2	0.1500	0.04995	69	rest of plant	0 (1) 0	<0.05 2.2
								ear	0 (1) 0	<0.01 1.0
								grain	**35**	<0.01
								straw	**35**	0.06
RA-2018/03 R 2003 0249/5 0249-03 GLP: yes 2003	Wheat Winnetou	Germany D-[redacted] [redacted]Europe, North	300 EC	2	0.1500	0.04995	69	rest of plant	0 (1) 0	0.06 0.65
								ear	0 (1) 0	0.04 0.62
								grain	**35** 46	<0.01 <0.01
								straw	**35** 46	0.05 0.05

***** determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) : before last application

In the 2 trials conducted in 2003 (report RA-2018/03), residues of prothioconazole-desthio in wheat grain at harvest were < 0.01 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.06 mg/kg.

Conclusion
Four wheat residue trials were conducted with fluoxastrobin & prothioconazole & trifloxystrobin EC 300 in northern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha. Residues of prothioconazole-desthio in wheat grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.06 mg/kg.

Report: KCA 6.3.1/11[redacted], 2004; M-067473-01-1
Title: Determination of residues of trifloxystrobin, CGA321113, and JAU 6476-desthio in/on wheat, soft following spray application of CGA279202 & JAU 6476 (325 SC) in the field in Northern France and Great Britain
Report No.: RA-2108/03
Document No.: M-067473-01-1
Guideline(s): EU-Ref, Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8
Residues in or on Treated Products, Food and Feed
Guideline deviation(s): not specified
GLP/GEP: **yes**

Materials and Methods

A total of two trials were conducted in northern Europe in 2002/2003, one in northern France and one in Great Britain. Two applications with prothioconazole & trifloxystrobin SC 325 were carried out at rate of 1.0 L product/ha corresponding to 0.175 kg a.s./ha of prothioconazole. The first application was done at BBCH growth stage 47-55/57 and the second at growth stage 69-71/73. The interval between applications was 28-29 days. The water rate was 300 L/ha, corresponding to an application concentration of 0.33 kg a.s./hL water. Samples of ear and rest of plant were taken on day 0 after the last application, and samples of grain and straw were taken at harvest (day 34-37).
The desired PHI was 35 days, but the application schedule called for last application at GS 69. If, 35 days after application, the grain could not be separated, grain and straw were then sampled later at harvest maturity (GS 89).

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 16.

Table 6.3.1- 16: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	229	7.6	RA-2108/03 M-067473-01-1
		grain	192	6.4	
		rest of plant	229	7.6	
		straw	199	6.6	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours; or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 17. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 17: RA-2108/03: Concurrent recoveries and validation recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Wheat grain / Ear	0.01	98; 103; 85	95	9.7	0.01
		0.1	*96; 96*	96	-	
		Overall (n = 5)		**96**	**6.9**	
Prothio-conazole-desthio	Rest of plant	0.05	109; 106; 98	104	5.5	0.05
		0.50	*98; 102*	100	-	
		Overall (n = 5)		**103**	**4.8**	
Prothio-conazole-desthio	Straw	0.05	95; 77; 96	89	12.0	0.05
		0.50	*90; 97*	94	-	
		Overall (n = 5)		**91**	**9.1**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation; LOQ: Limit of Quantitation; Concurrent recoveries in italics.

- Residue results:

The residue results are summarised in Table 6.3.1- 18.

Table 6.3.1- 18: Residues of prothioconazole-desthio in/on soft wheat applied with prothioconazole & trifloxystrobin SC 325

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2108/03 R 2003 0980/5 0980-03 GLP: yes 2003	Wheat soft Claire	United Kingdom GB-[redacted] Europe, North	325 SC	2	0.1750	0.05828	71	ear	0	0.25
								grain	34	0.02
								straw	34	0.79
								rest of plant	0	1.0
RA-2108/03 R 2003 0941/4 0941-03 GLP: yes 2003	Wheat, soft Trenie	France F-[redacted] Europe, North	325 SC	2	0.1750	0.05828	69	ear	0	0.32
								grain	37	<0.01
								straw	37	0.09
								rest of plant	0	0.70

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio

In the 2 trials conducted in 2003 (report RA-2108/03), residues of prothioconazole-desthio in wheat grain at harvest ranged from <0.01 mg/kg to 0.02 mg/kg. The residues in straw at harvest ranged between 0.09 mg/kg and 0.79 mg/kg.

Conclusion

Two wheat residue trials were conducted with prothioconazole & trifloxystrobin SC 325 in northern Europe. The product application corresponded to a prothioconazole rate of 2x175 g a.s./ha. A delay in application was observed in one trial: first treatment was carried out at the GS 55-57 (when at least half

of inflorescence emerged) instead of GS 47, and second application was conducted at the GS 71-73 during development of fruit instead of end of flowering (GS 69).
Residues of prothioconazole-desthio in wheat grain at harvest ranged from <0.01 mg/kg to 0.02 mg/kg.
The residues in straw at harvest ranged between 0.09 mg/kg and 0.79 mg/kg.

Report:	KCA 6.3.1/12[redacted]Z; 2006; M-268270-01-1
Title:	Determination of the residues of JAU 6476 and trifloxystrobin in/on winter wheat after spraying of JAU 6476 & CGA 279202 (263 SC) in the field in France, Sweden, United Kingdom and Germany
Report No.:	RA-2575/05
Document No.:	M-268270-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

A total of four trials were conducted in northern Europe in 2005, one in northern France, Sweden, Germany and in Great Britain. Two applications with prothioconazole & trifloxystrobin SC 263 were carried out at rate of 1.14 L product/ha corresponding to 0.200 kg a.s./ha of prothioconazole. The first application was done at BBCH growth stage 47 and the second at growth stage 69.
The interval between applications was 12-25 days. The water rate was 300 L/ha. Samples of green material was taken on day 0 before and after the last application and 28 and 35 days after the last application, and samples of grain and straw were taken at harvest (day 42-54).

Residues of prothioconazole-desthio were analysed according to method 00598/M001 by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution (please refer to Section 4.2.1). The LOQ for prothioconazole-desthio, defined as the lowest validated fortification level, was 0.01 mg/kg for wheat grain and 0.05 mg/kg for wheat rest of plant and straw.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 19.

Table 6.3.1- 19: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	grain	107	3.6	RA-2575/05 M-268270-01-1
		green material	133	4.4	
		straw	114	3.8	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of sample s ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 20. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 20: RA-2575/05: Concurrent recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Wheat green material	0.05	90; 93; 106	96	8.8	0.05
		0.50	89; 90; 91; 93	91	1.9	
		5	89; 91; 90; 93	91	1.9	
		Overall (n = 11)		**92**	**5.2**	
Prothio-conazole-desthio	Wheat grain	0.01	97; 81; 94	91	9.4	0.01
		0.10	72; 83; 89	81	10.6	
		Overall (n = 6)		**86**	**10.7**	
Prothio-conazole-desthio	Wheat straw	0.05	81; 86; 94	87	7.5	0.05
		0.50	90; 92; 91	91	1.1	
		Overall (n = 6)		**89**	**5.3**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:
The residue results are summarised in Table 6.3.1- 21.

Table 6.3.1- 21: Residues of prothioconazole-desthio in/on winter wheat applied with Prothioconazole & Trifloxystrobin SC 263

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2575/05 R 2005 0028/9 0028-05 GLP: yes 2005	Wheat, winter Apache	France F-[redacted] (Centre) Europe, North	263 SC	2	0.1995	0.06650	69	green material	0 0 28 35	0.06 0.62 0.28 0.10
								grain	42	<0.01
								straw	42	0.20
RA-2575/05 R 2005 0896/4 0896-05 GLP: yes 2005	Wheat, winter Winnetou	Germany D-[redacted] (Nordrhein-Westfalen) Europe, North	263 SC	2	0.1995	0.06650	69	green material	0 35	0.46 0.08
								grain	54	<0.01
								straw	54	0.12
RA-2575/05 R 2005 0895/6 0895-05 GLP: yes 2005	Wheat, winter Einstein	United Kingdom GB-[redacted] Europe, North	263 SC	2	0.1995	0.06650	69	green material	0(1) 0 28 35	0.17 0.61 0.14 0.11
								grain	50	<0.01
								straw	50	0.22
RA-2575/05 R 2005 0029/7 0029-05 GLP: yes 2005	Wheat, winter Ritmo	Sweden S-[redacted] Europe, North	263 SC	2	0.1995	0.06650	69	green material	0 35	1.3 0.85
								grain	54	<0.01
								straw	54	0.65

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) : before last application

In the 4 trials conducted in 2005 (report RA-2575/05), residues of prothioconazole-desthio in wheat grain at harvest were always <0.01 mg/kg. The residues in straw at harvest ranged between 0.12 mg/kg and 0.65 mg/kg.

Conclusion

Two wheat residue trials were conducted with prothioconazole & trifloxystrobin SC 263 in northern Europe. The product application corresponded to a prothioconazole rate of 2x200 g a.s./ha.
Residues of prothioconazole-desthio in wheat grain at harvest were always <0.01 mg/kg. The residues in straw at harvest ranged between 0.12 mg/kg and 0.65 mg/kg.

Report:	KCA 6.3.1/13[redacted],;[redacted]; 2008; M-298112-01-1
Title:	Determination of the residues of BYF 00587 and JAU 6476 in/on spring wheat and winter wheat after spraying of BYF 00587 & JAU 6476 (225 EC) in the field in the Netherlands, Northern France, the United Kingdom and Germany
Report No.:	RA-2037/07
Document No.:	M-298112-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

Four residue trials were carried out in 2007 on the combination product bixafen & prothioconazole EC 225 according to the supported use pattern. The trials were conducted on both spring wheat (3 trials) and winter wheat (1 trial) varieties. The test locations were in Germany, the United Kingdom, northern France and the Netherlands. Bixafen & prothioconazole EC 225 was applied twice at the required rates of 1.25 L product/ha corresponding to 0.188 kg a.s./ha of prothioconazole. The treatments were carried out at the growth stages "flag leaf sheath opening" (BBCH 47) and "end of flowering" (BBCH 69). Depending on the study, the spray interval ranged from 16-23 days. The water rate was 300 L/ha in all trials.

Samples of green material were taken just prior to and immediately after the final application in all trials. In northern Europe, 2 trials were designed as decline series, *i.e.* samples were collected further 7, 14, and 28 days after the final treatment.
The desired PHI was 35 days, but, as mentioned above, the application schedule called for application at GS 69. If, 35 days after application, the grain could not be separated, sample materials available at that time (ear and "rest of plant") were taken then, and grain and straw were then sampled later at harvest maturity (GS 89). Also, if grain samples could be taken on day 35, but the commodity had not yet reached final harvest maturity, a second set of grain and straw samples was taken at harvest. Thus, in the trials summarized here, first grain samples were taken on day 35-73; in one trial with early sampling of grain (GS 83), an additional sample was taken on day 61 at GS 89.

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 22.

Table 6.3.1- 22: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	164	5.5	RA-2037/07 M-298112-01-1
		grain	144	4.8	
		green material	199	6.6	
		rest of plant	164	5.5	
		straw	144	4.8	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 23. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 23: RA-2037/07: Concurrent recoveries for the determination of prothioconazole-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain #	0.01	97; 97; 96; 100; 93; 94; 99	97	2.6	0.01
		0.1	95; 93; 95; 91; 93; 100	95	3.3	
		1.0	92; 95; 95; 89; 93; 99	94	3.6	
		5.0	89	--	--	
		10	86	--	--	
		Overall Recovery (n = 21)		**94**	**3.9**	
Prothio-conazole-desthio	Cereal green material*	0.01	98; 94; 90; 97	95	3.8	0.01
		0.1	97; 90; 95; 88	93	4.5	
		1.0	87; 93; 95; 91; 89	91	3.9	
		5.0	89; 85; 82	85	4.1	
		10.0	83; 86; 85	85	1.8	
		Overall Recovery (n = 19)		**90**	**5.5**	
Prothio-conazole-desthio	Cereal straw	0.01	92; 88; 104; 93; 104	96	7.7	0.01
		0.1	95; 88; 94; 103	95	6.5	
		1.0	88; 93; 91; 93	91	2.6	
		5.0	88; 80; 87	85	5.1	
		10	84	--	--	
		Overall Recovery (n = 17)		**92**	**7.3**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole desthio
RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
These recoveries were performed with barley and wheat commodities during the conduct of the studies RA-2037/07, RA-2038/07, RA-2039/07, RA-2040/07, RA-2041/07, RA-2042/07, RA-2043/07, RA-2045/07, RA-2046/07, RA-2049/07 and RA-2050/07. Cereal summarises wheat and wheat. Recoveries for wheat are also valid for wheat.
* = Recoveries for rest of plant are covered by recoveries for green material.
# = Recoveries for ear are covered by recoveries for grain.

- Residue results:

The residue results are summarised in Table 6.3.1- 24.

Table 6.3.1- 24: Residues of prothioconazole-desthio in/on spring and winter wheat applied with Bixafen & Prothioconazole EC 225

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2037/07 R 2007 0418/6 0418-07 GLP: yes 2007	Wheat, spring Baldus	Netherlands NL-[redacted] (Noord-Holland) Europe, North	225 EC	2	0.1875	0.06255	69	green material	0[1] 0 7 14 28	0.07 0.86 0.09 0.04 <0.01
								straw	35 61	0.02 0.03
								grain	35 61	<0.01 <0.01
RA-2037/07 R 2007 0420/8 0420-07 GLP: yes 2007	Wheat, spring Belvoir	United Kingdom GB-[redacted] Europe, North	225 EC	2	0.1875	0.06255	69	green material	0[1] 0	0.14 1.1
								straw	73	0.03
								grain	73	<0.01
								ear	35	<0.01
								rest of plant	35	0.02
RA-2037/07 R 2007 0419/4 0419-07 GLP: yes 2007	Wheat, winter Mercato	France F-[redacted] (Centre) Europe, North	225 EC	2	0.1875	0.06255	69	green material	0[1] 0 7 14 28	0.08 1.7 0.32 0.12 0.06
								straw	55	0.10
								grain	55	<0.01
								ear	35	0.01
								rest of plant	35	0.07
RA-2037/07 R 2007 0421/6 0421-07 GLP: yes 2007	Wheat, spring Thasos	Germany D-[redacted] (Niedersachsen) Europe, North	225 EC	2	0.1875	0.06255	69	green material	0[1] 0	0.02 1.2
								straw	53	0.04
								grain	53	<0.01
								ear	35	0.01
								rest of plant	35	0.03

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
[1] before last application
GS = growth stage (BBCH code) at last application
FL = formulation

In the 4 trials conducted in 2007 (report RA-2037/07), residues of prothioconazole-desthio in wheat grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.03 mg/kg and 0.10 mg/kg.

Conclusion

Four wheat residue trials were conducted with bixafen & prothioconazole EC 225 in northern Europe. The product application corresponded to a prothioconazole rate of 2x187.5 g a.s./ha.

Residues of prothioconazole-desthio in wheat grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.03 mg/kg and 0.10 mg/kg.

Report:	KCA 6.3.1/14[redacted],[redacted]; 2008; M-298182-02-1
Title:	Determination of the residues of BYF 00587, JAU 6476 and KWG 4168 in/on winter wheat and spring wheat after spraying of BYF 00587 & JAU 6476 & KWG 4168 (400 EC) in the field in Northern France, the Netherlands, the United Kingdom and German
Report No.:	RA-2040/07
Document No.:	M-298182-02-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2007, a set of 4 residue trials on wheat was conducted in northern Europe. The trials were located in Germany, northern France, the Netherlands, and Great Britain.

In each trial, wheat was treated twice at a product rate of 1.5 L/ha BYF 00587 (bixafen) & JAU 6476 (prothioconazole) & KWG 4168 (spiroxamine) EC 400 corresponding to 0.150 kg/ha prothioconazole. The treatments were carried out at the growth stages "flag leaf sheath opening" (BBCH 47) and "end of flowering" (BBCH 69). Depending on the study, the spray interval ranged from 13-22 days. The water rate was 300 L/ha in all trials.

Samples were taken at the following intervals:
- prior to and immediately after the final application,
- at a pre-harvest interval of 35 days after the final treatment,
- at harvest (BBCH 89).

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 25.

Table 6.3.1- 25: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
Wheat	prothioconazole-desthio	ear	161	5.4	RA-2040/07 M-298182-02-1
		grain	152	5.1	
		green material	196	6.5	
		rest of plant	161	5.4	
		straw	152	5.1	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 26. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 26: RA-2040/07: Concurrent recoveries for the determination of prothioconazole-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain #	0.01	97; 97; 96; 100; 93; 94; 99	97	2.6	0.01
		0.1	95; 93; 95; 91; 93; 100	95	3.3	
		1.0	92; 95; 95; 89; 93; 99	94	3.6	
		5.0	89	--	--	
		10.0	86	--	--	
		Overall Recovery (n = 21)		**94**	**3.9**	
Prothio-conazole-desthio	Cereal green material*	0.01	98; 94; 90; 97	95	3.8	0.01
		0.1	97; 90; 95; 88	93	4.5	
		1.0	87; 93; 95; 91; 89	91	3.9	
		5.0	89; 85; 82	85	4.1	
		10.0	83; 86; 85	85	1.8	
		Overall Recovery (n = 19)		**90**	**5.5**	
Prothio-conazole-desthio	Cereal straw	0.01	92; 88; 104; 93; 104	96	7.7	0.01
		0.1	95; 88; 94; 103	95	6.5	
		1.0	88; 93; 91; 93	91	2.6	
		5.0	88; 80; 87	85	5.1	
		10.0	84	--	--	
		Overall Recovery (n = 17)		**92**	**7.3**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
These recoveries were performed with wheat and barley commodities during the conduct of the studies RA-2037/07, RA-2038/07, RA-2039/07, RA-2040/07, RA-2041/07, RA-2042/07, RA-2043/07, RA-2045/07, RA-2046/07, RA-2049/07 and RA-2050/07. Cereal summarises barley and wheat. Recoveries for wheat are also valid for barley.
* = Recoveries for rest of plant are covered by recoveries for green material.
# = Recoveries for ear are covered by recoveries for grain.

- Residue results:

The residue results are summarised in Table 6.3.1- 27.

Table 6.3.1- 27: Residues of prothioconazole-desthio in/on spring and winter wheat applied with bixafen & prothioconazole & spiroxamine EC 400

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	TAU 6476-desthio (mg/kg)
RA-2040/07 R 2007 0443/7 0443-07 GLP: yes 2007	Wheat, winter Mendel	France F-[redacted] (Centre) Europe, North	400 EC	2	0.1500	0.05000	69	green material	0 (1) 0	0.04 0.84
								straw	44	0.08
								grain	44	<0.01
								ear	35	0.02
								rest of plant	35	0.08
RA-2040/07 R 2007 0524/7 0524-07 GLP: yes 2007	Wheat, spring Thasos	Germany D-[redacted] (Nordrhein Westfalen) Europe, North	400 EC	2	0.1500	0.05000	69	green material	0 (1) 0	0.16 1.5
								straw	35 56	0.04 0.04
								grain	35 56	<0.01 <0.01
RA-2040/07 R 2007 0444/5 0444-07 GLP: yes 2007	Wheat, spring Baldus	Netherlands NL-[redacted] (Noord-Holland) Europe, North	400 EC	2	0.1500	0.05000	69	green material	0 (1) 0	0.03 0.92
								straw	35 61	0.02 0.02
								grain	35 61	<0.01 <0.01
RA-2040/07 R 2007 0523/9 0523-07 GLP: yes 2007	Wheat, spring Belvoir	United Kingdom GB-[redacted] Europe, North	400 EC	2	0.1500	0.05000	69	green material	0 (1) 0	0.04 0.92
								straw	73	0.03
								grain	73	<0.01
								ear	35	<0.01
								rest of plant	35	0.01

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) before last application
GS = growth stage (BBCH-code) at last application
FL = formulation

In the 4 trials conducted in 2007 (report RA-2040/07), residues of prothioconazole-desthio in wheat grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.02 mg/kg and 0.08 mg/kg.

Conclusion

Four wheat residue trials were conducted with bixafen & prothioconazole & spiroxamine EC 400 in northern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha. Residues of prothioconazole-desthio in wheat grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.02 mg/kg and 0.08 mg/kg.

Report:	KCA 6.3.1/15[redacted],;[redacted]; 2010; M-393820-01-1
Title:	Determination of the residues of BYF 00587 and prothioconazole in/on winter wheat after spraying of Bixafen & Prothioconazole EC 260 in the field in Germany and the Netherlands
Report No.:	09-2057
Document No.:	M-393820-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

Two wheat residue trials were carried out in the vegetation period of 2009. The trials were conducted on winter wheat varieties. The test locations were in Germany and the Netherlands. Bixafen & prothioconazole EC 260 was applied twice at the required rates of 1.0 L product/ha corresponding to 200 g a.s./ha of prothioconazole. The treatments were carried out at the growth stages BBCH 47 or 59 and BBCH 69 (end of flowering). Depending on the study, the spray interval was 21 or 22 days. The water rate was 300 L/ha in both trials.

Samples of green material were taken just prior to and immediately after the final application in both trials. The desired PHI was 35 days, but, as mentioned above, the application schedule called for application at GS 69. If, 35 days after application, grain samples could be taken, but the commodity had not yet reached final harvest maturity, a second set of grain and straw samples was taken at harvest. Thus, in trial 09-2057-01, first grain samples were taken on day 35 (BBCH 85); and an additional sample was taken on day 42 at BBCH 89.

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 28.

Table 6.3.1- 28: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	grain	426	14.2	09-2057 M-393820-01-1
		green material	321	10.7	
		straw	287	9.6	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 29. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 29: 09-2057: Concurrent recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Wheat grain	0.01	67; 108; 95; 95	91	19	0.01
		0.50	72	-	-	
		Overall Recovery (n = 5)		**87**	**20**	
	Wheat green material	0.01	98; 101	100	-	0.01
		0.50	87	-	-	
		2.0	85	-	-	
		Overall Recovery (n = 4)		**93**	**8.6**	
	Wheat straw	0.01	91	-	-	0.01
		0.50	89	-	-	
		Overall Recovery (n = 2)		**90**	**-**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification

- Residue results:
The residue results are summarised in Table 6.3.1- 30.

Table 6.3.1- 30: Residues of prothioconazole-desthio in/on winter wheat applied with Bixafen & prothioconazole EC 260

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
09-2057 09-2057-01 GLP: yes 2009	Wheat, winter Dekan	Germany [redacted] Europe, North	260 EC	2	0.200	0.067	69	green material	0* 0	0.07 0.76
								grain	35 42	<0.01 <0.01
								straw	35 42	0.04 0.05
09-2057 09-2057-02 GLP: yes 2009	Wheat, winter Tataros	Netherlands [redacted] Europe, North	260 EC	2	0.200	0.067	69	green material	0* 0	0.18 0.98
								grain	35	<0.01
								straw	35	0.06

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) before last application
GS = growth stage (BBCH code) at last application
FL = formulation

In the 2 trials conducted in 2009 (report 09-2057), residues of prothioconazole-desthio in wheat grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.06 mg/kg.

Conclusion

Two wheat residue trials were conducted with Bixafen & Prothioconazole EC 260 in northern Europe. The product application corresponded to a prothioconazole rate of 2x200 g a.s./ha.
Residues of prothioconazole-desthio in wheat grain at harvest were < 0.01 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.06 mg/kg.

Report: KCA 6.3.1/16 [redacted]; 2011; M-414652-01-1
Title: Determination of the residues of BYF 00587, HEC 5725 and prothioconazole in/on wheat, spring and wheat, winter after spray application of bixafen & fluoxastrobin & prothioconazole EC 190 in the field in the United Kingdom and Germany
Report No.: 10-2205
Document No.: M-414652-01-1
Guideline(s): EC-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8
Residues in or on Treated Products, Food and Feed
EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s): not specified
GLP/GEP: **yes**

Materials and Methods

In the vegetation period of 2010, a set of 2 residue trials on spring and winter wheat was conducted in northern Europe. The trials were located in the United Kingdom and Germany.

In each trial, wheat was treated twice at a product rate of 1.75 L/ha bixafen & fluoxastrobin & prothioconazole EC 190 corresponding to 0.175 kg a.s./ha of prothioconazole. The water rate was 200-400 L/ha. The spray interval was 14 days. The treatments were carried out at the growth stages BBCH 47 or 59 and BBCH 69 (end of flowering).

Samples were taken at the following intervals:
- prior to and immediately after the final application;
- at a pre-harvest interval of 35 days after the final treatment.

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).
Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio were determined according to method 00979/M001 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1-31.

Table 6.3.1- 31: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	grain	224	7.5	10-2205 M-414652-01-1
		green material	259	8.6	
		straw	224	7.5	
	prothioconazole-alpha-hydroxy-desthio prothioconazole-3-hydroxy-desthio prothioconazole-4-hydroxy-desthio prothioconazole-5-hydroxy-desthio prothioconazole-6-hydroxy-desthio	grain	258	8.6	
		green material	294	9.8	
		straw	258	8.6	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110% except for prothioconazole-desthio in green material at the LOQ level (112%) and for prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio and prothioconazole-6-hydroxy-desthio in straw at 0.50 mg/kg (68%, 69% and 68% respectively). The single and overall mean recoveries are shown in Table 6.3.1- 32 to Table 6.3.1- 37. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 32: 10-2205: Concurrent recoveries for the determination of prothioconazole-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain	0.01	108	-	-	0.01
		2.5	86; 99	93	-	
		Overall Recovery (n = 3)		**98**	**10.3**	
	Cereal green material	0.01	108; 108; 116; 111; 117	112[a]	3.8	0.01
		0.10	97	-	-	
		2.0	84; 104	94	-	
		2.5	92; 95; 94; 89	93	2.9	
		Overall Recovery (n = 12)		**101**	**10.7**	
	Cereal straw	2.5	73	-	-	0.01
		Overall Recovery (n = 1)		**73**	**-**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-desthio, determined as JAU 6476-desthio and calculated as JAU 6476-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat. [a] This value was accepted dueto a RSD below 20% and an overall mean in the range of 70-110%.

Table 6.3.1- 33: 10-2205: Concurrent recoveries for the determination of prothioconazole-alpha-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-alpha-hydroxy-desthio	Cereal grain	0.01	89; 90; 118; 120; 89; 89; 94; 100; 95; 96	98	11.9	0.01
		0.50	89; 98; 89; 95	93	4.9	
		Overall Recovery (n = 14)		**97**	**10.6**	
	Cereal green material	0.01	80; 99; 104; 99; 104; 93; 98; 100; 102; 85; 85; 91; 92	95	8.2	0.01
		0.50	101; 95; 100; 101; 83; 85	94	8.7	
		Overall Recovery (n = 19)		**95**	**8.1**	
	Cereal straw	0.01	77; 79; 88; 97; 81; 84	84	8.7	0.01
		0.50	72; 71; 88; 78; 92	80	11.8	
		Overall Recovery (n =11)		**82**	**9.9**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-alpha-hydroxy-desthio, determined as JAU 6476-alpha-hydroxy-desthio and calculated as JAU 6476-desthio. *These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207. Cereal summarises barley and wheat ** : mean of double injection

Table 6.3.1- 34: 10-2205: Concurrent recoveries for the determination of prothioconazole-3-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-3-hydroxy-desthio	Cereal grain	0.01	73; 82; 109; 113; 85; 88; 89; 90; 88; 94	91	13.1	0.01
		0.50	83; 93; 78; 81	84	7.8	
		Overall Recovery (n =14)		**89**	**12.3**	
	Cereal green material	0.01	70; 90; 101; 91; 103; 91; 94; 94; 94; 70; 71; 75; 77	86	13.8	0.01
		0.50	85; 82; 98; 104; 70; 74	86	15.5	
		Overall Recovery (n =19)		**86**	**14.0**	
	Cereal straw	0.01	80; 80; 83; 86; 98; 107	89	12.4	0.01
		0.50	62; 63; 81; 67; 76	70	12.0	
		Overall Recovery (n =11)		**80**	**17.2**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-3-hydroxy-desthio, determined as prothioconazole-3-hydroxy-desthio and calculated as prothioconazole-desthio *These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207. Cereal summarises barley and wheat ** : mean of double injection

Table 6.3.1- 35: 10-2205: Concurrent recoveries for the determination of prothioconazole-4-hydroxy-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-4-hydroxy-desthio	Cereal grain	0.01	65; 72; 99; 105; 60; 67; 68; 70; 71; 73	75	19.7	0.01
		0.50	87; 95; 72; 76	83	12.7	
		Overall Recovery (n = 14)		**77**	**17.8**	
	Cereal green material	0.01	68; 80; 99; 79; 83; 65; 66; 73; 74; 63; 64; 74; 76	74	13.3	0.01
		0.50	88; 84; 95; 102; 71; 75	86	13.7	
		Overall Recovery (n = 19)		**78**	**14.9**	
	Cereal straw	0.01	67; 64; 67; 77; 77; 81	72	9.7	0.01
		0.50	67; 62; 79; 62; 70	68***	10.3	
		Overall Recovery (n = 11)		**70**	**10.0**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-4-hydroxy-desthio, determined as prothioconazole-4-hydroxy-desthio and calculated as prothioconazole-desthio *These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207. Cereal summarises barley and wheat ** : mean of double injection *** This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%

Table 6.3.1- 36: 10-2205: Concurrent recoveries for the determination of prothioconazole-5-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-5-hydroxy-desthio	Cereal grain	0.01	74; 77; 108; 108; 75; 83; 86; 88; 78; 86	86	14.4	0.01
		0.50	80;86;83;83	83	3.0	
		Overall Recovery (n = 14)		**85**	**12.3**	
	Cereal green material	0.01	73; 93; 99; 95; 95; 76; 77; 78; 82; 72; 73; 78; 79	82	11.7	0.01
		0.50	90; 85; 92; 98; 70; 73	85	13.1	
		Overall Recovery (n = 19)		**83**	**11.9**	
	Cereal straw	0.01	80; 71; 79; 84; 78; 88	80	7.2	0.01
		0.50	62; 64; 80; 66; 75	69***	11.1	
		Overall Recovery (n = 11)		**75**	**11.2**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-5-hydroxy-desthio, determined as prothioconazole-5-hydroxy-desthio and calculated as prothioconazole-desthio *These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207 Cereal summarises barley and wheat ** : mean of double injection *** : This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%

Table 6.3.1- 37: 10-2205: Concurrent recoveries for the determination of prothioconazole-6-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-6-hydroxy-desthio	Cereal grain	0.01	69; 75; 99; 101; 94; 96; 97; 100; 93; 97	92	11.9	0.01
		0.50	79;84;79;82	81	3.0	
		Overall Recovery (n = 14)		**89**	**11.9**	
	Cereal green material	0.01	73; 102; 103; 92; 99; 87; 93; 95; 99; 69; 75; 77; 77	88	13.8	0.01
		0.50	79; 75; 89; 91; 70; 79	81	10.1	
		Overall Recovery (n = 19)		**85**	**13.2**	
	Cereal straw	0.01	82; 74; 75; 78; 75; 82	78	4.7	0.01
		0.50	68; 68; 80; 60; 62	68***	11.5	
		Overall Recovery (n = 11)		**73**	**10.5**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-6-hydroxy-desthio, determined as prothioconazole-6-hydroxy-desthio and calculated as prothioconazole-desthio *These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207 Cereal summarises barley and wheat **: mean of double injection *** : This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%

- Residue results:

The residue results are summarised in Table 6.3.1- 38.

Table 6.3.1- 38: Residues of prothioconazole-desthio in/on spring and winter wheat applied with Bixafen & fluoxastrobin & prothioconazole EC 190

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
10-2205 10-2205-02 GLP: yes 2010	Wheat, spring Kadrilj	Germany [redacted] Europe, North	190 EC	2	0.175	0.0438	69	green material	0(1) 0	0.38 1.4	0.14 0.13	0.16 0.16	0.12 0.12	0.13 0.13	0.02 0.02	0.95 2.0
								grain	35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	35	0.19	0.30	0.34	0.27	0.28	0.04	1.4
10-2205 10-2205-01 GLP: yes 2010	Wheat, winter Robigus	United Kingdom [redacted] Europe, North	190 EC	2	0.175	0.0875	69	green material	0(1) 0	0.41 1.2	0.10 0.11	0.07 0.08	0.08 0.09	0.08 0.10	0.02 0.02	0.76 1.6
								grain	35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	35	0.29	0.10	0.18	0.15	0.15	0.04	0.91

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg.
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

In the 2 trials conducted in 2010 (report 10-2205), residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in wheat grain at harvest were <0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg.
In straw, the residues at harvest ranged as follows :

- between 0.19 mg/kg and 0.29 mg/kg for prothioconazole-desthio
- between 0.10 mg/kg and 0.30 mg/kg for prothioconazole-alpha-hydroxy
- between 0.18 mg/kg and 0.34 mg/kg for prothioconazole-3-hydroxy-desthio
- between 0.15 mg/kg and 0.27 mg/kg for prothioconazole-4-hydroxy-desthio
- between 0.15 mg/kg and 0.28 mg/kg for prothioconazole-5-hydroxy-desthio
- at 0.04 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio
- between 0.91 mg/kg and 1.4 mg/kg for the total residue.

Conclusion

Two wheat residue trials were conducted with Bixafen & Fluoxastrobin & Prothioconazole EC 190 in northern Europe. The product application corresponded to a prothioconazole rate of 2x175 g a.s./ha.
Residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in wheat grain at harvest were <0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg.
In straw, the residues at harvest ranged as follows :

- between 0.19 mg/kg and 0.29 mg/kg for prothioconazole-desthio
- between 0.10 mg/kg and 0.30 mg/kg for prothioconazole-alpha-hydroxy
- between 0.18 mg/kg and 0.34 mg/kg for prothioconazole-3-hydroxy-desthio
- between 0.15 mg/kg and 0.27 mg/kg for prothioconazole-4-hydroxy-desthio
- between 0.15 mg/kg and 0.28 mg/kg for prothioconazole-5-hydroxy-desthio
- at 0.04 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio
- between 0.91 mg/kg and 1.4 mg/kg for the total residue.

Report:	KCA 6.3.1.17 [redacted]; 2015; M-501083-02-1
Title:	Determination of the residues of fluoxastrobin and prothioconazole in/on spring wheat after after spray application of fluoxastrobin & prothioconazole EC 200 in the field in Germany and United Kingdom
Report No.:	13-2138
Document No.:	M-501083-02-1
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC EC Guidance working document 7029/VI/95 rev.5 (1997-07-22), OECD 509 Adopted 2009-09-07, OECD GUIDELINE FOR THE TESTING OF CHEMICALS, Crop Field Trial US EPA OCSPP Guideline No. 860.1500
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.3.1/18[redacted],;[redacted]; 2014; M-501715-01-1
Title:	Determination of the residues of fluoxastrobin and prothioconazole in/on wheat and spring wheat after spray application of fluoxastrobin & prothioconazole EC 200 in France (North)
Report No.:	13-2159
Document No.:	M-501715-01-1
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC EC Guidance working document 7029/VI/95 rev 5 (1997-07-22) OECD 509 Adopted 2009-09-07, OECD GUIDELINE FOR THE TESTING OF CHEMICALS, Crop Field Trial US EPA OCSPP Guideline No. 860.1500
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2013, a set of 5 residue trials on spring and winter wheat was conducted in northern Europe. The trials were located in the northern France (2), the United Kingdom and Germany (2).

In each trial, wheat was treated twice at a product rate of 1.5 L/ha fluoxastrobin & prothioconazole EC 200 corresponding to 0.150 kg a.s./ha of prothioconazole. The water rate was 181-400 L/ha. The spray interval ranged from 14-21 days. The treatments were carried out at the growth stages BBCH 39-49 and BBCH 69 (end of flowering). In trial 13-2159-02, the first application was underdosed by 9.3% and the second application overdosed by 6.0%. In trial 13-2138-03, the first application overdosed by approximately 6%.

Samples were taken at the following intervals:
- prior to and immediately after the final application,
- at days 7, 14, 21, 28 and 31 after final application for 3 trials,
- at a pre-harvest interval of 34-35 days after the final application,
- at harvest (BBCH 89).

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days.
Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).
Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio

were determined according to method 00979/M001 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 39.

Table 6.3.1- 39: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	grain	316	10.5	13-2159 M-501715-01-1
		green material	365	12.2	
		straw	316	10.5	
	prothioconazole-alpha-hydroxy-desthio prothioconazole-3-hydroxy-desthio prothioconazole-4-hydroxy-desthio prothioconazole-5-hydroxy-desthio prothioconazole-6-hydroxy-desthio	grain	385	12.8	
		green material	436	14.5	
		straw	386	12.9	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown inTable 6.3.1- 40 to Table 6.3.1- 45. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 40: 13-2159 and 13-2138: Concurrent recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
Prothio-conazole-desthio	13-2159 and 13-2138	wheat grain	0.01	108; 110**	109	-	0.01
			0.10	97**; 102	100	-	
			Overall Recovery (n = 4)		**104**	**5.7**	
		wheat green material	0.01	107*; 114**	111	-	0.01
			0.10	101**	-	-	
			1.0	88**	-	-	
			2.0	88	-	-	
			Overall Recovery (n = 5)		**100**	**11.6**	
		wheat straw	0.01	103**; 104	104	-	0.01
			0.10	94**	-	-	
			1.0	89***			
			Overall Recovery (n = 4)		**98**	**7.4**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-desthio, determined as prothioconazole-desthio and calculated as prothioconazole-desthio
* value corrected for apparent residue in the control sample (76% of the LOQ). The uncorrected recovery value at FL 0.01 mg/kg was 183%. ** recovery value obtained with control sample of study 13-2138. *** The control sample of wheat straw used for recovery at 100 X LOQ level contained apparent residues at 144% of the LOQ. Recovery value obtained at 100 X LOQ level was not corrected for apparent residues in the control sample.

Table 6.3.1- 41: 13-2159 and 13-2138: Concurrent recoveries for the determination of prothioconazole-alpha-hydroxy-desthio in wheat

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
prothioconazole-alpha - hydroxy-desthio	13-2159	wheat grain	0.01	87; 93	90	-	0.01
			0.0	96		-	
			Overall recovery (n = 3)		**92**	**5.0**	
		wheat green material	0.01	80; 81; 97; 100	90	11.7	0.01
			0.10	105	-	-	
			0.53	95	-	-	
			Overall recovery (n = 6)		**93**	**11.0**	
		wheat straw	0.01	93; 101	97	-	0.01
			0.10	98	-	-	
			0.50	100	-	-	
			Overall recovery (n = 4)		**98**	**3.6**	
prothioconazole-alpha-hydroxy-desthio	13-2138	wheat green material	0.009	90; 92; 70	84	14.5	0.01
			0.11	89	-	-	
			0.09	82	-	-	
			0.10	91	-	-	
			0.30	104; 76	90	-	
			0.50	96; 101	99	-	
			Overall Recovery (n = 10)		**89**	**11.9**	
		wheat grain	0.01	76; 98; 104	93	15.9	0.01
			0.10	91	-	-	
			Overall Recovery (n = 4)		**92**	**13.1**	
		wheat straw	0.009	91	-	-	0.01
			001	67; 72; 96	78	19.8	
			0.09	86	-	-	
			0.10	66; 92	79	-	
			0.30	64	-	-	
			1.0	85; 88	87	-	
			Overall Recovery (n = 10)		**81**	**15.0**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-alpha-hydroxy-desthio, determined as prothioconazole-alpha-hydroxy-desthio and calculated as prothioconazole-desthio

Table 6.3.1- 42: 13-2159 and 13-2138: Concurrent recoveries for the determination of prothioconazole-3-hydroxy-desthio in wheat

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
prothioconazole-3-hydroxy-desthio	13-2159	wheat grain	0.01	90; 95	93	-	0.01
			0.10	91	-	-	
			Overall recovery (n = 3)		**92**	**2.9**	
		wheat green material	0.01	83; 86; 95; 96	90	7.2	0.01
			0.10	93	-	-	
			0.53	84	-	-	
			Overall recovery (n = 6)		**90**	**6.5**	
		wheat straw	0.01	79; 103	91	-	0.01
			0.10	85	-	-	
			0.50	93	-	-	
			Overall recovery (n = 4)		**90**	**11.5**	
prothioconazole-3-hydroxy-desthio	13-2138	wheat green material	0.009	89; 84; 89	87	3.3	0.01
			0.11	82	-	-	
			0.09	82	-	-	
			0.10	83	-	-	
			0.30	81; 89	85	-	
			0.50	89; 92	91	-	
			Overall Recovery (n = 10)		**86**	**4.6**	
		wheat grain	0.01	72; 101; 106	93	19.7	0.01
			0.10	90	-	-	
			Overall Recovery (n = 4)		**92**	**16.3**	
		wheat straw	0.009	96	-	-	0.01
			0.01	70; 75; 90	78	13.3	
			0.09	81	-	-	
			0.10	83; 73	78	-	
			0.30	74	-	-	
			1.0	77; 86	82	-	
			Overall Recovery (n = 10)		**81**	**10.3**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-hydroxy-desthio, determined as prothioconazole-hydroxy-desthio and calculated as prothioconazole-desthio

Table 6.3.1- 43: 13-2159 and 13-2138: Concurrent recoveries for the determination of prothioconazole-4-hydroxy-desthio in wheat

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
prothioconazole-4-hydroxy-desthio	13-2159	wheat grain	0.10	74; 98	86	-	0.01
			Overall recovery (n = 2)		**86**	**-**	
		wheat green material	0.01	66	-	-	0.01
			0.10	90	-	-	
			0.53	84	-	-	
			Overall recovery (n = 3)		**80**	**15.6**	
		wheat straw	0.01	71; 87	79	-	0.01
			0.10	80	-	-	
			0.50	98	-	-	
			Overall recovery (n = 4)		**82**	**10.3**	
prothioconazole-4-hydroxy desthio	13-2138	wheat green material	0.009	69; 82	76	-	0.01
			0.11	67	-	-	
			0.09	75	-	-	
			0.10	90	-	-	
			0.30	90; 76	83	-	
			0.50	89; 94	92	-	
			Overall Recovery (n = 9)		**81**	**12.3**	
		wheat grain	0.01	72; 79	76	-	0.01
			0.10	76	-	-	
			Overall Recovery (n = 3)		**76**	**4.6**	
		wheat straw	0.009	69	-	-	0.01
			0.01	64	-	-	
			0.09	82	-	-	
			0.10	83; 61	72	-	
			0.30	67	-	-	
			1.0	71; 74	-	-	
			Overall Recovery (n = 8)		**71**	**11.1**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-4-hydroxy-desthio, determined as prothioconazole-4-hydroxy-desthio and calculated as prothioconazole-desthio

Table 6.3.1- 44: 13-2159 and 13-2138: Concurrent recoveries for the determination of prothioconazole-5-hydroxy-desthio in wheat

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
prothioconazole-5-hydroxy-desthio	13-2159	wheat grain	0.01	81; 91	86	-	0.01
			0.10	89	-	-	
			Overall recovery (n = 3)		**87**	**6.1**	
		wheat green material	0.01	77; 77; 81; 88	81	6.4	0.01
			0.10	96	-	-	
			0.53	82	-	-	
			Overall recovery (n = 6)		**84**	**8.8**	
		wheat straw	0.01	86; 92	89	-	0.01
			0.10	90	-	-	
			0.50	101	-	-	
			Overall recovery (n = 4)		**92**	**6.9**	
prothioconazole-5-hydroxy desthio	13-2138	wheat green material	0.009	86; 91; 94	90	4.5	0.01
			0.11	91	-	-	
			0.09	85	-	-	
			0.10	77	-	-	
			0.30	96; 81	89	-	
			0.50	92; 95	94	-	
			Overall Recovery (n = 10)		**89**	**7.1**	
		wheat grain	0.01	73; 86; 101	87	16.2	0.01
			0.10	84	-	-	
			Overall Recovery (n = 4)		**86**	**13.4**	
		wheat straw	0.009	94	-	-	0.01
			0.01	82; 85	84	-	
			0.09	90	-	-	
			0.10	71; 90	81	-	
			0.30	70	-	-	
			1.0	82; 84	83	-	
			Overall Recovery (n = 8)		**83**	**9.9**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-5-hydroxy-desthio, determined as prothioconazole-5-hydroxy-desthio and calculated as prothioconazole-desthio

Table 6.3.1- 45: 13-2159 and 13-2138: Concurrent recoveries for the determination of prothioconazole-6-hydroxy-desthio in wheat

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
prothioconazole-6-hydroxy-desthio	13-2159	wheat grain	0.01	82; 91	87	-	0.01
			0.10	96		-	
			Overall recovery (n = 3)		**90**	**7.9**	
		wheat green material	0.01	67; 80; 89; 100	84	16.6	0.01
			0.10	101	-	-	
			0.53	85	-	-	
			Overall recovery (n = 6)		**87**	**14.7**	
		wheat straw	0.01	94; 95	95	-	0.01
			0.10	101	-	-	
			0.50	92	-	-	
			Overall recovery (n = 4)		**96**	**4.1**	
prothioconazole-6-hydroxy-desthio	13-2138	wheat green material	0.009	91; 102; 87	94	8.0	0.01
			0.11	98	-	-	
			0.09	86	-	-	
			0.10	78	-	-	
			0.30	90; 82	86	-	
			0.50	93; 95	94	-	
			Overall Recovery (n = 10)		**91**	**8.1**	
		wheat grain	0.01	79; 91; 105	92	14.2	0.01
			0.10	81	-	-	
			Overall Recovery (n = 4)		**89**	**13.4**	
		wheat straw	0.009	92	-	-	0.01
			0.01	82; 77; 92	84	9.1	
			0.09	82	-	-	
			0.10	89; 72	81	-	
			0.30	76	-	-	
			1.0	79; 79	79	-	
			Overall Recovery (n = 10)		**82**	**8.4**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-6-hydroxy-desthio, determined as prothioconazole-6-hydroxy-desthio and calculated as prothioconazole-desthio

- Residue results:

The residue results are summarised in Table 6.3.1- 46 and Table 6.3.1- 47.

Table 6.3.1- 46: Residues of prothioconazole-desthio in/on wheat applied with fluoxastrobin & prothioconazole EC 200

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
13-2159 13-2159-01 GLP: yes 2013	Wheat Siala	France [redacted] Europe, North	200 EC	2	0.15	0.075	69	green material	0 (1)	0.027	0.014	0.021	0.015	0.020	<0.01	0.11
									0	1.4	0.018	0.028	0.019	0.023	<0.01	1.5
									7	0.71	0.13	0.11	0.10	0.11	0.027	1.2
									14	0.25	0.13	0.12	0.10	0.10	0.025	0.73
									21	0.13	0.078	0.097	0.078	0.086	0.027	0.50
									28	0.075	0.062	0.096	0.086	0.086	0.023	0.43
									34	0.074	0.055	0.088	0.084	0.078	0.022	0.40
								grain	49	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	49	0.14/0.0 14**	0.052	0.12	0.091	0.11	0.029	0.54
13-2159 13-2159-02 GLP: yes 2013	Wheat, spring Valbona	France [redacted] Europe, North	200 EC	2	0.136-0.199	0.075	69	green material	0	1.1	<0.01	0.052	0.045	0.11	0.012	1.3
									15	0.46	<0.01	0.081	0.076	0.13	0.021	0.78
								grain	35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	35	0.61	0.014	0.22	0.15	0.26	0.053	1.3

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg. **residue in control
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

Table 6.3.1- 47: Residues of prothioconazole-desthio in/on spring wheat applied with fluoxastrobin & prothioconazole EC 200

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
13-2138 13-2138-02 GLP: yes 2013	Wheat, spring Kadrilj	Germany [redacted] [redacted]Europe, North	200 EC	2	0.15	0.038	69	green material	0(1)	0.25	0.13	0.11	0.096	0.11	0.018	0.71
									0	0.92	0.12	0.11	0.093	0.11	0.018	1.4
									7	0.51	0.24	0.14	0.13	0.13	0.023	1.2
									14	0.33	0.26	0.18	0.15	0.18	0.038	1.1
									21	0.19	0.32	0.21	0.17	0.19	0.046	1.1
									28	0.074	0.18	0.16	0.13	0.13	0.028	0.70
									35	0.11	0.21	0.15	0.13	0.12	0.031	0.75
								grain	54	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	54	0.11	0.17	0.11	0.12	0.13	0.017	0.66
13-2138 13-2138-01 GLP: yes 2013	Wheat, spring Taifun	Germany [redacted] Europe, North	200 EC	2	0.15	0.050	69	green material	0(1)	0.063	0.081	0.068	0.072	0.053	0.011	0.35
									0	0.90	0.094	0.079	0.089	0.073	0.015	1.3
									7	0.18	0.088	0.087	0.10	0.072	0.014	0.54
									14	0.055	0.075	0.064	0.10	0.070	0.012	0.41
									21	0.042	0.064	0.085	0.088	0.059	0.012	0.35
									28	0.023	0.051	0.086	0.091	0.063	0.012	0.33
									28	0.025	0.052	0.079	0.086	0.059	0.012	0.31
								grain	35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
									57	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	35	0.027	0.044	0.099	0.086	0.070	0.016	0.34
									57	0.038	0.020	0.035	0.035	0.031	<0.01	0.17

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg.
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

Table 6.3.1-47 (Cont'd): Residues of prothioconazole-desthio in/on spring wheat applied with fluoxastrobin & prothioconazole EC 200

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
13-2138 13-2138-03 GLP: yes 2013	Wheat, spring Alderon; (feed)	United Kingdom [redacted] Europe, North	200 EC	2	0.15-0.158	0.0749-0.075	69	green material	0[(1)]	0.45	0.061	0.15	0.083	0.089	<0.01	0.84
									0	1.8	0.065	0.14	0.069	0.070	<0.01	2.2
									7	1.1	0.13	0.21	0.14	0.14	0.018	1.7
									14	0.64	0.17	0.25	0.17	0.14	0.023	1.4
									21	0.37	0.14	0.22	0.14	0.12	0.021	1.0
									28	0.50	0.14	0.24	0.16	0.12	0.023	1.2
									24	0.44	0.18	0.27	0.18	0.14	0.028	1.2
								grain	35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	35	0.92	0.31	0.64	0.42	0.30	0.055	2.6

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg.
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

In the 5 trials conducted in 2013 (reports13-2138 and 13-2159), residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in wheat grain at harvest were <0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg. In straw, the residues at harvest ranged as follows :

- between 0.038 mg/kg and 0.92 mg/kg for prothioconazole-desthio,
- between 0.014 mg/kg and 0.31 mg/kg for prothioconazole-alpha-hydroxy,
- between 0.035 mg/kg and 0.64 mg/kg for prothioconazole-3-hydroxy-desthio,
- between 0.035 mg/kg and 0.42 mg/kg for prothioconazole-4-hydroxy-desthio,
- between 0.031 mg/kg and 0.30 mg/kg for prothioconazole-5-hydroxy-desthio,
- between < 0.01 mg/kg and 0.055 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio,
- between 0.17 mg/kg and 2.6 mg/kg for total residue.

Conclusion

Four wheat residue trials were conducted with Fluoxastrobin & Prothioconazole EC 200 in northern Europe. The product application corresponded to a prothioconazole rate of 2x175 g a.s./ha. In trial 13-2159-02, the first application was underdosed by 9.3% and the second application overdosed by 6.0%. In trial 13-2138-03, the first application overdosed by approximately 6%.

Residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in wheat grain at harvest were <0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg.

In straw, the residues at harvest ranged as follows :

- between 0.038 mg/kg and 0.92 mg/kg for prothioconazole-desthio,
- between 0.014 mg/kg and 0.31 mg/kg for prothioconazole-alpha-hydroxy,
- between 0.035 mg/kg and 0.64 mg/kg for prothioconazole-3-hydroxy-desthio,
- between 0.035 mg/kg and 0.42 mg/kg for prothioconazole-4-hydroxy-desthio,
- between 0.031 mg/kg and 0.30 mg/kg for prothioconazole-5-hydroxy-desthio,
- between < 0.01 mg/kg and 0.055 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio,
- between 0.17 mg/kg and 2.6 mg/kg for total residue.

EU - southern region

Table 6.3.1- 48: Number of residue trials conducted per geographical region and vegetation period

Year	GAP	Formulation	N° of trials	Study number Report No.	Dossier Reference
Wheat EU foliar spray residue trials – southern EU					
2000	2 x 150 g a.s./ha BBCH69	EC200 (100 g/L fluoxastrobin, 100 g/L prothioconazole)	4	RA-2012/00 M-078033-02-1	KCA 6.3.1/19
2000	2 x 200 g a.s./ha BBCH69	EC460 (160 g/L prothioconazole, 300 g/L spiroxamine)	2	RA-2092/00 M-087669-01-1	KCA 6.3.1/09
2003	2 x 150 g a.s./ha BBCH69	EC300 (75 g/L fluoxastrobin, 150 g/L prothioconazole, 75 g/L trifloxystrobin)	2	RA-2019/03 M-060549-02-1	KCA 6.3.1/20
2003	2 x 175 g a.s./ha BBCH69	SC325 (175 g/L prothioconazole, 150 g/L trifloxystrobin)	2	RA-2109/03 M-068785-01-1	KCA 6.3.1/21
2007	2 x 187 g a.s./ha BBCH69	EC225 (75 g/L bixafen, 150 g/L prothioconazole)	4	RA-2038/07 M-298110-02-1	KCA 6.3.1/22
2007	2 x 150 g a.s./ha BBCH69	EC400 (50 g/L bixafen, 100 g/L prothioconazole, 250 g/L spiroxamine)	4	RA-2041/07 M-298650-02-1	KCA 6.3.1/23
2010	2 x 200 g a.s./ha BBCH69	EC150 (50 g/L fluoxastrobin, 100 g/L prothioconazole)	7	10-2156 M-399682-02-1	KCA 6.3.1/24
2010	2 x 175 g a.s./ha BBCH69	EC190 (40 g/L bixafen, 50 g/L fluoxastrobin and 100 g/L prothioconazole)	2	10-2207 M-414694-01-1	KCA 6.3.1/25
TOTAL southern EU region			**27**		

Report: KCA 6.3.1/19 [redacted]; 2001; M-078033-02-1
Title: Determination of residues of HEC 5725 & JAU 6476-Desthio on winter wheat and hard wheat following spray application of HEC 5725 & JAU 6476 200 EC in Spain, Italy and France
Report No.: RA-2012/00
Document No.: M-078033-02-1
Guideline(s): EU-Ref: Council Directive 91/414/EEC of 15 July, 1991, Annex II, part A, point 6 and Annex III, part A, point 8
Residues in or on Treated Products, Food and Feed
Guideline deviation(s): not specified
GLP/GEP: **yes**

Materials and Methods

In the vegetation period of 1999/2000, a set of 4 residue trials was conducted in southern Europe. The trials were located in Spain, Italy and southern France (2 trials).

In each trial, wheat was treated twice at a product rate of 1.5 L/ha fluoxastrobin & prothioconazole EC 200 corresponding to 0.15 kg a.s./ha of prothioconazole. The water rate was 300 L/ha in all trials.

The spray interval ranged from 6 to 26 days. The time of application was:
1. when the flag leaf sheath was opening (BBCH 47) in 3 trials and at stage BBCH 61 in one trial,
2. at the end of flowering (BBCH 69) in 3 trials and at stage BBCH 71 in one trial.

Samples were taken at the following intervals:
- prior to and immediately after the final application;
- at a pre-harvest interval of 35 days as well as 47-57 days after the final treatment (BBCH stage 89-92).

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:
The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 49.

Table 6.3.1- 49: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	309	10.3	RA-2012/00 M-078033-02-1
		grain	274	9.1	
		Rest of plant	309	10.3	
		straw	274	9.1	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown inTable 6.3.1- 50. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 50: RA-2012/00: Concurrent recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Wheat grain / Ear	0.01	101; 101; 97	100	2.3	0.01
		0.10	105; 103; 105; 104; 104; 102; 106; 104; 108; 102; 102; 105; 102; 103; 100; 97; 103; 103; 102; 104; 104; 102; 102; 109; 100; 102; 99	103	2.4	
		Overall (n = 30)		**103**	**2.6**	
Prothio-conazole-desthio	Rest of plant	0.05	107; 98; 107	104	5.0	0.05
		0.50	105; 102; 102; 103; 106; 105; 102; 101; 104; 103; 100; 99; 120; 98; 102; 101; 99; 103	103	4.6	
		Overall (n = 21)		**103**	**4.6**	
Prothio-conazole-desthio	Straw	0.05	98; 97; 96	97	1.0	0.05
		0.50	106; 102; 102; 102; 103; 97; 100; 103; 103; 109; 103; 99; 105; 101; 101	102	2.8	
		Overall (n = 18)		**102**	**3.3**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:

The residue results are summarised in Table 6.3.1- 51.

Table 6.3.1- 51: Residues of prothioconazole-desthio in/on winter and durum wheat applied with fluoxastrobin & prothioconazole EC 200

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2012/00 R 2000 0149/5 0149-00 GLP: yes 2000	Wheat, durum Creso	Italy I-[redacted] Europe, South	200 EC	2	0.1500	0.05000	69	ear	0(1) 0	<0.01 2.3
								rest of plant	0(1) 0	<0.05 1.0
								straw	35 47	0.23 0.36
								grain	35 47	<0.01 <0.01
RA-2012/00 R 2000 0151/7 0151-00 GLP: yes 2000	Wheat, winter Sideral	France F-[redacted] Europe, South	200 EC	2	0.1500	0.05000	69	ear	0(1) 0	0.12 1.2
								rest of plant	0(1) 0	0.23 0.86
								straw	35 49	0.10 0.22
								grain	35 49	<0.01 <0.01
RA-2012/00 R 2000 0148/7 0148-00 GLP: yes 2000	Wheat, durum Soisson	Spain E-[redacted] Europe, South	200 EC	2	0.1500	0.05000	71 **	ear	0(1) 0	0.10 1.9
								rest of plant	0* 0	0.08 2.7
								straw	34 51	0.29 0.22
								grain	34 51	<0.01 <0.01
RA-2012/00 R 2000 0150/9 0150-00 GLP: yes 2000	Wheat, winter Tremie	France F-[redacted] Europe, South	200 EC	2	0.1500	0.05000	69	ear	0(1) 0 35	0.04 1.6 0.05
								rest of plant	0(1) 0 35	0.11 1.4 0.13
								straw	57	0.30
								grain	57	<0.01

FL = Formulation, DALT = Days after last treatment
* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) : before last application
** : the 2nd treatment was carried out at a later stage as originally intended (BBCH 71 instead of 69).

In the 4 trials conducted in 2000 (report RA-2012/00), residues of prothioconazole-desthio in wheat grain at harvest were always below the limit of quantification (LOQ) of 0.01 mg/kg. The residues in straw at harvest ranged between 0.12 mg/kg and 0.36 mg/kg.

Conclusion

Four wheat residue trials were conducted with fluoxastrobin & prothioconazole EC 200 in southern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha.

Residues of prothioconazole-desthio in wheat grain at harvest were always below the limit of quantification (LOQ) of 0.01 mg/kg. The residues in straw at harvest ranged between 0.12 mg/kg and 0.36 mg/kg.

Report:	KCA 6.3.1/09[redacted].;[redacted]; 2001; M-087669-01-1
Title:	Determination of residues of JAU 6476-desthio & KWG 4168 on spring wheat following spray application of JAU 6476 & KWG 4168 460 EC in Great Britain, France, Germany and Italy
Report No.:	RA-2092/00
Document No.:	M-087669-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of 15 July 1991, Annex II, part A, point 6 and Annex III, part A, point 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2000, a set of 2 residue trials was conducted on wheat in southern Europe. The studies were located in southern France and Italy.

In each trial, wheat was treated twice at a product rate of 1.25 L/ha JAU 6476 & KWG 4168 EC 460 corresponding to 200 g a.s./ha of prothioconazole. The water rate was 300 L/ha The spray interval ranged from 13 to 32 days. The time of application was:

1) during stem elongation (BBCH 32), and
2) at the end of flowering (BBCH 69),

with the last treatment performed 42 days prior to harvest.

Samples were taken at the following intervals:
– immediately after the final application,
– at a pre-harvest interval of 28 days, 35 days, 42 and at grain maturity, *i.e.* 49 days after the final treatment.

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 52.

Table 6.3.1- 52: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	318	10.6	RA-2092/00 M-087669-01-1
		grain	303	10.1	
		Rest of plant	318	10.6	
		Green material	345	11.5	
		straw	303	10.1	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).

Sample extracts were measured within 24 hours, or if not acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 53. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 53: RA-2092/00: Concurrent recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Wheat grain / Ear	0.01	101; 101; 97	100	2.3	0.01
		0.10	105; 103; 105; 104; 104; 102; 106; 104; 108; 102; 102; 105; 102; 103; 100; 97; 103; 103; 102; 104; 104; 102; 102; 109; 100; 102; 99	103	2.4	
		Overall (n = 30)		**103**	**2.6**	
Prothio-conazole-desthio	Rest of plant	0.05	107; 98; 107	104	5.0	0.05
		0.50	105; 102; 102; 105; 106; 105; 102; 101; 104; 103; 100; 99; 120; 98; 102; 101; 99; 103	103	4.6	
		Overall (n = 21)		**103**	**4.6**	
Prothio-conazole-desthio	Straw	0.05	98; 97; 96	97	1.0	0.05
		0.50	106; 102; 102; 102; 103; 97; 100; 103; 103; 109; 103; 99; 105; 101; 101	102	2.8	
		Overall (n = 18)		**102**	**3.3**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:

The residue results are summarised in Table 6.3.1- 54.

Table 6.3.1- 54: Residues of prothioconazole-desthio in/on spring wheat applied with prothioconazole & spiroxamine EC 460

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2092/00 R 2000 0434/6 0434-00 GLP: yes 2000	Wheat, spring Centauro	Italy [redacted] Europe, South	460 EC	2	0.2000	0.06672	69	green material	0	2.7
								ear	28 35	0.05 0.06
								rest of plant	28 35	0.15 0.18
								grain	42 49	<0.01 <0.01
								straw	42 49	0.18 0.17
RA-2092/00 R 2000 0082/0 0082-00 GLP: yes 2000	Wheat, spring Furio	France F-[redacted] Europe, South	460 EC	2	0.2000	0.06672	69	ear	0 28	1.5 0.06
								rest of plant	0 28	2.9 0.63
								grain	35 42 49	<0.01 <0.01 <0.01
								straw	35 42 49	1.1 0.90 1.2

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio

In the 2 trials conducted in 2000 (report RA-2092/00), residues of prothioconazole-desthio in wheat grain at harvest were always <0.01 mg/kg. The residues in straw at harvest ranged between 0.18 mg/kg and 1.2 mg/kg.

Conclusion

Four wheat residue trials were conducted with prothioconazole & spiroxamine EC 460 in southern Europe. The product application corresponded to a prothioconazole rate of 2x200 g a.s./ha.

Residues of prothioconazole-desthio in wheat grain at harvest were always <0.01 mg/kg. The residues in straw at harvest ranged between 0.18 mg/kg and 1.2 mg/kg.

Report:	KCA 6.3.1/20[redacted],;[redacted]; 2004; M-060549-02-1
Title:	Determination of residues of HEC 5725, JAU 6476 and trifloxystrobin in/on wheat following spray application of HEC 5725 & JAU 6476 & CGA 279202 300 EC in the field in Southern France and Spain
Report No.:	RA-2019/03
Document No.:	M-060549-02-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of 15 July, 1991, Annex II, part A, point 6 and Annex III, part A, point 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2003, a set of 2 residue trials was conducted in southern Europe. The trials were located in southern France and Spain.

In each trial, wheat was treated twice at a product rate of 1.0 L/ha fluoxastrobin & prothioconazole & trifloxystrobin EC 300 corresponding to 0.150 kg/ha of prothioconazole. The employed water rate was 300 L/ha in both trials. The applications were scheduled for growth stages BBCH 47 and 69. The applications were carried out at growth stages 47-61 (1st application) and 69 (2nd application; corresponding to intervals of 10-15 days).

Samples were taken at the following intervals:
- prior to and immediately after the final application for one trial,
- at a pre-harvest interval of 34-35 days,
- at harvest (BBCH 89), 41-46 days after the final treatment.

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2.1) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 55.

Table 6.3.1- 55: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	195	6.5	RA-2019/03 M-060549-02-1
		grain	168	5.6	
		rest of plant	195	6.5	
		straw	168	5.6	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 56. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 56: RA-2019/03: Concurrent recoveries and validation recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Wheat grain	0.01	94; 94; 92	93	1.2	0.01
		0.10	*100; 95; 88; 95*	95	5.2	
		Overall (n = 7)		**94**	**3.8**	
Prothio-conazole-desthio	Straw	0.05	98; 94; 108	100	7.2	0.05
		0.50	*89; 98; 97*	95	5.2	
		Overall (n = 6)		**97**	**6.4**	
Prothio-conazole-desthio	Rest of plant	0.05	101; 92; 98	97	4.7	0.05
		0.50	*101; 94*	98	-	
		Overall (n = 5)		**97**	**4.2**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation. Concurrent recoveries in italics.

- Residue results:
The residue results are summarised in Table 6.3.1-57.

Table 6.3.1- 57: Residues of prothioconazole-desthio in/on wheat applied with fluoxastrobin & prothioconazole & trifloxystrobin EC 300

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2019/03 R 2003 0134/0A 0134-03A GLP: yes 2003	Wheat Frelon	France F-[redacted] Europe, South	300 EC	2	0.15	0.050	69	rest of plant	0[1] 0	0.24 1.7
								ear	0[1] 0	0.21 2.1
								grain	35 41	<0.01 <0.01
								straw	35 41	<0.05 0.27
RA-2019/03 R 2003 0257/6 0257-03 GLP: yes 2003	Wheat Yecora	Spain E-[redacted] Europe, South	300 EC	2	0.1422	0.04740	69	grain	34 46	<0.01 <0.01
								straw	34 46	0.40 0.62

Residues determined as prothioconazole-desthio and calculated as prothioconazole-desthio
[1] : before last application

In the 2 trials conducted in 2003 (report RA-2019/03), residues of prothioconazole-desthio in wheat grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.27 mg/kg and 0.62 mg/kg.

Conclusion

Four wheat residue trials were conducted with fluoxastrobin & prothioconazole & trifloxystrobin EC 300 in northern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha. Residues of prothioconazole-desthio in wheat grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.27 mg/kg and 0.62 mg/kg.

Report: KCA 6.3.1/21[redacted]; 2004; M-068785-01-1
Title: Determination of residues of trifloxystrobin, CGA321113, and JAU 6476-desthio in/on wheat, soil following spray application of CGA279202 & JAU 6476 (325 SC) in the field in Italy and Portugal
Report No.: RA-2109/03
Document No.: M-068785-01-1
Guideline(s):
Guideline deviation(s): --
GLP/GEP: yes

Materials and Methods

A total of two trials were conducted in southern Europe in 2003, in Italy and Portugal.
Two applications were carried out with the formulation prothioconazole & trifloxystrobin SC 325. The first application was done at BBCH growth stage 47 and the second at growth stage 69. The interval between applications was 9 and 15 days for the two trials.

The application rate was 0.92-1.0 L product/ha corresponding to 0.161-0.175 kg a.s./ha of prothioconazole (the 1st application in trial R 2003 0942/2 was under-dosed by 6.3%, and the 2nd application in trial R 2003 0981/3 was under-dosed by 8%). The water rate was 277-300 L/ha.
In trial R 2003 0981/3, samples of ear and rest of plant were taken on day 0 after the last application, and samples of grain and straw were taken at harvest (day 35).
In trial R 2003 0942/2, samples of ear and rest of plant were taken day -9 (immediately after the 1st application). Samples of grain and straw were taken at harvest (day 35) and on day 44 after the last application when the commodity had reached final harvest maturity.

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 58.

Table 6.3.1- 58: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	268	8.9	RA-2109/03 M-068785-01-1
		grain	224	7.5	
		green material	268	8.9	
		straw	224	7.5	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.10).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of sample s ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 59. All results of the method validation are in accordance with the general requirements for residue analytical methods; therefore the method was validated successfully.

Table 6.3.1- 59: RA-2109/03: Concurrent recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Wheat grain / Ear	0.01	98; 103; 85	95	9.7	0.01
		0.10	96; 96	96	-	
		Overall (n = 5)		**96**	**6.9**	
Prothio-conazole-desthio	Green material / Rest of plant	0.05	109; 106; 98	104	5.5	0.05
		0.50	98; 102	100	-	
		Overall (n = 5)		**103**	**4.8**	
Prothio-conazole-desthio	Straw	0.05	95; 77; 96	89	12.0	0.05
		0.50	90; 97	94	-	
		Overall (n = 5)		**91**	**9.1**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:

The residue results are summarised in Table 6.3.1- 60.

Table 6.3.1- 60: Residues of prothioconazole-desthio in/on soft wheat applied with prothioconazole & trifloxystrobin SC 325

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2109/03 R 2003 0942/2 0942-03 GLP: yes 2003	Wheat soft Centauro	Italy I-[redacted] Europe, South	325 SC	2	0.1638 0.1750	0.05828	69	green material	-9	0.75
								ear	-9	0.11
								grain	35 44	<0.01 <0.01
								straw	35 44	0.64 0.75
RA-2109/03 R 2003 0981/3* 0981-03 GLP: yes 2003	Wheat soft Bancal	Portugal P-[redacted] Europe, South	325 SC	2	0.1614 0.1750	0.05828	69	green material	0	0.99
								ear	0	1.4
								grain	35	<0.01
								straw	35	1.9

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio

In the 2 trials conducted in 2003 (report RA-2109/03), residues of prothioconazole-desthio in wheat grain at harvest were < 0.01 mg/kg. The residues in straw at harvest ranged between 0.75 mg/kg and 1.9 mg/kg.

Conclusion

Two wheat residue trials were conducted with prothioconazole + trifloxystrobin SC 325 in southern Europe. The product application corresponded to a prothioconazole rate of 2x175 g a.s./ha.
(the 1st application in trial R 2003 0942/2 was under-dosed by 6.3%, and the 2nd application in trial R 2003 0981/3 was under-dosed by 8%).

Residues of prothioconazole-desthio in wheat grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.75 mg/kg and 1.9 mg/kg.

Report:	KCA 6.3.1/22[redacted],[redacted]; 2008; M-298110-02-1
Title:	Determination of the residues of BYF 00587 and JAU 6476 in/on winter wheat and wheat, durum after spraying of BYF 00587 & JAU 6476 (225 EC) in the field in Southern France, Spain, Portugal and Italy
Report No.:	RA-2038/07
Document No.:	M-298110-02-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

Four trials were conducted in the vegetation period of 2007, in southern France, Spain, Portugal and Italy. The trials were conducted on winter wheat (3 trials) and durum varieties (1 trial). Bixafen & prothioconazole EC 225 was applied twice at the required rates of 1.25 L product/ha corresponding to 0.188 kg/ha prothioconazole. The treatments were carried out at the growth stages "flag leaf sheath opening" (BBCH 47) and "end of flowering" (BBCH 69). Depending on the trial, the spray-interval ranged from 14 - 21 days. The water rate was 300 L/ha in all trials.
Samples of green material were taken just prior to and immediately after the final. Two trials were designed as decline series, i.e. samples were collected further 7, 14, and 28 days after the final treatment. First grain samples were taken on day 35-63; in one trial with early sampling of grain (GS 87), an additional sample was taken on day 53 at GS 89.

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 61.

Table 6.3.1- 61: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	105	3.5	RA-2038/07 M-298110-02-1
		grain	103	3.4	
		green material	141	4.7	
		rest of plant	105	3.5	
		straw	103	3.4	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 62. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 62: RA-2038/07: Concurrent recoveries for the determination of prothioconazole-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain #	0.01	97; 97; 96; 100; 93; 94; 99	97	2.6	0.01
		0.1	95; 93; 95; 91; 93; 100	95	3.3	
		1.0	92; 95; 95; 89; 93; 99	94	3.6	
		5.0	89	--	--	
		10.0	86	--	--	
		Overall Recovery (n = 21)		**94**	**3.9**	
Prothio-conazole-desthio	Cereal green material*	0.01	98; 94; 90; 97	95	3.8	0.01
		0.1	97; 90; 95; 88	93	4.5	
		1.0	87; 95; 95; 91; 89	91	3.9	
		5.0	89; 85; 82	85	4.1	
		10.0	83; 86; 85	85	1.8	
		Overall Recovery (n = 19)		**90**	**5.5**	
Prothio-conazole-desthio	Cereal straw	0.01	92; 88; 104; 93; 104	96	7.7	0.01
		0.1	95; 88; 94; 103	95	6.5	
		1.0	88; 93; 91; 93	91	2.6	
		5.0	88; 80; 87	85	5.1	
		10.0	84	--	--	
		Overall Recovery (n = 17)		**92**	**7.3**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
These recoveries were performed with barley and wheat commodities during the conduct of the studies RA-2037/07, RA-2038/07, RA-2039/07, RA-2040/07, RA-2041/07, RA-2042/07, RA-2043/07, RA-2045/07, RA-2046/07, RA-2049/07 and RA-2050/07. Cereal summarises wheat and wheat. Recoveries for wheat are also valid for wheat.
* = Recoveries for rest of plant are covered by recoveries for green material.
# = Recoveries for ear are covered by recoveries for grain.

- Residue results:
The residue results are summarised in Table 6.3.1- 63.

Table 6.3.1- 63: Residues of prothioconazole-desthio in/on winter and durum wheat applied with bixafen & prothioconazole EC 225

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2038/07 R 2007 0424/0 0424-07 GLP: yes 2007	Wheat, winter Galeira	Portugal P-[redacted] Europe, South	225 EC	2	0.1875	0.06255	69	green material	0 (1) 0	0.07 1.4
								straw	53	0.67
								grain	53	<0.01
								rest of plant	35	0.60
								ear	35	0.08
RA-2038/07 R 2007 0425/9 0425-07 GLP: yes 2007	Wheat, durum Simeto	Italy I-[redacted] Europe, South	225 EC	2	0.1875	0.06255	69	green material	0 (1) 0 7 14 28	0.78 2.7 2.2 1.4 1.4
								straw	35 53	1.5 1.6
								grain	35 53	0.02 <0.01
RA-2038/07 R 2007 0423/2 0423-07 GLP: yes 2007	Wheat, winter Bolido R1	Spain E-[redacted]) Europe, South	225 EC	2	0.1875	0.06255	69	green material	0 (1) 0	0.27 0.31
								straw	54	0.27
								grain	54	<0.01
								rest of plant	36	0.20
								ear	36	0.02
RA-2038/07 R 2007 0422/4 0422-07 GLP: yes 2007	Wheat, winter Andalou	France F-[redacted] ope, South	225 EC	2	0.1875	0.06255	69	green material	0 (1) 0 7 14 28	0.33 0.92 0.70 0.24 0.05
								straw	63	0.10
								grain	63	<0.01
								ear	35	<0.01
								rest of plant	35	0.07

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) before last application
GS = growth stage (BBCH code) at last application
FL = formulation

In the 4 trials conducted in 2007 (report RA-2038/07), residues of prothioconazole-desthio in wheat grain at harvest ranged from <0.01 mg/kg to 0.02 mg/kg. The residues in straw at harvest ranged between 0.10 mg/kg and 1.6 mg/kg.

Conclusion

Four wheat residue trials were conducted with bixafen & prothioconazole EC 225 in southern Europe. The product application corresponded to a prothioconazole rate of 2x187.5 g a.s./ha.

Residues of prothioconazole-desthio in wheat grain at harvest ranged from <0.01 mg/kg to 0.02 mg/kg. The residues in straw at harvest ranged between 0.10 mg/kg and 1.6 mg/kg.

Report:	KCA 6.3.1/23 [redacted]; [redacted]; 2008; M-298650-02-1
Title:	Determination of the residues of BYF 00587, JAU 6476 and KWG 4168 in/on winter wheat and wheat, durum after spraying of BYF 00587 & JAU 6476 & KWG 4168 (400 EC) in the field in Southern France, Italy, Spain and Greece
Report No.:	RA-2041/07
Document No.:	M-298650-02-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2007, a set of 4 residue trials on winter wheat (3 trials) and durum varieties (1 trial) was conducted in southern Europe. The trials were located in Italy, southern France, Spain and Greece.

In each trial, wheat was treated twice at a product rate of 1.5 L/ha BYF 00587 (bixafen) & JAU 6476 (prothioconazole) & KWG 4168 (spiroxamine) EC 400 corresponding to 0.150 kg/ha prothioconazole. The treatments were carried out at the growth stages "flag leaf sheath opening" (BBCH 47, except for one trial at BBCH52) and "end of flowering" (BBCH 69). Depending on the study, the spray interval ranged from 8-26 days. The water rate was 300 L/ha in all trials.

Samples were taken at the following intervals:
- prior to and immediately after the final application,
- at a pre-harvest interval of 35 days after the final treatment,
- at harvest (BBCH 89).

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 64.

Table 6.3.1- 64: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	ear	105	3.5	RA-2041/07 M-298650-02-1
		grain	97	3.2	
		green material	141	4.7	
		rest of plant	105	3.5	
		straw	97	3.2	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.1- 65. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 65: RA-2041/07: Concurrent recoveries for the determination of prothioconazole-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain #	0.01	97; 97; 96; 100; 93; 94; 99	97	2.6	0.01
		0.1	95; 93; 95; 91; 93; 100	95	3.3	
		1.0	92; 95; 95; 89; 93; 99	94	3.6	
		5.0	89	--	--	
		10.0	86	--	--	
		Overall Recovery (n = 21)		**94**	**3.9**	
Prothio-conazole-desthio	Cereal green material*	0.01	98; 94; 90; 97	95	3.8	0.01
		0.1	97; 90; 95; 88	93	4.5	
		1.0	87; 95; 95; 91; 89	91	3.9	
		5.0	89; 85; 82	85	4.1	
		10.0	83; 86; 85	85	1.8	
		Overall Recovery (n = 19)		**90**	**5.5**	
Prothio-conazole-desthio	Cereal straw	0.01	92; 88; 104; 93; 104	96	7.7	0.01
		0.1	95; 88; 94; 103	95	6.5	
		1.0	88; 93; 91; 93	91	2.6	
		5.0	88; 80; 87	85	5.1	
		10.0	84	--	--	
		Overall Recovery (n = 17)		**92**	**7.3**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
These recoveries were performed with wheat and barley commodities during the conduct of the studies RA-2037/07, RA-2038/07, RA-2039/07, RA-2040/07, RA-2041/07, RA-2042/07, RA-2043/07, RA-2045/07, RA-2046/07, RA-2049/07 and RA-2050/07. Cereal summarises barley and wheat. Recoveries for barley are also valid for wheat.
*= Recoveries for rest of plant are covered by recoveries for green material.
= Recoveries for ear are covered by recoveries for grain.

- Residue results:

The residue results are summarised in Table 6.3.1- 66.

Table 6.3.1- 66: Residues of prothioconazole-desthio in/on winter and durum wheat applied with bixafen & prothioconazole & spiroxamine EC 400

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2041/07 R 2007 0526/3 0526-07 GLP: yes 2007	Wheat, winter Moro (durum)	Greece GR-[redacted] Europe, South	400 EC	2	0.1500	0.05000	69	green material	0 (1) 0	1.1 2.7
								straw	35	1.2
								grain	35	0.01
RA-2041/07 R 2007 0525/5 0525-07 GLP: yes 2007	Wheat, winter Bolido R1	Spain E-[redacted] Europe, South	400 EC	2	0.1500	0.05000	69	green material	0 (1) 0	0.19 1.1
								straw	55	0.72
								grain	55	<0.01
								rest of plant	36	0.35
								ear	36	0.09
RA-2041/07 R 2007 0446/1 0446-07 GLP: yes 2007	Wheat, durum Simeto	Italy I-[redacted] Europe, South	400 EC	2	0.1500	0.05000	69	green material	0 (1) 0	0.13 1.4
								straw	44	0.73
								grain	44	<0.01
								ear	35	0.08
								rest of plant	35	0.63
RA-2041/07 R 2007 0445/3 0445-07 GLP: yes 2007	Wheat, winter Autan	France F-[redacted] Europe, South	400 EC	2	0.1500	0.05000	69	green material	0 (1) 0	0.17 0.99
								straw	44	0.05
								grain	44	<0.01
								ear	35	0.04
								rest of plant	35	0.05

Determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) before last application
GS = growth stage (BBCH code) at last application
FL = formulation

In the 4 trials conducted in 2007 (report RA-2041/07), residues of prothioconazole-desthio in wheat grain at harvest ranged from <0.01 mg/kg to 0.01 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 1.2 mg/kg.

Conclusion

Four wheat residue trials were conducted with bixafen & prothioconazole & spiroxamine EC 400 in southern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha. Residues of prothioconazole-desthio in wheat grain at harvest ranged from <0.01 mg/kg to 0.01 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 1.2 mg/kg.

Report:	KCA 6.3.1/24[redacted];[redacted]; 2011; M-399682-02-1
Title:	Determination of the residues of fluoxastrobin and prothioconazole in/on durum wheat and winter wheat after spraying of Fluoxastrobin & Prothioconazole EC 150 in the field in France (South), Spain, Italy, Portugal and Greece
Report No.:	10-2156
Document No.:	M-399682-02-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2010, a set of 7 residue trials on spring and winter wheat was conducted in southern Europe. The trials were located in France (2), Greece, Italy, Spain (2) and Portugal.
In each trial, wheat was treated twice at a product rate of 2 L/ha fluoxastrobin & prothioconazole EC 150 corresponding to 0.200 kg a.s./ha of prothioconazole. The water rate was 300-400 L/ha. The spray interval ranged between 14 and 19 days. The treatments were carried out at the growth stages BBCH 45-61 and BBCH 69 (end of flowering).

Samples were taken at the following intervals:
- prior to and immediately after the final application for all the trials and 7, 14, 21 and 28 days after for 4 trials,
- at a pre-harvest interval of 34-35 days after the final treatment,
- at maturity (BBCH 89), 41-53 after the last application.

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).
Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio were determined according to method 00979/M001 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 67.

Table 6.3.1- 67: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	grain	148	4.9	10-2156 M-399682-02-1
		ear	146	4.9	
		green material	187	6.2	
		rest of plant	146	4.9	
		straw	152	5.1	
	prothioconazole-alpha-hydroxy-desthio prothioconazole-3-hydroxy-desthio prothioconazole-4-hydroxy-desthio prothioconazole-5-hydroxy-desthio prothioconazole-6-hydroxy-desthio	grain	155	5.2	
		ear	154	5.1	
		green material	189	6.3	
		rest of plant	140	4.7	
		straw	160	5.3	

These storage periods are covered by the storage stability studies, i.e. analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio above 30% of the LOQ was found in the control samples except for prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio and prothioconazole-5-hydroxy-desthio in green material where the residues were between 0.02 and 0.10 mg/kg at day0 before the last application and in straw between 0.01 and 0.10 mg/kg at DALT34 and between 0.01 and 0.12 mg/kg at DALT 41.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110% except for prothioconazole-alpha-hydroxy-desthio in green material at 0.80 mg/kg (118%) and in straw at 0.01 and 0.10 mg/kg (116% and 111%, respectively) and for prothioconazole-3-hydroxy-desthio in green material at 0.80 mg/kg (117%) and in straw at 0.10 mg/kg (114%). The single and overall mean recoveries are shown in Table 6.3.1- 68 to Table 6.3.1- 73. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 68: 10-2156: Concurrent recoveries for the determination of prothioconazole-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	green material	0.01	83; 95; 104; 118; 120	104	15.0	0.01
		0.10	101; 101; 104	102	1.7	
		5	85; 86; 87	86	1.2	
		Overall Recovery (n = 11)		**99**	**13.0**	
	straw [a]	0.01	100; 109; 114; 77; 88; 113	100	14.9	0.01
		0.10	93; 94; 95; 95; 100	95	2.8	
		5	71; 80; 84; 87; 91; 94	85	9.8	
		Overall Recovery (n = 17)		**93**	**12.7**	
	ear	0.01	87; 99; 101; 103; 104	99	7.0	0.01
		0.10	97; 101; 102; 103; 104	101	2.7	
		5	83; 89; 90; 92; 97	90	5.6	
		Overall Recovery (n = 15)		**97**	**7.1**	
	grain	0.01	82; 94; 95; 96; 105; 116; 91;104; 106; 107; 110; 72; 74; 88	96	13.8	0.01
		0.10	91; 93; 103; 95; 96; 96; 100; 104; 99; 100; 104	98	4.8	
		5	86; 91; 92; 96; 97; 98; 95; 95; 95; 96; 97; 96; 98; 98	95	3.5	
		Overall Recovery (n = 39)		**96**	**8.8**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-desthio, determined as JAU 6476-desthio and calculated as JAU 6476-desthio [a] covers rest of plant

Table 6.3.1-69: 10-2156: Concurrent recoveries for the determination of prothioconazole-alpha-hydroxy-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
prothiocon azole-alpha-hydroxy-desthio	green material	0.01	78; 106; 106	97	16.7	0.01
		0.10	109; 116; 120; 94; 108	109	9.1	
		0.80	117; 119	118 [c]	-	
		Overall Recovery (n = 10)		**107**	**12.1**	
	straw [a]	0.01	117; 119; 113	116 [c]	2.6	0.01
		0.10	112; 119; 103	111 [c]	7.2	
		0.80	118	-	-	
		Overall Recovery (n = 7)		**114 [c]**	**5.0**	
	grain [b]	0.01	78; 81; 84	81	3.7	0.01
		0.10	86; 102; 116	101	14.8	
		Overall Recovery (n = 6)		**91**	**16.2**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-alpha-hydroxy-desthio, determined as JAU 6476-alpha-hydroxy-desthio and calculated as JAU 6476-desthio. [a] covers rest of plant [b] covers ear [c] Accepted by study director because of good repeatability. Recoveries per fortification level and overall are close to guideline requirements.

Table 6.3.1- 70: 10-2156: Concurrent recoveries for the determination of prothioconazole-3-hydroxy-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
prothioconazole-3-hydroxy-desthio	green material	0.01	62; 86; 110	86	27.9 [c]	0.01
		0.10	99; 118; 120; 90; 107	107	11.9	
		0.80	116; 118	117 [d]	-	
		Overall Recovery (n = 10)		**103**	**18.2**	
	straw [a]	0.01	105; 112; 108	108	3.2	0.01
		0.10	111; 119; 113	114	3.6	
		0.80	114	-	-	
		Overall Recovery (n = 7)		**112 [d]**	**4.0**	
	grain [b]	0.01	94; 95; 99	96	2.8	0.01
		0.10	97; 115; 115	109	9.5	
		Overall Recovery (n = 6)		**103**	**9.6**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-3-hydroxy-desthio, determined as JAU 6476-3-hydroxy-desthio and calculated as JAU 6476-desthio.
[a] covers rest of plant [b] covers ear [c] accepted because recoveries per fortification level and overall are close to guideline requirements. [d] Accepted by study director because of good repeatability.

Table 6.3.1- 71: 10-2156: Concurrent recoveries for the determination of prothioconazole-4-hydroxy-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
prothioconazole-4-hydroxy-desthio	green material	0.01	63; 65; 89	72	20.0	0.01
		0.10	89; 94; 97; 76; 89	89	9.0	
		0.80	89; 93	91	-	
		Overall Recovery (n = 10)		**84**	**14.3**	
	straw [a]	0.01	91; 113; 116	107	12.8	0.01
		0.10	91; 94; 91	92	1.9	
		0.80	99	-	-	
		Overall Recovery (n = 7)		**99**	**10.9**	
	grain [b]	0.01	80; 80; 84	81	2.8	0.01
		0.10	80; 96; 110	95	15.7	
		Overall Recovery (n = 6)		**88**	**13.9**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-4-hydroxy-desthio, determined as JAU 6476-4-hydroxy-desthio and calculated as JAU 6476-desthio.
[a] covers rest of plant [b] covers ear

Table 6.3.1- 72: 10-2156: Concurrent recoveries for the determination of prothioconazole-5-hydroxy-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
prothioconazole-5-hydroxy-desthio	green material	0.01	62; 65; 98	75	26.6 [c]	0.01
		0.10	89; 91; 107, 75; 94	91	12.6	
		0.80	94; 98	96	-	
		Overall Recovery (n = 10)		**87**	**17.1**	
	straw[a]	0.01	94; 115; 101	103	10.3	0.01
		0.10	93; 99; 92	95	4.0	
		0.80	101	-	-	
		Overall Recovery (n = 7)		**99**	**7.9**	
	grain[b]	0.01	80; 89; 85	83	5.0	0.01
		0.10	81; 98; 119	99	19.2	
		Overall Recovery (n = 6)		**91**	**16.7**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-5-hydroxy-desthio, determined as JAU 6476-5-hydroxy-desthio and calculated as JAU 6476-desthio.
[a] covers rest of plant [b] covers ear. [c] accepted because recoveries per fortification level and overall are close to guideline requirements.

Table 6.3.1- 73: 10-2156: Concurrent recoveries for the determination of prothioconazole-6-hydroxy-desthio in wheat

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
prothioconazole-6-hydroxy-desthio	green material	0.01	68; 79; 86	78	11.7	0.01
		0.10	81; 84; 84; 75; 79	81	4.7	
		0.80	80; 83	82	-	
		Overall Recovery (n = 10)		**80**	**6.6**	
	straw[a]	0.01	82; 96; 91	90	7.9	0.01
		0.10	85; 90; 102	92	9.5	
		0.80	90	-	-	
		Overall Recovery (n = 7)		**91**	**7.3**	
	grain[b]	0.01	74; 79; 80	78	4.1	0.01
		0.10	73; 88; 105	89	18.1	
		Overall Recovery (n = 6)		**83**	**14.4**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-6-hydroxy-desthio, determined as JAU 6476-6-hydroxy-desthio and calculated as JAU 6476-desthio.
[a] covers rest of plant [b] covers ear

- Residue results:

The residue results are summarised in Table 6.3.1- 74.

Table 6.3.1- 74: Residues of prothioconazole-desthio in/on wheat applied with fluoxastrobin & prothioconazole EC 150

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
10-2156 10-2156-06 GLP: yes 2010	Wheat, winter Poison	Portugal Europe, South	150 EC	2	0.20	0.050	69	green material	0[1]	0.62	0.10	0.08	0.06	0.05	<0.01	0.92
									0	2.9	0.14	0.12	0.10	0.10	0.02	3.4
									7	1.3	0.33	0.18	0.16	0.16	0.02	2.2
									14	1.0	0.28	0.19	0.15	0.14	0.02	1.8
									21	0.96	0.43	0.25	0.20	0.19	0.03	2.1
								ear	28	0.23	0.12	0.06	0.04	0.03	<0.01	0.49
								rest of plant	28	1.0	0.13	0.22	0.16	0.14	0.02	1.7
								grain	48	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	48	1.7	0.33	0.43	0.37	0.27	0.07	3.2
10-2156 10-2156-07 GLP: yes 2010	Wheat, winter Yecora	Greece Europe, South	150 EC	2	0.20	0.067	69	green material	0[1]	0.86	0.35	0.09	0.14	0.05	<0.01	1.5
									0	1.7	0.28	0.08	0.13	0.04	<0.01	2.2
									7	1.4	0.69	0.18	0.38	0.11	0.03	2.8
									14	1.3	0.43	0.12	0.18	0.07	0.01	2.1
									28	0.86	0.39	0.10	0.29	0.07	0.02	1.7
								grain	35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.06
								straw	35	1.1	0.66	0.29	0.77	0.15	0.03	3.0

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg.
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

Table 6.3.1-74 (Cont'd): Residues of prothioconazole-desthio in/on wheat applied with fluoxastrobin & prothioconazole EC 150

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
10-2156 10-2156-03 GLP: yes 2010	Wheat, durum Perseo	Italy [redacted] Europe, South	150 EC	2	0.20	0.067	69	green material	0 (1) 0 7 14 28	0.60 2.6 1.9 1.1 0.99	<0.01 <0.01 <0.01 <0.01 <0.01	0.06 0.06 0.10 0.13 0.14	0.04 0.03 0.06 0.07 0.07	0.09 0.08 0.12 0.14 0.12	<0.01 <0.01 0.01 0.02 0.03	0.81 2.8 2.2 1.5 1.4
								grain	35 42	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06
								straw	35 42	1.6 2.3	0.02 0.03	0.34 0.35	0.19 0.19	0.41 0.38	0.09 0.10	2.7 3.4
10-2156 10-2156-04 GLP: yes 2010	Wheat, durum Saragolla	Italy [redacted] Europe, South	150 EC	2	0.20	0.067	69	green material	0 (1) 0	0.58 1.9	<0.01 <0.01	0.05 0.06	0.03 0.04	0.05 0.06	<0.01 <0.01	0.73 1.9
								grain	35 44	0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.06 <0.06
								straw	35 44	1.8 1.2	0.03 0.02	0.32 0.22	0.18 0.12	0.29 0.23	0.07 0.05	2.7 1.8

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg.
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio

Table 6.3.1-74 (Cont'd): Residues of prothioconazole-desthio in/on wheat applied with fluoxastrobin & prothioconazole EC 150

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
10-2156 10-2156-05 GLP: yes 2010	Wheat, winter Aubusson	France [redacted] Europe, South	150 EC	2	0.20	0.067	69	green material	0[1] 0	0.09 0.93	0.03 0.03	0.05 0.07	0.03 0.05	0.03 0.05	0.01 0.02	0.24 1.2
								grain	34 53	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06
								straw	34 53	0.10 0.22	0.08 0.08	0.35 0.32	0.22 0.21	0.22 0.19	0.03 <0.01	1.0 1.0
10-2156 10-2156-01 GLP: yes 2010	Wheat Pescadou	France [redacted] Europe, South	150 EC	2	0.20	0.067	69	green material	0[1] 0 7 14 27	0.48 /0.10** 2.0 0.97 0.43 0.23	0.02 0.02 0.01 0.02 0.01	0.08/0.02** 0.08 0.06 0.10 0.08	0.06/0.02** 0.07 0.05 0.09 0.08	0.11/0.03** 0.11 0.08 0.12 0.08	0.01 0.01 0.01 0.02 0.01	0.76 2.3 1.2 0.78 0.49
								grain	34 41	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06
								straw	34 41	1.0/0.10** 1.6/0.10**	0.07 0.08	0.38/0.07** 0.37/0.07**	0.32/0.04** 0.32/0.04**	0.36/0.06** 0.33/0.07**	0.09/0.01** 0.08/0.01**	2.2 2.8

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg unless all the values were <0.01 mg/kg. **: residue in control
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

Table 6.3.1-74 (Cont'd): Residues of prothioconazole-desthio in/on wheat applied with fluoxastrobin & prothioconazole EC 150

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
10-2156 10-2156-02 GLP: yes 2010	Wheat Nogal	Spain [redacted] Europe, South	150 EC	2	0.20	0.050-0.067	71	green material	0 (1) 0	0.12 1.9	0.05 0.05	0.03 0.03	0.03 0.04	0.02 0.02	<0.01 <0.01	0.26 2.1
								grain	35 43	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06
								straw	35 43	0.85 0.84	0.32 0.35	0.29 0.27	0.26 0.24	0.15 0.14	0.03 0.03	2.0 1.9

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg.
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio
6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

In the 7 trials conducted in 2010 (report 10-2156), residues of prothioconazole-desthio ranged between <0.01 mg/kg and 0.01 mg/kg. Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in wheat grain at harvest were <0.01 mg/kg. In grain at harvest, the total residue ranged between <0.06 mg/kg and 0.06 mg/kg.
In straw, the residues at harvest ranged as follows :

- between 0.22 mg/kg and 2.3 mg/kg for prothioconazole-desthio
- between 0.02 mg/kg and 0.66 mg/kg for prothioconazole-alpha-hydroxy
- between 0.22 mg/kg and 0.43 mg/kg for prothioconazole-3-hydroxy-desthio
- between 0.12 mg/kg and 0.77 mg/kg for prothioconazole-4-hydroxy-desthio
- between 0.15 mg/kg and 0.38 mg/kg for prothioconazole-5-hydroxy-desthio
- between <0.01 mg/kg and 0.10 mg/kg for prothioconazole-6-hydroxy-desthio
- between 1.0 mg/kg and 3.4 mg/kg for the total residue.

Conclusion

Two wheat residue trials were conducted with fluoxastrobin & prothioconazole EC 150 in southern Europe. The product application corresponded to a prothioconazole rate of 2x200 g a.s./ha. Residues of prothioconazole-desthio ranged between <0.01 mg/kg and 0.01 mg/kg. Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in wheat grain at harvest were < 0.01 mg/kg. In grain at harvest, the total residue ranged between <0.06 mg/kg and 0.06 mg/kg.
In straw, the residues at harvest ranged as follows :

- between 0.22 mg/kg and 2.3 mg/kg for prothioconazole-desthio
- between 0.02 mg/kg and 0.66 mg/kg for prothioconazole-alpha-hydroxy
- between 0.22 mg/kg and 0.43 mg/kg for prothioconazole-3-hydroxy-desthio
- between 0.12 mg/kg and 0.77 mg/kg for prothioconazole-4-hydroxy-desthio
- between 0.15 mg/kg and 0.38 mg/kg for prothioconazole-5-hydroxy-desthio
- between <0.01 mg/kg and 0.10 mg/kg for prothioconazole-6-hydroxy-desthio
- between 1.0 mg/kg and 3.4 mg/kg for the total residue.

Reports:	KCA 6.3.1/25 [redacted]; 2011; M-414694-01-1
Title:	Determination of the residues of BYF 00587, HEC 5725 and prothioconazole in/on wheat after spray application of bixafen & fluoxastrobin & prothioconazole EC 190 in the field in France (South) and Portugal
Report No.:	10-2207
Document No.:	M-414694-01-1
Guideline(s):	EU-Ref. Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2010, a set of 2 residue trials on wheat was conducted in southern Europe. The trials were located in southern France and Portugal.
In each trial, wheat was treated twice at a product rate of 1.75 L/ha bixafen & fluoxastrobin & prothioconazole EC 190 corresponding to 0.175 kg a.s./ha of prothioconazole. The water rate was 300 L/ha. The spray interval was 14-21 days. The treatments were carried out at the growth stages BBCH 45 or 57 and BBCH 69 (end of flowering).

Samples were taken at the following intervals:
- prior to and immediately after the final application,
- at a pre-harvest interval of 35 days after the final treatment (one trial), at maturity (BBCH 89), 38-63 after the last application.

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).
Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio were determined according to method 00979/M001 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in wheat samples from the supplementary residue field trials are presented in Table 6.3.1- 75.

Table 6.3.1-75: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
wheat	prothioconazole-desthio	grain	260	8.7	10-2207 M-414694-01-1
		green material	323	10.8	
		straw	260	8.7	
	prothioconazole-alpha-hydroxy-desthio prothioconazole-3-hydroxy-desthio prothioconazole-4-hydroxy-desthio prothioconazole-5-hydroxy-desthio prothioconazole-6-hydroxy-desthio	grain	291	9.7	
		green material	354	11.8	
		straw	291	9.7	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).

Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio above 30% of the LOQ was found in the control samples except for prothioconazole-desthio in green material where the residues were at 0.01 mg/kg at day0 before the last application.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110% except for prothioconazole-desthio in green material at the LOQ level (112%) and for prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio and prothioconazole-6-hydroxy-desthio in straw at 0.50 mg/kg (68%, 69% and 68% respectively). The single and overall mean recoveries are shown in Table 6.3.1- 76 to Table 6.3.1- 81. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.1- 76: 10-2207: Concurrent recoveries for the determination of prothioconazole-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain	0.01	108	-	-	0.01
		2.5	86 ; 99	93	-	
		Overall Recovery (n = 3)		**98**	**11.3**	
	Cereal green material	0.01	108; 108; 116; 111; 117	112[a]	3.8	0.01
		0.10	93	-	-	
		2.0	84; 104	94	-	
		2.5	92; 95; 94; 89	93	2.9	
		Overall Recovery (n = 12)		**101**	**10.7**	
	Cereal straw	2.5	73	-	-	0.01
		Overall Recovery (n = 1)		**73**	**-**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-desthio, determined as JAU 6476-desthio and calculated as JAU 6476-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises wheat and wheat.
[a]This value was accepted dueto a RSD below 20% and an overall mean in the range of 70-110%.

Table 6.3.1- 77: 10-2207: Concurrent recoveries for the determination of prothioconazole-alpha-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-alpha-hydroxy-desthio	Cereal grain	0.01	89; 90; 118; 120; 89; 89; 94; 100; 95; 96	98	11.9	0.01
		0.50	89; 98; 89; 95	93	4.9	
		Overall Recovery (n = 14)		**97**	**10.6**	
	Cereal green material	0.01	80; 99; 104; 99; 104; 93; 98; 100; 102; 85; 85; 91; 92	95	8.2	0.01
		0.50	101; 95; 100; 101; 83; 85	94	8.7	
		Overall Recovery (n = 19)		**95**	**8.1**	
	Cereal straw	0.01	77; 79; 88; 97; 81; 84	84	8.7	0.01
		0.50	72; 71; 88; 78; 92	80	11.8	
		Overall Recovery (n =11)		**82**	**9.9**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-alpha-hydroxy-desthio, determined as JAU 6476-alpha-hydroxy-desthio and calculated as JAU 6476-desthio. *These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207. Cereal summarises wheat and wheat ** : mean of double injection

Table 6.3.1- 78: 10-2207: Concurrent recoveries for the determination of prothioconazole-3-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-3-hydroxy-desthio	Cereal grain	0.01	73; 82; 109; 113; 85; 88; 89; 90; 88; 94	91	13.1	0.01
		0.50	83; 93; 78; 81	84	7.8	
		Overall Recovery (n =14)		**89**	**12.3**	
	Cereal green material	0.01	70; 90; 101; 91; 103; 91; 94; 94; 94; 70; 71; 75; 77	86	13.8	0.01
		0.50	85; 82; 98; 104; 70; 74	86	15.5	
		Overall Recovery (n =19)		**86**	**14.0**	
	Cereal straw	0.01	80; 80; 83; 86; 98; 107	89	12.4	0.01
		0.50	62; 63; 81; 67; 76	70	12.0	
		Overall Recovery (n =11)		**80**	**17.2**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-3-hydroxy-desthio, determined as prothioconazole-3-hydroxy-desthio and calculated as prothioconazole-desthio *These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207. Cereal summarises wheat and wheat ** : mean of double injection

Table 6.3.1- 79: 10-2207: Concurrent recoveries for the determination of prothioconazole-4-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-4-hydroxy-desthio	Cereal grain	0.01	65; 72; 99; 105; 60; 67; 68; 70; 71; 73	75	19.7	0.01
		0.50	87; 95; 72; 76	83	12.7	
		Overall Recovery (n = 14)		**77**	**17.8**	
	Cereal green material	0.01	68; 80; 99; 79; 83; 65; 66; 73; 74; 63; 64; 74; 76	74	13.3	0.01
		0.50	88; 84; 95; 102; 71; 75	86	13.7	
		Overall Recovery (n = 19)		**78**	**14.9**	
	Cereal straw	0.01	67; 64; 67; 77; 77; 81	72	9.7	0.01
		0.50	67; 62; 79; 62; 70	68***	10.3	
		Overall Recovery (n = 11)		**70**	**10.0**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-4-hydroxy-desthio, determined as prothioconazole-4-hydroxy-desthio and calculated as prothioconazole-desthio *These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207. Cereal summarises wheat and wheat ** : mean of double injection *** This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%

Table 6.3.1- 80: 10-2207: Concurrent recoveries for the determination of prothioconazole-5-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-5-hydroxy-desthio	Cereal grain	0.01	74; 77; 108; 108; 75; 83; 86; 88; 78; 86	86	14.4	0.01
		0.50	80;86;83;83	83	3.0	
		Overall Recovery (n = 14)		**85**	**12.3**	
	Cereal green material	0.01	73; 93; 99; 95; 95; 76; 77; 78; 82; 72; 73; 78; 79	82	11.7	0.01
		0.50	90; 85; 92; 98; 70; 73	85	13.1	
		Overall Recovery (n = 19)		**83**	**11.9**	
	Cereal straw	0.01	80; 71; 79; 84; 78; 88	80	7.2	0.01
		0.50	62; 64; 80; 66; 75	69***	11.1	
		Overall Recovery (n = 11)		**75**	**11.2**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-5-hydroxy-desthio, determined as prothioconazole-5-hydroxy-desthio and calculated as prothioconazole-desthio *These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207. Cereal summarises wheat and wheat ** : mean of double injection *** : This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%

Table 6.3.1- 81: 10-2207: Concurrent recoveries for the determination of prothioconazole-6-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-6-hydroxy-desthio	Cereal grain	0.01	69; 75; 99; 101; 94; 96; 97; 100; 93; 97	92	11.9	0.01
		0.50	79;84;79;82	81	3.0	
		Overall Recovery (n = 14)		**89**	**11.9**	
	Cereal green material	0.01	73; 102; 103; 92; 99; 87; 93; 95; 99; 69; 75; 77; 77	88	13.8	0.01
		0.50	79; 75; 89; 91; 70; 79	81	10.1	
		Overall Recovery (n = 19)		**85**	**13.2**	
	Cereal straw	0.01	82; 74; 75; 78; 75; 82	78	4.7	0.01
		0.50	68; 68; 80; 60; 62	68***	11.5	
		Overall Recovery (n = 11)		**73**	**10.5**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-6-hydroxy-desthio, determined as prothioconazole-6-hydroxy-desthio and calculated as prothioconazole-desthio *These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207. Cereal summarises wheat and wheat ***: mean of double injection *** : This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%

- Residue results:

The residue results are summarised in Table 6.3.1- 82.

Table 6.3.1- 82: Residues of prothioconazole-desthio in/on wheat applied with Bixafen & Fluoxastrobin & Prothioconazole EC 190

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
10-2207 10-2207-01 GLP: yes 2010	Wheat Cezanne	France [redacted] Europe, South	190 EC	2	0.175	0.0583	69	green material	0[(1)] 0	0.80 2.0	0.01** 0.07	0.12 0.12	0.10 0.11	0.09 0.09	0.02 0.02	1.2 2.4
								grain	35 38	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06
								straw	35 38	0.41 0.40	0.25 0.18	0.45 0.40	0.34 0.30	0.25 0.20	0.06 0.05	1.8 1.5
10-2207 10-2207-02 GLP: yes 2010	Wheat Jordao	Portugal [redacted] Europe, South	190 EC	2	0.175	0.0583	69	green material	0[(1)] 0	0.02 1.2	0.01 0.02	0.04 0.04	0.03 0.04	0.02 0.03	<0.01 <0.01	0.13 1.3
								grain	63	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	63	0.37	0.21	0.25	0.26	0.15	0.03	1.3

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg. ** residue in control
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

In the 2 trials conducted in 2010 (report 10-2207), residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in wheat grain at harvest were < 0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg.
In straw, the residues at harvest ranged as follows :

- between 0.37 mg/kg and 0.41 mg/kg for prothioconazole-desthio
- between 0.18 mg/kg and 0.25 mg/kg for prothioconazole-alpha-hydroxy
- between 0.25 mg/kg and 0.45 mg/kg for prothioconazole-3-hydroxy-desthio
- between 0.26 mg/kg and 0.34 mg/kg for prothioconazole-4-hydroxy-desthio
- between 0.15 mg/kg and 0.25 mg/kg for prothioconazole-5-hydroxy-desthio
- between 0.03 mg/kg and 0.06 mg/kg for prothioconazole-6-hydroxy-desthio
- between 1.27 mg/kg and 1.76 mg/kg for the total residue.

Conclusion

Two wheat residue trials were conducted with Bixafen & Fluoxastrobin & Prothioconazole EC 190 in southern Europe. The product application corresponded to a prothioconazole rate of 2x175 g a.s./ha.
Residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in wheat grain at harvest were < 0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg.
In straw, the residues at harvest ranged as follows :

- between 0.37 mg/kg and 0.41 mg/kg for prothioconazole-desthio
- between 0.18 mg/kg and 0.25 mg/kg for prothioconazole-alpha-hydroxy
- between 0.25 mg/kg and 0.45 mg/kg for prothioconazole-3-hydroxy-desthio
- between 0.26 mg/kg and 0.34 mg/kg for prothioconazole-4-hydroxy-desthio
- between 0.15 mg/kg and 0.25 mg/kg for prothioconazole-5-hydroxy-desthio
- between 0.03 mg/kg and 0.06 mg/kg for prothioconazole-6-hydroxy-desthio
- between 1.27 mg/kg and 1.76 mg/kg for the total residue.

Overall conclusion of the wheat trials

A total of 60 wheat trials were performed in Europe, supporting the representative GAP. An overall summary of the obtained residues in feed and food items is presented in the table below.
When two sets of values were obtained from one given trial (at a 35-d PHI and at a later date), the values obtained at maturity (BBCH89 or later) was taken.
Table 6.3.1- 83 summarises the values for prothioconazole-desthio and Table 6.3.1- 84 summarises the values for the calculated "total residue" for prothioconazole, i.e. the sum of prothioconazole-desthio and its hydroxylated metabolites when they were all determined (*i.e.* for 7 trials in northern Europe and 9 trials in southern Europe). Finally, Table 6.3.1- 85 summarises the values for the hydroxylated metabolites of prothioconazole-desthio (when they were measured, *i.e.* for 7 trials in northern Europe and 9 trials in southern Europe).

Table 6.3.1- 83: Summary of residue data from wheat trials with prothioconazole: prothioconazole-desthio

Com-modity	Region	No. of trials	Values (mg/kg)		
			Residue level of prothioconazole-desthio	Maximum (HR)	STMR
Wheat, grain	N-EU	33	32x <0.01; 0.02	0.02	<0.01
	S-EU	27	25x <0.01; 2x0.01	0.01	<0.01
Wheat, straw	N-EU	33	0.02; 3x0.03; 0.038; 2x0.04; 3x0.05; 4x0.06; 2x0.07; 0.08; 2x0.09; 0.10; 2x0.11; 0.12; 2x0.14; 0.19; 0.20; 0.22; 0.29; 0.61; 0.65; 0.79; 0.92	0.92	0.08
	S-EU	27	0.05; 0.10; 0.18; 3x0.22; 2x0.27; 0.30; 0.36; 0.37; 0.40; 0.62; 0.67; 0.72; 0.73; 0.75; 0.85; 1.1; 3x1.2, 2x1.6; 1.7; 1.9; 2.3	2.3	0.67

N-EU = northern Europe; S-EU = southern Europe

Important note: in EFSA's reasoned opinion on the review of the existing MRLs for prothioconazole according to Article 12 of Regulation (EC) N° 396/2005 (EFSA Journal 2014; 12(5):3689), a Highest Residue (HR) of 2.4 mg/kg is reported for wheat straw in southern Europe. This value was not considered in this Annex I Renewal dossier, as it corresponds to a trial (RA-23237/06-0455/6) performed at much more critical GAP than the representative GAP supported in this dossier. Indeed the trial was performed with 3 applications of Bixafen & prothioconazole EC 225 (containing 150 g/L prothioconazole) at the rates of 1.25 L product/ha corresponding to 3 x 0.188 kg/ha prothioconazole.

Table 6.3.1- 84: Summary of residue data from wheat trials with prothioconazole: total residue (sum of the 6 analytes)

Com-modity	Region	No. of trials	Values (mg/kg)		
			Calculated sum of prothioconazole-desthio and its hydroxy metabolites	Maximum (HR)	STMR
Wheat, grain	N-EU	7	7x <0.06	0.06	0.06
	S-EU	9	8x<0.06, 0.06	0.06	0.06
Wheat, straw	N-EU	7	0.17; 0.54; 0.66; 0.91; 1.3; 1.4; 2.6	2.6	0.91
	S-EU	9	1.0; 1.3; 1.5; 1.8; 2.0; 2.8; 3.0; 3.2; 3.4	3.4	2.0

N-EU = northern Europe; S-EU = southern Europe

Table 6.3.1- 85: Summary of residue data from wheat trials with prothioconazole: hydroxy metabolites

Com-modity	Region	n	Analyte	Values for the hydroxy metabolites		
				Residue level(mg/kg)	Maximum (HR)	STMR
Wheat, grain	N-EU	7	alpha-hydroxy-PTZ-DT	7x<0.01	<0.01	<0.01
			3-hydroxy-PTZ-DT	7x<0.01	<0.01	<0.01
			4-hydroxy-PTZ-DT	7x<0.01	<0.01	<0.01
			5-hydroxy-PTZ-DT	7x<0.01	<0.01	<0.01
			6-hydroxy-PTZ-DT	7x<0.01	<0.01	<0.01
	S-EU	9	alpha-hydroxy-PTZ-DT	9x<0.01	<0.01	<0.01
			3-hydroxy-PTZ-DT	9x<0.01	<0.01	<0.01
			4-hydroxy-PTZ-DT	9x<0.01	<0.01	<0.01
			5-hydroxy-PTZ-DT	9x<0.01	<0.01	<0.01
			6-hydroxy-PTZ-DT	9x<0.01	<0.01	<0.01
Wheat, straw	N-EU	7	alpha-hydroxy-PTZ-DT	0.014; 0.02; 0.052; 0.10; 0.17; 0.30; 0.31	0.31	0.10
			3-hydroxy-PTZ-DT	0.035; 0.11; 0.12; 0.18; 0.22; 0.34; 0.64	0.64	0.18
			4-hydroxy-PTZ-DT	0.035; 0.091; 0.12; 2x0.15; 0.27; 0.42	0.42	0.15
			5-hydroxy-PTZ-DT	0.031; 0.11; 0.13; 0.15; 0.26; 0.28; 0.30	0.30	0.15
			6-hydroxy-PTZ-DT	<0.01; 0.017; 0.029; 2x0.04; 0.053; 0.055	0.055	0.04
	S-EU	9	alpha-hydroxy-PTZ-DT	0.02; 0.03; 2x0.08; 0.18; 0.21; 0.33; 0.37; 0.66	0.66	0.18
			3-hydroxy-PTZ-DT	0.22; 0.25; 2x0.29; 0.32; 0.35; 0.37; 0.40; 0.43	0.43	0.32
			4-hydroxy-PTZ-DT	0.12; 0.19; 0.21; 2x0.26; 0.30; 0.32; 0.37; 0.77	0.77	0.26
			5-hydroxy-PTZ-DT	3x0.15; 0.19; 0.20; 0.23; 0.27; 0.33; 0.38	0.38	0.20
			6-hydroxy-PTZ-DT	<0.01;3x0.03; 2x0.05; 0.07; 0.08; 0.10	0.10	0.05

N-EU = northern Europe; S-EU = southern Europe. PTZ-DT: prothioconazole-desthio

CA 6.3.2 Barley

The barley uses and GAPs supported for this AIR dossier are summarised in Table 6.3.2-1 and Table 6.3.2-2.

Foliar use (Bixafen + Prothioconazole EC 225 (75 + 150 g/L)) Aviator

Table 6.3.2-1: Summary of the critical GAP for the representative foliar uses for prothioconazole*

Crop	Region	FT**	F, G, I***	Timing of application	Max. n° of applic.	Minimum Application interval [days]	Maximum application rate [g a.s./ha]	PHI [days]
Barley, Oat	EU-N EU-S	EC225	F	Up to BBCH 61	2	14	150	†

EU-S = Southern Europe EU-N = northern Europe
* justification of the critical GAP for each crop (all zones) applied for is provided in documents D1 and D2.
** FormulationType EC225 = EC formulation containing 75 g/L bixafen and 150 g/L prothioconazole.
*** F Field; G Greenhouse; I Indoor †As the application in cereals is driven purely by growth stage, no PHI is necessary.

Seed treatment use (Prothioconazole FS 100 (100 g/L)) Redigo

Table 6.3.2-2: Summary of the critical GAP for the representative seed treatment uses for prothioconazole*

Crop	Region	FT**	F, I***	G Mode of application	n° of applic.	Maximum application rate		PHI
						[g a.s./dt seeds]	[g a.s./ha]	
Barley, Oat	EU-N EU-S	FS100	F	Seed treatement	1	PTZ: 10.0	PTZ: 18.0[a]	†

EU-S = Southern Europe EU-N = northern Europe
* justification of the critical GAP for each crop (all zones) applied for is provided in documents D1 and D2.
** FormulationType FS100 Seed treatment formulation containing 100 g/L prothioconazole
*** F Field; G Greenhouse; I Indoor †As the application in cereals is driven purely by growth stage, no PHI is necessary.
[a] Based on a seeding rate of 180 kg seed/ha

According to EU guidance document SANCO 7525/VI/95-rev.9 of March 2011 ("Guidelines on comparability, extrapolation, group tolerances and data requirements for setting MRLs") the data obtained from trials conducted on barley can be extrapolated to oat.

The current EU MRL for barley (0.3 mg/kg) will be replaced by an MRL of 0.2 mg/kg in spring 2016 following the review of the existing MRLs for prothioconazole according to Article 12 of Regulation (EC) N° 396/2005. Please refer to Regulation (EU) 843/2013 as well as draft Regulation SANCO 11481/2014. The current EU MRL for oat is 0.05 mg/kg.

Summary of the trials from the Annex I:

The residue trials on barley that have already been evaluated during the EU review of prothioconazole are summarised in Table 6.3.2-3. Since these residue reports have been previously evaluated by the RMS UK (refer to Draft Assessment Report, Annex B.7.6.1, Table B.7.15) They are only presented for reference purposes. The critical supported GAP consisted in one seed treatment application with 15 g a.s./dt seed followed by 2 sprayings at rate of 200 g a.s./ha with last treatment performed at BBCH growth stage 61 at the latest.

Table 6.3.2-3: Number of residue trials conducted per geographical region and vegetation period

Crop	GAP	Region	Formu-lation*	Number of Trials				Report No.	Dossier Reference
				Vegetation period			Total		
				1998	1999	2000			
Annex II data – seed treatment plus foliar									
Barley, spring	ST 15 g a.s./dt and SPI 2 x 200 g a.s./ha	N-EU	FS 200 and 250 EC	5	-	-	10	RA-2139/98 RA-2140/98	KCA 6.3.2.1.3
Barley, spring	SPI 2 x 200 g a.s./ha		250 EC	-	-	5		RA-2100/00	
Barley, winter	ST 15 g a.s./dt and SPI 2 x 200 g a.s./ha	S-EU	FS 200 and 250 EC	1	-	-		RA-2079/98	
Barley, winter	SPI 2 x 200 g a.s./ha		250 EC	3	-	2		RA-2144/98 RA-2103/00	
Annex II data – seed treatment use									
Cereal (wheat)	ST 19.5-22 g/ha (15 g a.s./dt)	N-EU	FS 200	-	2	2	4	RA-2010/99 RA-2091/00	KCA 6.3.2.1.1
Cereal (wheat)		S-EU	FS 200	2	-	2	4	RA-2010/99 RA-2090/00	

N-EU: northern Europe S-EU: southern Europe ST: seed treatment SPI: spray
* FS 200: flowable concentrate formulations containing 200 g/L of prothioconazole
250 EC: emulsifiable concentrate containing 250 g/L of prothioconazole

Seed treatment + foliar use:

With the Annex II dossier on Prothioconazole complete residue data packages on barley were submitted. In northern and southern Europe, a total of 15 residue trials were carried out, in which the compound was either applied:
- as seed dressing followed by two spray applications of 'prothioconazole EC 250' straight formulation at 200 g a.s/ha (6 trials)
- as two spray applications of 'prothioconazole EC 250' at 200 g a.s/ha only (12 trials).

The sprayings were conducted at growth stages 37-49 and 59-71 with the last treatment performed 35 (36) days prior to harvest (recommended pre-harvest interval). It is demonstrated in Annex II, Section 4, Point 6.1 that seed treatment has no effect on the residue levels determined at harvest.
The GAP supported for barley in the Annex II dossier comprises seed treatment with 15 g a.s./dt followed by 2 sprayings at 200 g a.s./ha. This use pattern is considered to represent the EU critical GAP and can be used as a basis for setting the EU MRLs.

For the Annex II data, the desired PHI was 35 days. If 35 days after application, the grain could not be separated, sample materials available at that time (ear and "rest of plant") were taken, and grain and straw were then sampled later at harvest maturity (growth stage 89). Also, if grain samples could be taken on day 35, but the commodity had not yet reached final harvest maturity, a second set of grain and straw samples was taken at harvest.

For data gathering purposes the samples taken from these trials were analysed for prothioconazole-desthio using methods 00598/M001 or 00647 with a limit of quantitation of 0.01 mg/kg for grain and 0.05 mg/kg for cereal green material and straw.

No residues above the LOQ could be detected in any of the control samples.
An overall summary of the obtained residues in feed and food items is presented in Table 6.3.2-4.

Table 6.3.2-4: Overall summary of residue of prothioconazole-desthio in barley with foliar treatment or seed treatment followed by foliar

Application Rate	Region	Com-modity	No. of trials	Residue level of prothioconazole-desthio (mg/kg)			Reference
				Minimum	Maximum (HR)	STMR	
2 x 200 g a.s./ha or seed treatment (15 g a.s./dt seed) followed by 2 x 200 g a.s./ha PHI 35 days)	N-EU	Barley, grain	9	< 0.01	< 0.01	< 0.01	EFSA Scientific Report (2007) 106
	S-EU		8	< 0.01	0.02	0.01	
	N-EU	Barley, straw	9	0.25	0.30	0.13	
	S-EU			0.1	1.1	0.4	

N-EU = northern Europe; S-EU = southern Europe

Seed treatment:

For seed treatment in cereals, 8 trials in wheat (4 in northern Europe and 4 in Southern Europe) were carried out and evaluated during Annex I inclusion. The product was a flowable concentrate for seed treatment, containing 200 g/L JAU 6476. The seed was dressed at a rate of 75 mL product per 100 kg of seed, corresponding to 15 g JAU 6476 per 100 kg of seed. The actual dressing rate was determined with method 00598/M001 by taking seed samples after seed dressing prior to drilling.

For data gathering purposes the samples taken from these trials were analysed for prothioconazole-desthio using methods 00598/M001 or 00647 with a limit of quantitation of 0.01 mg/kg for grain and 0.05 mg/kg for cereal green material and straw.

No residues above the LOQ could be detected in any of the control samples.
An overall summary of the obtained residues in feed and food items is presented in Table 6.3.2-5.

Table 6.3.2-5: Overall summary of residue of prothioconazole-desthio in cereal seed treatment

Application Rate	Region	Com-modity	No. of trials	Residue level of prothioconazole-desthio (mg/kg)			Reference
				Minimum	Maximum (HR)	STMR	
19.5-22 g/hl (15 g a.s./dt seed)	N-EU and S-EU	Wheat grain	8	< 0.01	< 0.01	0.01	EFSA Scientific Report (2007) 106
		Wheat, straw	8	< 0.05	< 0.05	0.05	

N-EU = northern Europe; S-EU = southern Europe

Supplementary trials

Since the initial Annex I inclusion, a number of barley trials have been performed with prothioconazole, in various formulations, including the representative formulation (Bixafen + Prothioconazole EC 225 (75 + 150 g/L)) Aviator. All these trials are supporting the representative use pattern and are summarised below. For the sake of clarity and for the purpose of this Annex I renewal dossier, only results for prothioconazole residues are presented.
In some trials, on top of the normal sampling performed at maturity (BBCH stage 89), an earlier sampling was also performed at a fixed PHI (pre harvest interval) of 35 days. Nevertheless, in this dossier for cereal, the supported use pattern is driven by growth stage, and no PHI is necessary (BBCH 89).

EU - northern region

Table 6.3.2-6: Number of residue trials conducted per geographical region and vegetation period

Year	GAP	Formulation	N° of trials	Study number Report No.	Dossier Reference
Barley EU foliar spray residue trials – northern EU					
2000	2 x 125 g a.s./ha BBCH 61	EC200 (100 g/L fluoxastrobin, 100 g/L prothioconazole)	4	RA-2013/00 M-083920-01-1	KCA 6.3.2/07
2002	2 x 125 g a.s./ha BBCH 61	EC250 (125 g/L tebuconazole 125 g/L prothioconazole)	4	RA-2103/02 M-086877-01-1	KCA 6.3.2/08
2003	2 x 150 g a.s./ha BBCH 61	EC300 (75 g/L fluoxastrobin, 150 g/L prothioconazole, 75 g/L trifloxystrobin)	4	RA-2016/03 M-001702-01-1	KCA 6.3.2/09
2003	2 x 175 g a.s./ha BBCH 61	SC 325 (175 g/L prothioconazole, 150 g/L trifloxystrobin)	4	RA-2106/03 M-060905-02-1	KCA 6.3.2/10
2005	2 x 125 g a.s./ha BBCH 61	EC450 (100 g/L prothioconazole, 250 g/L spiroxamine, 100 g/L tebuconazole)	4	RA-2571/05 M-272012-01-1	KCA 6.3.2/11
2006	2 x 150 g a.s./ha BBCH 61	EC225 (75 g/L bixafen, 150 g/L prothioconazole)	4	RA-2328/06 M-294779-02-1	KCA 6.3.2/12
2007	2 x 150 g a.s./ha BBCH 61	EC225 (75 g/L bixafen, 150 g/L prothioconazole)	2	RA-2039/07 M-298114-03-1	KCA 6.3.2/13
2007	2 x 150 g a.s./ha BBCH 61	EC400 (50 g/L bixafen, 100 g/L prothioconazole, 250 g/L spiroxamine)	4	RA-2042/07 M-298147-01-1	KCA 6.3.2/14
2010	2 x 150 g a.s./ha BBCH 61	EC290 (40 g/L bixafen, 50 g/L fluoxastrobin and 100 g/L prothioconazole)	2	10-2204 M-414691-01-1	KCA 6.3.2/15
2013	2 x 125 g a.s./ha BBCH 61	EC200 (100 g/L fluoxastrobin, 100 g/L prothioconazole)	2	13-2137 M-501711-03-1	KCA 6.3.2/16
2013	2 x 125 g a.s./ha BBCH 61	EC200 (100 g/L fluoxastrobin, 100 g/L prothioconazole)	2	13-2158 M-501503-01-1	KCA 6.3.2/17
TOTAL northern EU region			**36**		

Report:	KCA 6.3.2/07 [redacted],; [redacted]; 2001; M-083920-01-1
Title:	Determination of residues of HEC5725 & JAU6476-Desthio on winter barley following spray application of HEC5725 & JAU6476 200 EC in Sweden, France, Great Britain and Germany
Report No.:	RA-2013/00
Document No.:	M-083920-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of 15 July, 1991, Annex II, part A, point 6 and Annex III, part A, point 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 1999/2000, a set of 4 residue trials was conducted in northern Europe. The trials were located in Sweden, northern France, Great Britain and Germany.

In each trial, barley was treated twice at a product rate of 1.25 L/ha fluoxastrobin & prothioconazole EC 200 corresponding to 0.125 kg a.s./ha of each mixing partner. The employed water rate was 300 L/ha in all trials. The spray interval ranged from 10 - 22 days. The time of application was:

1. during the time when the flag leaf sheath of the barley plants was just visible until fully unrolled (BBCH 37 - 39), and
2. at the beginning of flowering (BBCH 61) in 3 trials and at the end of flowering (BBCH 69) in one trial.

Samples were taken at the following intervals:
- prior to and immediately after the final application;
- at a pre-harvest interval of 35 (36) days
- at harvest (BBCH 89): 47 - 71 days after the final treatment.

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-7.

Table 6.3.2-7: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	ear	313	10.4	RA-2013/00 M-083920-01-1
		grain	257	8.6	
		Rest of plant	313	10.4	
		straw	257	8.6	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples except for one trial where the residues were at 0.01 mg/kg in ear at day0 before the last application.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown Table 6.3.2-8. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-8: RA-2013/00: Concurrent recoveries and validation recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Barley grain / Ear	0.01	102 ; 100 ; 103	102	1.5	0.01
		0.10	103 ; 103 ; 102 ; 103 ; 102 ; 103 ; 104 ; 103 ; 102 ; 103 ; 105 ; 104 ; 115 ; 99 ; 103 ; 106 ; 102 ; 94 ; 102 ; 101 ; 101	103	3.6	
		Overall (n = 24)		**103**	**3.4**	
Prothio-conazole-desthio	Rest of plant	0.05	102 ; 107 ; 102	104	2.8	0.05
		0.50	106 ; 103 ; 106 ; 103 ; 104 ; 104 ; 104 ; 105 ; 103 ; 103 ; 103 ; 97 ; 104 ; 115 ; 105 ; 102 ; 103 ; 99	104	3.4	
		Overall (n = 21)		**104**	**3.3**	
Prothio-conazole-desthio	Straw	0.05	94 ; 102 ; 96	97	4.3	0.05
		0.50	103 ; 104 ; 102 ; 102 ; 98 ; 100 ; 96 ; 103	101	2.7	
		Overall (n = 11)		**100**	**3.4**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:

The residue results are summarised in Table 6.3.2-9.

Table 6.3.2-9: Residues of prothioconazole-desthio in/on winter barley applied with fluoxastrobin & prothioconazole EC 200

Study Trial No. Trial SubID GLP Year	Crop Variety	Country	Application					Residues (mg/kg)*		
			FL [a]	No	kg/ha (a.s.) [b]	kg/hL (a.s.) [b]	GS	Portion analysed	DALT (days)	Prothioconazole-desthio
RA-2013/00 R 2000 0152 5 0152-00 GLP yes 2000	Barley, winter Jura	Sweden S-[redacted] Europe, North	200 EC	2	0.125	0.042	61	ear	0[(1)] 0 35	0.01 2.5 <0.01
								rest of plant	0[(1)] 0 35	0.45 1.4 0.14
								straw	47	0.07
								grain	47	0.01
RA-2013/00 R 2000 0153 3 0153-00 GLP yes 2000	Barley, winter Nickel	France F-[redacted] Europe, North	200 EC	2	0.125	0.042	61	ear	0[(1)] 0 35	0.01 2.0 <0.01
								rest of plant	0[(1)] 0 35	0.44 0.96 0.06
								straw	54	0.06
								grain	54	<0.01
RA-2013/00 R 2000 0154 1 0154-00 GLP yes 2000	Barley, winter Regina	United Kingdom GB-[redacted] Europe, North	200 EC	2	0.125	0.042	69 **	ear	0[(1)] 0 36	<0.01/0.01[(2)] 2.4 <0.01
								rest of plant	0[(1)] 0 36	0.11 1.2 0.05
								straw	56	0.08
								grain	56	<0.01
RA-2013/00 R 2000 0156 8 0156-00 GLP yes 2000	Barley, winter Theresa	Germany D-[redacted] Europe, North	200 EC	2	0.125	0.042	61	ear	0[(1)] 0 35	0.01 1.6 <0.01
								rest of plant	0[(1)] 0 35	0.32 1.6 0.13
								straw	71	<0.05
								grain	71	<0.01

FL = Formulation, DALT = Days after last treatment
* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) : before last application
(2) : residue in control sample
** : the 2nd treatment was carried out at a later stage as originally intended (BBCH 69 instead of 61).

In the 4 trials conducted in 2000 (report RA-2013/00), residues of prothioconazole-desthio in barley grain at harvest were always below the limit of quantification (LOQ) of 0.01 mg/kg except for trial 0152-00 where the residues reached 0.01 mg/kg. The residues in straw at harvest ranged between <0.05 mg/kg and 0.08 mg/kg.

Conclusion

Four barley residue trials were conducted with fluoxastrobin & prothioconazole EC 200 in northern Europe. The product application corresponded to a prothioconazole rate of 2x125 g a.s./ha.
Residues of prothioconazole-desthio in barley grain at harvest were always below the limit of quantification (LOQ) of 0.01 mg/kg except for trial 0152-00 where the residues reached 0.01 mg/kg. The residues in straw at harvest ranged between <0.05 mg/kg and 0.08 mg/kg.

Report:	KCA 6.3.2/08 [redacted] カ; 2003; M-086877-01-1
Title:	Determination of residues of tebuconazole and prothioconazole-desthio in/on barley after spray application of HWG 1608 & JAU 6476 250 EC in the field in France, Germany and Great Britain
Report No.:	RA-2103/02
Document No.:	M-086877-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of 15 July, 1991, Annex II, part A, point 6 and Annex III, part A, point 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2002, a set of 4 residue trials was conducted in northern Europe. The trials were located in northern France, Germany and Great Britain.

In each trial, barley was treated twice at a product rate of 1.0 L/ha tebuconazole & prothioconazole EC 250 corresponding to 0.125 kg a.s./ha of each mixing partner. The employed water rate was 300 L/ha in all trials. The spray interval ranged from 7 - 30 days. The time of application was:

1. during the time when the flag leaf is fully unrolled (ligule just visible) in 3 trials and when the first awns are visible (in awned forms only) in one trial (BBCH 39 - 49), and
2. at the beginning of flowering in 3 trials and at the late milk stage in one trial. (BBCH 61-77).

Samples were taken at the following intervals:
- prior to the last application only for control samples of ear and rest of plant for 2 trials and immediately after the final application,
- at a pre-harvest interval of 35 days,
- at harvest (BBCH 89), 56/57 days after the final treatment .

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-10.

Table 6.3.2-10: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	ear	233	7.8	RA-2103/02, M-088877-01-1
		grain	183	6.1	
		Rest of plant	233	7.8	
		straw	183	6.1	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-11. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-11: RA-2103/02: Concurrent recoveries and validation recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%] Single Values	Mean	RSD [%]	LOQ [mg/kg]
Prothio-conazole-desthio	Barley grain / Ear	0.01	89 ; 100 ; 96	95	5.9	0.01
		0.10	94 ; 90 ; 95	93	2.8	
		Overall (n = 6)		**94**	**4.3**	
Prothio-conazole-desthio	Rest of plant	0.05	94 ; 91 ; 93	93	1.6	0.05
		0.50	95 ; 95 ; 95	96	1.2	
		Overall (n = 6)		**94**	**2.2**	
Prothio-conazole-desthio	Straw	0.05	100 ; 86 ; 102	96	9.1	0.05
		0.50	94 ; 96	95	-	
		Overall (n = 5)		**96**	**6.5**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:
The residue results are summarised in Table 6.3.2-12.

Table 6.3.2-12: Residues of prothioconazole-desthio in/on spring barley applied with tebuconazole & prothioconazole EC 250

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	Prothioconazole-desthio (mg/kg)
RA-2103/02 R 2002 0529/5 0529-02 GLP: yes 2002	Barley, spring Nevada	France F-[redacted] Europe, North	250 EC	2	0.125	0.042	61	ear	0	0.94
								rest of plant	0	1.2
								grain	35	0.02
								straw	35	0.29
RA-2103/02 R 2002 0541/4 0541-02 GLP: yes 2002	Barley, spring Prisma	United Kingdom GB-[redacted] Europe, North	250 EC	2	0.125	0.042	77	ear	0	0.86
								rest of plant	0	2.2
								grain	35	0.01
								straw	35	0.21
RA-2103/02 R 2002 0539/2 0539-02 GLP: yes 2002	Barley, spring Jacinta	Germany D-[redacted] Europe, North	250 EC	2	0.125	0.042	61	ear	0 35	1.0 <0.01
								rest of plant	0 35	1.7 0.10
								grain	56	<0.01
								straw	56	0.05
RA-2103/02 R 2002 0540/6 0540-02 GLP: yes 2002	Barley, spring Extract	Germany D-[redacted] Europe, North	250 EC	2	0.125	0.042	61	ear	0 35	1.4 <0.01
								rest of plant	0 35	2.8 0.16
								grain	57	<0.01
								straw	57	0.05

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio

In the 4 trials conducted in 2002 (report RA-2103/02), residues of prothioconazole-desthio in barley grain at harvest ranged from < 0.01 mg/kg to 0.02 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.29 mg/kg.

Conclusion

Four barley residue trials were conducted with tebuconazole & prothioconazole EC 250 in northern Europe. The product application corresponded to a prothioconazole rate of 2x125 g a.s./ha.
Residues of prothioconazole-desthio in barley grain at harvest ranged from < 0.01 mg/kg to 0.02 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.29 mg/kg.

Report:	KCA 6.3.2/09[redacted]; [redacted]; 2004; M-001702-01-1
Title:	Determination of residues of fluoxastrobin (HEC 5725), prothioconazole (JAU 6476) and trifloxystrobin in/on barley following spray application of HEC 5725 & JAU 6476 & CGA 279202 300 EC in the field in Germany, Northern France, Great Brit..
Report No.:	RA-2016/03
Document No.:	M-001702-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2002/2003, a set of 4 residue trials was conducted in northern Europe. The trials were located in Germany, northern France, Great Britain and Sweden.

In each trial, barley was treated twice at a product rate of 1.0 L/ha fluoxastrobin & prothioconazole & trifloxystrobin EC 300 corresponding to 0.150 kg a.s./ha of prothioconazole with the exception of trial R 2003 0250/9 (France) where both applications were 11 % overdosed (1.11 L product/ha corresponding to 0.167 kg a.s./ha of prothioconazole). The employed water rate was 300 L/ha in all trials. The spray interval ranged from 13 - 25 days. The time of application was:

1. during the time when the flag leaf is just visible (still rolled) in 3 trials and when the flat leaf sheath is opening in one trial (BBCH – 37-47), and
2. at the beginning of flowering in 3 trials and at the end of flowering in one trial (BBCH 61-65).

Samples were taken at the following intervals:
- prior to the last application only for control samples of ear and rest of plant and immediately after the final application;
- at a pre-harvest interval of 35 days
- at harvest (BBCH 89), 43-52 days after the final treatment.

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-13.

Table 6.3.2-13: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	ear	147	4.9	RA-2016/03 M-001702-01-1
		grain	112	3.7	
		Rest of plant	147	4.9	
		straw	112	3.7	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples except for one trial where the residues were at 0.01 mg/kg in ear at day0 before the last application.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-14. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-14: RA-2016/03: Concurrent recoveries and validation recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single values	Mean		
Prothio-conazole-desthio	Barley grain + ear	0.01	*94 ; 97 ; 94*	95	1.8	0.01
		0.1	95 ; 93 ; 100 ; 93 ; 94	95	3.1	
		1.0	92	92	-	
		2.0	94	94	-	
		4.0	94	94	-	
		Overall (n = 11)		**95**	**2.3**	
Prothio-conazole-desthio	Straw	0.05	*97 ; 91 ; 98*	95	4.0	0.05
		0.50	98 ; 97 ; 96 ; 97 ; 99	97	1.2	
		5.0	94	94	-	
		Overall (n = 9)		**96**	**2.5**	
Prothio-conazole-desthio	Rest of plant	0.05	*100 ; 97 ; 99*	99	1.5	0.05
		0.50	96 ; 91 ; 95 ; 93 ; 97	94	2.6	
		5.0	93	93	-	
		Overall (n = 9)		**96**	**3.1**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
Recoveries from method validation in italics

- Residue results:
The residue results are summarised in Table 6.3.2-15.

Table 6.3.2-15: Residues of prothioconazole-desthio in/on barley applied with fluoxastrobin & prothioconazole & trifloxystrobin EC 300

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	Prothioconazole-desthio (mg/kg)
RA-2016/03 R 2003 0252/5 0252-03 GLP: yes 2003	Barley Pasadena	Sweden S-[redacted] Europe, North	300 EC	2	0.150	0.050	61	rest of plant	0(1) 0 35	1.2 2.3 0.16
								ear	0(1) 0 35	<0.01 1.4 <0.01
								grain	52	<0.01
								straw	52	0.19
RA-2016/03 R 2003 0250/9 0250-03 GLP: yes 2003	Barley Esterel	France F-[redacted] Europe, North	300 EC	2	0.167**	0.0555	65	rest of plant	0(1) 0	0.11 0.53
								ear	0(1) 0	0.03 2.8
								grain	35 48	<0.01 <0.01
								straw	35 48	0.05 <0.05
RA-2016/03 R 2003 0251/7 0251-03 GLP: yes 2003	Barley Caret	United Kingdom GB-[redacted] Europe, North	300 EC	2	0.150	0.050	61	rest of plant	0(1) 0 35	0.37 1.1 0.06
								ear	0(1) 0 35	0.01/0.01(2) 3.0 0.02
								grain	43	<0.01
								straw	43	0.09
RA-2016/03 R 2003 0131/6 0131-03 GLP: yes 2003	Barley Theresa	Germany D-[redacted] Europe, North	300 EC	2	0.150	0.050	61	rest of plant	0(1) 0	0.30 0.96
								ear	0(1) 0	<0.01 1.2
								grain	35 51	<0.01 <0.01
								straw	35 51	<0.05 0.05

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
** 11% overdosed
(1) : before last application
(2) : residue in control sample

In the 4 trials conducted in 2003 (report RA-2016/03), residues of prothioconazole-desthio in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between <0.05 mg/kg and 0.19 mg/kg.

Conclusion

Four barley residue trials were conducted with fluoxastrobin & prothioconazole & trifloxystrobin EC 300 in northern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha with the exception of trial R 2003 0250/9 (France) where both applications were 11 % overdosed (1.11 L product/ha corresponding to 0.167 kg a.s./ha of prothioconazole). Residues of prothioconazole-

desthio in barley grain at harvest was <0.01 mg/kg. The residues in straw at harvest ranged between <0.05 mg/kg and 0.19 mg/kg.

Report:	KCA 6.3.2/10[redacted]; 2004; M-060905-02-1
Title:	Determination of residues of trifloxystrobin, CGA 321113, and JAU 6476-desthio in/on barley following spray application of CGA 279202 & JAU 6476 (325 SC) in the field in Northern France, Germany, and Great Britain
Report No.:	RA-2106/03
Document No.:	M-060905-02-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2003, a set of 4 residue trials was conducted in northern Europe. The trials were located in Germany, northern France and Great Britain.

In each trial, barley was treated twice at a product rate of 1.0 L/ha prothioconazole & trifloxystrobin SC 325 corresponding to 0.175 kg a.s./ha of prothioconazole with the exception of trial R2003 0939/2 (France) where both applications were 12.4 % overdosed (1.12 L product/ha corresponding to 0.197 kg a.s./ha of prothioconazole). The employed water rate was 300 L/ha in all trials except for trial R2003 0939/2 (France) where water rate was 12.4% overdosed (337 L/ha).The spray interval ranged from 10 - 25 days. The time of application was:

1. during the time when the flag leaf sheath is opening and when the first awns are visible (in awned forms only) (BBCH 47 - 49), and
2. at the beginning of flowering and at the full flowering (BBCH 61-65).

Samples were taken at the following intervals:
- prior to the last application only for control samples of ear and rest of plant for 2 trials and immediately after the final application;
- at a pre-harvest interval of 35 days after the final treatment.
- at harvest (BBCH 89)

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-16.

Table 6.3.2-16: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	ear	180	6	RA-2106/03 M-060905-02-1
		grain	131	4.4	
		green material	162	5.4	
		rest of plant	180	6	
		straw	131	4.4	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of sample s ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-17. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-17: RA-2106/03: Concurrent recoveries and validation recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Barley grain / Ear	0.01	93 ; 100 ; 97	97	3.6	0.01
		0.10	101 ; 95 ; 95 ; 92 ; 96 ; 98	96	3.2	
		Overall (n = 9)		**96**	**3.1**	
Prothio-conazole-desthio	Green material	0.05	101 ; 99 ; 97	99	2.0	0.05
		0.50	101 ; 98 ; 103 ; 104 ; 95	100	3.7	
		Overall (n = 8)		**100**	**3.1**	
Prothio-conazole-desthio	Straw	0.05	101 ; 101 ; 103	102	1.1	0.05
		0.50	99 ; 98 ; 97 ; 95	97	1.8	
		Overall (n = 7)		**99**	**2.8**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio

RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:

The residue results are summarised in Table 6.3.2-18.

Table 6.3.2-18: Residues of prothioconazole-desthio in/on barley applied with prothioconazole & trifloxystrobin SC 325

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed**	DALT (days)	prothioconazole-desthio
RA-2106/03 R 2003 0975/9 0975-03 GLP: yes 2003	Barley Caret	United Kingdom GB-[redacted] Europe, North	325 SC	2	0.175	0.058	61	ear	0 35	2.5 0.02
								grain	43	<0.01
								straw	43	0.11
								rest of plant	0 35	1.1 0.12
RA-2106/03 R 2003 0976/7 0976-03 GLP: yes 2003	Barley Nickel	France F-[redacted] Europe, North	325 SC	2	0.175	0.058	61	ear	0 35	2.9 <0.01
								grain	49	<0.01
								straw	49	0.10
								rest of plant	0 35	1.1 0.09
RA-2106/03 R 2003 0939/2 0939-03 GLP: yes 2003	Barley Esterel	France F-[redacted] Europe, North	325 SC	2	0.1965 ***	0.058	61	green material	0	0.57
								ear	0	2.3
								grain	35 48	<0.01 <0.01
								straw	35 48	<0.05 <0.05
RA-2106/03 R 2003 0974/0 0974-03 GLP: yes 2003	Barley Tafeno	Germany D-[redacted] Europe, North	325 SC	2	0.175	0.058	61	green material	0	0.87
								ear	0	1.8
								grain	35 42	<0.01 <0.01
								straw	35 42	0.06 0.08

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
** portion analysed are in accordance to the appendix 4 (sampling) of the study report
*** : 12.5% overdosed

In the 4 trials conducted in 2003 (report RA-2106/03), residues of prothioconazole-desthio in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between <0.05 mg/kg and 0.11 mg/kg.

Conclusion

Four barley residue trials were conducted with Prothioconazole + Trifloxystrobin SC 325 in northern Europe. The product application corresponded to a prothioconazole rate of 2x175 g a.s./ha with the exception of trial R2003 0939/2 (France) where both applications were 12.4 % overdosed (1.12 L product/ha corresponding to 0.197 kg a.s./ha of prothioconazole). Residues of prothioconazole-desthio

in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between <0.05 mg/kg and 0.11 mg/kg.

Report:	KCA 6.3.2/11 [redacted] E; [redacted]; 2006; M-272012-01-1
Title:	Determination of the residues of JAU 6476, tebuconazole and KWG 4168 in/on spring barley after spraying of JAU 6476 & HWG 1608 & KWG 4168 (450 EC) in the field in Northern France, United Kingdom, Germany and Sweden
Report No.:	RA-2571/05
Document No.:	M-272012-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2005, a set of 4 residue trials was conducted in northern Europe. The trials were located in Germany, Sweden, northern France and Great Britain.

In each trial, barley was treated twice at a product rate of 1.25 L/ha JAU 6476 (prothioconazole) & HWG 1608 (tebuconazole) & KWG 4168 (spiroxamine) EC450 corresponding to 0.125 kg a.s./ha of prothioconazole. The water rate was 300 L/ha. The spray interval ranged from 6 - 12 days. The time of application was:
1. when the flag leaf sheath of the plants was opening (BBCH 47), and
2. at the beginning of flowering (BBCH 61).

Samples were taken at the following intervals:
- prior to the last application only for all control samples and one treated sample of green material and immediately after the final application as well at a PHI of 28 and 35 (38) days for green material
- at harvest (BBCH 89).

Residues of prothioconazole-desthio were determined according to method 00598/M001 with a limit of quantitation of 0.01 mg/kg for grain and 0.05 mg/kg for cereal green material and straw (cf. original Annex II, Section 2, Point 4.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-19.

Table 6.3.2-19: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	grain	111	3.7	RA-2571/05 M-272012-01-1
		Green material	123	4.1	
		straw	113	3.8	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-20. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-20: RA-2571/05: Concurrent recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Barley grain / Ear	0.01	90 ; 81 ; 94	88	7.5	0.01
		0.10	90 ; 91	91	-	
		Overall (n = 5)		**89**	**5.5**	
Prothio-conazole-desthio	Green material	0.05	79 ; 86 ; 93	86	8.1	0.05
		0.50	75 ; 90 ; 104 ; 99	92	13.8	
		5.0	93 ; 84 ; 100	92	8.7	
		Overall (n = 10)		**90**	**10.4**	
Prothio-conazole-desthio	Straw	0.05	90 ; 88 ; 91	90	1.7	0.05
		0.50	88 ; 91	90	-	
		Overall (n = 5)		**90**	**1.7**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
These recoveries were assigned to the studies RA-2571/05 and RA-2572/05.

- Residue results:
The residue results are summarised in Table 6.3.2-21.

Table 6.3.2-21: Residues of prothioconazole-desthio in/on spring barley applied with JAU 6476 (prothioconazole) & HWG 1608 (tebuconazole) & KWG 4168 (spiroxamine) EC450

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	Prothioconazole-desthio
RA-2571/05 R 2005 0031/9 0031-05 GLP: yes 2005	Barley, spring Optic	United Kingdom GB-[redacted] Europe, North	450 EC	2	0.125	0.042	61	green material	0 35	1.8 0.06
								grain	47	<0.01
								straw	47	0.10
RA-2571/05 R 2005 0802/6 0802-05 GLP: yes 2005	Barley, spring Pasadena	Sweden SE-[redacted] Europe, North	450 EC	2	0.125	0.042	61	green material	0 35	1.6 0.16
								grain	51	<0.01
								straw	51	0.22
RA-2571/05 R 2005 0030/0 0030-05 GLP: yes 2005	Barley, spring CARAFE	France F-[redacted] Europe, North	450 EC	2	0.125	0.042	61	green material	0[1] 0 28 35	0.48 1.5 0.28 0.19
								grain	39	<0.01
								straw	39	0.75
RA-2571/05 R 2005 0801/8 0801-05 GLP: yes 2005	Barley, spring Adonis	Germany D-[redacted] Europe, North	450 EC	2	0.125	0.042	61	green material	0 38	1.6 <0.05
								grain	49	<0.01
								straw	49	<0.05

(1) Prior to last treatment
* determined as prothioconazole-desthio and calculated as prothioconazole-desthio

In the 4 trials conducted in 2005 (report RA-2571/05), residues of prothioconazole-desthio in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between <0.05 mg/kg and 0.75 mg/kg.

Conclusion

Four barley residue trials were conducted with JAU 6476 & KWG 4168 & HWG 1608 EC 450 in northern Europe. The product application corresponded to a prothioconazole rate of 2x125 g a.s./ha. Residues of prothioconazole-desthio in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between <0.05 mg/kg and 0.75 mg/kg.

Report:	KCA 6.3.2/12[redacted],[redacted]; 2013; M-294779-02-1
Title:	Amendment no.1 to report no: RA-2328/06 - Determination of the residues of BYF 00587 and JAU 6476 in/on spring barley and winter barley after spraying of BYF 00587 & JAU 6476 (225 EC) in the field in Northern France, Sweden, the United Kingdom and Germany
Report No.:	RA-2328/06
Document No.:	M-294779-02-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed Pre-Registration: SANCO/3029/99 Rev. 4, 2000-07-11
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.3.2/13[redacted]; 2008; M-298114-03-1
Title:	Determination of the residues of BYF 00587 and JAU 6476 in/on spring barley after spraying of BYF 00587 & JAU 6476 (225 EC) in the field in Northern France and Germany
Report No.:	RA-2039/07
Document No.:	M-298114-03-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2006 and 2007, a set of 6 residue trials was conducted in northern Europe. The trials were located in Germany, Sweden, northern France and Great Britain.

In each trial, barley was treated twice at a product rate of 1 L/ha BYF 00587 (bixafen) & JAU 6476 (prothioconazole) EC 225 corresponding 0.150 kg a.s./ha of prothioconazole. The water rate was 300 L/ha. The spray interval ranged from 12 - 27 days. The timing of application was:

1. when the flag leaf is just visible (still rolled) and at the flag leaf stage (flag leaf fully unrolled, ligule just visible) (BBCH 37-39), and
2. at the beginning of flowering (BBCH 61).

Samples of green material were taken just prior to and immediately after the final application. Two trials were designed as decline series, *i.e.* samples were collected further 7, 14, and 28 days after the final treatment. Following the second and final application, samples of grain and straw were collected after 34-62 days at full maturity of the crop (BBCH 89).

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-22.

Table 6.3.2-22: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	grain	182	6.1	RA-2328/06 M-294779-02-1
		green material	174	5.8	
		straw	187	6.2	
		ear	111	3.7	RA-2039/07 M-298114-03-1
		rest of plant	111	3.7	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples except for 3 trials where the residues were at 0.02 and 0.04 mg/kg in green material at day0 before the last application and at 0.01 mg/kg in straw at DALT 62.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-23 and Table 6.3.2-24. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-23: RA-2039/07 : Concurrent recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain#	0.01	97 ; 97 ; 96 ; 100 ; 93 ; 94 ; 99	97	2.6	0.01
		0.10	95 ; 93 ; 95 ; 91 ; 93 ; 100	95	3.3	
		1.0	92 ; 95 ; 95 ; 89 ; 93 ; 99	94	3.6	
		5.0	89	89	-	
		10	86	86	-	
		Overall (n = 21)		**94**	**3.9**	
Prothio-conazole-desthio	Cereal green material*	0.01	98 ; 94 ; 90 ; 97	95	3.8	0.01
		0.10	97 ; 90 ; 95 ; 88	93	4.5	
		1.0	87 ; 95 ; 95 ; 91 ; 89	91	3.9	
		5.0	89 ; 85 ; 82	85	4.1	
		10	83 ; 86 ; 85	85	1.8	
		Overall (n = 19)		**90**	**5.5**	
Prothio-conazole-desthio	Cereal straw	0.01	92 ; 88 ; 104 ; 93 ; 104	96	7.7	0.01
		0.10	95 ; 88 ; 94 ; 103	95	6.5	
		1.0	88 ; 93 ; 91 ; 93	91	2.6	
		5.0	88 ; 80 ; 87	85	5.1	
		10	84	84	-	
		Overall (n = 17)		**92**	**7.3**	

Final determination as: Prothioconazole-desthio; Residues calculated as: Prothioconazole-desthio
RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
These recoveries were performed with barley and wheat commodities during the conduct of the studies RA-2037/07, RA-2038/07, RA-2039/07, RA-2040/07, RA-2041/07, RA-2042/07, RA-2043/07, RA-2045/07, RA-2046/07, RA-2047/07, RA-2048/07, RA-2049/07 and RA-2050/07. Cereal summarises barley and wheat.
* = Recoveries for rest of plant are covered by recoveries for green material.
# = Recoveries for ear are covered by recoveries for grain.

Table 6.3.2-24: RA-2328/06 : Concurrent recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain	0.01	103 ; 115 ; 110	109	5.5	0.01
		0.10	111 ; 105 ; 96	104	7.3	
		Overall (n = 6)		**107**	**6.3**	
Prothio-conazole-desthio	Cereal green material	0.01	106 ; 98 ; 91	98	7.6	0.01
		0.10	100 ; 95 ; 100 ; 113 ; 92 ; 109 ; 105 ; 106 ; 100	102	6.5	
		5.0	86 ; 86	86	-	
		6.0	79	79	-	
		Overall (n = 15)		**98**	**9.8**	
Prothio-conazole-desthio	Cereal straw	0.01	105	105	-	0.01
		0.10	97 ; 101 ; 111	103	7.0	
		9.0	90	90	-	
		Overall (n = 5)		**101**	**7.9**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
These recoveries were performed with barley and wheat commodities during the conduct of the studies RA-2326/06, RA-2327/06, RA-2328/06 and RA-2329/06. Cereal summarises barley and wheat.

- Residue results:

The residue results are summarised in Table 6.3.2-25.

Table 6.3.2-25: Residues of prothioconazole-desthio in/on spring and winter barley applied with BYF 00587 (bixafen) & JAU 6476 (prothioconazole) EC 225

Study Trial No. Trial SubID GLP Year	Crop Variety	Country	Application					Residues (mg/kg)*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	Prothioconazole-desthio
RA-2328/06 R 2006 0457 2 0457-06 GLP yes 2006	Barley, spring Carafe	France F-[redacted] Europe, North	225 EC	2	0.15	0.05	61	green material	0(1) 0	0.06/0.04(2) 0.84
								straw	34	0.36
								grain	34	<0.01
RA-2328/06 R 2006 0458 0 0458-06 GLP yes 2006	Barley, spring Prestige	Sweden S-[redacted] Europe, North	225 EC	2	0.15	0.05	61	green material	0(1) 7 14 28	1.8 1.5 0.59 0.53
								straw	40	0.56
								grain	40	<0.01
RA-2328/06 R 2006 0459 9 0459-06 GLP yes 2006	Barley, winter Sequel	United Kingdom GB-[redacted] Europe, North	225 EC	2	0.15	0.05	61	green material	0(1) 0	0.21 0.71
								straw	62	0.12/0.01(2)
								grain	62	<0.01
RA-2328/06 R 2006 0460 2 0460-06 GLP yes 2006	Barley, winter Duet	Germany D-[redacted] (Nordrhein-Westfalen) Europe, North	225 EC	2	0.15	0.05	61	green material	0(1) 0 7 14 28	0.15/0.02(2) 0.81 0.38 0.17 0.06
								straw	51	0.04
								grain	51	<0.01
RA-2039/07 R 2007 0426 7 0426-07 GLP yes 2007	Barley, spring Heidley	France F-[redacted] Europe, North	225 EC	2	0.15	0.05	61	green material	0(1) 0	0.27 1.3
								straw	58	0.03
								grain	58	<0.01
								ear	35	<0.01
								rest of plant	35	0.03
RA-2039/07 R 2007 0427 5 0427-07 GLP yes 2007	Barley, spring Annabell	Germany D-[redacted] (Nordrhein-Westfalen) Europe, North	225 EC	2	0.15	0.05	61	green material	0(1) 0	0.21 0.82
								straw	35 49	0.11 0.04
								grain	35 49	<0.01 <0.01

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) before last application
(2): residue in control sample
GS = growth stage (BBCH code)
FL = formulation

In the 6 trials conducted in 2006 and 2007 (report RA-2328/06 and RA-2039/07), residues of prothioconazole-desthio in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.03 mg/kg and 0.56 mg/kg.

Conclusion

Six barley residue trials were conducted with bixafen & prothioconazole EC 225 in northern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha.

In the 6 trials conducted in 2006 and 2007 (report RA-2328/06 and RA-2039/07), residues of prothioconazole-desthio in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.03 mg/kg and 0.56 mg/kg.

Report:	KCA 6.3.2/14[redacted]; 2008; M-298147-01-1
Title:	Determination of the residues of BYF 00587, JAU 6476 and KWG 4168 in/on spring barley and winter barley after spraying of BYF 00587 & JAU 6476 & KWG 4168 (400 EC) in the field in Northern France, Germany, the United Kingdom and the Netherla
Report No.:	RA-2042/07
Document No.:	M-298147-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/A/I/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2007, a set of 4 residue trials on spring and winter barley was conducted in northern Europe. The trials were located in Germany, northern France, the Netherlands and Great Britain.

In each trial, barley was treated twice at a product rate of 1.5 L/ha BYF 00587 (bixafen) & JAU 6476 (prothioconazole) & KWG 4168 (spiroxamine) EC 400, corresponding to 0.150 kg/ha prothioconazole. The second application in one trial was 6% overdosed (0.160 kg/ha of prothioconazole were applied). The water rate was 300 - 318 L/ha. The spray interval ranged from 14 - 19 days. The time of application was:

1. when the flag leaf is just visible (still rolled) (BBCH 37), and
2. at the beginning of flowering (BBCH 61).

Samples were taken at the following intervals:
- prior to and immediately after the final application,
- at a pre-harvest interval of 35 (34) days after the final treatment,
- at harvest (BBCH 89).

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-26.

Table 6.3.2-26: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	ear	136	4.5	RA-2042/07 M-298147-01-1
		grain	116	3.9	
		green material	171	5.7	
		rest of plant	136	4.5	
		straw	116	3.9	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-27. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-27: RA-2042/07: Concurrent recoveries for the determination of prothioconazole-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain #	0.01	97; 97; 96; 100; 93; 94; 99	97	2.6	0.01
		0.10	95; 93; 95; 91; 93; 100	95	3.3	
		1.0	92; 95; 95; 89; 93; 99	94	3.6	
		5.0	89	--	--	
		10	86	--	--	
		Overall Recovery (n = 21)		**94**	**3.9**	
Prothio-conazole-desthio	Cereal green material*	0.01	98; 94; 90; 97	95	3.8	0.01
		0.1	97; 90; 95; 88	93	4.5	
		1.0	87; 93; 95; 91; 89	91	3.9	
		5.0	89; 85; 82	85	4.1	
		10.0	83; 86; 85	85	1.8	
		Overall Recovery (n = 19)		**90**	**5.5**	
Prothio-conazole-desthio	Cereal straw	0.01	92; 88; 104; 93; 104	96	7.7	0.01
		0.10	95; 88; 94; 103	95	6.5	
		1.0	88; 93; 91; 93	91	2.6	
		5.0	88; 80; 87	85	5.1	
		10	84	--	--	
		Overall Recovery (n = 17)		**92**	**7.3**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
These recoveries were performed with wheat and barley commodities during the conduct of the studies RA-2037/07, RA-2038/07, RA-2039/07, RA-2040/07, RA-2041/07, RA-2042/07, RA-2043/07, RA-2045/07, RA-2046/07, RA-2049/07 and RA-2050/07. Cereal summarises barley and wheat. Recoveries for wheat are also valid for barley.
* = Recoveries for rest of plant are covered by recoveries for green material.
# = Recoveries for ear are covered by recoveries for grain.

- Residue results:

The residue results are summarised in Table 6.3.2-28.

Table 6.3.2-28: Residues of prothioconazole-desthio in/on spring and winter barley applied with Bixafen & Prothioconazole & Spiroxamine EC 400

Study Trial No. Trial SubID GLP Year	Crop, Variety	Country	Application FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Residues (mg/kg)* Portion analysed	DALT (days)	Prothioconazole-desthio
RA-2042/07 R 2007 0448 8 0448-07 GLP: yes 2007	barley, spring Heinley	France F-[redacted] northern EU	400 EC	2	0.150	0.05	61	green material	0(1) 0	0.13 1.3
								straw	58	0.02
								grain	58	<0.01
								ear	35	<0.01
								rest of plant	35	0.02
RA-2042/07 R 2007 0449 6 0449-07 GLP: yes 2007	barley, winter Naomi	Germany D-[redacted] (Nordrhein-Westfalen) northern EU	400 EC	2	0.150	0.05	61	green material	0(1) 0	0.31 1.4
								straw	55	0.06
								grain	55	<0.01
								ear	35	0.01
								rest of plant	55	0.16
RA-2042/07 R 2007 0527 1 0527-07 GLP: yes 2007	barley, spring Cocktail	United Kingdom GB-[redacted] northern EU	400 EC	2	0.150-0.160	0.05	61	green material	0(1) 0	0.07 1.3
								straw	96	0.02
								grain	96	<0.01
								rest of plant	34	0.02
								ear	34	<0.01
RA-2042/07 R 2007 0529 8 0529-07 GLP: yes 2007	barley, spring Prestige	Netherlands NL-[redacted] (Noord-Holland) northern EU	400 EC	2	0.150	0.05	61	green material	0(1) 0	0.21 1.2
								straw	35 71	0.02 0.02
								grain	35 71	<0.01 <0.01

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) before last application
GS = growth stage (BBCH code) at last application
FL = formulation

In the 4 trials conducted in 2007 (report RA-2042/07), residues of prothioconazole-desthio in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.02 mg/kg and 0.06 mg/kg.

Conclusion

Four barley residue trials were conducted with Bixafen & Prothioconazole & Spiroxamine EC 400 in northern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha. Residues of prothioconazole-desthio in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.02 mg/kg and 0.06 mg/kg.

Report:	KCA 6.3.2/15[redacted],;[redacted];[redacted]; 2011; M-414691-01-1
Title:	Determination of the residues of BYF 00587, HEC 5725 and prothioconazole in/on barley after spray application of bixafen & fluoxastrobin & prothioconazole EC 190 in the field in the Netherlands and Germany
Report No.:	10-2204
Document No.:	M-414691-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2010, a set of 2 residue trials on spring barley was conducted in northern Europe. The trials were located in the Netherlands and Germany.

In each trial, barley was treated twice at a product rate of 1.5 L/ha Bixafen & Fluoxastrobin & Prothioconazole EC 190 corresponding to 0.150 kg a.s./ha of prothioconazole. The water rate was 300 L/ha. The spray interval was 14 days. The time of application was:

1. when the flag leaf is just visible (still rolled) and at the flag leaf stage (flag leaf fully unrolled, ligule just visible) (BBCH 37-39), and
2. at the beginning of flowering (BBCH 61).

Samples were taken at the following intervals:
- prior to and immediately after the final application;
- at a pre-harvest interval of 35 (34) days after the final treatment.
- at harvest (BBCH 89).

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days.

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).
Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio were determined according to method 00979/M001 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-29.

Table 6.3.2-29: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	grain	352	11.7	10-2204 M-514691-01-1
		green material	387	12.9	
		straw	352	11.7	
	prothioconazole-alpha-hydroxy-desthio prothioconazole-3-hydroxy-desthio prothioconazole-4-hydroxy-desthio prothioconazole-5-hydroxy-desthio prothioconazole-6-hydroxy-desthio	grain	281	9.4	
		green material	301	10	
		straw	282	9.4	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110% except for prothioconazole-desthio in green material at the LOQ level (112%) and for prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio and prothioconazole-6-hydroxy-desthio in straw at 0.50 mg/kg (68%, 69% and 68% respectively). The single and overall mean recoveries are shown in Table 6.3.2-30 to Table 6.3.2-35. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-30: 10-2204: Concurrent recoveries for the determination of prothioconazole-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain	0.01	108		-	0.01
		2.5	86 ; 99	93	-	
		Overall Recovery (n = 3)		**98**	**11.3**	
	Cereal green material	0.01	108; 108; 116; 111; 117	112[a]	3.8	0.01
		0.10	97	-	-	
		2.0	84; 104	94	-	
		2.5	92; 95; 94; 89	93	2.9	
		Overall Recovery (n = 12)		**101**	**10.7**	
	Cereal straw	2.5	73	-	-	0.01
		Overall Recovery (n = 1)		**73**	**-**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-desthio, determined as JAU 6476-desthio and calculated as JAU 6476-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
[a]This value was accepted due to a RSD below 20% and an overall mean in the range of 70-110%.

Table 6.3.2-31: 10-2204: Concurrent recoveries for the determination of prothioconazole-alpha-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-alpha-hydroxy-desthio	Cereal grain	0.01	89; 90; 118; 120; 89; 89; 94; 100; 95; 96	98	11.9	0.01
		0.50	89; 98; 89; 95	93	4.9	
		Overall Recovery (n = 14)		**97**	**10.6**	
	Cereal green material	0.01	80; 99; 104; 99; 104; 93; 98; 100; 102; 85; 85; 91; 92	95	8.2	0.01
		0.50	101; 95; 100; 101; 83; 85	94	8.7	
		Overall Recovery (n = 19)		**95**	**8.1**	
	Cereal straw	0.01	77; 79; 88; 97; 81; 84	84	8.7	0.01
		0.50	72; 71; 88; 78; 92	80	11.8	
		Overall Recovery (n = 11)		**82**	**9.9**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-alpha-hydroxy-desthio, determined as JAU 6476-alpha-hydroxy-desthio and calculated as JAU 6476-desthio.
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
** : mean of double injection

Table 6.3.2-32: 10-2204: Concurrent recoveries for the determination of prothioconazole-3-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-3-hydroxy-desthio	Cereal grain	0.01	73; 82; 109; 113; 85; 88; 89; 90; 88; 94	91	13.1	0.01
		0.50	83; 93; 78; 81	84	7.8	
		Overall Recovery (n =14)		**89**	**12.3**	
	Cereal green material	0.01	70; 90; 101; 91; 103; 91; 94; 94; 94; 70; 71; 75; 77	86	13.8	0.01
		0.50	85; 82; 98; 104; 70; 74	86	15.5	
		Overall Recovery (n =19)		**86**	**14.0**	
	Cereal straw	0.01	80; 80; 83; 86; 98; 107	89	12.4	0.01
		0.50	62; 63; 81; 67; 76	70	12.0	
		Overall Recovery (n =11)		**80**	**17.2**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-3-hydroxy-desthio, determined as prothioconazole-3-hydroxy-desthio and calculated as prothioconazole-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
**: mean of double injection

Table 6.3.2-33: 10-2204: Concurrent recoveries for the determination of prothioconazole-4-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-4-hydroxy-desthio	Cereal grain	0.01	65; 72; 99; 105; 60; 67; 68; 70; 71; 73	75	19.7	0.01
		0.50	87; 95; 72; 76	83	12.7	
		Overall Recovery (n = 14)		**77**	**17.8**	
	Cereal green material	0.01	68; 80; 99; 79; 83; 65; 66; 73; 74; 63; 64; 74; 76	74	13.3	0.01
		0.50	88; 84; 95; 102; 71 ;75	86	13.7	
		Overall Recovery (n = 19)		**78**	**14.9**	
	Cereal straw	0.01	67; 64; 67; 77; 77; 81	72	9.7	0.01
		0.50	67; 62; 79; 62; 70	68***	10.3	
		Overall Recovery (n = 11)		**70**	**10.0**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-4-hydroxy-desthio, determined as prothioconazole-4-hydroxy-desthio and calculated as prothioconazole-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
**: mean of double injection
***: This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%.

Table 6.3.2-34: 10-2204: Concurrent recoveries for the determination of prothioconazole-5-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-5-hydroxy-desthio	Cereal grain	0.01	74; 77; 108; 108; 75; 83; 86; 88; 78; 86	86	14.4	0.01
		0.50	80;86;83;83	83	3.0	
		Overall Recovery (n = 14)		**85**	**12.3**	
	Cereal green material	0.01	73; 93; 99; 95; 95; 76; 77; 78; 82; 72; 73; 78; 79	82	11.7	0.01
		0.50	90; 85; 92; 98; 70; 73	85	13.1	
		Overall Recovery (n = 19)		**83**	**11.9**	
	Cereal straw	0.01	80; 71; 79; 84; 78; 88	80	7.2	0.01
		0.50	62; 64; 80; 66; 75	69***	11.1	
		Overall Recovery (n = 11)		**75**	**11.2**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-5-hydroxy-desthio, determined as prothioconazole-5-hydroxy-desthio and calculated as prothioconazole-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
**: mean of double injection
***: This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%.

Table 6.3.2-35: Concurrent recoveries for the determination of prothioconazole-6-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-6-hydroxy-desthio	Cereal grain	0.01	69; 75; 99; 101; 94; 96; 97; 100; 93; 97	92	11.9	0.01
		0.50	79;84;79;82	81	3.0	
		Overall Recovery (n = 14)		**89**	**11.9**	
	Cereal green material	0.01	73; 102; 103; 92; 99; 87; 93; 95; 99; 69; 75; 77; 77	88	13.8	0.01
		0.50	79; 75; 89; 91; 70; 79	81	10.1	
		Overall Recovery (n = 19)		**85**	**13.2**	
	Cereal straw	0.01	82; 74; 75; 78; 75; 82	78	4.7	0.01
		0.50	68; 68; 80; 60; 62	68***	11.5	
		Overall Recovery (n = 11)		**73**	**10.5**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-6-hydroxy-desthio, determined as prothioconazole-6-hydroxy-desthio and calculated as prothioconazole-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
**: mean of double injection
***: This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%.

- Residue results:

The residue results are summarised in Table 6.3.2-36.

Table 6.3.2-36: Residues of prothioconazole-desthio in/on spring barley applied with bixafen & fluoxastrobin & prothioconazole EC 190

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
10-2204 10-2204-01 GLP: yes 2010	Barley, spring Tipple	Nether-lands [redacted] Europe, North	190 EC	2	0.150	0.050	61	green material	0 0	0.36 0.51	<0.01 <0.01	0.05 0.05	0.07 0.08	0.03 0.03	<0.01 <0.01	0.53 0.69
								grain	35 43	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06
								straw	35 43	0.11 0.05	<0.01 <0.01	0.10 0.04	0.12 0.04	0.06 0.02	<0.01 <0.01	0.41 0.17
10-2204 10-2204-02 GLP: yes 2010	Barley, spring Quench	Germany [redacted] Europe, North	190 EC	2	0.150	0.050	61	green material	0(1) 0	0.53 1.5	<0.01 <0.01	0.09 0.10	0.09 0.09	0.05 0.06	<0.01 <0.01	0.78 1.8
								grain	34 46	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06
								straw	34 46	0.54 0.17	<0.01 <0.01	0.32 0.17	0.25 0.12	0.10 0.08	0.01 <0.01	1.2 0.56

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg.
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

In the 2 trials conducted in 2010 (report 10-2204), residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in barley grain at harvest were <0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg.
In straw, the residues at harvest ranged as follows :

- between 0.05 mg/kg and 0.17 mg/kg for prothioconazole-desthio
- <0.01 mg/kg mg/kg for prothioconazole-alpha-hydroxy,
- between 0.04 mg/kg and 0.17 mg/kg for prothioconazole-3-hydroxy-desthio,
- between 0.04 mg/kg and 0.12 mg/kg for prothioconazole-4-hydroxy-desthio,
- between 0.02 mg/kg and 0.08 mg/kg for prothioconazole-5-hydroxy-desthio,
- <0.01 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio,
- between 0.17 mg/kg and 0.56 mg/kg for the total residue.

Conclusion

Two barley residue trials were conducted with Bixafen & Fluoxastrobin & Prothioconazole EC 190 in northern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha.
Residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in barley grain at harvest were <0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg.
In straw, the residues at harvest ranged as follows :

- between 0.05 mg/kg and 0.17 mg/kg for prothioconazole-desthio,
- <0.01 mg/kg mg/kg for prothioconazole-alpha-hydroxy,
- between 0.04 mg/kg and 0.17 mg/kg for prothioconazole-3-hydroxy-desthio,
- between 0.04 mg/kg and 0.12 mg/kg for prothioconazole-4-hydroxy-desthio,
- between 0.02 mg/kg and 0.08 mg/kg for prothioconazole-5-hydroxy-desthio,
- <0.01 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio,
- between 0.17 mg/kg and 0.56 mg/kg for the total residue.

Report:	KCA 6.3.2/16 [redacted], 2015; M-501711-03-1
Title:	Determination of the residues of fluoxastrobin and prothioconazole in/on spring barley after spray application of fluoxastrobin & prothioconazole EC 200 in Germany
Report No.:	13-2137
Document No.:	M-501711-03-1
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC EC Guidance working document 7029/VI/95 rev.5 (1997-07-22), OECD 509 Adopted 2009-09-07, OECD GUIDELINE FOR THE TESTING OF CHEMICALS, Crop Field Trial US EPA OCSPP Guideline No. 860.1500
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.3.2/17 [redacted],; [redacted]; 2014; M-501503-01-1
Title:	Determination of the residues of fluoxastrobin and prothioconazole in/on barley and spring barley after spray application of Fluoxastrobin & Prothioconazole EC 200 in France (North)
Report No.:	13-2158
Document No.:	M-501503-01-1
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC EC Guidance working document 7029/VI/95 rev.5 (1997-07-22) OECD 509 Adopted 2009-09-07, OECD GUIDELINE FOR THE TESTING OF CHEMICALS, Crop Field Trial US EPA OCSPP Guideline No. 860.1500
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2013, a set of 4 residue trials on spring and winter barley was conducted in northern Europe. The trials were located in northern France and Germany.

In each trial, barley was treated twice at a product rate of 1.25 L/ha fluoxastrobin & prothioconazole EC 200 corresponding to 0.125 kg a.s./ha of prothioconazole. In trial 13-2158-02, the two applications were overdosed by 6.3% and 8.3%, respectively. The water rate was 200-400 L/ha. The spray interval ranged from 6-20 days. The time of application was:

1. when node 2 is at least 2 cm above node 1until 70% of inflorescence emerged (BBCH 32-57), and
2. at the beginning of flowering (BBCH 61).

Samples were taken at the following intervals:
- prior to and immediately after the final application,
- at days 7, 14, 21 and 28 (except for the trial 13-2158-02) after final application,
- at a pre-harvest interval of 35 days (except for the trial 13-2137-02) after the final application,
- at harvest (BBCH 89). Additionnal samples were taken at days 17 (trial 13-2158-02), 31 (trial 13-2158-01) and 42 (trial 13-2137-02) after final application.

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days.

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).
Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio were determined according to method 00979/M001 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-37.

Table 6.3.2-37: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	grain	337	11.2	13-2158 M-501503-01-1
		green material	366	12.2	
		straw	337	11.2	
	prothioconazole-alpha-hydroxy-desthio prothioconazole-3-hydroxy-desthio prothioconazole-4-hydroxy-desthio prothioconazole-5-hydroxy-desthio prothioconazole-6-hydroxy-desthio	grain	415	13.8	
		green material	451	15.0	
		straw	424	14.1	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110% except for prothioconazole-3-hydroxy-desthio in green material at the LOQ level (68%), for prothioconazole-4-hydroxy-desthio at the LOQ level in grain (68%) and in straw (64%) and prothioconazole-6-hydroxy-desthio in grain at the LOQ level (117%). The single and overall mean recoveries are shown in Table 6.3.2-38 to Table 6.3.2-43. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-38: 13-2158 and 13-2137: Concurrent recoveries for the determination of prothioconazole-desthio in barley

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
Prothio-conazole-desthio	13-2158	barley grain	0.01	103	-	-	0.01
			0.10	104	-	-	
			Overall Recovery (n = 2)		**104**		
		barley green material	0.01	104*	-	-	0.01
			0.10	113	-	-	
			1.0	88	-	-	
			Overall Recovery (n = 3)		**102**	**12.5**	
		barley straw	0.01	107*	-	-	0.01
			0.10	108	-	-	
			1.0	91	-	-	
			Overall Recovery (n = 3)		**102**	**9.4**	
Prothio-conazole-desthio	13-2137	barley grain	0.01	99	-	-	0.01
			0.10	98	-	-	
			Overall Recovery (n = 2)		**99**	-	
		barley green material	0.01	118	-	-	0.01
			0.10	100	-	-	
			Overall Recovery (n = 2)		**109**		
		barley straw	0.01	97	-	-	0.01
			0.10	96	-	-	
			Overall Recovery (n = 2)		**97**	**-**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-desthio, determined as prothioconazole-desthio and calculated as prothioconazole-desthio
* : corrected for residue level in control sample (29% of the LOQ for green material and 24% of the LOQ for straw). Uncorrected values are 133% (green material) and 132% (straw).

Table 6.3.2-39: 13-2158 and 13-2137: Concurrent recoveries for the determination of prothioconazole-alpha-hydroxy-desthio in barley

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
prothioconazole-alpha - hydroxy-desthio	13-2158	barley grain	0.01	81; 82	82	-	0.01
			0.10	76; 80	78	-	
			Overall recovery (n = 4)		**80**	**3.3**	
		barley green material	0.01	78; 80	79	-	0.01
			0.10	95; 99	97	-	
			0.30	99; 100	100		
			Overall recovery (n = 6)		**92**	**11.0**	
		barley straw	0.01	63; 69; 76; 81	72	10.9	0.01
			0.10	87; 88	88	-	
			0.60	87; 100	94		
			Overall recovery (n = 8)		**81**	**14.5**	
prothioconazole-alpha-hydroxy-desthio	13-2137	barley grain	0.01	77 ; 78	78	-	0.01
			0.10	93 ; 93	93	-	
			Overall Recovery (n = 4)		**85**	**10.5**	
		barley green material	0.01	85 ; 94 ; 109	96	12.6	0.01
			0.10	85 ; 86	86	-	
			Overall Recovery (n = 5)		**92**	**11.3**	
		barley straw	0.01	85 ; 86	86	-	0.01
			0.20	88 ; 90 ; 97; 100	94	6.1	
			Overall Recovery (n = 6)		**91**	**6.7**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-alpha-hydroxy-desthio, determined as prothioconazole-alpha-hydroxy-desthio and calculated as prothioconazole-desthio

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Table 6.3.2-40: 13-2158 and 13-2137: Concurrent recoveries for the determination of prothioconazole-3-hydroxy-desthio in barley

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
prothioconazole-3-hydroxy-desthio	13-2158	barley grain	0.01	83; 86	85	-	0.01
			0.10	74; 75	75	-	
			Overall recovery (n = 4)		**80**	**7.4**	
		barley green material	0.01	68; 68	68*	-	0.01
			0.10	88; 91	90	-	
			0.30	96; 97	97	-	
			Overall recovery (n = 6)		**85**	**15.7**	
		barley straw	0.01	67; 67; 78; 86	75	12.4	0.01
			0.10	79; 86	83	-	
			0.60	86; 96	91	-	
			Overall recovery (n = 8)		**81**	**12.4**	
prothioconazole-3-hydroxy-desthio	13-2137	barley grain	0.01	86 ; 87	87	-	0.01
			0.10	87 ; 87	87	-	
			Overall Recovery (n = 4)		**87**	**0.6**	
		barley green material	0.01	81 ; 96 ; 108	95	14.2	0.01
			0.10	85 ; 93	89	-	
			Overall Recovery (n = 5)		**93**	**11.3**	
		barley straw	0.01	89 ; 89	89	-	0.01
			0.20	85 ; 85 ; 91 ; 97	90	6.4	
			Overall Recovery (n = 6)		**89**	**5.0**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-hydroxy-desthio, determined as prothioconazole-hydroxy-desthio and calculated as prothioconazole-desthio *: This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%.

Table 6.3.2-41: 13-2158 and 13-2137: Concurrent recoveries for the determination of prothioconazole-4-hydroxy-desthio in barley

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
prothioconazole-4-hydroxy-desthio	13-2158	barley grain	0.01	65; 70	68*	-	0.01
			0.10	70; 76	73	-	
			Overall recovery (n = 4)		**70**	**6.4**	
		barley green material	0.01	69; 70	70	-	0.01
			0.10	91; 96	94	-	
			0.30	90; 95	93	-	
			Overall recovery (n = 6)		**85**	**14.5**	
		barley straw	0.01	62; 65; 71	66*	6.9	0.01
			0.10	71; 80	76	-	
			0.60	83; 96	90	-	
			Overall recovery (n = 7)		**75**	**15.6**	
prothioconazole-4-hydroxy desthio	13-2137	barley grain	0.01	66 ; 67	67	-	0.01
			0.10	83 ; 85	84	-	
			Overall Recovery (n = 4)		**75**	**13.5**	
		barley green material	0.01	83 ; 95	89	-	0.01
			0.10	76 ; 80	78	-	
			Overall Recovery (n = 4)		**84**	**9.8**	
		barley straw	0.01	63 ; 64	64	-	0.01
			0.20	86 ; 87 ; 84 ; 86	86	1.5	
			Overall Recovery (n = 6)		**78**	**14.7**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-4-hydroxy-desthio, determined as prothioconazole-4-hydroxy-desthio and calculated as prothioconazole-desthio
* : This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%.

Table 6.3.2-42: 13-2158 and 13-2137: Concurrent recoveries for the determination of prothioconazole-5-hydroxy-desthio in barley

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
prothioconazole-5-hydroxy-desthio	13-2158	barley grain	0.01	73; 77	75	-	0.01
			0.10	77; 82	80	-	
			Overall recovery (n = 4)		**77**	**4.8**	
		barley green material	0.01	67; 83	75	-	0.01
			0.10	91; 94	93	-	
			0.30	96; 98	97	-	
			Overall recovery (n = 6)		**88**	**13.2**	
		barley straw	0.01	76; 77; 86	80	6.9	0.01
			0.10	81; 87	84	-	
			0.60	81; 94	88	-	
			Overall recovery (n = 7)		**83**	**7.6**	
prothioconazole-5-hydroxy desthio	13-2137	barley grain	0.01	87 ; 87	87	-	0.01
			0.10	85 ; 91	88	-	
			Overall Recovery (n = 4)		**88**	**2.9**	
		barley green material	0.01	79 ; 81 ; 92	84	8.3	0.01
			0.10	78 ; 85	82	-	
			Overall Recovery (n = 5)		**83**	**6.9**	
		barley straw	0.01	82 ; 85	84	-	0.01
			0.20	85 ; 86 ; 91 ; 97	90	6.1	
			Overall Recovery (n = 6)		**88**	**6.2**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-5-hydroxy-desthio, determined as prothioconazole-5-hydroxy-desthio and calculated as prothioconazole-desthio

Table 6.3.2-43: 13-2158 and 13-2137: Concurrent recoveries for the determination of prothioconazole-6-hydroxy-desthio in barley

Analyte	Study	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
				Single Values	Mean		
prothioconazole-6-hydroxy-desthio	13-2158	barley grain	0.01	115; 118	117*	-	0.01
			0.10	87; 92	90	-	
			Overall recovery (n = 4)		**103**	**15.3**	
		barley green material	0.01	73; 79	76	-	0.01
			0.10	88; 89	89	-	
			0.30	85; 91	88	-	
			Overall recovery (n = 6)		**84**	**8.2**	
		barley straw	0.01	70; 77; 78; 83	77	7.0	0.01
			0.10	78; 84	81	-	
			0.60	86; 95	91	-	
			Overall recovery (n = 8)		**81**	**9.1**	
prothioconazole-6-hydroxy-desthio	13-2137	barley grain	0.01	90 ; 92	91	-	0.01
			0.10	83 ; 88	86	-	
			Overall Recovery (n = 4)		**88**	**4.4**	
		barley green material	0.01	93; 101 ; 109	101	7.9	0.01
			0.10	87 ; 93	90	-	
			Overall Recovery (n = 5)		**97**	**8.8**	
		barley straw	0.01	87 ; 87	87	-	0.01
			0.20	90 ; 93 ; 93 ; 96	93	2.6	
			Overall Recovery (n = 6)		**91**	**4.0**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-6-hydroxy-desthio, determined as prothioconazole-6-hydroxy-desthio and calculated as prothioconazole-desthio * c) This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%

- Residue results:

The residue results are summarised in Table 6.3.2-44 and Table 6.3.2-45.

Table 6.3.2-44: Residues of prothioconazole-desthio in/on spring barley applied with Fluoxastrobin & Prothioconazole EC 200

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
13-2137 13-2137-01 GLP: yes 2013	Barley, spring Conchita	Germany [redacted] Europe, North	200 EC	2	0.125	0.042	61	green material	0(1)	0.27	<0.01	0.023	0.032	0.018	<0.01	0.36
									0	0.94	<0.01	0.032	0.044	0.024	<0.01	1.1
									7	0.88	<0.01	0.062	0.075	0.044	<0.01	1.1
									14	0.42	<0.01	0.093	0.091	0.059	<0.01	0.68
									21	0.15	<0.01	0.084	0.080	0.053	<0.01	0.39
									28	0.067	<0.01	0.074	0.066	0.039	<0.01	0.27
									35	0.049	<0.01	0.079	0.064	0.038	<0.01	0.25
								grain	35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
									69	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	35	0.087	<0.01	0.14	0.12	0.067	<0.01	0.43
									69	0.036	<0.01	0.029	0.030	0.020	<0.01	0.14
13-2137 13-2137-02 GLP: yes 2013	Barley, spring Grace	Germany [redacted] Europe, North	200 EC	2	0.125	0.031-0.042	61	green material	0(1)	0.50	<0.01	0.023	0.010	0.013	<0.01	0.57
									0	1.1	<0.01	0.028	0.012	0.013	<0.01	1.2
									7	0.47	0.010	0.070	0.025	0.030	<0.01	0.62
									14	0.22	0.013	0.080	0.027	0.032	<0.01	0.38
									21	0.098	0.013	0.092	0.028	0.029	<0.01	0.27
									28	0.067	0.011	0.078	0.025	0.024	<0.01	0.22
									42	0.041	<0.01	0.038	0.012	0.013	<0.01	0.12
								grain	68	0.011	<0.01	<0.01	<0.01	<0.01	<0.01	0.061
								straw	68	0.044	<0.01	0.020	<0.01	<0.01	<0.01	0.10

(1): before last treatment *for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg.
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

Table 6.3.2-45: Residues of prothioconazole-desthio in/on barley applied with Fluoxastrobin & Prothioconazole EC 200

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
13-2158 13-2158-01 GLP: yes 2013	Barley Esterel	France [redacted] Europe, North	200 EC	2	0.125	0.063	61	green material	0[1] 0 7 14 21 28 31	0.17 1.5 0.46 0.14 0.078 0.067 0.079	<0.01 <0.01 0.012 <0.01 <0.01 <0.01 <0.01	0.051 0.056 0.076 0.080 0.088 0.057 0.067	0.047 0.049 0.068 0.065 0.074 0.046 0.057	0.044 0.044 0.065 0.059 0.058 0.039 0.048	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.33 1.7 0.69 0.36 0.32 0.23 0.27
								grain	35 43	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06
								straw	35 43	0.039 0.15	<0.01 <0.01	0.051 0.11	0.036 0.083	0.034 0.092	<0.01 <0.01	0.18 0.46
13-2158 13-2158-02 GLP: yes 2013	Barley, spring Sébastian	France [redacted] Europe, North	200 EC	2	0.133-0.135	0.062-0.063	61	green material	0[1] 0 7 14 21 42	0.33 0.89 0.52 0.49 0.40 0.34	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.11 0.13 0.17 0.16 0.15 0.16	0.15 0.16 0.21 0.20 0.16 0.17	0.078 0.091 0.10 0.082 0.065 0.077	<0.01 <0.01 <0.01 0.010 0.010 <0.01	0.69 1.3 1.2 0.95 0.80 0.87
								grain	35	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	35	0.81	0.023	0.55	0.55	0.23	0.042	2.2

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg unless all the values were <0.01 mg/kg.
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

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In the 4 trials conducted in 2013 (reports 13-2137 and 13-2158), residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in barley grain at harvest were < 0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg except for trial 13-2137-02 where the total residue reached 0.061 mg/kg. In straw, the residues at harvest ranged as follows :

- between 0.036 mg/kg and 0.81 mg/kg for prothioconazole-desthio
- between < 0.01 mg/kg and 0.023 mg/kg for prothioconazole-alpha-hydroxy
- between 0.020 mg/kg and 0.55 mg/kg for prothioconazole-3-hydroxy-desthio
- between <0.01 mg/kg and 0.55 mg/kg for prothioconazole-4-hydroxy-desthio
- between <0.01 mg/kg and 0.23 mg/kg for prothioconazole-5-hydroxy-desthio
- between < 0.01 mg/kg and 0.042 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio
- between 0.10 mg/kg and 2.2 mg/kg for total residue calc.

Conclusion

Four barley residue trials were conducted with Fluoxastrobin & Prothioconazole EC 200 in northern Europe. The product application corresponded to a prothioconazole rate of 2x125 g a.s./ha with the exception of trial 13-2158-02 where the two applications were overdosed by 6.3% and 8.3%, respectively.

Residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in barley grain at harvest were < 0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg.

In straw, the residues at harvest ranged as follows:

- between 0.036 mg/kg and 0.81 mg/kg for prothioconazole-desthio
- between < 0.01 mg/kg and 0.023 mg/kg for prothioconazole-alpha-hydroxy
- between 0.020 mg/kg and 0.55 mg/kg for prothioconazole-3-hydroxy-desthio
- between <0.01 mg/kg and 0.55 mg/kg for prothioconazole-4-hydroxy-desthio
- between <0.01 mg/kg and 0.23 mg/kg for prothioconazole-5-hydroxy-desthio
- between < 0.01 mg/kg and 0.042 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio
- between 0.10 mg/kg and 2.2 mg/kg for total residue calc.

EU - southern region

Table 6.3.2-46: Number of residue trials conducted per geographical region and vegetation period

Year	GAP	Formulation	N° of trials	Study number Report No.	Dossier Reference
Barley EU foliar spray residue trials – southern EU					
2000	2 x 125 g a.s./ha BBCH61	EC200 (100 g/L fluoxastrobin, 100 g/L prothioconazole)	3	RA-2014/00 M-137478-01-1	KCA 6.3.2/18
2002	2 x 125 g a.s./ha BBCH61	EC250 (125 g/L tebuconazole 125 g/L prothioconazole)	3	RA-2104/02 M-086880-01-1	KCA 6.3.2/19
2003	2 x 150 g a.s./ha BBCH61	EC300 (75 g/L fluoxastrobin, 150 g/L prothioconazole, 75 g/L trifloxystrobin)	4	RA-2017/03 M-062669-03-1	KCA 6.3.2/20
2003	2 x 175 g a.s./ha BBCH61	SC325 (175 g/L prothioconazole, 150 g/L trifloxystrobin)	4	RA-2107/03 M-060760-01-1	KCA 6.3.2/21
2005	2 x 125 g a.s./ha BBCH61	EC450 (100 g/L prothioconazole, 250 g/L spiroxamine, 100 g/L tebuconazole)	3	RA-2572/05 M-232115-01-1	KCA 6.3.2/22
2006	2 x 150 g a.s./ha BBCH61	EC225 (75 g/L bixafen, 150 g/L prothioconazole)	4	RA-2329/06 M-294526-01-1	KCA 6.3.2/23
2007	2 x 150 g a.s./ha BBCH61	EC400 (50 g/L bixafen, 100 g/L prothioconazole, 250 g/L spiroxamine)	4	RA-2043/07 M-298412-01-1	KCA 6.3.2/24
2010	2 x 150 g a.s./ha BBCH61	EC190 (40 g/L bixafen, 50 g/L fluoxastrobin and 100 g/L prothioconazole)	2	10-2206 M-414709-01-1	KCA 6.3.2/25
2011	2 x 175 g a.s./ha BBCH61	EC150 (50 g/L fluoxastrobin, 100 g/L prothioconazole)	5	11-2111 M-434980-04-1	KCA 6.3.2/26
TOTAL southern EU region			**31**		

Report: KCA 6.3.2/18 [redacted]; 2001; M-137478-01-1
Title: Determinaton of residues of HEC 5725 & JAU 6476-Desthio on barley following spray application of HEC 5725 & JAU 6476 200 EC in Italy, France and Spain
Report No.: RA-2014/00
Document No.: M-137478-01-1
Guideline(s): EU-Ref: Council Directive 91/414/EEC of 15 July, 1991, Annex II, part A, point 6 and Annex III, part A, point 8
Residues in or on Treated Products, Food and Feed
Guideline deviation(s): not specified
GLP/GEP: **yes**

Materials and Methods

Important note: In this report, a trial (Spain R 2000 0160/6) had to be discarded as the good agricultural practice was not met: the second treatment was carried out at a later stage as originally intended (BBCH 85 instead of 61). Indeed, in order to keep the pre-harvest interval of 35 days, barley was treated at a

rather late growth stage (soft dough stage, BBCH 85, 42 days after the first application). In this dossier, we no longer support a PHI of 35 days. As the application in cereals is driven purely by growth stage, no PHI is deemed necessary (last application at BBCH61). The resulting residue values are not considered relevant for the purpose of MRL setting or related assessments, because the treatment of cereals at such a late growth stage as BBCH 85 is neither recommended nor common or good agricultural practice. This trial is not included in the summary below.

In the vegetation period of 1999/2000, a set of 3 residue trials were planned in southern Europe. The trials were located in Italy, and southern France.

In each trial, barley was treated twice at a product rate of 1.25 L/ha fluoxastrobin & prothioconazole EC 200 corresponding to 0.125 kg a.s./ha of prothioconazole. The employed water rate was 280 or 300 L/ha. The spray interval ranged from 13-18 days. The time of application was:

1. during the time when the flag leaf sheath of the barley plants was just visible (BBCH 37), and
2. at the beginning of flowering (BBCH 61).

Samples were taken at the following intervals:
- prior to and immediately after the final application,
- at a pre-harvest interval of 35 days as well as 49-55 days after the final treatment.

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-47.

Table 6.3.2-47: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	ear	322	10.7	RA-2014/00 M-137478-01-1
		grain	287	9.6	
		Rest of plant	479	16.0	
		straw	287	9.6	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-48. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-48: RA-2014/00: Concurrent recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Barley grain / Ear	0.01	102 ; 100 ; 103	102	1.5	0.01
		0.10	103 ; 103 ; 102 ; 103 ; 102 ; 103 ; 104 ; 103 ; 102 ; 103 ; 105 ; 104 ; 115 ; 99 ; 103 ; 106 ; 102 ; 94 ; 102 ; 101 ; 101	103	3.6	
		Overall (n = 24)		**103**	**3.4**	
Prothio-conazole-desthio	Rest of plant	0.05	102 ; 107 ; 102	104	2.8	0.05
		0.50	106 ; 103 ; 106 ; 103 ; 104 ; 104 ; 104 ; 105 ; 103 ; 103 ; 103 ; 97 ; 104 ; 115 ; 105 ; 102 ; 103 ; 99	104	3.4	
		Overall (n = 21)		**104**	**3.3**	
Prothio-conazole-desthio	Straw	0.05	94 ; 102 ; 96	97	4.3	0.05
		0.50	103 ; 104 ; 102 ; 102 ; 98 ; 100 ; 96 ; 103	101	2.7	
		Overall (n = 11)		**100**	**3.4**	

Final determination as: Prothioconazole-desthio. Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:
The residue results are summarised in Table 6.3.2-49.

Table 6.3.2-49: Results of residue trials conducted on fluoxastrobin & prothioconazole EC 200 in barley; results for prothioconazole-desthio

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2014/00 R 2000 0159/2 0159-00 GLP: yes 2000	Barley, winter Pastoral	France F-[redacted] Europe, South	200 EC	2	0.1250	0.04170 - 0.04460	61	ear	0[(1)] 0 35	<0.01 3.4 0.01
								rest of plant	0[(1)] 0 35	0.24 1.0 0.10
								straw	49	0.05
								grain	49	<0.01
RA-2014/00 R 2000 0157/6 0157-00 GLP: yes 2000	Barley Ganora	Italy I-[redacted] Europe, South	200 EC	2	0.1250	0.04170	61	ear	0[(1)] 0	0.02 2.0
								rest of plant	0[(1)] 0	0.56 1.7
								straw	35 55	0.14 0.11
								grain	35 55	<0.01 0.02
RA-2014/00 R 2000 0158/4 0158-00 GLP: yes 2000	Barley, winter Univer.	France F-[redacted] Europe, South	200 EC	2	0.1250	0.04170	61	ear	0[(1)] 0	<0.01 2.9
								rest of plant	0[(1)] 0	0.55 1.4
								straw	35	0.14
								grain	35	0.01

FL = Formulation, DALT = Days after last treatment,
* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) : before last application

In the three trials conducted in 2000 (report RA-2014/00), residues of prothioconazole-desthio in barley grain at harvest ranged between below the limit of quantification (LOQ) of 0.01 mg/kg and 0.02 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.14 mg/kg.

Conclusion

Three barley residue trials were conducted with fluoxastrobin & prothioconazole EC 200 in southern Europe. The product application corresponded to a prothioconazole rate of 2x125 g a.s./ha. Residues of prothioconazole-desthio in barley grain at harvest ranged between below the limit of quantification (LOQ) of 0.01 mg/kg and 0.02 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.14 mg/kg.

Report:	KCA 6.3.2/19[redacted],; 2003; M-086880-01-1
Title:	Determination of residues of tebuconazole and prothioconazole-desthio in/on barley after spray application of HWG 1608 & JAU 6476 250 EC in the field in Italy and Spain
Report No.:	RA-2104/02
Document No.:	M-086880-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of 15 July, 1991, Annex II, part A, point 6 and Annex III, part A, point 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2002, a set of 3 residue trials was conducted in southern Europe. The trials were conducted in Italy (2) and Spain (1).

'Prothioconazole + tebuconazole EC 250 (125+125 g/L)' was applied to barley by spraying two times at a product rate of 1.0 L/ha and 300 L/ha water (product concentration of 0.333%). The amount of tebuconazole and prothioconazole was 0.125 kg/ha.

The applications were performed at BBCH growth stages 39 and 55-59, except for 1 trial:

- R 2002 0542/2 (Italy) where the 2nd application was performed at BBCH growth stage 75 instead of 61 due to wet and windy weather conditions as well.

Spray intervals were from 14-22 days with the last treatment 35-42 days prior to the date of harvest.

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-50.

Table 6.3.2-50: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	ear	273	9.1	RA-2104/02 M-086880-01-1
		grain	238	7.9	
		Rest of plant	273	9.1	
		straw	238	7.9	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-51. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-51: RA-2104/02: Concurrent recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Barley grain / Ear	0.01	89 ; 100 ; 96	95	5.9	0.01
		0.10	94 ; 90 ; 95	93	2.8	
		Overall (n = 6)		**94**	**4.3**	
Prothio-conazole-desthio	Rest of plant	0.05	94 ; 91 ; 93	93	1.6	0.05
		0.50	97 ; 95 ; 95	96	1.2	
		Overall (n = 6)		**94**	**2.2**	
Prothio-conazole-desthio	Straw	0.05	100 ; 86 ; 102	96	9.1	0.05
		0.50	94 ; 96	95	-	
		Overall (n = 5)		**96**	**6.5**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:
The residue results are summarised in Table 6.3.2-52.

Table 6.3.2-52: Residues of prothioconazole-desthio in/on spring barley applied with tebuconazole & prothioconazole EC 250

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	Prothioconazole-desthio (mg/kg)
RA-2104/02 R 2002 0544/9 0544-02 GLP: yes 2002	Barley, spring Grafit	Spain E-[redacted] Europe, South	250 EC	2	0.1250	0.04163	55	ear	0	1.5
								rest of plant	0	2.2
								grain	35 42	0.01 <0.01
								straw	35 42	1.2 1.2
RA-2104/02 R 2002 0530/9 0530-02 GLP: yes 2002	Barley, spring Pandas	Italy I-[redacted] Europe, South	250 EC	2	0.1250	0.04163	59	ear	0	1.5
								rest of plant	0	0.85
								grain	35 41	<0.01 <0.01
								straw	35 41	0.08 0.08
RA-2104/02 R 2002 0542/2 0542-02 GLP: yes 2002	Barley, spring Klaxon	Italy I-[redacted] Europe, South	250 EC	2	0.1250	0.04163	75	ear	0 35	0.86 0.03
								rest of plant	0 35	1.1 0.13
								grain	42	0.01
								straw	42	0.24

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio

In the 3 trials conducted in 2002 (report RA-2104/02), residues of prothioconazole-desthio in barley grain at harvest ranged from <0.01 mg/kg to 0.01 mg/kg. The residues in straw at harvest ranged between 0.08 mg/kg and 1.2 mg/kg.

Conclusion

Four barley residue trials were conducted with tebuconazole & prothioconazole EC 250 in southern Europe. The product application corresponded to a prothioconazole rate of 2x125 g a.s./ha. Residues of prothioconazole-desthio in barley grain at harvest ranged from <0.01 mg/kg to 0.01 mg/kg. The residues in straw at harvest ranged between 0.08 mg/kg and 1.2 mg/kg.

Report: KCA 6.3.2/20[redacted]; 2015; M-062669-03-1
Title: Amendment No. 2 to report no: RA-2017/03 - Determination of residues of fluoxastrobin (HEC5725), prothioconazole (JAU6476) and trifloxystrobin (CGA279202 in/on barley following spray application of HEC 5725 & JAU 6476 & CGA279202 (300 EC) in France, Italy and Spain
Report No.: RA-2017/03
Document No.: M-062669-03-1
Guideline(s): EU-Ref: Council Directive 91/414/EEC of 15 July, 1991, Annex II, part A, point 6 and Annex III, part A, point 8
Residues in or on Treated Products, Food and Feed
Guideline deviation(s): not specified
GLP/GEP: **yes**

Materials and Methods

In the vegetation period of 2003, a set of 4 residue trials was conducted in southern Europe. The trials were located in Italy (2), southern France and Spain.

In each trial, barley was treated twice at a product rate of 1.0 L/ha fluoxastrobin & prothioconazole & trifloxystrobin EC 300 corresponding to 0.150 kg a.s./ha of prothioconazole. The employed water rate was 300-320 L/ha in all trials. The spray interval ranged from 13 - 18 days. The time of application was:
1. during the time when the flag leaf is just visible until the flag leaf sheath is extended (BBCH37-41),
2. at the beginning of flowering (BBCH 61) (except one trial at BBCH55-65).

Samples were taken at the following intervals:
- prior to and immediately after the final application,
- at a pre-harvest interval of 35 days
- at harvest (BBCH 89), 40-69 days after the final treatment.

Plant material was collected at a pre-harvest interval of 35 days as well as at one later date to obtain harvest values. This additional date was needed to ensure that samples of mature plants were available independent of the growth stage reached following the proposed pre-harvest interval of 35 days. Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-53.

Table 6.3.2-53: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	ear	196	6.5	RA-2017/03 M-062669-03-1
		grain	138	4.6	
		Rest of plant	196	6.5	
		straw	138	4.6	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-54. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-54: RA-2017/03: Concurrent recoveries and validation recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Barley grain / Ear	0.01	94 ; 97 ; 94	95	1.8	0.01
		0.10	*95 ; 93 ; 100 ; 93 ; 94*	95	3.1	
		1.0	*92*	92	-	
		2.0	94	94	-	
		4.0	94	94	-	
		Overall (n = 11)		**95**	**2.3**	
Prothio-conazole-desthio	Straw	0.05	97 ; 91 ; 98	95	4.0	0.05
		0.50	*98 ; 97 ; 96 ; 97 ; 99*	97	1.2	
		5.0	*94*	94	-	
		Overall (n = 9)		**96**	**2.5**	
Prothio-conazole-desthio	Rest of plant	0.05	100 ; 97 ; 99	99	1.5	0.05
		0.50	*96 ; 91 ; 95 ; 93 ; 95*	94	2.6	
		5.0	93	93	-	
		Overall (n = 9)		**96**	**3.1**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
Concurrent recoveries in italics

- Residue results:
The residue results are summarised in Table 6.3.2-55.

Table 6.3.2-55: Residues of prothioconazole-desthio in/on barley applied with fluoxastrobin & prothioconazole & trifloxystrobin EC 300

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2017/03 R 2003 0253/3 0253-03 GLP: yes 2003	Barley Aliseo	Italy I-[redacted] Europe, South	300 EC	2	0.1500	0.04995	61	rest of plant	0(1) 0	0.19 0.74
								ear	0(1) 0	<0.01 2.4
								grain	35 45	<0.01 <0.01
								straw	35 45	<0.05 0.05
RA-2017/03 R 2003 0254/1 0254-03 GLP: yes 2002	Barley Hispanic	Spain E-[redacted] Europe, South	300 EC	2	0.1500 0.1598	0.04995	61	rest of plant	0(1) 0 35	0.30 1.5 0.21
								ear	0(1) 0 35	0.02 2.6 0.03
								grain	69	<0.01
								straw	69	0.31
RA-2017/03 R 2003 0132/4 0132-03 GLP: yes 2003	Barley Print	France F-[redacted] Europe, South	300 EC	2	0.1500	0.04995	55-65	rest of plant	0(1) 0	0.67 2.1
								ear	0(1) 0	0.05 1.3
								grain	35	<0.01
								straw	35	0.62
RA-2017/03 R 2003 0256/8 0256-03 GLP: yes 2003	Barley Sonora	Italy I-[redacted] Europe, South	300 EC	2	0.1500	0.04995	61	rest of plant	0(1) 0	0.20 4.2
								ear	0(1) 0	<0.01 3.5
								grain	35 40	<0.01 <0.01
								straw	35 40	0.10 0.14

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) : before last application

In the 4 trials conducted in 2003 (report RA-2017/03), residues of prothioconazole-desthio in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.62 mg/kg.

Conclusion

Four barley residue trials were conducted with fluoxastrobin & prothioconazole & trifloxystrobin EC 300 in southern Europe. The product application corresponded to a prothioconazole rate of 2x125 g a.s./ha. Residues of prothioconazole-desthio in barley grain at harvest were <0.01 mg/kg. The residues in straw at harvest ranged between 0.05 mg/kg and 0.62 mg/kg.

Report:	KCA 6.3.2/21[redacted]; 2004; M-060760-01-1
Title:	Determination of residues of trifloxystrobin, CGA 321113, and JAU 6476-desthio in/on barley following spray application of CGA 279202 & JAU 6476 (325 SC) in the field in Southern France, Italy, Spain, and Greece
Report No.:	RA-2107/03
Document No.:	M-060760-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2003, a set of 4 residue trials was conducted in southern Europe. The trials were located in southern France, Italy, Spain and Greece.

In each trial, barley was treated twice at a product rate of 1.0 L/ha prothioconazole & trifloxystrobin SC 325 corresponding to 0.175 kg a.s./ha of prothioconazole. The first application was done at BBCH growth stage 47 and the second at growth stage 61 except for trial R 2003 0978/3 where treatments were carried out at the growth stages BBCH 57-58 and 69, respectively. The interval between applications ranged from 7 to 13 days.
The water rate was 300 L/ha, corresponding to a concentration of test substance in the spray liquid of 0.333%.
Samples of ear and rest of plant were taken on day 0 after the last application, and samples of grain and straw were taken as close as possible to recommended PHI of 35 days. However, if grain had not yet reached final harvest maturity 35 days after last application, a second set of grain and straw samples was taken at maturity (BBCH 89).
Among the 4 trials conducted in southern Europe, grain reached its maturity stage at PHI 35 only in Spain and Greece. In France and Italy, grain and straw were sampled at PHI 44 days.

Residues of prothioconazole-desthio were determined according to method 00647 (cf. original Annex II, Section 2, Point 4.2) by HPLC-MS/MS in the multiple-reaction-monitoring mode (MRM) using an electrospray interface (ESI) after extraction, filtration and dilution. The LOQ with this method was 0.05 mg/kg for forage/rest of plant and straw and 0.01 mg/kg for grain/ear.

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-56.

Table 6.3.2-56: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	ear	207	6.9	RA-2107/03 M-060760-01-1
		grain	172	5.7	
		rest of plant	207	6.9	
		straw	172	5.7	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of sample s ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-57. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-57: RA-2107/03: Concurrent recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Barley grain, Ear	0.01	93 ; 100 ; 97	97	3.6	0.01
		0.10	101 ; 93 ; 95 ; 92 ; 96 ; 98	96	3.2	
		Overall (n = 9)		**96**	**3.1**	
Prothio-conazole-desthio	Green material	0.05	101 ; 99 ; 97	99	2.0	0.05
		0.50	101 ; 98 ; 103 ; 104 ; 95	100	3.7	
		Overall (n = 8)		**100**	**3.1**	
Prothio-conazole-desthio	Straw	0.05	101 ; 101 ; 103	102	1.1	0.05
		0.50	99 ; 98 ; 97 ; 95	97	1.8	
		Overall (n = 7)		**99**	**2.8**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

- Residue results:
The residue results are summarised in Table 6.3.2-58.

Table 6.3.2-58: Residues of prothioconazole-desthio in/on barley applied with prothioconazole & trifloxystrobin SC 325

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2107/03 R 2003 0979/1 0979-03 GLP: yes 2003	Barley Scarlet	Greece GR-[redacted] Europe, South	325 SC	2	0.1750	0.05828	61	ear	0	1.3
								grain	35	<0.01
								straw	35	1.1
								rest of plant	0	2.8
RA-2107/03 R 2003 0940/6 0940-03 GLP: yes 2003	Barley Esterel	France F-[redacted] Europe, South	325 SC	2	0.1750	0.05828	61	ear	0 35	3.6 0.02
								grain	44	0.01
								straw	44	0.10
								rest of plant	0 35	0.94 0.09
RA-2107/03 R 2003 0978/3 0978-03 GLP: yes 2003	Barley Hispania	Spain E-[redacted] Europe, South	325 SC	2	0.1750	0.05828	69	ear	0	2.3
								grain	34	0.03
								straw	34 42	2.5 1.3
								rest of plant	0	4.3
RA-2107/03 R 2003 0977/5 0977-03 GLP: yes 2003	Barley Alisier	Italy I-[redacted] Europe, South	325 SC	2	0.1750	0.05828	61	ear	0 35	3.0 0.02
								grain	44	<0.01
								straw	44	0.06
								rest of plant	0 35	0.96 0.08

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio

In the 4 trials conducted in 2003 (report RA-2107/03), residues of prothioconazole-desthio in barley grain at harvest ranged between < 0.01 mg/kg and 0.03 mg/kg. The residues in straw at harvest ranged between 0.06 mg/kg and 2.5 mg/kg. This relatively high value of 2.5 mg/kg can be explained by the fact that the last application was performed at BBCH stage 69 (instead of 61) and with a short PHI.

Conclusion

Four barley residue trials were conducted with prothioconazole & trifloxystrobin SC325 in southern Europe. The product application corresponded to a prothioconazole rate of 2x175 g a.s./ha. Residues of prothioconazole-desthio in barley grain at harvest ranged between < 0.01 mg/kg and 0.03 mg/kg. The residues in straw at harvest ranged between 0.06 mg/kg and 2.5 mg/kg. This relatively high value of 2.5 mg/kg can be explained by the fact that the last application was performed at BBCH stage 69 (instead of 61) and with a short PHI (34 days).

Report:	KCA 6.3.2/22[redacted],;[redacted]; 2006; M-272115-01-1
Title:	Determination of the residues of JAU 6476, Tebuconazole and KWG 4168 in/on winter barley after spraying of JAU 6476 & HWG 1608 & KWG 4168 (450 EC) in the field in Southern France and Spain
Report No.:	RA-2572/05
Document No.:	M-272115-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2005, a set of 2 residue trials was conducted in southern Europe. The trials were located in Spain and southern France.

In each trial, barley was treated twice at a product rate of 1.25 L/ha JAU 6476 (prothioconazole) & HWG 1608 (tebuconazole) & KWG 4168 (spiroxamine) EC450 corresponding to 0.125 kg a.s./ha of prothioconazole. The water rate was 300 L/ha. The spray interval ranged from 8-9 days. The time of application was:

1. when the flag leaf sheath of the plants was opening (BBCH 47), and
2. at the beginning of flowering (BBCH 61).

A deviation to the described spray program occurred in trial R2005 0469/1 in Spain, where both treatments were carried out at a different stage as originally intended (BBCH 51 instead of BBCH 47, and BBCH 59 instead of BBCH 61).

Samples were taken at the following intervals:
In one trial,
- prior to and immediately after the final application as well at a PHI of 28 and 35 days for green material
- for grain and straw: at harvest (BBCH 89)

In the other trial, green material was sampled immediately after the final application and grain and straw at a 35-d PHI and at harvest (BBCH 89).

Residues of prothioconazole-desthio were determined according to method 00598/M001 with a limit of quantitation of 0.01 mg/kg for grain and 0.05 mg/kg for cereal green material and straw (cf. original Annex II, Section 2, Point 4.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-59.

Table 6.3.2-59: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	grain	146	4.9	RA-2572/05 M-272115-01-1
		Green material	175	5.8	
		straw	148	4.9	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-60. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-60: RA-2572/05: Concurrent recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Barley grain / Ear	0.01	90 ; 81 ; 94	88	7.5	0.01
		0.1	90 ; 91	91	-	
		Overall (n = 5)		**89**	**5.5**	
Prothio-conazole-desthio	Green material	0.05	79 ; 86 ; 93	86	8.1	0.05
		0.50	75 ; 90 ; 104 ; 99	92	13.8	
		5.0	93 ; 84 ; 100	92	8.7	
		Overall (n = 10)		**90**	**10.4**	
Prothio-conazole-desthio	Straw	0.05	90 ; 88 ; 91	90	1.7	0.05
		0.50	88 ; 91	90	-	
		Overall (n = 9)		**90**	**1.7**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
These recoveries were assigned to the studies RA-2571/05 and RA-2572/05.

- Residue results:

The residue results are summarised in Table 6.3.2-61.

Table 6.3.2-61: Residues of prothioconazole-desthio in/on winter barley applied with JAU 6476 (prothioconazole) & HWG 1608 (tebuconazole) & KWG 4168 (spiroxamine) EC 450

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2572/05 R 2005 0468/3 0468-05 GLP: yes 2005	Barley, winter BARAKA	France F-[redacted] (Provence-Cote D'azur) Europe, South	450 EC	2	0.1250	0.04170	61	green material	0 (1) 0 28 35	0.00 2.1 0.06 0.05
								grain	61	<0.01
								straw	61	0.19
RA-2572/05 R 2005 0469/1 0469-05 GLP: yes 2005	Barley, winter Graphic	Spain E-[redacted] (Cataluña) Europe, South	450 EC	2	0.1250	0.04170	59	green material	0	2.4
								grain	35 49	<0.01 <0.01
								straw	35 49	1.6 1.2

(1) Prior to last treatment
* determined as prothioconazole-desthio and calculated as prothioconazole-desthio

In the 2 trials conducted in 2005 (report RA-2572/05), residues of prothioconazole-desthio in barley grain at harvest were < 0.01 mg/kg. The residues in straw at harvest ranged between 0.19 mg/kg and 1.2 mg/kg.

Conclusion

Two barley residue trials were conducted with JAU 6476 & KWG 4168 & HWG 1608 EC 450 in southern Europe. The product application corresponded to a prothioconazole rate of 2x125 g a.s./ha. Residues of prothioconazole-desthio in barley grain at harvest were < 0.01 mg/kg. The residues in straw at harvest ranged between 0.19 mg/kg and 1.2 mg/kg.

Report: KCA 6.3.2/23[redacted]; [redacted]; 2007; M-294526-01-1
Title: Determination of the residues of BYF 00587 and JAU 6476 in/on Spring barley and Winter barley after spraying of BYF 00587 & JAU 6476 (225 EC) in the field in Southern France, Italy, Spain and Portugal
Report No.: RA-2329/06
Document No.: M-294526-01-1
Guideline(s): EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8
Residues in or on Treated Products, Food and Feed
Guideline deviation(s): not specified
GLP/GEP: **yes**

Materials and Methods

In the vegetation period of 2007, a set of 4 residue trials was conducted in southern Europe. The trials were located in Spain, Italy, southern France and Portugal.

The trials were conducted on both spring barley (1 trial) and winter barley (3 trials). In each trial, barley was treated twice at a product rate of 1.25 L/ha BYF 00587 (bixafen) & JAU 6476 (prothioconazole) EC 225 corresponding to 0.150 kg a.s./ha of prothioconazole. All applications were made at the specified rates with the exception of the 1st application that was overdosed by 7% in 2 trials.
Two trials were designed as decline series and the remaining 2 trials yielded harvest values only. The water rate was 300-321 L/ha. Depending on the study, the spray interval ranged from 9-27 days. The timing of application was:

1. when the flag leaf is just visible (still rolled) (BBCH 39), and
2. at the beginning of flowering (BBCH 61).

Samples of green material were taken just prior to and immediately after the final. Two trials were designed as decline series, *i.e.* samples were collected further 7, 14, and 28 days after the final treatment. Grain and straw were sampled 34-35 days after the final treatment in all trials and, if BBCH 89 has not yet been reached 35 days post treatment, additionally at one later sampling date (43-48 days after final treatment).

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-62.

Table 6.3.2-62: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	grain	217	7.2	RA-2329/06 M-294526-01-1
		green material	133	4.4	
		straw	222	7.4	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance:

Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-63. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-63: RA-2329/06 : Concurrent recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothioconazole -desthio	Cereal grain	0.01	103 ; 115 ; 110	109	5.5	0.01
		0.1	114 ; 105 ; 96	104	7.3	
		Overall (n = 6)		**107**	**6.3**	
Prothioconazole -desthio	Cereal green material	0.01	106 ; 98 ; 91	98	7.6	0.01
		0.10	100 ; 95 ; 100 ; 113 ; 92 ; 109 ; 105 ; 106 ; 100	102	6.5	
		5.0	86 ; 86	86	-	
		6.0	79	79	-	
		Overall (n = 15)		**98**	**9.8**	
Prothioconazole -desthio	Cereal straw	0.01	105	105	-	0.01
		0.1	97 ; 101 ; 111	103	7.0	
		9.0	90	90	-	
		Overall (n = 5)		**101**	**7.9**	

Final determination as: Prothioconazole-desthio, Residues calculated as: Prothioconazole-desthio
RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
These recoveries were performed with barley and wheat commodities during the conduct of the studies RA-2326/06, RA-2327/06, RA-2328/06 and RA-2329/06. Cereal summarises barley and wheat.

- Residue results:

The residue results are summarised in Table 6.3.2-64.

Table 6.3.2-64: Residues of prothioconazole-desthio in/on spring and winter barley applied with BYF 00587 (bixafen) & JAU 6476 (prothioconazole) EC225

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2329/06 R 2006 0464/5 0464-06 GLP: yes 2006	Barley, winter Scarlet	Portugal P-[redacted] Europe, South	225 EC	2	0.1500 - 0.1605	0.04995	61	green material	0* 0 7 14 28	0.02 0.88 0.26 0.12 0.15
								straw	35 43	0.32 0.33
								grain	35 43	0.01 0.01
RA-2329/06 R 2006 0461/0 0461-06 GLP: yes 2006	Barley, spring Scarlett	France F-[redacted] Europe, South	225 EC	2	0.1500	0.04995	61	green material	0* 0	0.38 1.3
								straw	34	1.0
								grain	34	0.01
RA-2329/06 R 2006 0463/7 0463-06 GLP: yes 2006	Barley, winter GRAPH IC	Spain E-[redacted] Europe, South	225 EC	2	0.1500 - 0.1605	0.04995	61	green material	0* 0	0.38 1.3
								straw	35 48	0.67 0.79
								grain	35 48	<0.01 <0.01
RA-2329/06 R 2006 0462/9 0462-06 GLP: yes 2006	Barley, winter Margior y	Italy I-[redacted] Europe, South	225 EC	2	0.1500	0.04995	61	green material	0* 0 7 14 28	0.53 1.7 1.2 0.57 0.36
								straw	35 43	0.64 0.56
								grain	35 43	0.01 0.02

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) before last application
GS = growth stage (BBCH code)
FL = formulation

In the 4 trials conducted in 2006 (report RA-2329/06), residues of prothioconazole-desthio in barley grain at harvest (34-48 days after the last treatment) ranged between < 0.01 mg/kg and 0.02 mg/kg. The residues in straw at harvest ranged between 0.33 mg/kg and 1.0 mg/kg.

Conclusion

Four barley residue trials were conducted with bixafen & prothioconazole EC 225 in southern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha. Residues of prothioconazole-desthio in barley grain at harvest (34-48 days after the last treatment) ranged between <0.01 mg/kg and 0.02 mg/kg. The residues in straw at harvest ranged between 0.33 mg/kg and 1.0 mg/kg.

Report:	KCA 6.3.2/24[redacted]B;[redacted]; 2008; M-298412-01-1
Title:	Determination of the residues of BYF 00587, JAU 6476 and KWG 4168 in/on winter barley and spring barley after spraying of BYF 00587 & JAU 6476 & KWG 4168 (400 EC) in the field in Southern France, Spain, Italy and Portugal
Report No.:	RA-2043/07
Document No.:	M-298412-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2007, a set of 4 residue trials on spring and winter barley was conducted in southern Europe. The trials were located in southern France, Spain, Italy and Portugal.

In each trial, barley was treated twice at a product rate of 1.5 L/ha BYF 00587 (bixafen) & JAU 6476 (prothioconazole) & KWG 4168 (spiroxamine) EC 400 corresponding to 0.150 kg a.s./ha of prothioconazole. The water rate was 300 L/ha. The spray interval ranged from 8 - 20 days. The time of application was:

1. when the flag leaf is just visible (still rolled) until is fully unrolled (ligule just visible) (BBCH 37 - 39), and
2. at the beginning of flowering (BBCH 61).

Samples were taken at the following intervals:
- prior to and immediately after the final application,
- at a pre-harvest interval of 35-36 days after the final treatment,
- at harvest (BBCH 89), 35-58 days after the final application.

Depending on the growth stage at sampling, cereal plants were divided into "ear" and "rest of plant", or "grain" and "straw".

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-65.

Table 6.3.2-65: Maximum storage periods of field samples from supplementary residue trials

Crop	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	ear	176	5.9	RA-2043/07 M-298412-04-1
		grain	176	5.9	
		green material	212	7.1	
		rest of plant	176	5.9	
		straw	176	5.9	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-66. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-66: RA-2043/07: Concurrent recoveries for the determination of prothioconazole-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain #	0.01	97; 97; 96; 100; 93; 94; 99	97	2.6	0.01
		0.1	95; 93; 95; 91; 93; 100	95	3.3	
		1.0	92; 95; 95; 89; 93; 99	94	3.6	
		5.0	89	--	--	
		10.0	86	--	--	
		Overall Recovery (n = 21)		**94**	**3.9**	
Prothio-conazole-desthio	Cereal green material*	0.01	98; 94; 90; 97	95	3.8	0.01
		0.1	97; 90; 95; 88	93	4.5	
		1.0	87; 93; 95; 91; 89	91	3.9	
		5.0	89; 85; 82	85	4.1	
		10.0	83; 86; 85	85	1.8	
		Overall Recovery (n = 19)		**90**	**5.5**	
Prothio-conazole-desthio	Cereal straw	0.01	92; 88; 104; 93; 104	96	7.7	0.01
		0.1	95; 88; 94; 103	95	6.5	
		1.0	88; 93; 91; 93	91	2.6	
		5.0	88; 80; 87	85	5.1	
		10.0	84	--	--	
		Overall Recovery (n = 17)		**92**	**7.3**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
These recoveries were performed with wheat and barley commodities during the conduct of the studies RA-2037/07, RA-2038/07, RA-2039/07, RA-2040/07, RA-2041/07, RA-2042/07, RA-2043/07, RA-2045/07, RA-2046/07, RA-2049/07 and RA-2050/07. Cereal summarises barley and wheat. Recoveries for wheat are also valid for barley. * = Recoveries for rest of plant are covered by recoveries for green material.
= Recoveries for ear are covered by recoveries for grain.

- Residue results:

The residue results are summarised in Table 6.3.2-67.

Table 6.3.2-67: Residues of prothioconazole-desthio in/on spring barley and winter barley applied with bixafen & prothioconazole & spiroxamine EC 400

Study Trial No. Plot No. GLP Year	Crop Variety	Country	Application					Residues*		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	JAU 6476-desthio (mg/kg)
RA-2043/07 R 2007 0532/8 0532-07 GLP: yes 2007	Barley, winter Scarlet	Portugal P-[redacted] Europe, South	400 EC	2	0.1500	0.05000	61	green material	0 (1) 0	0.12 0.95
								straw	35	0.67
								grain	35	<0.01
RA-2043/07 R 2007 0530/1 0530-07 GLP: yes 2007	Barley, spring Tunica	Italy I-[redacted] Europe, South	400 EC	2	0.1500	0.05000	61	green material	0 (1) 0	0.38 2.0
								straw	39 55	0.14 0.04
								grain	39 55	<0.01 <0.01
RA-2043/07 R 2007 0452/6 0452-07 GLP: yes 2007	Barley, winter County R2	Spain E-[redacted] Europe, South	400 EC	2	0.1500	0.05000	61	green material	0 (2) 0	0.15 1.8
								straw	36 42	0.65 0.69
								grain	36 42	0.01 0.01
RA-2043/07 R 2007 0451/8 0451-07 GLP: yes 2007	Barley, winter Esterel	France F-[redacted] Europe, South	400 EC	2	0.1500	0.05000	61	green material	0 (1) 0	0.34 0.97
								straw	58	0.09
								grain	58	<0.01
								ear	35	0.01
								rest of plant	35	0.33

* determined as prothioconazole-desthio and calculated as prothioconazole-desthio
(1) before last application
GS = growth stage (BBCH code) at last application
FL = formulation

In the 4 trials conducted in 2007 (report RA-2043/07), residues of prothioconazole-desthio in barley grain at harvest ranged between < 0.01 mg/kg and 0.01 mg/kg. The residues in straw at harvest ranged between 0.04 mg/kg and 0.69 mg/kg.

Conclusion

Four barley residue trials were conducted with Bixafen & Prothioconazole & Spiroxamine EC 400 in southern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha. Residues of prothioconazole-desthio in barley grain at harvest ranged between < 0.01 mg/kg and 0.01 mg/kg. The residues in straw at harvest ranged between 0.04 mg/kg and 0.69 mg/kg.

Report:	KCA 6.3.2/25[redacted],;[redacted];[redacted]; 2011; M-414709-01-1
Title:	Determination of the residues of BYF 00587, HEC 5725 and prothioconazole in/on barley after spray application of bixafen & fluoxastrobin & prothioconazole EC 190 in the field in France (south) and Italy
Report No.:	10-2206
Document No.:	M-414709-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2010, a set of 2 residue trials on barley was conducted in southern Europe. The trials were located in Italy and southern France.

In each trial, barley was treated twice at a product rate of 1.5 L/ha bixafen & fluoxastrobin & prothioconazole EC 190 corresponding to 0.150 kg a.s./ha of prothioconazole. The water rate was 300-400 L/ha. The spray interval ranged from 14-15 days. The time of application was:

1. at BBCH stage 38-51
2. at the beginning of flowering (BBCH 61).

Samples were taken at the following intervals:
- prior to and immediately after the final application,
- at a pre-harvest interval of 34 days after the final treatment,
- at harvest (BBCH 89), 47-52 days after the last application.

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio were determined according to method 00979/M001 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-68.

Table 6.3.2-68: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	grain	259	8.6	10-2206 M-414709-01-1
		green material	302	10.1	
		straw	259	8.6	
	prothioconazole-alpha-hydroxy-desthio prothioconazole-3-hydroxy-desthio prothioconazole-4-hydroxy-desthio prothioconazole-5-hydroxy-desthio prothioconazole-6-hydroxy-desthio	grain	323	10.8	
		green material	351	11.7	
		straw	324	10.8	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).
Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio above 30% of the LOQ was found in the control samples.

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110% except for prothioconazole-desthio in green material at the LOQ level (112%) and for prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio and prothioconazole-6-hydroxy-desthio in straw at 0.50 mg/kg (68%, 69% and 68% respectively). The single and overall mean recoveries are shown in Table 6.3.2-69 to Table 6.3.2-74. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-69: 10-2206: Concurrent recoveries for the determination of prothioconazole-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	Cereal grain	0.01	108	-	-	0.01
		2.5	86 ; 99	93	-	
		Overall Recovery (n = 3)		**98**	**11.3**	
	Cereal green material	0.01	108; 108; 116; 111; 117	112[a]	3.8	0.01
		0.10	97	-	-	
		2.0	84; 104	94	-	
		2.5	92; 95; 94; 89	93	2.9	
		Overall Recovery (n = 12)		**101**	**10.7**	
	Cereal straw	2.5	73	-	-	0.01
		Overall Recovery (n = 1)		**73**	**-**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-desthio, determined as JAU 6476-desthio and calculated as JAU 6476-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
[a]This value was accepted due to a RSD below 20% and an overall mean in the range of 70-110%.

Table 6.3.2-70: 10-2206: Concurrent recoveries for the determination of prothioconazole-alpha-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothiocon azole-alpha-hydroxy-desthio	Cereal grain	0.01	89; 90; 118; 120; 89; 89; 94; 100; 95; 96	98	11.9	0.01
		0.50	89; 98; 89; 95	93	4.9	
		Overall Recovery (n = 14)		**97**	**10.6**	
	Cereal green material	0.01	80; 99; 104; 99; 104; 93; 98; 100; 102; 85; 85; 91; 92	95	8.2	0.01
		0.50	101; 95; 100; 101; 83; 85	94	8.7	
		Overall Recovery (n = 19)		**95**	**8.1**	
	Cereal straw	0.01	79; 79; 88; 97; 81; 84	84	8.7	0.01
		0.50	72; 71; 88; 78; 92	80	11.8	
		Overall Recovery (n = 11)		**82**	**9.9**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with JAU 6476-alpha-hydroxy-desthio, determined as JAU 6476-alpha-hydroxy-desthio and calculated as JAU 6476-desthio.
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
** : mean of double injection

Table 6.3.2-71: 10-2206: Concurrent recoveries for the determination of prothioconazole-3-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-3-hydroxy-desthio	Cereal grain	0.01	73; 82; 109; 113; 85; 88; 89; 90; 88; 94	91	13.1	0.01
		0.50	83; 93; 78; 81	84	7.8	
		Overall Recovery (n =14)		**89**	**12.3**	
	Cereal green material	0.01	70; 90; 101; 91; 103; 91; 94; 94; 94; 70; 71; 75; 77	86	13.8	0.01
		0.50	85; 82; 98; 104; 70; 74	86	15.5	
		Overall Recovery (n =19)		**86**	**14.0**	
	Cereal straw	0.01	80; 80; 83; 86; 98; 100	89	12.4	0.01
		0.50	62; 63; 81; 67; 76	70	12.0	
		Overall Recovery (n =11)		**80**	**17.2**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-3-hydroxy-desthio, determined as prothioconazole-3-hydroxy-desthio and calculated as prothioconazole-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
** : mean of double injection

Table 6.3.2-72: 10-2206: Concurrent recoveries for the determination of prothioconazole-4-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-4-hydroxy-desthio	Cereal grain	0.01	65; 72; 99; 105; 60; 67; 68; 70; 71; 73	75	19.7	0.01
		0.50	87; 95; 72; 76	83	12.7	
		Overall Recovery (n = 14)		**77**	**17.8**	
	Cereal green material	0.01	68; 80; 99; 79; 83; 65; 66; 73; 74; 63; 64; 74; 76	74	13.3	0.01
		0.50	88; 84; 95; 102; 71 ;75	86	13.7	
		Overall Recovery (n = 19)		**78**	**14.9**	
	Cereal straw	0.01	67; 64; 67; 77; 77; 81	72	9.7	0.01
		0.50	67; 62; 79; 62; 70	68***	10.3	
		Overall Recovery (n = 11)		**70**	**10.0**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-4-hydroxy-desthio, determined as prothioconazole-4-hydroxy-desthio and calculated as prothioconazole-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
** : mean of double injection
*** This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%

Table 6.3.2-73: 10-2206: Concurrent recoveries for the determination of prothioconazole-5-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-5-hydroxy-desthio	Cereal grain	0.01	74; 77; 108; 108; 75; 83; 86; 88; 78; 86	86	14.4	0.01
		0.50	80;86;83;83	83	3.0	
		Overall Recovery (n = 14)		**85**	**12.3**	
	Cereal green material	0.01	73; 93; 99; 95; 95; 76; 77; 78; 82; 72; 73; 78; 79	82	11.7	0.01
		0.50	90; 85; 92; 98; 70; 73	85	13.1	
		Overall Recovery (n = 19)		**83**	**11.9**	
	Cereal straw	0.01	80; 71; 79; 84; 78; 88	80	7.2	0.01
		0.50	62; 64; 80; 66; 75	69***	11.1	
		Overall Recovery (n = 11)		**75**	**11.2**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-5-hydroxy-desthio, determined as prothioconazole-5-hydroxy-desthio and calculated as prothioconazole-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
** : mean of double injection
*** : This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%

Table 6.3.2-74: 10-2206: Concurrent recoveries for the determination of prothioconazole-6-hydroxy-desthio in cereal

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
			Single Values**	Mean		
prothioconazole-6-hydroxy-desthio	Cereal grain	0.01	69; 75; 99; 101; 94; 96; 97; 100; 93; 97	92	11.9	0.01
		0.50	79;84;79;82	81	3.0	
		Overall Recovery (n = 14)		**89**	**11.9**	
	Cereal green material	0.01	73; 102; 103; 92; 99; 87; 93; 95; 99; 69; 75; 77; 77	88	13.8	0.01
		0.50	79; 75; 89; 91; 70; 79	81	10.1	
		Overall Recovery (n = 19)		**85**	**13.2**	
	Cereal straw	0.01	82; 74; 75; 78; 75; 82	78	4.7	0.01
		0.50	68; 68; 80; 60; 62	68***	11.5	
		Overall Recovery (n = 11)		**73**	**10.5**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-6-hydroxy-desthio, determined as prothioconazole-6-hydroxy-desthio and calculated as prothioconazole-desthio
*These recoveries were performed with sample material from studies 10-2204, 10-2205, 10-2206 and 10-2207.
Cereal summarises barley and wheat.
** : mean of double injection
*** : This value was accepted due to a RSD below 20% and an overall mean value in the range of 70-110%

- Residue results:

The residue results are summarised in Table 6.3.2-75.

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Table 6.3.2-75: Residues of prothioconazole-desthio in/on barley applied with bixafen & fluoxastrobin & prothioconazole EC 190

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
10-2206 10-2206-01 GLP: yes 2010	Barley Ketos	France [redacted] Europe, South	190 EC	2	0.15	0.050	61	green material	0(1) 0	0.45 1.7	<0.01 <0.01	0.06 0.05	0.10 0.09	0.05 0.05	<0.01 <0.01	0.68 1.9
								grain	34 47	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06
								straw	34 47	0.25 0.33	<0.01 <0.01	0.16 <0.01	0.22 0.02	0.10 0.01	<0.01 <0.01	0.75 0.39
10-2206 10-2206-02 GLP: yes 2010	Barley Ketos	Italy [redacted] Europe, South	190 EC	2	0.15	0.038	61	green material	0 0	0.15 1.5	<0.01 <0.01	0.03 0.04	0.05 0.06	0.03 0.03	<0.01 <0.01	0.28 1.7
								grain	52	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	52	<0.01	<0.01	0.12	0.21	0.10	<0.01	0.46

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg.
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

In the 2 trials conducted in 2010 (report 10-2206), residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in barley grain at harvest were < 0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg.
In straw, the residues at harvest ranged as follows :

- between <0.01 mg/kg and 0.33 mg/kg for prothioconazole-desthio
- < 0.01 mg/kg mg/kg for prothioconazole-alpha-hydroxy
- between <0.01 mg/kg and 0.12 mg/kg for prothioconazole-3-hydroxy-desthio
- between 0.02 mg/kg and 0.21 mg/kg for prothioconazole-4-hydroxy-desthio
- between 0.01 mg/kg and 0.10 mg/kg for prothioconazole-5-hydroxy-desthio
- < 0.01 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio
- between 0.39 mg/kg and 0.46 mg/kg for the total residue calc.

Conclusion

Two barley residue trials were conducted with Bixafen & Fluoxastrobin & Prothioconazole EC 190 in southern Europe. The product application corresponded to a prothioconazole rate of 2x150 g a.s./ha.
In the 2 trials conducted in 2010 (report 10-2206), residues of prothioconazole-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in barley grain at harvest were < 0.01 mg/kg. In grain at harvest, the total residue was always <0.06 mg/kg.
In straw, the residues at harvest ranged as follows :

- between <0.01 mg/kg and 0.33 mg/kg for prothioconazole-desthio
- < 0.01 mg/kg mg/kg for prothioconazole-alpha-hydroxy
- between <0.01 mg/kg and 0.12 mg/kg for prothioconazole-3-hydroxy-desthio
- between 0.02 mg/kg and 0.21 mg/kg for prothioconazole-4-hydroxy-desthio
- between 0.01 mg/kg and 0.10 mg/kg for prothioconazole-5-hydroxy-desthio
- < 0.01 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio
- between 0.39 mg/kg and 0.46 mg/kg for the total residue calc.

Report:	KCA 6.3.2/26 [redacted]; 2013; M-434980-04-1
Title:	Amendment no. 3 to report no. 11-2111 - Determination of the residues of fluoxastrobin and prothioconazole in/on winter barley after spray application of fluoxastrobin & prothioconazole EC 150 in southern France, Italy and Spain
Report No.:	11-2111
Document No.:	M-434980-04-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22) US EPA OCSPP Guideline No. 860.1500
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

In the vegetation period of 2011, a set of 5 residue trials on winter barley was conducted in southern Europe. The trials were located in southern France, Spain and Italy.

In each trial, barley was treated twice at a product rate of 1.75 L/ha fluoxastrobin & prothioconazole EC 150 corresponding to 0.175 kg a.s./ha of prothioconazole. The water rate was 300-400 L/ha. The spray interval ranged from 13-20 days. The time of application was:
1. at BBCH 37-43 and,
2. at the beginning of flowering (BBCH 61) except in 2 trials where the application was done slightly later (BBCH 65 or 69).

Samples were taken at the following intervals:
- prior to the last application for 2 trials and immediately after the final application,
- at days 7, 14, and 28 days after final application for green material for 2 trials and at days 10-13 or 22 days for the other trials,
- at harvest (BBCH 89).

Residues of prothioconazole-desthio were determined according to method 01013 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).
Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio were determined according to method 00979/M001 with a limit of quantitation of 0.01 mg/kg for grain, green material and straw (cf. KCA 4.1.2).

Findings

- Storage stability:

The maximum storage periods for prothioconazole-desthio in barley samples from the supplementary residue field trials are presented in Table 6.3.2-76.

Table 6.3.2-76: Maximum storage periods of field samples from supplementary residue trials

Crops	Substance	Sample Material	Storage period (days)	Storage period (months)	Study No.
barley	prothioconazole-desthio	grain	139	4.6	11-2111 M-434980-04-1
		green material	195	6.5	
		straw	139	4.6	
	prothioconazole-alpha-hydroxy-desthio prothioconazole-3-hydroxy-desthio prothioconazole-4-hydroxy-desthio prothioconazole-5-hydroxy-desthio prothioconazole-6-hydroxy-desthio	grain	131	4.4	
		green material	186	6.2	
		straw	131	4.4	

These storage periods are covered by the storage stability studies, *i.e.* analytes were shown to be stable for a period of at least 24 months (cf. KCA 6.1).

Sample extracts were measured within 24 hours, or if not, acceptable recoveries measured concurrently with each set of samples ensured integrity of the sample extracts during the period of time between extraction and analysis.

No residue of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio above 30% of the LOQ was found in the control samples. Residues were found in control samples from 2 trials for JAU-6476-desthio (green material: 0.013-0.051 mg/kg; straw: 0.014-0.021 mg/kg).

- Method performance: Recovery rates were determined concurrently with the sample analysis in order to check the accuracy of the residue levels. Recovery means by fortification levels were within the acceptable range of 70-110%. The single and overall mean recoveries are shown in Table 6.3.2-77 to Table 6.3.2-82. All results of the method validation are in accordance with the general requirements for residue analytical methods, therefore the method was validated successfully.

Table 6.3.2-77: 11-2111: Concurrent recoveries for the determination of prothioconazole-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-desthio	barley grain	0.01	113	-	-	0.01
		3.0	99; 104	102	-	
		Overall Recovery (n = 3)		**105**	**6.7**	
	barley green material	0.01	120	-	-	0.01
		0.10	100; 106	103	-	
		3.0	88; 89	89	-	
		Overall Recovery (n = 5)		**101**	**13.1**	
	barley straw	0.01	92; 97; 119	103	14.0	0.01
		1.0	92	-	-	
		3.0	85; 111	98	-	
		Overall Recovery (n = 6)		**99**	**13.1**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-desthio, determined as prothioconazole-desthio and calculated as prothioconazole-desthio

Table 6.3.2-78: 11-2111: Concurrent recoveries for the determination of prothioconazole-alpha-hydroxy-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-alpha-hydroxy-desthio	barley grain	0.01	84	-	-	0.01
		0.30	94	-	-	
		Overall Recovery (n = 2)		**89**	-	
	barley green material	0.01	72; 86; 89	82	11.0	0.01
		0.10	86; 87	87		
		0.50	96; 98	97		
		Overall Recovery (n = 7)		**88**	9.6	
	barley straw	0.01	74	-	-	0.01
		0.30	69	-	-	
		0.80	74; 84	79	-	
		Overall Recovery (n = 4)		**75**	8.4	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-alpha-hydroxy-desthio, determined as prothioconazole-alpha-hydroxy-desthio and calculated as prothioconazole-desthio

Table 6.3.2-79: 11-2111: Concurrent recoveries for the determination of prothioconazole-3-hydroxy-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-3-hydroxy-desthio	barley grain	0.01	75	-	-	0.01
		0.30	88	-	-	
		Overall Recovery (n = 2)		**82**	-	
	barley green material	0.01	81; 84; 93	86	6.6	0.01
		0.10	82; 84	83	-	
		0.50	88; 90	89	-	
		Overall Recovery (n = 7)		**86**	**4.9**	
	barley straw	0.30	64	-	-	0.01
		0.80	70; 78	74	-	
		Overall Recovery (n = 3)		**71**	**9.9**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-3-hydroxy-desthio, determined as prothioconazole-3-hydroxy-desthio and calculated as prothioconazole-desthio

Table 6.3.2-80: 11-2111: Concurrent recoveries for the determination of prothioconazole-4-hydroxy-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-4-hydroxy-desthio	barley grain	0.01	74	-	-	0.01
		0.30	85	-	-	
		Overall Recovery (n = 2)		**80**	-	
	barley green material	0.01	78; 88; 95	87	9.8	0.01
		0.10	79; 79	79	-	
		0.50	89; 89	89	-	
		Overall Recovery (n = 7)		**85**	**7.7**	
	barley straw	0.30	65	-	-	0.01
		0.80	68; 74	71	-	
		Overall Recovery (n = 3)		**70**	**8.9**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-4-hydroxy-desthio, determined as prothioconazole-4-hydroxy-desthio and calculated as prothioconazole-desthio

Table 6.3.2-81: 11-2111: Concurrent recoveries for the determination of prothioconazole-5-hydroxy-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-5-hydroxy-desthio	barley grain	0.01	82	-	-	0.01
		0.30	82	-	-	
		Overall Recovery (n = 2)		**82**	-	
	barley green material	0.01	81; 83; 84	83	1.8	0.01
		0.10	82; 82	82	-	
		0.50	88; 116	102	-	
		Overall Recovery (n = 7)		**88**	**14.3**	
	barley straw	0.30	65	-	-	0.01
		0.80	69;77	73	-	
		Overall Recovery (n = 3)		**70**	**8.7**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-5-hydroxy-desthio, determined as prothioconazole-5-hydroxy-desthio and calculated as prothioconazole-desthio

Table 6.3.2-82: 11-2111: Concurrent recoveries for the determination of prothioconazole-6-hydroxy-desthio in barley

Analyte	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Prothio-conazole-6-hydroxy-desthio	barley grain	0.01	60	-	-	0.01
		0.30	79	-	-	
		Overall Recovery (n = 2)		**70**	-	
	barley green material	0.01	87; 92; 92	90	3.2	0.01
		0.10	83; 84	84	-	
		0.50	81; 83	82	-	
		Overall Recovery (n = 7)		**86**	**5.2**	
	barley straw	0.30	65	-	-	0.01
		0.80	75; 78	77	-	
		Overall Recovery (n = 3)		**73**	**9.4**	

RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification
Fortified with prothioconazole-6-hydroxy-desthio, determined as prothioconazole-6-hydroxy-desthio and calculated as prothioconazole-desthio

- Residue results:

The residue results are summarised in Table 6.3.2-83.

Table 6.3.2-83: Residues of prothioconazole-desthio in/on winter barley applied with fluoxastrobin & prothioconazole EC 150

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
11-2111 11-2111-01 GLP: yes 2011	Barley, winter Ketos	France [redacted] Europe, South	150 EC	2	0.175	0.0583	61	green material	0(1) 0 7 14 28	0.79/0.051** 2.6 1.4 0.97 0.49	<0.01 <0.01 <0.01 <0.01 <0.01	0.057 0.052 0.10 0.14 0.15	0.096 0.086 0.14 0.21 0.18	0.050 0.046 0.088 0.11 0.072	<0.01 <0.01 <0.01 <0.01 0.01	1.0 2.2 1.7 1.5 0.91
								grain	51	0.014	<0.01	<0.01	<0.01	<0.01	<0.01	0.064
								straw	51	1.7/0.021**	0.016	0.45	0.68	0.20	0.042	3.1
11-2111 11-2111-02 GLP: yes 2011	Barley, winter Campanil	France [redacted] Europe, South	150 EC	2	0.175	0.0583	61	green material	0 22	1.9/0.013** 0.36	<0.01 <0.01	0.051 0.14	0.079 0.19	0.038 0.074	<0.01 <0.01	2.1 0.78
								grain	55	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.06
								straw	55	0.54/0.014**	0.015	0.16	0.32	0.11	<0.01	1.2
11-2111 11-2111-03 GLP: yes 2011	Barley, winter Lutece	Italy [redacted] Europe, South	150 EC	2	0.175	0.0438	65	green material	0(1) 0 7 14 28	0.82 1.8 1.6 0.74 0.51	<0.01 <0.01 <0.01 <0.01 <0.01	0.044 0.050 0.085 0.12 0.16	0.060 0.063 0.11 0.15 0.19	0.047 0.056 0.077 0.10 0.12	<0.01 <0.01 <0.01 0.011 0.011	0.99 2.0 1.9 1.1 1.0
								grain	63	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	63	0.34	<0.01	0.15	0.21	0.15	<0.01	0.87
11-2111 11-2111-04 GLP: yes 2011	Barley, winter Ketos	Italy [redacted] Europe, South	150 EC	2	0.175	0.0583	61	green material	0 13	3.0 1.1	<0.01 <0.01	0.096 0.23	0.13 0.23	0.072 0.13	<0.01 <0.01	3.3 1.7
								grain	43	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.06
								straw	43	0.93	0.025	0.33	0.47	0.19	0.018	2.0

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)								
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	PTZ-desthio	PTZ-alpha-hydroxy-desthio	PTZ-3-hydroxy-desthio	PTZ-4-hydroxy-desthio	PTZ-5-hydroxy-desthio	PTZ-6-hydroxy-desthio	Total residue calc.*
11-2111 11-2111-05 GLP: yes 2011	Barley, winter Graphic	Spain [redacted] Europe, South	150 EC	2	0.175	0.0583	69	green material	0 10	2.4 1.1	0.015 0.022	0.052 0.10	0.060 0.11	0.040 0.069	<0.01 <0.01	2.6 1.4
								grain	49	<0.01	<0.01	<0.01	<0.01	<0.01	<0.06
								straw	49	0.79	0.014	0.18	0.17	0.079	<0.01	1.2

(1): before last treatment * for the sum, values <0.01 mg/kg were considered to be equal to 0.01 mg/kg, unless all the values were <0.01 mg/kg ** residue in control
Residues for PTZ-desthio were determined as PTZ-desthio and calculated as PTZ-desthio
Residues for PTZ-alpha-hydroxy-desthio were determined as PTZ-alpha-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-3-hydroxy-desthio were determined as PTZ-3-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-4-hydroxy-desthio were determined as PTZ-4-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-5-hydroxy-desthio were determined as PTZ-5-hydroxy-desthio and calculated as PTZ-desthio
Residues for PTZ-6-hydroxy-desthio were determined as PTZ-6-hydroxy-desthio and calculated as PTZ-desthio

In the 5 trials conducted in 2011 (report 11-2111), residues of prothioconazole-desthio in grain ranged between <0.01 mg/kg and 0.014. Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in barley grain at harvest were always < 0.01 mg/kg. Residues in grain for the total residues of prothioconazole ranged between <0.06 mg/kg and 0.064 mg/kg.

In straw, the residues at harvest ranged as follows :

- between 0.34 mg/kg and 1.7 mg/kg for prothioconazole-desthio
- between < 0.01 mg/kg and 0.025 mg/kg for prothioconazole-alpha-hydroxy
- between 0.15 mg/kg and 0.45 mg/kg for prothioconazole-3-hydroxy-desthio
- between 0.17 mg/kg and 0.68 mg/kg for prothioconazole-4-hydroxy-desthio
- between 0.079 mg/kg and 0.20 mg/kg for prothioconazole-5-hydroxy-desthio
- between < 0.01 mg/kg and 0.042 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio
- between 0.87 mg/kg and 3.1 mg/kg for total residue calc.

Conclusion

Five barley residue trials were conducted with Fluoxastrobin & Prothioconazole EC 150 in southern Europe. The product application corresponded to a prothioconazole rate of 2x175 g a.s./ha. Residues of prothioconazole-desthio in grain ranged between <0.01 mg/kg and 0.014. Residues of prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio, prothioconazole-6-hydroxy-desthio and prothioconazole-alpha-hydroxy-desthio in barley grain at harvest were always < 0.01 mg/kg. Residues in grain for the total residues of prothioconazole ranged between <0.06 mg/kg and 0.064 mg/kg.

In straw, the residues at harvest ranged as follows :

- between 0.34 mg/kg and 1.7 mg/kg for prothioconazole-desthio
- between < 0.01 mg/kg and 0.025 mg/kg for prothioconazole-alpha-hydroxy
- between 0.15 mg/kg and 0.45 mg/kg for prothioconazole-3-hydroxy-desthio
- between 0.17 mg/kg and 0.68 mg/kg for prothioconazole-4-hydroxy-desthio
- between 0.079 mg/kg and 0.20 mg/kg for prothioconazole-5-hydroxy-desthio
- between < 0.01 mg/kg and 0.042 mg/kg mg/kg for prothioconazole-6-hydroxy-desthio
- between 0.87 mg/kg and 3.1 mg/kg for total residue calc.

Overall conclusion of the barley trials

A total of 67 barley trials were performed in Europe, supporting the representative GAP . An overall summary of the obtained residues in feed and food items is presented in the tables below.

When two sets of values were obtained from one given trial (at a 35-d PHI and at a later date), the values obtained at maturity (BBCH89 or later) was taken.

Table 6.3.2-84 summarises the values for prothioconazole-desthio and Table 6.3.2-85 summarises the values for the calculated "total residue" for prothioconazole, i.e. the sum of prothioconazole-desthio and its hydroxylated metabolites when they were all determined (*i.e.* for 6 trials in northern Europe and 7 trials in southern Europe). Finally, Table 6.3.2-86 summarises the values for the hydroxylated

metabolites of prothioconazole-desthio (when they were measured, *i.e.* for 6 trials in northern Europe and 7 trials in southern Europe).

Table 6.3.2-84: Summary of residue data from barley trials with prothioconazole: prothioconazole-desthio

Com-modity	Region	No. of trials	Values (mg/kg)		
			Residue level of prothioconazole-desthio	Maximum (HR)	STMR
Barley, grain	N-EU	36	32x <0.01; 2 x 0.01; 0.011; 0.02	0.02	<0.01
	S-EU	31	19x<0.01; 8x0.01; 0.014; 2x0.02; 0.03	0.03	<0.01
Barley, straw	N-EU	36	4x<0.05; 3x0.02; 0.03; 0.036; 0.04; 0.044; 4x0.05; 2x0.06; 0.07; 2x0.08; 0.09; 2x0.10; 2x0.11; 0.12; 0.15; 0.17; 0.19; 0.21; 0.22; 0.29; 0.36; 0.56, 0.75, 0.81	0.81	0.075
	S-EU	31	<0.01, 0.04; 2x0.05; 0.06; 0.08; 0.09; 0.10; 0.11; 2x0.14; 0.19; 0.24; 0.31; 2x0.33; 0.34; 0.54; 0.56; 0.62; 0.67; 0.69; 2x0.79; 0.93; 1.0; 1.1; 2x1.2; 1.7; 2.5	2.5	0.33

N-EU = northern Europe; S-EU = southern Europe

Table 6.3.2-85: Summary of residue data from barley trials with prothioconazole: total residue (sum of the 6 analytes)

Com-modity	Region	No. of trials	Values (mg/kg)		
			Calculated sum of prothioconazole-desthio and its hydroxy metabolites	Maximum (HR)	STMR
Barley, grain	N-EU	6	5x<0.06; 0.061	0.061	<0.06
	S-EU	7	4x<0.06; 2x0.06; 0.064	0.064	<0.06
Barley, straw	N-EU	6	0.10; 0.14; 0.17; 0.46; 0.56; 2.2	2.2	0.32
	S-EU	7	0.39; 0.46; 0.87; 2x1.2; 2.0; 3.1	3.1	1.2

N-EU = northern Europe; S-EU = southern Europe

Table 6.3.2-86: Summary of residue data from barley trials with prothioconazole: hydroxy metabolites

Com-modity	Region	n	Analyte	Values for the hydroxy metabolites		
				Residue level(mg/kg)	Maximum (HR)	STMR
Barley, grain	N-EU	6	alpha-hydroxy-PTZ-DT	6x<0.01	<0.01	<0.01
			3-hydroxy-PTZ-DT	6x<0.01	<0.01	<0.01
			4-hydroxy-PTZ-DT	6x<0.01	<0.01	<0.01
			5-hydroxy-PTZ-DT	6x<0.01	<0.01	<0.01
			6-hydroxy-PTZ-DT	6x<0.01	<0.01	<0.01
	S-EU	7	alpha-hydroxy-PTZ-DT	7x<0.01	<0.01	<0.01
			3-hydroxy-PTZ-DT	7x<0.01	<0.01	<0.01
			4-hydroxy-PTZ-DT	7x<0.01	<0.01	<0.01
			5-hydroxy-PTZ-DT	7x<0.01	<0.01	<0.01
			6-hydroxy-PTZ-DT	7x<0.01	<0.01	<0.01
Barley, straw	N-EU	6	alpha-hydroxy-PTZ-DT	5x<0.01; 0.023	0.023	0.01
			3-hydroxy-PTZ-DT	0.02; 0.029; 0.04; 0.11; 0.17; 0.55	0.55	0.075
			4-hydroxy-PTZ-DT	<0.01; 0.03; 0.04; 0.083; 0.12; 0.55	0.55	0.062
			5-hydroxy-PTZ-DT	<0.01; 2x0.02; 0.08; 0.092; 0.23	0.23	0.05
			6-hydroxy-PTZ-DT	5x<0.01; 0.042	0.042	<0.01
	S-EU	7	alpha-hydroxy-PTZ-DT	3x<0.01; 0.014; 0.015; 0.016; 0.025	0.025	0.014
			3-hydroxy-PTZ-DT	0.01; 0.12; 0.15; 0.16; 0.18; 0.33; 0.45	0.45	0.16
			4-hydroxy-PTZ-DT	0.02; 0.17; 2x0.21; 0.32; 0.47; 0.68	0.68	0.21
			5-hydroxy-PTZ-DT	0.01; 0.079; 0.10; 0.11; 0.15; 0.19; 0.20	0.20	0.11
			6-hydroxy-PTZ-DT	5x<0.01; 0.018; 0.042	0.042	<0.01

N-EU = northern Europe; S-EU = southern Europe. PTZ-DT: prothioconazole-desthio

CA 6.3.3 Oilseed rape

Not applicable, see Table 6.3-1.

CA 6.4 Feeding studies

Prothioconazole is sought for use on cereal which parts are fed to livestock.
The maximum dietary burdens were therefore calculated for different groups of livestock as described in the OECD Guidance Document on Residues in Livestock (ENV/JM/MONO(2013)8 dated of 04-Sep-2013). As the uses are on cereal for grain production, only grain and straw values were used.

Scenario 1
Some of the residue trials were analysed only for prothioconazole-desthio and do not provide data on other metabolites which are included in the risk assessment residue definition (sum of prothioconazole-desthio and all metabolites containing the 2-(1 chlorocyclopropyl)-3-(2-chlorophenyl)-2 hydroxypropyl-2H-1,2,4-triazole moiety). To address this issue, for cereal grain an appropriate conversion factor of 2 between enforcement and risk assessment was derived by EFSA. For cereal straw, a conversion factor of 3 was set by EFSA based on the metabolism studies.

Scenario 2
Nevertheless, as a significant number of trials were determined for all 6 analytes, the "total prothioconazole" values were taken and no conversion factor was used.

Table 6.4-1 Input values for the dietary burden calculation of Prothioconazole with values from Prothioconazole-desthio only

Analytes	Commodity	Input value (mg/kg)	Comment	Reference
Prothioconazole-desthio only **Scenario 1**	Barley and oat Grain	0.02	STMR x CF(2)	Table 6.3.2-84
	Barley and oat Straw	7.5	HR x CF(3)	
	Wheat, Rye, Grain	0.02	STMR x CF(2)	Table 6.3.1- 83
	Wheat, Rye, Straw	6.9	HR x CF(3)	
"total residue" of prothioconazole (6 analytes) **Scenario 2**	Barley and oat Grain	0.06	STMR	Table 6.3.2-85
	Barley and oat Straw	1.155	HR	
	Wheat, Rye, Grain	0.06	STMR	Table 6.3.1- 84
	Wheat, Rye, Straw	3.35	HR	

CF: Conversion factor proposed in EFSA Scientific Report (2007)

The results of the dietary burden calculations are reported in Table 6.4- 2 and Table 6.4-3.

Table 6.4- 2: Scenario 1: Results of the dietary burden calculations - OECD methodology

	Maximum dietary burden (mg/kg bw/day)	Highest contributing commodity	Max dietary burden (mg/kg DM)	Trigger exceeded (Y/N)
Risk assessment residue definition: Sum of prothioconazole-desthio and all metabolites containing the 2-(1-chlorocyclopropyl)-3-(2-chloro-phenyl)-2-hydroxypropyl-2H-1,2,4-triazole moiety) expressed as prothioconazole-desthio.				
Cattle - Beef	**0.061**	Barley Straw	2.544	**Y**
Cattle - Dairy	**0.098**	Barley Straw	2.537	**Y**
Sheep – Ram/Ewe	**0.169**	Barley Straw	5.065	**Y**
Sheep – Lambs	**0.215**	Barley Straw	5.065	**Y**
Swine - Breeding	0.000	Barley Grain	0.018	N
Swine - Finishing	0.001	Barley Grain	0.018	N
Poultry - Broiler	0.001	Barley Grain	0.016	N
Poultry - Layer	**0.055**	Wheat Straw	0.805	**Y**
Poultry - Turkey	0.001	Rye grain	0.014	N

Table 6.4- 3: Scenario 2: Results of the dietary burden calculations - OECD methodology

	Maximum dietary burden (mg/kg bw/day)	Highest contributing commodity	Max dietary burden (mg/kg DM)	Trigger exceeded (Y/N)
Risk assessment residue definition: Sum of prothioconazole-desthio and all metabolites containing the 2-(1-chlorocyclopropyl)-3-(2-chloro-phenyl)-2-hydroxypropyl-2H-1,2,4-triazole moiety) expressed as prothioconazole-desthio.				
Cattle - Beef	**0.049**	Rye Straw	0.809	**Y**
Cattle - Dairy	**0.030**	Rye Straw	0.789	**Y**
Sheep – Ram/Ewe	**0.052**	Rye Straw	1.550	**Y**
Sheep – Lambs	**0.066**	Rye Straw	1.564	**Y**
Swine - Breeding	0.001	Barley grain	0.055	N
Swine - Finishing	0.002	Barley grain	0.058	N
Poultry - Broiler	0.003	Barley grain	0.048	N
Poultry - Layer	**0.030**	Wheat Straw	0.442	**Y**
Poultry - Turkey	0.003	Rye grain	0.041	N

The calculated dietary burdens exceed the trigger value of 0.004 mg/kg bw/day for cattle, sheep and layer poultry.

Cattle and sheep:

Calculation of the values based on the dietary burden and the existing feeding study with prothioconazole-desthio (please refer to KCA 6.4.2). These calculations were made with the worst case scenario, *i.e.* the "Scenario 1" above (Table 6.4-1 and Table 6.4-2) where prothioconazole-desthio values were used with conversion factors to cover the risk assessment residue definition.

Table 6.4.4: Highest residue concentrations (mg/kg) in the edible tissues of cattle and sheep

Type of animal Max dietary burden (mg/kg bw per d)	Dose level * (mg/kg bw per d)	Matrix	Maximum (mg/kg)	Highest residue (mg/kg)	Conversion Factor for Risk Asesssment
Cattle **0.098**	0.19	Muscle	<0.01	<0.01	1.0
	1.05		<0.01		
	4.54		<0.01		
	0.19	Fat	<0.01	<0.01	1.0
	1.05		0.01		
	4.54		0.09*		
	0.19	Liver	0.03	0.015	2.0
	1.05		0.18		
	4.54		1.20		
	0.19	Kidney	<0.01	<0.01	4.0**
	1.05		0.03		
	4.54		0.24		
	0.19	Milk	N/A	<0.004	1.0
	1.05		N/A		
	4.54		N/A		
Sheep **0.215**	0.19	Muscle	<0.01	<0.01	1.0
	1.05		<0.01		
	4.54		<0.01		
	0.19	Fat	<0.01	<0.01	1.0
	1.05		0.01		
	4.54		0.09**		
	0.19	Liver	0.03	0.034	2.0
	1.05		0.18		
	4.54		1.20		
	0.19	Kidney	<0.01	<0.01	4.0**
	1.05		0.03		
	4.54		0.24		

*please refer to 6.4.2

**based on the metabolism study. In EFSA Journal 2014;12(5):3689, the conversion factor (CF) of 9 for kidney is erroneous. Indeed, in the goat metabolism study with prothioconazole-desthio, the quantitative evaluation of radioactivity was done before and after acid treatment. A hydrolysis was necessary as there were a lot of conjugates. A conversion factor of 9 is derived if the values are taken before acidic treatment, but the CF is 4 if determined after the acidic treatment. Because all of the methods for the determination of prothioconazole-desthio include also a harsh hydrolysis step, the CF to be used for enforcement to risk assessment should be 4*, i.e.* derived from the values of the metabolism study after acidic treatment.

Based on these calculations, there is no need to change the existing MRLs in commodities of animal origin.

Poultry:

Please refer to the section below (KCA 6.4.1).

CA 6.4.1 Poultry

In EFSA's most recent Reasoned Opinion (EFSA, Journal 2014;12(5):3689) on the review of the existing maximum residue levels (MRLs), it is stated :
For poultry, although the maximum dietary burden exceeded the threshold of 0.1 mg/kg DM, no residues above the LOQ were expected in poultry matrices at the calculated dietary burden and no feeding study was triggered. Therefore, MRLs can be established at the LOQ in all poultry commodities and no default conversion factors for risk assessment need to be derived.

This statement is based on the poultry metabolism studies already submitted. To comfort this statement, the poultry feeding study performed with parent prothioconazole (conducted in the USA, report RAJAL001) is presented below. It shows that the transfers in poultry are extremely low.
Although it was performed with parent and not prothioconazole-desthio, it is believed to be comparable with a study performed with prothioconazole-desthio on the overall transfers. It is also written in EFSA, Journal 2014;12(5):3689, regarding livestock studies *"... the available studies indicate similar metabolic patterns for the different compounds and moieties investigated. Additional studies addressing these requirements are therefore not expected to provide different results."*
The study is summarised below.

Report:	KCA 6.4.1/01 [redacted]; [redacted]; 2008; M-304664-01-1
Title:	Prothioconazole - Magnitude of the residue in laying hens
Report No.:	RAJAL001
Document No.:	M-304664-01-1
Guideline(s):	OPPTS 860.1480; DACO 7.5.1
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Materials and Methods

The magnitude of the residue of prothioconazole and its metabolites prothioconazole-desthio and prothioconazole-4-hydroxy were studied in laying hens. This corresponds to the risk assessment residue definition for animal matrices in the United States of America.
Forty two laying hens (one control group of six hens, and three dose groups, each with three sub-groups of four hens noted A, B and C in the table below) were dosed orally, via capsule, for 29 consecutive days with prothioconazole at dose rates corresponding to 0 mg/kg feed (control animal dosed with only empty gelatin capsules), 0.263 mg/kg feed/day (1X dose group), 0.788 mg/kg feed/day (3X dose group), or 2.591 mg/kg feed/day (10X dose group).
Eggs were collected twice daily during the dosing period. Egg samples from the 10X dose group were analyzed for prothioconazole residue on study days 24 and 29. On day 29, the hens were sacrificed and liver, muscle (thigh and breast), and fat (abdominal and subcutaneous) tissues were collected and pooled by subgroup for analysis.

Tissue and egg samples were analyzed for the total prothioconazole residue (prothioconazole, prothioconazole-desthio, prothioconazole-4-hydroxy and their acid-hydrolyzable conjugates) by high performance liquid chromatography-electrospray ionization / tandem mass spectrometry (LC-MS/MS) using isotopically labeled internal standards. This method included a hydrolysis step for cleavage of conjugates to agylcones and conversion of the metabolites with diene structure back to aromatic compounds. This step is suitable for analysing the glucuronide conjugate of prothioconazole-desthio. Method validation was performed prior to sample analysis and concurrent recoveries were performed during sample analysis to demonstrate acceptable method performance. The limit of quantitation (LOQ) was 0.005 ppm each for the analytes of prothioconazole, prothioconazole-desthio, and prothioconazole-4-hydroxy in eggs. The LOQ was 0.010 ppm each for the analytes of prothioconazole, prothioconazole desthio, and prothioconazole-4-hydroxy in poultry tissues.
Egg, liver, muscle, and fat samples were analyzed within maximum 7 days of collection. Therefore, freezer storage stability studies on poultry tissues are not required.

Prothioconazole residues were <LOQ of 0.005 ppm in the egg samples analyzed from the 10X dose group. Therefore, egg samples from the 3X and 1X dose groups were not analyzed. In the 10X dose group, the prothioconazole residues in liver, muscle, and fat were <LOQ of 0.010 ppm, in all tissue samples. Therefore, tissue samples from the 3X and 1X dose groups were not analyzed.

Findings

A summary of the prothioconazole residues found in the tissues is given the table below.

Residue concentrations (mg/kg) in the edible tissues of laying hens after 29 days of dosing with prothioconazole (10X dose rate)

10 X dose group *	Study Day	Residue (mg/kg)		
		Prothioconazole-desthio	prothioconazole	prothioconazole-4 hydroxy
Eggs				
10X-A	24	<LOQ	<LOQ	<LOQ
10X-B	24	<LOQ	<LOQ	<LOQ
10X-C	24	<LOQ	<LOQ	<LOQ
10X-A	29	<LOD	<LOQ	<LOD
10X-B	29	<LOQ	<LOQ	<LOQ
10X-C	29	<LOQ	<LOQ	<LOQ
Liver				
10X-A	29	<LOD	<LOD	<LOD
10X-B	29	<LOD	<LOD	<LOD
10X-C	29	<LOD	<LOD	<LOD
Muscle				
10X-A	29	<LOD	<LOD	<LOD
10X-B	29	<LOD	<LOD	<LOD
10X-C	29	<LOD	<LOD	<LOD
Fat				
10X-A	29	<LOD	<LOD	<LOD
10X-B	29	<LOD	<LOD	<LOD
10X-C	29	<LOD	<LOD	<LOD

* samples from the 3X and 1X dose groups were not analyzed
** sum of individual prothioconazole, prothioconazole-desthio, and prothioconazole-4-hydroxy residues (in mg/kg parent equivalent)

Conclusion

Feed consumption, body weights, and egg production were not adversely affected by daily oral administration of prothioconazole to laying hens for 29 consecutive days at the average dose rates of 0.263 mg/kg feed (1X), 0.788 mg/kg feed (3X), and 2.591 mg/kg feed (10X).
For 24 and 29 day egg samples from the 10X dose group, prothioconazole residue was <LOQ of 0.005 ppm. Therefore, eggs from poultry fed crops containing prothioconazole would not be expected to contain prothioconazole residues greater than the LOQ of 0.005 ppm.
For all liver, muscle, and fat samples from the 10X dose group, the prothioconazole residues were <LOQ of 0.010 ppm. Therefore, tissue samples from poultry fed crops containing prothioconazole would not be expected to have prothioconazole residues greater than the LOQ of 0.010 ppm.

CA 6.4.2 Ruminants

During the peer review under Directive 91/414/EEC, the magnitude of prothioconazole residues in ruminants was investigated in a feeding study with lactating cows (EFSA Scientific Report (2007) 106, 1-98 and EFSA Journal 2014; 12(5):3689). Three groups of lactating cows, each consisting of three animals, were meant to be dosed for 28 consecutive days with prothioconazole-desthio at levels of 4, 25, and 100 mg/kg in the diet (1X, 6.25X and 25X). The actual average dose rates were 1.3X, 7.3X, and 31X the anticipated maximum dietary burden**, *i.e.* 5.1 ppm, 29 ppm and 125 mg/kg in the diet**. Please note in EFSA Journal 2014; 12(5):3689, the intended values of 4, 25 and 100 mg/kg were erroneously utilised for the ruminant livestock values.

The samples were analysed for prothioconazole-desthio, 3-hydroxy-prothioconazole-desthio (M19) and 4-hydroxy-prothioconazole-desthio (M15). Indeed, these 3 analytes were found to represent the major portion of the TRR in the goat metabolism study after acidic treatment (please refer to Fate Annex II dossier, 6.2.2.2), in particular in liver, kidney, muscle and fat. They do not however, completely cover the residue definition for risk assessment. Therefore, only the measured average and maximum concentrations for prothioconazole-desthio are compiled in the table below. This covers the residue definition for enforcement.

Table 6.4.2-1: Highest and mean residue concentrations (mg/kg) in the edible tissues of dairy cattle after 28 days of dosing with JAU 6476-desthio

Dose group	Dose level * (mg/kg bw per d)	Matrix	Mean (mg/kg)	Maximum (mg/kg)
1.3X (5.1 ppm feed)	**0.19**	**Muscle**	<0.01	<0.01
7.3X (29 ppm feed)	**1.05**		<0.01	<0.01
31X (125 ppm feed)	**4.54**		<0.01	<0.01
1.3X (5.1 ppm feed)	**0.19**	**Fat**	<0.01	<0.01
7.3X (29 ppm feed)	**1.05**		0.01	0.01
31X (125 ppm feed)	**4.54**		0.05**	0.09**
1.3X (5.1 ppm feed)	**0.19**	**Liver**	0.02	0.03
7.3X (29 ppm feed)	**1.05**		0.14	0.18
31X (125 ppm feed)	**4.54**		0.68	1.20
1.3X (5.1 ppm feed)	**0.19**	**Kidney**	<0.01	<0.01
7.3X (29 ppm feed)	**1.05**		0.03	0.03
31X (125 ppm feed)	**4.54**		0.13	0.24
1.3X (5.1 ppm feed)	**0.19**	**Milk**	<0.004	N/A
7.3X (29 ppm feed)	**1.05**		<0.004	N/A
31X (125 ppm feed)	**4.54**		<0.004	N/A

* Based on a 500 kg animal consuming 20 kg feed DM day

** in EFSA Journal 2014; 12(5):3689, the values are lower because one value (at 0.0905) mg/kg was erroneously reported as 0.01 mg/kg instead of 0.09 mg/kg in the study report.

Conversions factors for enforcement to risk assessment:

According to EFSA Journal 2014; 12(5):3689, the animal enforcement residue definition is proposed as prothioconazole-desthio (sum of isomers) only. Indeed, as can be seen in the feeding study, prothioconazole-desthio is a valid marker for prothioconazole in animal matrices (even in milk where transfers are extremely low). Prothioconazole-desthio was not detected in milk in the metabolism study because of the very low transfer of prothioconazole in this matrix. This was confirmed by the feeding study.

Based on the feeding study performed with prothioconazole-desthio, it was possible to calculate conversion factors for enforcement to risk assessment of 2 and 3 respectively for liver and kidney. Based on the metabolism study, the conversion factors (after acidic treatment) were in good agreement (2 and 4 respectively for liver and kidney).

In EFSA Journal 2014; 12(5):3689, the conversion factor (CF) of 9 for kidney is erroneous.
Indeed, in the goat metabolism study with prothioconazole-desthio, the quantitative evaluation of radioactivity was done before and after acid treatment. A hydrolysis was necessary as there were a lot of conjugates. A conversion factor of 9 is derived if the values are taken before acidic treatment, but the CF is 4 if determined after the acidic treatment. Because all of the methods for the determination of prothioconazole-desthio include also a harsh hydrolysis step, the CF to be used for enforcement to risk assessment should be 4, i.e. derived from the values of the metabolism study after acidic treatment.

As stated in EFSA Journal 2014; 12(5):3689, no conversion was derived for milk, muscle and fat since the residue levels in these matrices are expected to be negligible (< 0.01 mg/kg) at the calculated dietary burden.

CA 6.4.3 Pigs

A pig feeding study is not triggered. No study was conducted as calculations show that the dietary burden is under the trigger value, see Table 6.4-2 and 3.

CA 6.4.4 Fish

No metabolism study or feeding study in fish was conducted.
Currently, no test method or guidance document is available for conducting a feeding study in fish. Also, no feeding table with plant commodities for fish feeding is available. Therefore, it cannot be decided whether fish might be exposed to residues of prothioconazole or prothioconazole-desthion in parts of plants that have been treated with prothioconazole. In these cases, waiving of this particular data requirement is considered acceptable according to the "Guidance document for applicants on preparing dossiers for the approval of a chemical new active substance and the renewal of approval of the chemical active substance according to regulation (EU) No. 283/2013 and regulation (EU) No. 284/2013" (SANCO/10181/2013-rev. 3 of 12-December-2014).

CA 6.5 Effects of processing

CA 6.5.1 Nature of the residue

A hydrolysis study was perfomed with parent prothioconazole and was evaluated during the initial Annex I inclusion. The study demonstrated that prothioconazole is stable under all three sets of conditions representative for typical food processing operations.

Another hydrolysis study was conducted on prothioconazole-desthio and is presented below.

Report:	KCA 6.5.1/02 [redacted]; 2001; M-035293-01-1
Title:	Hydrolysis of JAU 6476-desthio under conditions of processing
Report No.:	MR-106/00
Document No.:	M-035293-01-1
Guideline(s):	ECPA Residue Task Force, March 1993
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

The hydrolytic degradation of the metabolite prothioconazole-desthio in buffered drinking water has been investigated in order to determine whether the nature of the residue found in processed agricultural commodities is likely to be different from that in raw agricultural commodities. The test substance was exposed to three sets of conditions each of them being representative for a typical food processing operation.

Test system

Radiolabelled [phenyl-UL-14C]prothioconazole-desthio was dissolved at concentrations of approximately 5 mg/L in citrate buffers which were incubated at 90 °C for 20 min (pH 4.0), 100 °C for 60 min (pH 5.0) and 120 °C for 20 min (pH 6.0). After incubation the samples were analysed by liquid scintillation counting to establish recoveries of radioactivity and by HPLC for determination of concentrations of test substance and hydrolysis products. A set of control solutions was used to obtain zero-time values.

Findings

Recoveries of radioactivity were found to be 99.4%, 99.9% and 100.3% of applied radioactivity for the three experiments. Prothioconazole-desthio did not show any sign of decomposition if exposed to the sets of conditions representing pasteurisation (pH 4, 90 °C), baking, brewing and boiling (pH 5, 100 °C) and sterilisation (pH 6, 120 °C).

Conclusion

For prothioconazole-desthio, the nature of the residue in the processed commodity is expected to be identical with that in the raw agricultural commodity for all three types of food processing operations examined.

Other metabolites included in the residue definition for risk assessment

Regarding the investigation of the effects of processing of the nature of all the metabolites included in the residue definition for risk assessment in plant commodities, *i.e.* on the monohydroxylated derivatives of prothioconazole-desthio, there is no need to perform a hydrolysis study.
Indeed, the data generation method used to determine these analytes (method 00979/M001) contains a drastic hydrolysis step (reflux for 2 h in 5*N* hydrochloric acid). This step is performed after the extraction to cleave the conjugates to aglycones and to convert the metabolites with diene structure back to aromatic compounds.
Because the method was fully validated in 5 different matrices (wheat grain, potato tuber, tomato fruit, rape seed and orange fruit), this proves that the 5 metabolites of prothioconazole-desthio are stable to acid hydrolysis even under much harsher conditions (2 h refluxing in 5*N* hydrochloric acid) than the representative hydrolytic conditions (*i.e.* pasteurisation (20 min, 90 °C, pH 4); baking, brewing and boiling (60 min, 100 °C, pH 5) and sterilisation (20 min, 120 °C, pH 6)).
This method has been utilised for the determination of these analytes in many different types of residue trials.
The detailed description of the method 00979/M001 is in section KCA 4.1.2 (documents M-267072-01-1 and M-328686-01-1).

CA 6.5.2 Distribution of the residue in peel and pulp

Not applicable for cereals.

CA 6.5.3 Magnitude of residues in processed commodities

During the previous Annex I inclusion, based on the residue data on seed dressing and spray application, processing studies were not considered necessary.
For this submission, as residues of active substance do not exceed the trigger values defined in Regulation (EU) No 283/2013, there is no need to investigate the effect of industrial and/or household processing. Nevertheless, 5 processing studies were performed (4 on barley, 1 on wheat) and are presented below.

- Barley

Report:	KCA 6.5.3/01 [redacted]; [redacted]; 2015; M-513062-03-1
Title:	Determination of the residues of fluoxastrobin and prothioconazole in/on barley, spring and the processed fractions (malt sprouts; brewer's malt; brewer's grain; hops draff; brewer's yeast; beer; pearl barley rub off; pearl barley) after spraying of fluoxastrobin & prothioconazole EC 200 in the field in Germany and France (South)
Report No.:	13-3401
Document No.:	M-513062-03-1
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC EC guidance working document 7029/VI/95-rev. 5 (July 22, 1997) OECD 509 Adopted 2009-09-07, OECD GUIDELINE FOR THE TESTING OF CHEMICALS, Crop Field Trial OECD 508 adopted 3 October 2008, OECD Guideline for the testing of chemicals, magnitude of the pesticide residues in processed commodities US EPA OCSPP Guideline No. 860.1520 US EPA OCSPP Guideline No. 860.1500, Crop Field Trial
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Two processing studies were performed in Germany and France in order to determine the transfer residues of fluoxastrobin residues and prothioconazole residues in unprocessed spring barley grain and in the primary processed products beer and pearl barley; as well as in intermediate fractions, including malt. For the sake of clarity, only results for prothioconazole residues are presented in this dossier.

Material and methods

Two field trials were conducted in southern France during the 2013 growing season. Spring barley was treated twice with fluoxastrobin & prothioconazole EC 200 (EC emulsifiable concentrate containing 100 g/L fluoxastrobin and 100 g/L prothioconazole) at an exaggerated dose of 375 g/ha (total of 750 g/ha) with an interval of 14 days. Barley grain for processing was harvested 50 or 69 days after the last application. The design of the field trials and the residues in the raw agricultural commodity are summarised in Table 6.5.3-1.

Table 6.5.3-1: Field trials conducted to generate barley samples for processing – overview of trial design

Study Trial No. Year	Crop Variety	Location	Application of prothioconazole				
			FL	No	g a.s./ha	g as/hL	GS
13-3401 13-3401-01 2013	Spring barley Conchita	Germany [redacted]	EC 200 g/L	2	375	125	61
13-3401 13-3401-02 2013	Spring barley Henley	France [redacted]	EC 200 g/L	2	375	125	61

FL: Formulation; GS : BBCH growth stage at last application

After processing (described below), residue analysis was performed by LC/MS/MS according to method 01013 for prothioconazole-desthio and method 00979/M001 for 3-hydroxy-prothioconazole-desthio, 4-hydroxy-prothioconazole-desthio, 5-hydroxy-prothioconazole-desthio, 6-hydroxy-prothioconazole-desthio and alpha-hydroxy-prothioconazole-desthio (for more information, cf. see chapter 4, point KCA 4.1.2). The limits of quantitation were 0.01 mg/kg expressed as prothioconazole-desthio equivalents. Prior and parallel to the residue analysis, the methods were validated by recovery experiments.

Processing procedures

The processing was designed to simulate industrial procedures of grain into the processed fractions (malt sprouts, brewer's malt, brewer's grain, hops draff, brewer's yeast, and beer, as well as in pearl barley and pearl barley rub-off).

Cleaning

The field specimens for processing were cleaned. In order to obtain acceptable results, grain with a moisture content of approx. 14% was required. The optimal moisture content was already reached at the sampling date, therefore no drying of the field specimens was necessary before cleaning.

Malting

Sieving and steeping

Before malting was started, the grain was sieved (sieve mesh 2.5 mm). After sieving, the steeping process was conducted as a combined wet and dry steeping. Sieved barley grain was transferred in a special steeping vessel. During steeping water is supplied to the interior of the kernel. As a result the enzymes become active and germination begins. Water uptake depends on the steeping time, steeping temperature, kernel size, barley variety and harvest year. The final steeping degree was in the range of 44.6 to 44.8%.

Germination and kiln drying

The processes during germination can be divided into growth processes, enzyme formation and metabolic changes. Towards the end of steeping the rootlets break through the base of the corn and become visible. Activation of enzymes and formation of new enzymes are essential processes during germination (starch degrading enzymes, cytolytic enzymes, protein degrading enzymes and phosphoric

acid splitting enzymes). For proper performance of germination it is necessary to control the duration of germination, the mean temperature of wet air and the relative humidity of the air around the kernels. During the intensive respiration the steeped good was turned over continuously. After germination, the life processes are terminated by kilning. During kilning the water content of green malt is lowered down to < 10%, germination and modification are stopped; colour and flavour compounds are formed. The malt becomes stable and storable. Kiln-drying was conducted in a dry chamber. After kiln-drying the germs (= malt sprouts) were removed mechanically by a trimmer. Brewer's malt and malt sprouts were sampled immediately after end of malting.

Brewing (processing of malt to beer)

Mashing and lautering (wort extraction and separation)
Before mashing, the brewer's malt was dry milled in a special malt mill. The crushed malt was mixed with brew water. To produce Pilsener beer, mashing was started in a heatable tun.

After mash boiling, the wort was separated from the insoluble malt components (brewer's grain). The extract remaining in the brewer's grain was extracted by washing with hot water (first filter runnings). The wort separation was done using a refining vat. After separation, the brewer's grain was sampled.

Wort boiling and conditioning
Hop pellets were added and the separated wort was boiled (about 90 minutes at normal pressure). After boiling, the flocs (hops draff) were separated in a whirlpool, causing the sludge to deposit on the bottom in the shape of a cone. For cooling and ventilating the wort, an intra-plant circulation was used. By adding oxygen (intra-plant circulation), the conditions for the start of the fermentation were prepared. Hops draff was sampled.

Fermentation and maturation
In the pilot plant, the classical primary fermentation (low fermentation) was carried out in bottom-fermentation containers. Fermentation heat was dissipated by means of room ventilation. As soon as the extract content of the fermented young beer was 2% higher than the final attenuation, the storing time began. Before maturation, the young beer was cooled down. During the main fermentation, the yeast deposited on the tank bottom and was sampled as brewer's yeast.

At the beginning of maturation, the young beer was stored at room temperature (warm maturation to break down the diacetyl) in casks. Then the young beer was stored under pressure (approx. 0.7-2.1 bar) at 2°C (cold maturation) for about 4 weeks. In this time, the remaining extract was fermented. Unwanted flavour and odorous substances were decomposed or expelled. Sludge particles and yeast settle at the bottom. The rack beer was filtered using a special filter combination. During filtration, all organisms harming the beer (bacteria and yeast) were removed and sludge particles were separated. The final product, beer, was sampled.

The processes are illustrated in figures 6.5.3-1 to 6.5.3-3

Figure 6.5.3-1: Industrial "processing" of grain to malt

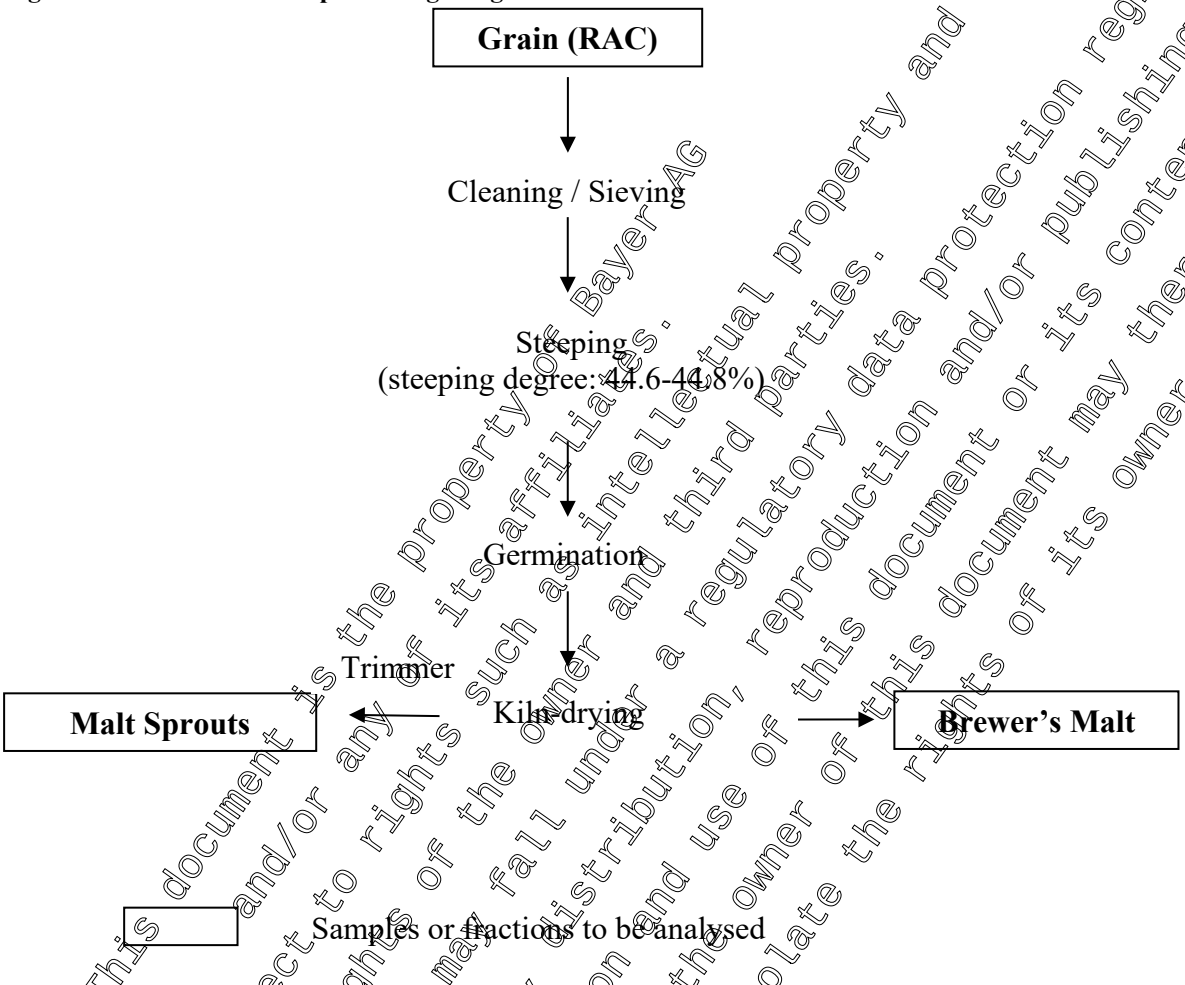
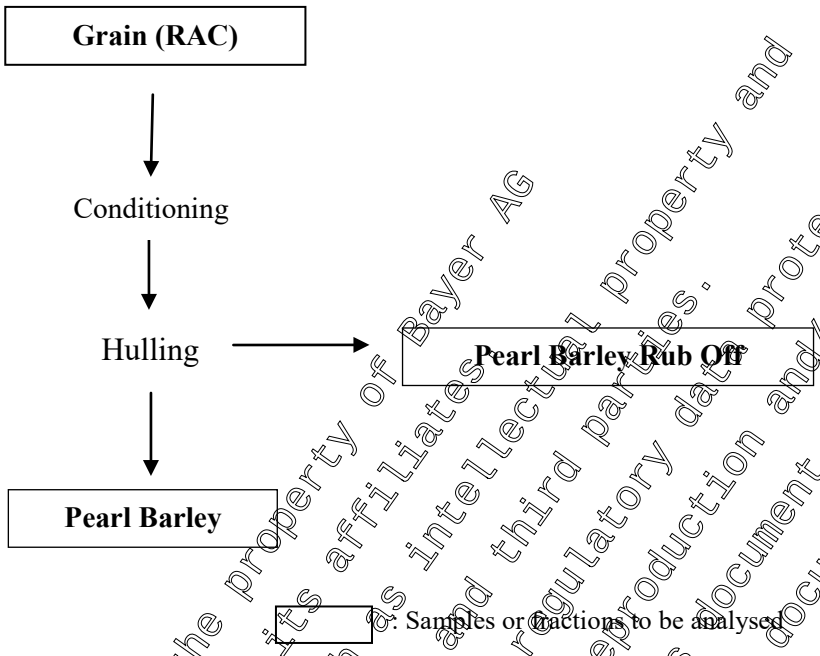


Figure 6.5.3-2: Industrial "processing" of malt to beer

Figure 6.5.3-3: Industrial "processing" of spring barley to pearl barley



Findings

The validation of the method for the determination of residues in the sample materials hops draff, brewer´s grain and beer was conducted within this study.
The recoveries for the sample material brewer´s grain are considered to also validate the residue determination in the sample materials grain, brewer´s malt, malt sprouts, pearl barley and stored grain. In the same way, the sample material hops draff is considered to be representative for sample material brewer´s yeast.
In all mentioned matrices, recovery samples for parent compound were spiked at levels of 0.01 mg/kg and 0.10 mg/kg. Mean recoveries for all matrices were in an acceptable range from 70-110%, with RSDs below 20%.

Table 6.5.3.2 gives an overview of the concurrent recoveries for the determination of prothioconazole-desthio in barley grain, brewer's grain, beer and hops draff.. Table 6.5.3-3 gives an overview of the concurrent recoveries for the determination of the hydroxy metabolites of prothioconazole-desthio in brewer's grain, beer and hops draff. The overall average recoveries per analyte and matrix were within the acceptable range of 70-110% and the corresponding RSDs were below 20%.

Table 6.5.3-2: 13-3401: Concurrent recoveries for the determination of prothioconazole-desthio in barley and barley processed commodities

Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
		Single Values	Mean		
Barley grain (stored)	0.01	94, 96, 96, 97	96	1.3	0.01
	0.10	96, 98, 100	98	2.0	
	Overall (n = 7)		**97**	**2.0**	
Barley brewer's grain	0.01	86, 88, 90	88	2.3	0.01
	0.10	99, 99, 101	100	1.2	
	Overall (n = 6)		**94**	**7.0**	
Beer	0.01	91, 97, 100	96	4.8	0.01
	0.10	98, 98, 99	98	0.6	
	Overall (n = 6)		**97**	**3.3**	
Barley hops draff	0.01	88, 94, 101	94	6.9	0.01
	0.10	98, 100, 102	100	2.0	
	Overall (n = 6)		**97**	**5.5**	

RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
Fortified with prothioconazole-desthio, determined as prothioconazole-desthio and calculated as prothioconazole-desthio

Table 6.5.3-3: 13-3401: Concurrent recoveries for the determination of the hydroxy metabolites of prothioconazole-desthio in barley grain and processed barley fractions

Analyte	Sample Material	Fortification level [mg/kg]*	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
prothioconazole-alpha-hydroxy-desthio	Barley brewer's grain	0.01	70, 74, 77, 78, 78, 79	76	4.5	0.01
		0.10	86, 89, 92, 93, 96	91	4.2	
		Overall (n = 11)		**83**	**10.4**	
	Beer	0.01	84, 85, 88, 89	87	2.8	0.01
		0.10	89, 92, 94, 97	93	3.6	
		Overall (n = 8)		**90**	**4.9**	
	Barley hops draff	0.01	78, 79, 86, 87	83	5.6	0.01
		0.10	85, 86, 90, 92	88	3.7	
		Overall (n = 8)		**85**	**5.7**	
prothioconazole-3-hydroxy-desthio	Barley brewer's grain	0.01	69, 75, 76, 77, 78, 79	76	4.7	0.01
		0.10	87, 89, 91, 91, 94	90	2.9	
		Overall (n = 11)		**82**	**10.0**	
	Beer	0.01	86, 89, 90, 92	89	2.8	0.01
		0.10	82, 83, 83, 84	83	1.0	
		Overall (n = 8)		**86**	**4.4**	
	Barley hops draff	0.01	73, 76, 85, 91	81	10.2	0.01
		0.10	79, 83, 85, 87	84	4.1	
		Overall (n = 8)		**82**	**7.3**	
prothioconazole-4-hydroxy-desthio	Barley brewer's grain	0.01	66, 68	66	-	0.01
		0.10	75, 75, 84, 88, 90	82	8.6	
		Overall (n = 7)		**78**	**12.8**	
	Beer	0.01	68, 74, 76, 78	74	5.8	0.01
		0.10	81, 81, 84, 95	85	7.8	
		Overall (n = 8)		**80**	**10.0**	
	Barley hops draff	0.01	67, 70, 73, 75	70	8.9	0.01
		0.10	69, 72, 77, 82	75	7.6	
		Overall (n = 8)		**72**	**8.5**	

RSD: Relative Standard Deviation, LOQ: Limit of Quantitation

* the individual hydroxy metabolites are fortified and determined as themselves and calculated as prothioconazole-desthio

Table 6.5.3-3 (cont'd): 13-3401: Concurrent recoveries for the determination of the hydroxy metabolites of prothioconazole-desthio in barley grain and processed barley fractions

Analyte	Sample Material	Fortification level [mg/kg]*	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
prothioconazole-5-hydroxy-desthio	Barley brewer's grain	0.01	66, 68, 84, 77, 82, 84	77	10.5	0.01
		0.10	85, 86, 86, 90, 90	87	2.8	
		Overall (n = 11)		**82**	**9.9**	
	Beer	0.01	82, 87, 91, 94	89	5.9	0.01
		0.10	81, 86, 92, 92	88	6.1	
		Overall (n = 8)		**88**	**5.5**	
	Barley hops draff	0.01	78, 78, 88, 91	83	7.4	0.01
		0.10	79, 83, 83, 85	83	3.1	
		Overall (n = 8)		**83**	**5.3**	
prothioconazole-6-hydroxy-desthio	Barley brewer's grain	0.01	70, 80, 83, 83, 91, 94	84	10.2	0.01
		0.10	86, 87, 87, 89, 94	89	3.6	
		Overall (n = 11)		**86**	**8.0**	
	Beer	0.01	89, 90, 90, 95	91	3.0	0.01
		0.10	85, 91, 93, 94	91	4.4	
		Overall (n = 8)		**91**	**3.5**	
	Barley hops draff	0.01	82, 82, 89, 94	87	6.7	0.01
		0.10	78, 85, 85, 86	84	4.4	
		Overall (n = 8)		**85**	**5.7**	

RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
* the individual hydroxy metabolites are fortified and determined as themselves and calculated as prothioconazole-desthio

The residues of prothioconazole-desthio and its hydroxy metabolites determined in the barley raw agricultural commodity and in the processed fractions are shown in Table 6.5.3-4. The corresponding transfer factors are shown in Table 6.5.3-5.

In both trials, except for pearl barley rub off (for prothioconazole-desthio and 3-hydroxy-prothioconazole-desthio), no processing factor could be derived since all residues were below the limit of quantification in the raw agricultural commodities and in the processed fractions. For pearl barley rub off (in trial 13-3401-02), a concentration of prothioconazole-desthio and 3-hydroxy-prothioconazole-desthio was observed.

Table 6.5.3-4: Residue levels (mg/kg) of prothioconazole-desthio and its metabolites in barley commodities

Trial	Analyte	Barley Grain	Grain, stored (RAC)*	Malt sprouts	Brewer's malt	Brewer's grain	Hops draff	Brewer's yeast	Beer	Grain, stored (RAC)*	Pearl barley rub off	Pearl barley
13-3401-01 (DALT 69 days)	PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	α-hydroxy-PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	3-hydroxy-PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	4-hydroxy-PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	5-hydroxy-PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	6-hydroxy-PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
13-3401-02 (DALT 50 days)	PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.032	<0.01
	α-hydroxy-PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	3-hydroxy-PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.020	<0.01
	4-hydroxy-PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	5-hydroxy-PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	6-hydroxy-PTZ-desthio	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

RAC : raw agricultural commodity; PTZ: prothioconazole. The individual hydroxy metabolites are determined as themselves and calculated as prothioconazole-desthio *mean of duplicate for all analytes. The samples "grain, stored" served as RAC samples for the beer and pearl barley processing, which was done in fresh state. They are samples which were taken in the field at the same time as the samples for processing, stored and shipped under the same conditions as the samples for processing and deep frozen at ≤ -18°C at the very time when the processing started.

Table 6.5.3-5: Transfer factors of prothioconazole-desthio and its metabolites in barley commodities

Trial	Analyte	Barley Grain	Grain, stored (RAC)	Malt sprouts	Brewer's malt	Brewer's grain	Hops draff	Brewer's yeast	Beer	Grain, stored (RAC)	Pearl barley rub off	Pearl barley
13-3401-01 (DALT 69 days)	PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*
	α-hydroxy-PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*
	3-hydroxy-PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*
	4-hydroxy-PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*
	5-hydroxy-PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*
	6-hydroxy-PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*
13-3401-02 (DALT 50 days)	PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	>3.2	-*
	α-hydroxy-PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*
	3-hydroxy-PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	>2.0	-*
	4-hydroxy-PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*
	5-hydroxy-PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*
	6-hydroxy-PTZ-desthio	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*	-*

RAC : raw agricultural commodity; PTZ: prothioconazole

* The transfer factor cannot be determined due to residues in the raw agricultural commodity and processed fraction being below the limit of quantification.

Conclusion

In order to determine processing factors for prothioconazole-desthio and its hydroxy metabolites (3-hydroxy-prothioconazole-desthio, 4-hydroxy-prothioconazole-desthio, 5-hydroxy-prothioconazole-desthio, 6-hydroxy-prothioconazole-desthio and alpha-hydroxy-prothioconazole-desthio) from spring barley grain in malt, beer, pearl barley and other processed fractions, two processing studies have been conducted.
In both trials, except for pearl barley rub off (for prothioconazole-desthio and 3-hydroxy-prothioconazole-desthio), no processing factor could be derived since all residues were below the limit of quantification in the raw agricultural commodities and in the processed fractions. For pearl barley rub off (in trial 13-3401-02), a concentration of prothioconazole-desthio and 3-hydroxy-prothioconazole-desthio was observed.

Report:	KCA 6.5.3/02 [redacted] p; 2008; M-303475-01-1
Title:	Determination of the residues of JAU 6476 in/on winter barley and spring barley after spraying of JAU 6476 (250 EC) in the field in Northern France
Report No.:	RA-3669/07
Document No.:	M-303475-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.5.3/03 [redacted] ; 2011; M-412497-01-1
Title:	Determination of the residues of JAU 6476-a-hydroxy-desthio, JAU 6476-3-hydroxy-desthio, JAU 6476-4-hydroxy-desthio, JAU 6476-5-hydroxy-desthio, and JAU 6476-6-hydroxy-desthio in/on/on materials of plant origin by HPLC-MS/MS
Report No.:	MR-08/025
Document No.:	M-412497-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Processing studies were performed in France in order to determine the residue transfer of prothioconazole its metabolites from winter and spring barley into brewer's malt, brewer's grain, yeast and beer generated during the brewing of barley grain from field trials conducted with an EC formulation containing 250 g/L of prothioconazole. Only results for prothioconazole-desthio and its hydroxy metabolites will be presented in this dossier.
Information on the field phase of the study may be found in the report. The processing phase was not conducted under GLP and is not described in the reports. Therefore, the study is only submitted as supplementary information and the processing factors derived for barley brewing are only considered indicative.

Material and methods

Two field trials were conducted in the northern part of France during the 2007 growing season to generate barley grain for a processing study. The crop received two spray applications with an emulsifiable concentrate (EC) containing 250 g/L of prothioconazole. Both applications were conducted at the recommended label use rate of 200 g a.s./ha. The first application was carried out at the growth stage BBCH 37 (flag leaf just visible, still rolled) and the second application at the growth stage BBCH 61 (beginning of flowering). The interval between the two applications was in the range of 13-17 days. The barley grain was harvested at maturity (BBCH 89) 53-63 days after the last application and stored frozen until processing. The design of the field trials is summarised in Table 6.5.3.6.

Table 6.5.3-6: Field trials conducted to generate barley samples for processing – overview of trial design

Study Trial No. Year	Crop Variety	Location	Application of prothioconazole				
			FL	No	g a.s./ha	g as/hL	GS
RA-3669/07 R 2007 0781/9 2007	Winter barley Vanessa	France [redacted]	EC 250 g/L	2	200	67	61
RA-3669/07 R 2007 0782/7 2007	Spring barley Scarlette	France [redacted]	EC 250 g/L	2	200	67	61

FL: Formulation; GS : BBCH growth stage at last application

The processing phase (barley brewing) was carried out at IFBM (French Institute for Brewing and Malting) using 31 kg samples of barley grain from each trial. The processed commodities generated during this phase consisted of brewer's malt, brewer's grain, brewer's yeast and beer. Since the processing was not conducted under GLP, it is not described in the study reports and no more detailed information on the brewing procedure is available.

The raw agricultural commodity (barley grain) and the barley processed fractions (brewer's malt, brewer's grain, brewer's yeast and beer were analysed for:

- the residues of prothioconazole-desthio according to the method 01013 (cf. KCA 4.1.2). The residues were extracted from the samples with acetonitrile/water (4/1; v/v, containing cysteine hydrochloride) using a blender. After filtration of the extract, the stable isotopically labeled analytes was added.. The solution was made up to volume, diluted and subjected to reversed phase HPLC-MS/MS without a further clean-up step. The limit of quantification (LOQ), defined as the lowest validated fortification level, was 0.010 mg/kg for prothioconazole-desthio in all the investigated matrices.

The unprocessed barley grain samples and the various fractions from barley processing were stored deep-frozen for a maximum of 242 days before analysis.

- and for the residues of the hydroxy metabolites of prothioconazole-desthio according to method 00979/M001.

The analytical method 00979/M001 was developed in order to determine residues of prothioconazole-α-hydroxy-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio,

prothioconazole-5-hydroxy-desthio and prothioconazole-6-hydroxy-desthio expressed as prothioconazole-desthio equivalent for all single compounds in/on matrices of plant origin by LC-MS/MS using matrix matched standards. The homogenised sample material of plant origin (approx. 5 g) is extracted with a mixture of acetonitrile/water (4/1; v/v) by high-speed blending. After filtration and evaporation to the aqueous remainder the extract is diluted and acidified with 5 N hydrochloric acid and refluxed for 2 hours. This hydrolysis step is performed to convert glycosidic bound analogues into the respective hydroxy analytes. An aliquot is neutralised with sodium hydrogen carbonate and purified on a Chromabond® XTR cartridge. The analytes are eluted with cyclohexane/ethyl acetate (85/15; v/v). The eluate is evaporated to dryness and the remainder is re-dissolved in acetonitrile. For quantitative analysis the extract is diluted with acetonitrile and water and subjected to LC-MS/MS.

Findings

Table 6.5.3-7 gives an overview of the concurrent recoveries for the determination of prothioconazole-desthio in barley grain and brewer's malt. Table 6.5.3-8 gives an overview of the concurrent recoveries for the determination of the hydroxy metabolites of prothioconazole-desthio in barley grain and barley processed commodities. The average recoveries per analyte and matrix were within the acceptable range of 70-110% and the corresponding RSDs were below 20%.

Table 6.5.3-7: RA-3669/07: Concurrent recoveries for the determination of prothioconazole-desthio in brewer's malt and barley grain

Sample Material	Fortification level [mg/kg]	Recovery rates [%]* Single Values	Mean	RSD [%]	LOQ [mg/kg]
Barley grain	0.01	85	85	-	0.01
	0.20	82	82	-	
	Overall (n = 2)		**84**	**-**	
Barley grain for processing	0.01	91; 85	88	-	0.01
	0.20	84;86	85	-	
	Overall (n = 4)		**87**	**3.6**	
Brewer's malt**	0.01	89;86	88	-	0.01
	0.20	89;86	88	-	
	Overall (n = 4)		**88**	**2.0**	

RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
Fortified with prothioconazole-desthio, determined as prothioconazole-desthio and calculated as prothioconazole-desthio
*These recoveries were performed during the conduct of the studies RA-3062/07 and RA-3669/07.
** = recoveries for barley beer, barley brewer's grain, barley brewer's yeast are covered by recoveries for brewer's malt.

Table 6.5.3-8: RA-3669/07: Concurrent recoveries for the determination of the hydroxy metabolites of prothioconazole-desthio in barley grain and processed barley fractions

Sample Material	Analyte	Fortification level [mg/kg]**	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
Barley grain for processing*	α-hydroxy-PTZ-desthio	0.01	108; 108; 112	109	2.1	0.01
		0.10	96	-	-	
		Overall (n = 4)		**106**	**6.5**	
	3-hydroxy-PTZ-desthio	0.01	105; 101; 105	104	2.2	0.01
		0.10	95	-	-	
		Overall (n = 4)		**102**	**4.7**	
	4-hydroxy-PTZ-desthio	0.01	97; 97; 102	99	2.9	0.01
		0.10	93	-	-	
		Overall (n = 4)		**97**	**3.8**	
	5-hydroxy-PTZ-desthio	0.01	100; 97; 101	99	2.1	0.01
		0.10	95	-	-	
		Overall (n = 4)		**98**	**2.8**	
	6-hydroxy-PTZ-desthio	0.01	96; 94; 101	97	3.7	0.01
		0.10	102	-	-	
		Overall (n = 4)		**98**	**3.9**	
Beer	α-hydroxy-PTZ-desthio	0.01	92;95;96	94	2.2	0.01
	3-hydroxy-PTZ-desthio	0.01	95; 100; 97	97	2.6	0.01
	4-hydroxy-PTZ-desthio	0.01	93; 95; 95	94	1.2	0.01
	5-hydroxy-PTZ-desthio	0.01	91;93;95	93	2.2	0.01
	6-hydroxy-PTZ-desthio	0.01	97; 101; 100	99	2.1	0.01
Brewer's yeast	α-hydroxy-PTZ-desthio	0.01	93	-	-	0.01
	3-hydroxy-PTZ-desthio	0.01	93	-	-	0.01
	4-hydroxy-PTZ-desthio	0.01	92	-	-	0.01
	5-hydroxy-PTZ-desthio	0.01	93	-	-	0.01
	6-hydroxy-PTZ-desthio	0.01	97	-	-	0.01
Brewer's grain	α-hydroxy-PTZ-desthio	0.01	97	-	-	0.01
	3-hydroxy-PTZ-desthio	0.01	97	-	-	0.01
	4-hydroxy-PTZ-desthio	0.01	94	-	-	0.01
	5-hydroxy-PTZ-desthio	0.01	96	-	-	0.01
	6-hydroxy-PTZ-desthio	0.01	97	-	-	0.01
Brewer's malt	α-hydroxy-PTZ-desthio	0.01	99	-	-	0.01
	3-hydroxy-PTZ-desthio	0.01	98	-	-	0.01
	4-hydroxy-PTZ-desthio	0.01	92	-	-	0.01
	5-hydroxy-PTZ-desthio	0.01	95	-	-	0.01
	6-hydroxy-PTZ-desthio	0.01	95	-	-	0.01

RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
PTZ: prothioconazole * These recoveries were performed during the conduct of studies RA-2618/07 and RA-2619/07.
** The individual hydroxy metabolites are fortified and determined as themselves and calculated as prothioconazole-desthio.

The residues of prothioconazole-desthio and its hydroxy metabolites determined in the barley raw agricultural commodity and in the barley processed fractions are shown in Table 6.5.3-9.
In both trials, since all residues were below the limit of quantification both in the raw agricultural commodity and in the processed fraction, no processing factor could be derived.

Table 6.5.3-9: Residue levels of prothioconazole-desthio and its metabolites in barley commodities

Trial	Analyte	Barley Grain (RAC)	Grain for processing	Brewer's malt	Beer	Brewer's grain	Brewer's yeast
R 2007 0781/9	Prothioconazole-desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
	α-hydroxy-PTZ -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
	3-hydroxy-PTZ -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
	4-hydroxy-PTZ -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
	5-hydroxy-PTZ -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
	6-hydroxy-PTZ -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
R 2007 0782/7	Prothioconazole -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
	α-hydroxy-PTZ -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
	3-hydroxy-PTZ -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
	4-hydroxy-PTZ -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
	5-hydroxy-PTZ -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
	6-hydroxy-PTZ -desthio	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010

RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
RAC: raw agricultural commodity; PTZ: prothioconazole. The individual hydroxy metabolites are determined as themselves and calculated as prothioconazole-desthio.

Conclusion

A barley processing study was conducted using barley grain from two field trials in which the crop was treated with prothioconazole at the normal GAP rate. The field and analytical phases were conducted under GLP but not the processing phase. The residues of prothioconazole-desthio and its hydroxy metabolites were measured in grain, brewer's malt, brewer's grain, brewer's yeast and beer. In both trials,

since the residues were below the limit of quantification both in the raw agricultural commodity and in the processed fraction, no processing factor could be derived.

Report:	KCA 6.5.3/04[redacted],[redacted];[redacted]; 2008; M-304125-01-1
Title:	Determination of the residues of fluoxastrobin and JAU 6476 in/on winter barley grain and grain for processing and the processed fractions (brewer's malt; beer; brewer's grain; brewer's yeast) and spring barley grain and grain for processin
Report No.:	RA-3062/07
Document No.:	M-304125-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Processing studies were performed in France in order to determine the residue transfer of prothioconazole and its desthio- metabolite from winter and spring barley into brewer's malt, brewer's grain, yeast and beer generated during the brewing of barley grain from field trials conducted with an EC formulation containing 50 g/L fluoxastrobin and 100 g/L of prothioconazole. Only results for prothioconazole-desthio will be presented in this dossier.
Information on the field phase of the study may be found in the report. The processing phase was not conducted under GLP and is not described in the reports. Therefore, the study is only submitted as supplementary information and the processing factors derived for barley brewing are only considered indicative.

Material and methods

Two field trials were conducted in the northern part of France during the 2007 growing season to generate barley grain for a processing study. The crop received two spray applications with an emulsifiable concentrate (EC) containing 100 g/L of prothioconazole. Both applications were conducted at the recommended label use rate of 175 g a.s./ha. The first application was carried out at the growth stage BBCH 37 (flag leaf just visible, still rolled) and the second application at the growth stage BBCH 61 (beginning of flowering). The interval between the two applications was in the range of 13-17 days. The barley grain was harvested at maturity (BBCH 89) 53-63 days after the last application and stored frozen until processing. The design of the field trials is summarised in Table 6.5.3-10.

Table 6.5.3-10: Field trials conducted to generate barley samples for processing – overview of trial design

Study Trial No. Year	Crop Variety	Location	Application of prothioconazole				
			FL	No	g a.s./ha	g as/hL	GS
RA-3062/07 R 2007 0779/7 2007	Winter barley Vanessa	France [redacted]	EC 150 g/L	2	175	58	61
RA-3062/07 R 2007 0780/0 2007	Spring barley Scarlet	France [redacted]	EC 150 g/L	2	175	58	61

FL: Formulation; GS : BBCH growth stage at last application

The processing phase (barley brewing) was carried out at IFBM (French Institute for Brewing and Malting) using 31 kg samples of barley grain from each trial. The processed commodities generated during this phase consisted of brewer's malt, brewer's grain, brewer's yeast and beer. Since the processing was not conducted under GLP, it is not described in the study reports and no more detailed information on the brewing procedure is available.

The raw agricultural commodity (barley grain) and the barley processed fractions (brewer's malt, brewer's grain, brewer's yeast and beer were analysed for the residues of prothioconazole-desthio according to the method 01013 (cf. KCA 4.1.2). The residues were extracted from the samples with acetonitrile/water (4/1; v/v, containing cysteine hydrochloride) using a blender. After filtration of the extract, the stable isotopically labeled analytes was added. The solution was made up to volume, diluted and subjected to reversed phase HPLC-MS/MS without a further clean-up step. The limit of quantification (LOQ), defined as the lowest validated fortification level, was 0.010 mg/kg for prothioconazole-desthio in all the investigated matrices.
The unprocessed barley grain samples and the various fractions from barley processing were stored deep-frozen for a maximum of 238 days before analysis.

Findings

Table 6.5.3-11 gives an overview of the concurrent recoveries for the determination of prothioconazole-desthio in barley grain and brewer's malt. The average recoveries per analyte and matrix were within the acceptable range of 70-110% and the corresponding RSDs were below 20%.

Table 6.5.3-11: RA-3062/07: Concurrent recoveries for the determination of prothioconazole-desthio in brewer's malt and barley grain

Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
		Single Values	Mean		
Barley grain	0.01	85	85	-	0.01
	0.20	82	82	-	
	Overall (n = 2)		**84**	**-**	
Barley grain for processing	0.01	91; 85	88	-	0.01
	0.20	84;86	85	-	

Sample Material	Fortification level [mg/kg]	Recovery rates [%]*		RSD [%]	LOQ [mg/kg]
		Single Values	Mean		
	Overall (n = 4)		**87**	**3.6**	
Brewer's malt**	0.01	89;86	88	-	0.01
	0.20	89;86	88	-	
	Overall (n = 4)		**88**	**2.0**	

RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
Fortified with prothioconazole-desthio, determined as prothioconazole-desthio and calculated as prothioconazole-desthio
*These recoveries were performed during the conduct of the studies RA-3062/07 and RA-3669/07
** = recoveries for barley beer, barley brewer's grain, barley brewer's yeast are covered by recoveries for brewer's malt.

The residues of prothioconazole-desthio determined in the barley raw agricultural commodity and in the barley processed fractions are shown in Table 6.5.3-12.
In both trials, since the residues were below the limit of quantification both in the raw agricultural commodity and in the processed fraction, no processing factor could be derived.

Table 6.5.3-12: Residue levels of prothioconazole-desthio in barley commodities

Commodity	Residues (mg/kg)*	
	Trial R 2007 0779/7	**Trial R 2007 0780/0**
	Prothioconazole-desthio	**Prothioconazole-desthio**
Barley grain (RAC)	< 0.010	< 0.010
Grain for processing	< 0.010	< 0.010
Brewer's malt	< 0.010	< 0.010
Beer	< 0.010	< 0.010
Brewer's grain	< 0.010	< 0.010
Brewer's yeast	< 0.010	< 0.010

RAC : raw agricultural commodity

Conclusion

A barley processing study was conducted using barley grain from two field trials in which the crop was treated with prothioconazole at the normal GAP rate. The field and analytical phases were conducted under GLP but not the processing phase. The residues of prothioconazole-desthio were measured in grain, brewer's malt, brewer's grain, brewer's yeast and beer. In both trials, since the residues were below the limit of quantification both in the raw agricultural commodity and in the processed fraction, no processing factor could be derived.

-Wheat

Report:	KCA 6.5.3/05 [redacted]; 2004; M-000665-01-1
Title:	JAU 6476 480 SC - Magnitude of the residue in/on wheat grain, wheat aspirated grain fractions, and wheat processed commodities
Report No.:	200521
Document No.:	M-000665-01-1
Guideline(s):	EPA Ref.: OPPTS 860.1500, Crop Field Trials OPPTS 860.1520, Processed Food/Feed PMRA Ref.: DACO 7.4.1, Supervised Residue Trial Study DACO 7.4.5, Processed Food/Feed
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Material and methods

A field trial was conducted in the USA (Kansas) during the 2000 growing season to generate wheat grain for a processing study. The crop received two spray applications with a suspension concentrate (SC) containing 480 g/L of prothioconazole. The first application was conducted at the rate of 632 g a.s./ha when the crop had reached the growth stage BBCH 57 (70% of inflorescence emerged). The second application was conducted 11 days later, at the rate of 1010 g a.s./ha and at the growth stage BBCH 69 (end of flowering). The total applied rate was about five-fold (5X) the maximum recommended US label use rate. The wheat grain was harvested at maturity (BBCH 89) 47 days after the last application and stored deep frozen for 12.4 months until processing. The design of the field trial is summarised in Table 6.5.3-13.

Table 6.5.3-13: Field trial conducted to generate wheat samples for processing – overview of trial design

Study Trial No. Year	Crop Variety	Location	Application of prothioconazole				
			FL	No	g a.s./ha	g as/hL	GS
J619WH02 J6065-00P 2000	Winter wheat Karl 92	USA [redacted]	SC 480 g/L	2	632-1010	658-1060	69

FL: Formulation; GS: BBCH growth stage at last application

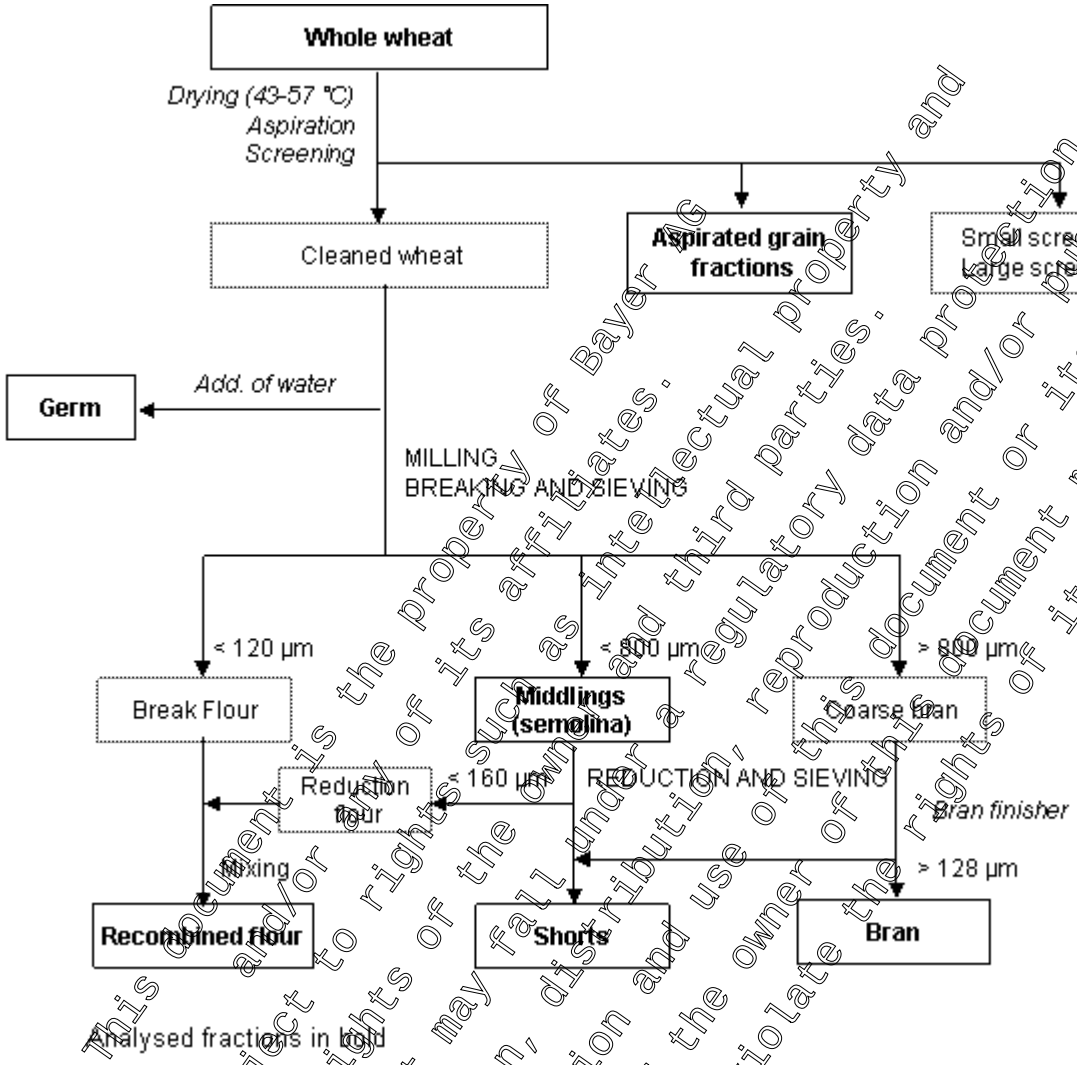
The processing was conducted at a laboratory scale in a way that simulated industrial practice as closely as possible. A simplified flowchart of the procedure is shown in Figure 6.5.4-5. About 260 kg of wheat grain were dried (43-57°C) and cleaned by aspiration of light impurities followed by screening. A 68 kg portion of cleaned wheat grain was used for germ production. The grain was moisture adjusted to 16% and then passed in two different mills in order to separate the bran and the endosperm. The procedure yielded 320 g of germ. A 9.1 kg portion of cleaned wheat grain was used flour milling. The grain was

moisture adjusted to 16.5-17.5%, passed through break rolls and screened to separate break flour (< 120 µm), middlings (< 800 µm) and coarse bran (> 800 µm). The middlings were passed through reduction rolls and screened to separate reduction flour (< 160 µm) and shorts (> 160 µm). The coarse bran was passed in a finisher t o produce shorts (< 128 µm) and bran (> 128 µm). The break flour and the reduction flour were combined to produce flour. The shorts obtained from middlings and the shorts obtained from bran were also combined to produce shorts.

The raw agricultural commodity (wheat grain) and selected wheat processed fractions (aspirated grain fractions, bran, flour, germ, middlings, and shorts) were analysed for the residues of prothioconazole-desthio according to method RPA JA/03/01 (please refer to section KCA 4.1.2). Residues of parent prothioconazole were also determined but for sake of clarity, are not presented in this summary as it is not relevant for the EU. Only results for prothioconazole-desthio will be presented.The analytical procedure involves extraction with with a mixture methanol (MeOH), 30% hydrogen peroxide (H_2O_2), and aqueous sodium bicarbonate ($NaHCO_3$) at about 64°C for 2 hours. Following the addition of a mixture of isotopically labelled prothioconazole-desthio internal standard, the sample extracts are purified by octadecyl solid phase extraction (C-18 SPE). The purified analyte is analysed by high performance liquid chromatography-electrospray/tandem mass spectrometry (LC-MS/MS).
The limit of quantification (LOQ) for prothioconazole-desthio was 0.02 mg/kg in all the analysed wheat commodities except aspirated grain fractions (0.25 mg/kg).

The wheat grain samples were stored deep-frozen for a maximum of 1285 days between sampling in the field and analysis. The various fractions from wheat processing were stored deep-frozen for a maximum of 909 days before analysis.

Figure 6.5.3-4: Processing of wheat – simplified flow chart



Findings

Table 6.5.3-14 gives an overview of the method validation data and the concurrent recoveries for the determination of prothioconazole-desthio. The average recoveries were within the acceptable range of 70-110% and the corresponding RSDs were below 20%.

Table 6.5.3-14: Validation data and concurrent recoveries for the determination of prothioconazole-desthio in wheat grain and wheat processed commodities

Report	Sample Material	Fortification level [mg/kg]	Recovery rates [%]		RSD [%]	LOQ [mg/kg]
			Single Values	Mean		
M-000665-01-1 (200521)	Wheat grain	0.02	116	-	-	0.02
		0.20	108, 108, 109	108	0.5	
		Overall (n = 4)		**110**	**3.5**	
	Aspirated grain fractions	0.25	98, 98, 102	99	2.3	0.25
		12.5	97, 96, 99	97	1.6	
		Overall (n = 6)		**98**	**2.1**	
	Bran	0.02	104, 103, 103	103	0.6	0.02
		0.20	105, 105, 106	105	0.5	
		Overall (n = 6)		**104**	**1.2**	
	Flour	0.02	101, 103, 104	103	1.5	0.02
		0.20	107, 106, 106	106	0.5	
		Overall (n = 6)		**105**	**2.2**	
	Germ	0.02	90, 96, 98	95	4.4	0.02
		0.20	100, 100, 101	100	0.6	
		Overall (n = 6)		**98**	**4.2**	
	Middlings	0.02	102, 100, 102	101	1.1	0.02
		0.20	106, 104, 110	107	2.9	
		Overall (n = 6)		**104**	**3.4**	
	Shorts	0.02	103, 101, 104	103	1.5	0.02
		0.20	111, 108, 101	107	4.8	
		Overall (n = 6)		**105**	**3.9**	

RSD: Relative Standard Deviation, LOQ: Limit of Quantitation
Note : The above table shows both the recovery rates from method validation and the concurrent recoveries determined alongside sample analysis.

The residues of prothioconazole-desthio determined in the wheat raw agricultural commodity and in the wheat processed fractions are shown in Table 6.5.3-15. The table also provides estimates of the respective processing factors.

The raw agricultural commodity (wheat grain) was found to contain measurable residues of prothioconazole-desthio (0.048 mg/kg). The residues concentrated in aspirated grain fractions , bran, germ and shorts, and were present at lower levels than in the raw agricultural commodity in flour and middlings.

Table 6.5.3-15: Residue levels and processing factors for prothioconazole-desthio in wheat commodities

Commodity	Residues (mg/kg)*	Processing factors
Grain (RAC)	0.047	N/A
Aspirated grain fractions	10.25	218
Bran	0.114	2.4
Flour	<0.02	0.3
Germ	0.101	2.1
Middlings	0.029	0.6
Shorts	0.050	1

RAC : raw agricultural commodity N/A : not applicable * average of triplicate

Conclusion

A wheat processing study was conducted using wheat grain from a field trial involving a five-fold exaggerated application rate of prothioconazole. The residues of prothioconazole-desthio were measured in grain, aspirated grain fractions, bran, flour, germ, middlings and shorts. The raw agricultural commodity (wheat grain) was found to contain measurable residues of prothioconazole-desthio (0.047 mg/kg). The residues concentrated in aspirated grain fractions , bran, germ and shorts, and were present at lower levels than in the raw agricultural commodity in flour and middlings.

CA 6.6 Residues in rotational crops

CA 6.6.1 Metabolism in rotational crops

Two confined rotational crop studies were conducted in wheat, Swiss chard and turnips using phenyl- and triazole-labelled parent compound. The study using phenyl-labelled prothioconazole was summarised in the EU Basic Dossier submitted in March 2002 and was deemed acceptable following evaluation and peer review at EU level (2007). For completeness, an executive summary of this study is provided below. An additional study using triazole-labelled prothioconazole was conducted in 2004 based on requests of EPA (USA). This study has also been evaluated by JMPR in 2008 and was added to the amended Annex II Tier 2 summary of the Section 4, Points IIA 6.2 and 6.6.2 (reference M-301848-01-1).

Report: KCA 6.6.1/01 [redacted]; 2001; M-049955-01-1
Title: Confined rotational crop study with JAU6476
Report No.: MR-159/00
Document No.: M-049955-01-1
Guideline(s): US EPA OPPTS 860.1850; EU 91/414/EEC (Final 11/97)
Guideline deviation(s): not specified
GLP/GEP: **yes**

Executive Summary

The metabolism of the prothioconazole was investigated in rotational crops (spring wheat, Swiss chard and turnips). Bare soil was treated with [phenyl-UL-^{14}C]prothioconazole dissolved in pure acetonitrile. The amount applied corresponded to the maximum annual field rate of 660 g a.s./ha. Crops of the first, second and third rotation were sown at day 28, 146 and 269, respectively. Seven raw agricultural commodities (RACs) were sampled, sampling dates (days after application) and total radioactive residues (TRRs, expressed as mg/kg a.s. equivalents) are given below:

	1st rotation		2nd rotation		3rd rotation	
	Sampling day	TRR (mg/kg)	Sampling day	TRR (mg/kg)	Sampling day	TRR (mg/kg)
Swiss chard	80	0.039	188	0.055	348	0.021
Turnip leaves	94	0.046	202	0.028	349	0.036
Turnip roots	94	0.043	201	0.031	349	0.015
Wheat forage	73	0.021	178	0.061	327	0.040
Wheat hay	111	0.114	211	0.435	372	0.166
Wheat straw	145	0.450	269	0.507	417	0.192
Wheat grain	145	0.007	269	n.d.	412	n.d.

n.d.: not determined , because already TRR value of rotation one was below the trigger value of 0.01 mg/kg

With the exception of wheat hay, total radioactive residue (TRR) levels in the different RACs either declined or remained at a constant but low level during the test period. The maximum concentration (0.450 mg/kg) was determined for wheat straw in the first rotation. The TRR observed for grain (0.007 mg/kg) was below the threshold value (0.01 mg/kg) for metabolite identification, hence grain samples of rotation II and III were discarded.

In general, with the exception of Swiss chard of rotation 3 and grain of rotation 1, more than 70% of the TRR was extracted using acetonitrile/water (80:20) and if necessary, subsequent Accelerated Solvent Extraction. In addition, hay of rotation 3 and straw of all rotations were exhaustively extracted using dioxan/HCl (9:1, v/v) under reflux.

More than 20 metabolites were detected in the different RACs, 16 of which were already known from the metabolism studies performed for spring wheat and peanuts. Prothioconazole-desthio was the main metabolite in all crops of rotation 1 (up to 20.8% of the TRR in Swiss chard). Obviously, prothioconazole-desthio was degraded further in soil and plants as higher amounts of derivatives (e.g. mono- and dihydroxylated metabolites and glucoside conjugates thereof) were found in all rotations.

In the subsequent rotations additional highly polar metabolites were detected, which were probably formed in soil and taken up by the plants. All of those metabolites were detected in amounts well below the identification threshold of 0.01 mg/kg.

The quantitative distribution of prothioconazole and its metabolites detected in rotational crops after application of prothioconazole (phenyl- and triazole-label) to soil - in mg/kg equivalents a.s. - is provided in Tables 6.6.2-3 to 6.6.2- 9 below.

Report:	KCA 6.6.1/02[redacted]し;[redacted]; 2004; M-000784-01-1
Title:	The accumulation of [triazole-3,5-14C] JAU6476 in confined rotational crops
Report No.:	200623
Document No.:	M-000784-01-1
Guideline(s):	OPPTS 860.1850; DACO 7.4.3
Guideline deviation(s):	--
GLP/GEP:	**yes**

Executive Summary

The nature and accumulation of [triazole-3,5-^{14}C] prothioconazole residues was studied in confined rotational crops following four applications to soil in a large trough at an average rate of 204 g a.s./ha/application. Rotational crops, wheat (small grain), Swiss chard (leafy vegetable), and turnips (root crop), were planted at 30, 125, and 366 days after treatment (DAT). Raw agricultural commodities (RACs) were harvested from each crop during each time interval.

The total radioactive residues (TRR) ranged from 0.059 mg/kg in turnip roots to 3.806 mg/kg in wheat grain for the 30-DAT crop samples from 0.047 mg/kg in Swiss chard to 4.136 mg/kg in wheat grain for the 125-DAT samples, and from 0.062 mg/kg in turnip roots to 5.875 mg/kg in wheat grain for the 366-DAT samples.

The major residues found in all rotational crop matrices were triazolylalanine (TA), triazolylhydroxypropionic acid (THPA), and triazolylacetic acid (TAA). TA represented 44% to 51% (0.120 mg/kg to 0.252 mg/kg) of the TRR in wheat forage, 33% to 36% (0.719 mg/kg to 0.846 mg/kg) in wheat hay, 15% to 28% (0.197 mg/kg to 0.401 mg/kg) in wheat straw, 58% to 68% (2.264 mg/kg to 3.940 mg/kg) in wheat grain, 49% to 56% (0.023 mg/kg to 0.096 mg/kg) in Swiss chard, 74% to 92% (0.077 mg/kg to 0.377 mg/kg) in turnip tops, and 81% to 93% (0.048 mg/kg to 0.411 mg/kg) in turnip roots.

THPA represented 32% to 35% (0.087 mg/kg to 0.184 mg/kg) of the TRR in wheat forage, 24% to 39% (0.562 mg/kg to 0.871 mg/kg) in wheat hay, 28% to 33% (0.382 mg/kg to 0.498 mg/kg) in wheat straw, <1% to 1% (0.023 mg/kg to 0.047 mg/kg) in wheat grain, 30% to 32% (0.038 mg/kg to 0.060 mg/kg) in Swiss chard, 7% (0.035 mg/kg) in turnip tops (at 125-DAT), and 1 to 5% (0.003 mg/kg to 0.005 mg/kg at 30-DAT and 125-DAT) in turnip roots.

TAA represented 1% to 6% (0.003 mg/kg to 0.034 mg/kg) of the TRR in wheat forage, 10% to 22% (0.222 mg/kg to 0.578 mg/kg) in wheat hay, 15% to 20% (0.233 mg/kg to 0.437 mg/kg) in wheat straw, 23% to 38% (0.958 mg/kg to 1.485 mg/kg) in wheat grain, 1% to <18% (0.001 mg/kg to <0.008 mg/kg) in Swiss chard, 2% to <7% (<0.009 mg/kg or less) in turnip tops, and <5% (<0.005 mg/kg) in turnip roots.

The minor residues found in the rotational crops were JAU6476-desthio, <1% to 4% (0.001 mg/kg to 0.020 mg/kg) of the TRR in all matrices except wheat grain; triazolylethanol-glucoside, <1% to 5% (0.001 mg/kg to 0.003 mg/kg) in all matrices except wheat grain and Swiss chard; triazolyl-ethanol, <1% to 7% (0.001 mg/kg to 0.030 mg/kg) in all matrices except wheat grain; and JAU6476-a-hydroxy-desthio, 1% to 2% (0.002 mg/kg to 0.026 mg/kg) in all matrices except wheat grain and Swiss chard. No prothioconazole was detected in any matrix at any planting interval.

A total of 94% to 100% of the residues in all matrices were extractable. The identified residues represented 72% to 96% of the TRRs in all matrices.

Overall summary of the metabolism of prothioconazole in confined rotational crops

A comparison of the two studies on the behaviour of phenyl- and triazole-labelled prothioconazole in confined rotational crops after application to soil shows that the studies are based on different designs, different sites and different analytical methods. Differences in the results of these studies can be attributed to these facts.

In both studies the metabolism of prothioconazole was investigated in the same confined rotational crops (wheat, Swiss chard and turnips) at three rotations after application to bare soil. In the study using phenyl-labelled prothioconazole, the parent compound was applied once at an application rate of 578 g a.s./ha. Crops of the first, second and third rotation were sown at day 28, 149 and 269, respectively. In contrast, the metabolism of triazole-labelled prothioconazole was investigated in confined rotational crops following four applications to the soil at an average rate of 204 g a.s./ha per application. The rotational crops were planted at 30, 125, and 366 days after treatment.

Additionally the growing conditions were different in the two studies. The study using the phenyl-label was performed in the green house where the plants were protected from rainfall and the soil was irrigated. In the study using the triazole-label the plant tubs were placed outside under natural weather conditions including rainfall. The different application scenarios are summarised in **Table 6.6.2- 1**.

Table 6.6.2- 1: Confined rotational crop studies: Application scenarios of phenyl- and triazole-labelled prothioconazole after spray application to soil

Label	Application rate (g a.s./ha)	planting of rotation I / II / III (days after the last application)	Growing conditions
Phenyl	1 x 578	28 / 149 / 269[a]	greenhouse
Triazole	204, 213, 201 and 193 (total: 815)	30 / 125 / 366[b]	outside

a) = days after treatment

b) = days after the 4th and final treatment

The total radioactive residue values found in the different matrices of the rotational crops after application of phenyl- and triazole-labelled prothioconazole are summarised in Table 6.6.2- 2.

Table 6.6.2- 2: Comparison of the total radioactive residue (TRR) in rotational crop matrices following soil treatment with phenyl- and triazole-labelled prothioconazole

Label		Rotation 1 TRR (mg/kg) phenyl	Rotation 1 TRR (mg/kg) triazole	Ratio triazole / phenyl	Rotation 2 TRR (mg/kg) phenyl	Rotation 2 TRR (mg/kg) triazole	Ratio triazole / phenyl	Rotation 3 TRR (mg/kg) phenyl	Rotation 3 TRR (mg/kg) triazole	Ratio triazole / phenyl
Wheat	forage	0.021	0.251	12	0.062	0.575	9	0.040	0.439	11
	hay	0.151	2.224	15	0.135	2.580	19	0.160	2.016	13
	straw	0.430	1.695	4	0.307	1.361	4	0.312	1.597	5
	grain	0.007	3.808	544	n.d.	4.136	-	n.d.	5.875	-
Swiss chard	leaves	0.038	0.188	5	0.053	0.047	5	0.021	0.129	6
Turnip	tops	0.060	1.131	19	0.028	0.507	18	0.036	0.084	2
	roots	0.043	0.059	1	0.031	0.442	14	0.015	0.061	4

The results of the two confined rotational crop studies revealed that prothioconazole was intensively metabolised.

In the study using phenyl-labelled prothioconazole the unchanged parent compound represented –if detected at all- only < 0.005 mg/kg or ≤ 1% of the TRR. The main metabolite JAU 6476-desthio *(M04)* was detected in all RACs and amounted up to 0.045 mg/kg in wheat straw of the 1st rotation. The hydroxy metabolites *(M14-M17)* also occurred in the rotational crop species. Furthermore, conjugation played an important role in the degradation of prothioconazole. The occurrence of some new but very minor polar metabolites (no single component > 0.05 mg/kg) indicates that the composition of the TRR in rotational crops was also influenced by additional soil metabolites of prothioconazole.

In the study using the triazole-labelled prothioconazole the major metabolites found in all matrices were triazolylalanine (TA, *M31*), triazolylhydroxypropionic acid (THP, *M30)* and triazolylacetic acid (TAA, *M29*). No free 1,2,4-triazole was detected in any matrix. This strongly supports the assumption that 1,2,4-triazole (M13) was taken up from the soil and immediately conjugated with serine to form triazolylalanine and the subsequent metabolites. Major metabolites detected in most matrices were JAU 6476-desthio *(M04)*, JAU 6476-desthio-α-hydroxy *(M18)*, JAU 6476-triazolyl-ethanol *(M15)*, and JAU 6476-triazolyl-ethanol-glucoside *(M19)*. Prothioconazole was not detected in any matrix at any plant-back interval. Due to the high concentrations of the label specific metabolites in the study with triazole-labelled prothioconazole, detection and identification of minor metabolites common to both labels received less attention since their levels were lower than both identification triggers (< 10% of TRR and < 0.05 mg/kg).

A comparison of prothioconazole and its metabolites detected in rotational crops after application of prothioconazole (phenyl- and triazole-label) to soil - in mg/kg equivalents a.s. - is provided in Tables 6.6.2- 3 to 6.6.2- 9. The proposed metabolic pathway of prothioconazole in the rotational crops is shown in Figure 6.6.2- 2.

Table 6.6.2- 3: Distribution of the active substance and metabolites in wheat forage of rotation 1 to 3 from phenyl- and triazole-labelled prothioconazole confined rotational crop studies (mg a.s. equivalents/kg)

Parent compound / metabolite	Rotation 1		Rotation 2		Rotation 3	
label	phenyl	triazole	phenyl	triazole	phenyl	triazole
Metabolites common to both labels:						
JAU 6476-sulfonic acid *(M02)*	< 0.001		0.002			
JAU 6476-triazolinone *(M03)*	< 0.001		< 0.001		< 0.001	
JAU 6476-disulfide *(M11)*	< 0.001		< 0.001		< 0.001	
JAU 6476-desthio *(M04)*	0.003		0.001	0.001	< 0.001	
JAU 6476-desthio-3-hydroxy *(M14)*	0.001		< 0.001		< 0.001	
JAU 6476-desthio-4-hydroxy *(M15)*	< 0.001		< 0.001			
JAU 6476-desthio-6-hydroxy *(M17)*			< 0.001			
JAU 6476-desthio-hydroxy-glucoside *(M21-23)*	0.001		0.001			
JAU 6476-desthio-α-hydroxy *(M18)*	< 0.001		< 0.001	0.004	< 0.001	
JAU 6476-desthio-α-acetoxy *(M19)*	< 0.001		0.002		< 0.001	
JAU 6476-desthio-dihydroxy-olefin-glucoside *(M64)*	0.003		0.010		0.017	
Metabolite specific to the phenyl-label:						
JAU 6476-benzylpropyldiol-glucoside *(M43)*	< 0.001		< 0.001			
Metabolites specific to the triazole-label:						
THP *(M30)*		0.087		0.084		0.155
Σ: TAA *(M29)* and THP *(M30)*				0.003		
TA *(M31)*		0.180		0.252		0.225
TAA *(M29)*		0.008		0.004		0.006
JAU 6476-triazolyl-ethanol *(M45)*		0.003		0.007		
JAU 6476-triazolyl-ethanol-glucoside *(M46)*		0.004		0.015		0.003
Total identified	0.010	0.222	0.040	0.508	0.021	0.389
Characterised	0.004	0.020	0.003	0.015	0.002	0.042
Unextracted	0.004	0.010	0.003	0.015	0.011	0.006

Table 6.6.2- 4: Distribution of the active substance and metabolites in wheat hay of rotation 1 to 3 from phenyl- and triazole-labelled prothioconazole confined rotational crop studies (mg a.s. equivalents/kg)

Parent compound / metabolite	Rotation 1		Rotation 2		Rotation 3	
label	phenyl	triazole	phenyl	triazole	phenyl	triazole
Metabolites common to both labels:						
JAU 6476-sulfonic acid *(M02)*	0.013		0.009			
JAU 6476-triazolinone *(M03)*	0.001		0.001			
JAU 6476-disulfide *(M11)*	0.002		0.002		0.001	
JAU 6476-desthio *(M04)*	0.014	0.020	0.016	0.020	0.032[a)]	
JAU 6476-desthio-3-hydroxy *(M14)*	0.003		0.001		0.002	
JAU 6476-desthio-4-hydroxy *(M15)*	0.004		0.002		0.003	
JAU 6476-desthio-hydroxy-glucoside *(M21-23)*	0.011		0.007		0.010	
JAU 6476-desthio-α-hydroxy *(M18)*	0.004	0.019	0.002	0.023	0.004	
JAU 6476-desthio-α-acetoxy *(M19)*	0.002		0.003		0.006	
JAU 6476-desthio-dihydroxy-olefin-glucoside *(M64)*	0.020		0.039		0.053	
Metabolites specific to the triazole-label:						
TA *(M31)*		0.720		0.846		0.719
THP *(M30)*		0.871		0.627		0.562
TAA *(M29)*		0.222		0.578		0.441
JAU 6476-triazolyl-ethanol *(M45)*		0.030		0.029		0.021
JAU 6476-triazolyl-ethanol-glucoside *(M46)*		0.045		0.060		0.030
Total identified	0.073	1.908	0.082	2.179	0.111	1.773
Additional characterised	0.006	0.238	0.009	0.329	0.016	0.068
Unextracted	0.020	0.076	0.029	0.011	0.009[a)]	0.045

a) = after treatment with dioxane/HCl (9:1)

Table 6.6.2- 5: Distribution of the active substance and metabolites in wheat straw of rotation 1 to 3 from phenyl- and triazole-labelled prothioconazole confined rotational crop studies (mg a.s. equivalents/kg)

Parent compound / metabolite label	Rotation 1		Rotation 2		Rotation 3	
	phenyl	triazole	phenyl	triazole	phenyl	triazole
Prothioconazole	0.004		0.001			
Metabolites common to both labels:						
JAU 6476-sulfonic acid *(M02)*	0.002		0.024		0.040	
JAU 6476-triazolinone *(M03)*	0.004		0.002		0.001	
JAU 6476-disulfide *(M11)*	0.007		0.0[illegible]			
JAU 6476-desthio *(M04)*	0.045	0.014	0.[illegible]6		0.0[illegible]	
JAU 6476-desthio-3-hydroxy *(M14)*	0.017		0.004		0.003	
JAU 6476-desthio-4-hydroxy *(M15)*	0.0[illegible]		0.004		0.007	
JAU 6476-desthio-6-hydroxy *(M17)*	0.007		0.00[illegible]			
JAU 6476-desthio-hydroxy-glucoside *(M21-23)*	0.097		0.[illegible]43		0.02[illegible]	
JAU 6476-desthio-α-hydroxy *(M18)*	0.030	0.007	0.008	0.0[illegible]	0.009	0.016
JAU 6476-desthio-α-acetoxy *(M19)*	0.0[illegible]		0.01[illegible]		0.009	
JAU 6476-desthio-dihydroxy-olefin-glucoside *(M54)*	0.055		0.044		0.05[illegible]	
Metabolites specific to the phenyl-label:						
JAU 6476-benzylpropyldiol *(M09)*	0.01[illegible]		0.002			
JAU 6476-benzylpropyldiol-glucoside *(M10)*					0.003	
Metabolites specific to the triazole-label:						
TA *(M31)*		0.3[illegible]		0.197		0.407
THP *(M30)*		0.[illegible]98		0.[illegible]		0.481
TAA *(M29)*		0.437		0.233		0.302
Σ: TAA *(M29)* and THP *(M30)*				0.041		
JAU 6476-triazolyl-ethanol *(M45)*		0.023		0.02[illegible]		
JAU 6476-triazolyl-ethanol-glucoside *(M46)*		0.042		0.0[illegible]		0.029
Total identified	0.[illegible]9	0.389	0.1[illegible]0	0.[illegible]89	0.171	1.235
Additional characterised	0.076	0.267	0.[illegible]3	0.290	0.100	0.192
Unextracted	0.010[illegible]	0.0[illegible]	0.020[a)]	0.011	0.007[a)]	0.006

a) = unextracted (solids after dioxan hydrolysis)

Table 6.6.2- 6: Distribution of the active substance and metabolites in wheat grain of rotation 1 to 3 from phenyl- and triazole-labelled prothioconazole confined rotational crop studies (mg a.s. equivalents/kg)

Parent compound / metabolite label	Rotation 1		Rotation 2		Rotation 3	
	phenyl	triazole	phenyl[a)]	triazole	phenyl[a)]	triazole
Metabolites common to both labels:						
JAU 6476-desthio *(M04)*	< 0.001					
JAU 6476-desthio-3-hydroxy *(M14)*	< 0.001					
JAU 6476-desthio-4-hydroxy *(M15)*						
JAU 6476-desthio-α-hydroxy *(M18)*	0.001					
Metabolites specific to the triazole-label:						
TA *(M31)*		2.264		2.33		3.940
THP *(M30)*		0.047		0.023		
TAA *(M29)*		1.116		0.957		1.235
Total identified	0.001	3.427		3.351		5.425
Additional characterised	0.004	0.285		0.1		0.285
Unextracted	0.003	0.011		0.021		0.014

a) = grain sample material of rotation 2 and 3 was not analysed, because TRR value of rotation 1 was already well below the trigger value of 0.01 mg/kg

Table 6.6.2- 7: Distribution of the active substance and metabolites in Swiss chard of rotation 1 to 3 from phenyl- and triazole-labelled prothioconazole confined rotational crop studies (mg a.s. equivalents/kg)

Parent compound / metabolite label	Rotation 1		Rotation 2		Rotation 3	
	phenyl	triazole	phenyl	triazole	phenyl	triazole
Prothioconazole			< 0.001			
Metabolites common to both labels:						
JAU 6476-sulfonic acid *(M02)*	< 0.001		0.001			
JAU 6476-triazolinone *(M03)*	0.001		0.001		< 0.001	
JAU 6476-disulfide *(M11)*	0.001		0.001		< 0.001	
JAU 6476-desthio *(M04)*	0.014	0.005	0.010	0.001	< 0.001	
JAU 6476-desthio-3-hydroxy *(M14)*	0.003		0.002		< 0.001	
JAU 6476-desthio-4-hydroxy *(M15)*	< 0.001		< 0.001			
JAU 6476-desthio-hydroxy-glucoside *(M21-23)*	0.004		0.002		0.002	
JAU 6476-desthio-α-hydroxy *(M18)*	< 0.001		< 0.001		< 0.001	
JAU 6476-desthio-dihydroxy-olefin-glucoside *(M64)*	0.007		0.006		0.004	
Metabolite specific to the phenyl-label:						
JAU 6476-benzylpropyldiol *(M09)*			< 0.001		< 0.001	
Metabolites specific to the triazole-label:						
TA *(M31)*		0.096		0.028		0.072
THP *(M30)*		0.060				0.038
Σ: TAA *(M29)* and THP *(M30)*				0.008		
TAA *(M29)*						0.001
JAU 6476-triazolyl-ethanol *(M45)*		0.014		0.001		0.002
Total identified	0.028	0.174	0.025	0.034	0.007	0.113
Additional characterised	0.005	0.008	0.014	0.001	0.005	
Unextracted	0.005	0.007	0.008	0.005	0.008	0.002

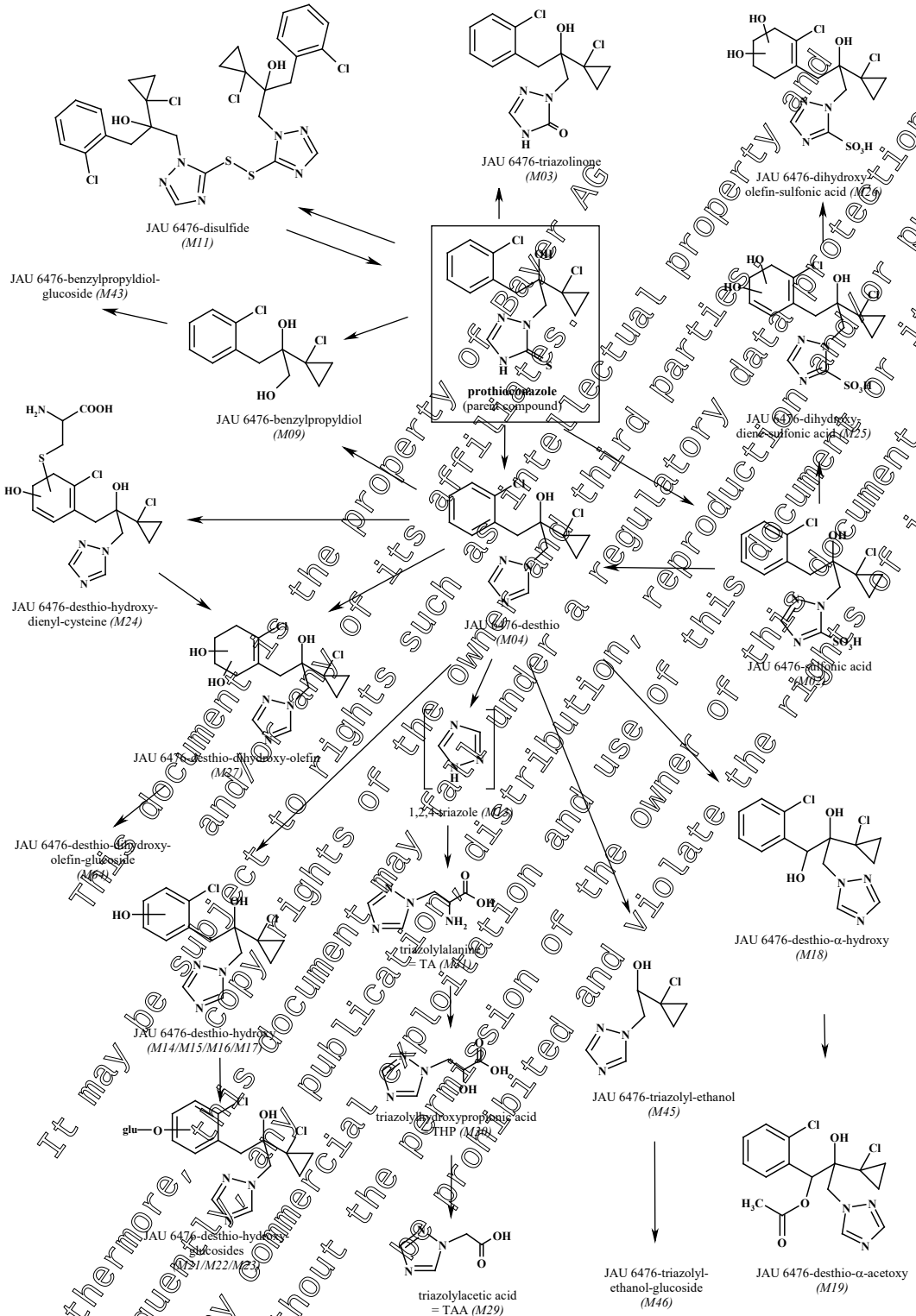
Table 6.6.2- 8: Distribution of the active substance and metabolites in turnip tops of rotation 1 to 3 from phenyl- and triazole-labelled prothioconazole confined rotational crop studies (mg a.s. equivalents/kg)

Parent compound / metabolite label	Rotation 1		Rotation 2		Rotation 3	
	phenyl	triazole	phenyl	triazole	phenyl	triazole
Prothioconazole			< 0.001			
Metabolites common to both labels:						
JAU 6476-sulfonic acid *(M02)*	< 0.001					
JAU 6476-triazolinone *(M03)*	0.001		< 0.001			
JAU 6476-disulfide *(M11)*			< 0.001		0.001	
JAU 6476-desthio *(M04)*	0.008		0.002	0.005	0.001	
JAU 6476-desthio-3-hydroxy *(M14)*	0.001		< 0.001			
JAU 6476-desthio-4-hydroxy *(M15)*	0.002		0.001		0.001	
JAU 6476-desthio-6-hydroxy *(M17)*	0.001		< 0.001		< 0.001	
JAU 6476-desthio-hydroxy-glucoside *(M21-23)*	0.009		0.004		0.002	
JAU 6476-desthio-α-hydroxy *(M18)*	0.003	0.002	0.002	0.003	0.001	
JAU 6476-desthio-dihydroxy-olefin-glucoside *(M64)*	0.009		0.006		0.006	
Metabolites specific to the phenyl-label:						
JAU 6476-benzylpropyldiol *(M09)*	< 0.001					
JAU 6476-benzylpropyldiol-glucoside *(M10)*	0.004		0.001			
Metabolites specific to the triazole-label:						
TA *(M31)*		0.106		0.377		0.077
THP *(M30)*				0.035		
Σ: TAA *(M29)* and THP *(M30)*		0.009				
TAA *(M29)*				0.009		
JAU 6476-triazolyl-ethanol *(M45)*		0.004		0.020		
JAU 6476-triazolyl-ethanol-glucoside *(M46)*		0.004		0.011		
Total identified	0.037	0.119	0.016	0.468	0.018	0.077
Additional characterised	0.002	0.005	0.006	0.020	0.009	
Unextracted	0.008	0.005	0.006	0.020	0.009	0.003

Table 6.6.2- 9: Distribution of the active substance and metabolites in turnip roots of rotation 1 to 3 from phenyl- and triazole-labelled prothioconazole confined rotational crop studies (mg/kg a.s. equivalents/kg)

Parent compound / metabolite label	Rotation 1		Rotation 2		Rotation 3	
	phenyl	triazole	phenyl	triazole	phenyl	triazole
Prothioconazole	< 0.001					
Metabolites common to both labels:						
JAU 6476-triazolinone *(M03)*	< 0.001		< 0.001		< 0.001	
JAU 6476-disulfide *(M11)*			0.005		< 0.001	
JAU 6476-desthio *(M04)*	0.009	0.002	0.009	0.007	0.005	< 0.001
JAU 6476-desthio-3-hydroxy *(M14)*	0.005		< 0.001		< 0.001	
JAU 6476-desthio-4-hydroxy *(M15)*			< 0.001		< 0.001	
JAU 6476-desthio-6-hydroxy *(M17)*	< 0.001		< 0.001		< 0.001	
JAU 6476-desthio-hydroxy-glucoside *(M21-23)*	0.006		0.002		0.002	
JAU 6476-desthio-α-hydroxy *(M18)*	0.004		0.002	0.002	< 0.001	
JAU 6476-desthio-dihydroxy-olefin-glucoside *(M64)*	0.005		0.001		0.001	
Metabolites specific to the phenyl-label:						
JAU 6476-benzylpropyldiol *(M09)*	0.004		< 0.001			
JAU 6476-benzylpropyldiol-glucoside *(M41)*	0.011					
Metabolites specific to the triazole-label:						
Triazolylalanine = TA *(M31)*		0.003		0.41		0.052
Σ: TAA *(M29)* and THP *(M30)*		0.003		0.03		
JAU 6476-triazolyl-ethanol *(M45)*				0.002		
JAU 6476-triazolyl-ethanol-glucoside *(M46)*				0.001		
Total identified	0.031	0.053	0.017	0.45	0.009	0.053
Additional characterised	0.004	0.001	0.005	0.002	0.002	
Unextracted	0.008	0.003	0.008	0.012	0.003	0.002

Figure 6.6.2- 2: Proposed metabolic pathway of prothioconazole in rotational crops



Comparison of the metabolism of prothioconazole in target plants and confined rotational crops

The metabolism in rotational crops is qualitatively identical to that in the target plants peanuts, sugar beet and wheat, as the same metabolic routes were detected. It appears that conjugation is an important step in the metabolism. A smaller number of conjugates was detected in the confined rotational crop studies than in the target plant metabolism studies, but in total these conjugates represented a higher percentage of the respective TRR's.

Irrespective of the crop or application mode, the major metabolites found in all crops were JAU 6476-desthio *(M04)*, triazolylalanine (TA, *M31*), triazolylhydroxypropionic acid (THPA, *M30*) and triazolylacetic acid (TAA, *M29*). Based on the results of the studies with the triazole-label it can be assumed that 1,2,4-triazole *(M13)* was taken up from the soil and immediately transformed in the plants to the metabolites triazolylalanine (TA, *M31*), triazolylhydroxypropionic acid (THPA, *M30*) and triazolylacetic acid (TAA, *M29*). No free 1,2,4-triazole was detected in any matrix, neither in the confined rotational crops study nor in the target plant metabolism studies.

Hydroxylation was the major metabolic process of prothioconazole in plants, as described before (Section 6.2.1), prothioconazole has potentially five carbons (four on the phenyl ring and one at the benzylic position) that could undergo hydroxylation. Consequently, the majority of these metabolites are simply multiple structural isomers of monohydroxylated JAU 6476-desthio and their conjugates.

In summary, the metabolic pattern in rotational crops is similar to that observed in primary crops. The residue definitions proposed for primary crops are evenly valid. Under the experimental conditions of the confined rotational crop studies, residues of prothioconazole-desthio were present in edible part of Swiss chard and turnip at a level of 0.01 mg/kg, when these plants were sown 28 and 146 days after application of the highest possible annual dose of prothioconazole on bare soil. The total amount of all metabolites containing the prothioconazole-desthio common structural moiety amounted to ca. 0.03 mg/kg for the same time intervals. Therefore, under practical conditions of use of prothioconazole according to the representative uses and considering that a fraction of the applied amount is intercepted by the target crop, no residue of any metabolite above 0.01 mg/kg is expected in rotational crops and no plant back restriction needs to be proposed.

The proposed metabolic pathway of prothioconazole summarising the metabolism in the various crops, including rotational crops, is shown in Figure 6.2.1- 1.

CA 6.6.2 Magnitude of residues in rotational crops

New residue data for prothioconazole in rotational crops are available from field rotational crop trials conducted in/on carrot & turnip (root crop), lettuce (leafy crop) and barley (cereal).
As these studies were performed with the aim to address the assessment of consumer exposure to triazole metabolites derivatives (TDMs) in rotational crops, prothioconazole-desthio was also determined but the hydroxy metabolites of prothioconazole-desthio were not.
Indeed, in the confined rotational crop study, it was concluded that prothioconazole residue levels in food and feed rotational commodities are expected to be covered by the residue levels in primary crops.
In this dossier, only the results of prothioconazole-desthio are presented. The results of these studies for the TDMs as well as data and information about the common triazole metabolites resulting from the various triazole active substances were submitted in March 2011, June 2012 and July 2013 by the

industry task force TDMG (Triazole Derivative Metabolites Group) for an assessment by UK CRD as the RMS.

– Carrot & Turnip, Lettuce, Barley

Report:	KCA 6.6.2/01 [redacted]; [redacted]; 2012; M-426697-01-1
Title:	Determination of the residues of prothioconazole in/on the field rotational crops carrot, lettuce, spring barley and winter barley after either a single application of JAU 6476 EC 250 on bare soil or sowing of spring wheat treated with JAU 6476 FS 100 followed by three spray applications with JAU 6476 EC 250 in the field in Germany
Report No.:	09-2500
Document No.:	M-426697-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22) OECD Guideline for testing of Chemicals; Residues in rotational crops (limited field studies), No. 504, 8 Jan. 2007
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.6.2/02 [redacted]; 2012; M-426699-01-1
Title:	Determination of the residues of prothioconazole in/on the field rotational crops carrot, lettuce, spring barley and winter barley after either a single application of JAU 6476 EC 250 on bare soil or sowing of spring wheat treated with JAU 6476 FS 100 followed by three spray applications with JAU 6476 EC 250 in the field in the Netherlands
Report No.:	09-2501
Document No.:	M-426699-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.6.2/03 [redacted]; 2012; M-426710-01-1
Title:	Determination of the residues of prothioconazole in/on the field rotational crops turnip, lettuce, spring barley and winter barley after either a single application of JAU 6476 EC 250 on bare soil or sowing of winter wheat treated with JAU 6476 FS 100 followed by spray application with JAU 6476 EC 250 in the field in southern France
Report No.:	09-2502
Document No.:	M-426710-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22) OECD Guideline for testing of Chemicals; Residues in rotational crops (limited field studies), No. 504, 8 Jan. 2007
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.6.2/04[redacted],[redacted]; 2012; M-426705-01-1
Title:	Determination of the residues of prothioconazole in/on the field rotational crops carrot, lettuce and winter barley after either a single application of JAU 6476 EC 250 on bare soil or sowing of winter wheat treated with JAU 6476 FS 100 followed by three spray applications with JAU 6476 EC 250 in the field in Spain
Report No.:	09-2503
Document No.:	M-426705-01-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22) OECD Guideline for testing of Chemicals, Residues in rotational crops (limited field studies), No. 504, 8 Jan. 2007
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

The reports 09-2500, 09-2501, 09-2502 and 09-2503 describe field rotational crop studies with prothioconazole that were conducted at four different sites (one site per study and report). At each site the potential uptake of residues after use of prothioconazole in the preceding crop was investigated for three types of rotational crops (root crops, leafy crops and cereals) and for three standard plant-back intervals (20-35 days, 60-200 days and 270-365 days). In these studies, the following prothioconazole-derived residues were measured: prothioconazole-desthio, 1,2,4-triazole, triazole acetic acid, triazole alanine and triazole lactic acid. For the sake of clarity, only the results of prothioconazole-desthio will be presented below.

Material and methods

Supervised field trials to investigate the residues in rotational crops after application of prothioconazole were conducted at four test sites located in Germany, the Netherlands, the southern part of France and Spain. The critical GAP to be investigated was defined as a maximum seasonal application rate of 630 g a.s./ha. This rate is achieved in grain cereals (wheat and rye) if seed treatment at the rate of 15 g as/dt and a seeding rate of 200 kg seed/ha is followed by 3 spray treatments at the rate of 200 g a.s./ha. At each test site three ranges of plant-back intervals (20-35 days, 60-200 days and 270-365 days) and three crop groups (root crops represented by turnip and carrot, leafy crops represented by lettuce, cereals represented by barley) were investigated. The design of the trials is summarised in Table 6.6.2-1.

Plant back interval of 20-35 days

In order to simulate an emergency crop rotation in case of a crop failure, the EC formulation containing 250 g/L of prothioconazole was applied to bare soil at the nominal rate of 2.52 L/ha (equivalent to 630 g a.s./ha of prothioconazole). The rotational crops (turnip/carrot, lettuce and barley) were sown or planted on separate plots 21-34 days after the application.

Plant back interval of 60-200 days

In order to simulate normal crop rotation wheat seed was treated at a nominal rate of 15 g as/dt with the FS formulation containing 100 g/L of prothioconazole. The seed was sown at a nominal rate of 200 kg seed/ha and the wheat plants received 3 spray treatments at the rate of 200 g a.s./ha with the EC formulation containing 250 g/L of prothioconazole. The treatments were conducted at the growth stages BBCH 32, BBCH 39 and BBCH 65-69, respectively, with intervals of 7-30 days between subsequent treatments. At harvest the wheat straw was ploughed in (so as to simulate a worst case scenario in terms of residues) and the plots were left bare until the rotational crops were sown or planted (*i.e.* no cover crop was sown).

In order to determine the residues in rotational crops after a short crop rotation, the rotational crops (turnip/carrot, lettuce and barley) were sown or planted on separate subplots about 0.3 to 5 months after the harvest of wheat. The plant back-intervals with respect to the last test item application were 56-129 days for carrot/turnip and lettuce, and 190-200 days for barley. However, on the Dutch test site the carrots were destroyed by wild geese. Therefore no samples of carrot root and leaves could be taken for the short crop rotation scenario.

In order to determine the residues in rotational crops after an annual crop rotation, the rotational crops (turnip/carrot, lettuce and barley) were sown or planted on separate subplots about 7 to 10 months after the harvest of wheat. The plant back-intervals with respect to the last test item application were 296-345 days for carrot/turnip and lettuce, and 277-296 days for barley.

Sampling

Samples of carrot or turnip root and leaf were collected about 14 days prior to main harvest (BBCH 47-48) and at main harvest (BBCH 49). Samples of lettuce head were collected about 14 days prior to main harvest (BBCH 41-49) and at main harvest (BBCH 49). Samples of barley plant (green material) were taken at the growth stage BBCH 29-32 while samples of barley grain and straw were collected at the growth stage BBCH 89 (mature harvest).

Table 6.6.2-1: Application summary of residue trials conducted on rotational crops following the use of FS and EC formulations containing 100 g/L and 250 g/L of prothioconazole

Study No. Trial No. Plot No. Year	Crop Variety	Country	Application of prothioconazole					PBI (days)
			FI	No	g a.s./ha	g as/hL	GS	
Plant-back interval of 20-35 days								
09-2500 09-2500-01 2009	Carrot Cestos F1	Germany	EC (250 g/L)	1 (to bare soil)	630	210	-	26
09-2500 09-2500-02 2009	Lettuce Argentinas	Germany	EC (250 g/L)	1 (to bare soil)	630	210	-	29
09-2500 09-2500-03 2009	Winter barley Laverda	Germany	EC (250 g/L)	1 (to bare soil)	630	210	-	25
09-2501 09-2501-01 2009	Carrot Nerac F1	Netherlands	EC (250 g/L)	1 (to bare soil)	630	210	-	25

Study No. Trial No. Plot No. Year	Crop Variety	Country	Application of prothioconazole					PBI (days)
			FL	No	g a.s./ha	g as/hL	GS	
09-2501 09-2501-02 2009	Lettuce Butter head	Netherlands	EC (250 g/L)	1 (to bare soil)	630	210	-	25
09-2501 09-2501-03 2009	Winter barley Naomie	Netherlands	EC (250 g/L)	1 (to bare soil)	630	158	-	25
09-2502 09-2502-01 2009	Turnip Rubin	Southern France	EC (250 g/L)	1 (to bare soil)	630	210	-	21
09-2502 09-2502-02 2009	Lettuce Belino	Southern France	EC (250 g/L)	1 (to bare soil)	630	210	-	27
09-2502 09-2502-03 2009	Winter barley Orelie	Southern France	EC (250 g/L)	1 (to bare soil)	630	210	-	21
09-2503 09-2503-01 2009	Carrot Nantesa 5 Coral	Spain	EC (250 g/L)	1 (to bare soil)	630	210	-	34
09-2503 09-2503-02 2009	Lettuce Arena tipo Maravilla	Spain	EC (250 g/L)	1 (to bare soil)	630	210	-	34
09-2503 09-2503-03 2009	Winter barley Graphic	Spain	EC (250 g/L)	1 (to bare soil)	630	210	-	28

FL: Formulation; GS : BBCH growth stage at last application PBI : plant back interval
[#] Corresponds to an application rate of 15 g as/dt with a seeding rate of 200 kg seed/ha.
* Deviation from study plan. The intended plant-back intervals were 60-150 days for carrot and lettuce and 120-200 days for winter barley.

Table 6.6.2-1 (cont'd): Application summary of residue trials conducted on rotational crops following the use of FS and EC formulations containing 100 g/L and 250 g/L of prothioconazole

Study No. Trial No. Plot No. Year	Crop Variety	Country	Application of prothioconazole					PBI (days)
			FL	No	g a.s./ha	g as/hL	GS	
Plant-back interval of 60-200 days								
09-2500 09-2500-04-2A 2009	Carrot Nantes Fancy	Germany	FS (100 g/L)	1 (to wheat seed)	30#	-	-	56*
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2500 09-2500-04-2B 2009	Lettuce Argentinos	Germany	FS (100 g/L)	1 (to wheat seed)	30#	-	-	56*
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2500 09-2500-04-2C 2009	Winter barley Laverda	Germany	FS (100 g/L)	1 (to wheat seed)	30#	-	-	93*
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2501 09-2501-04-2A 2009	Carrot Napoli	Netherlands	FS (100 g/L)	1 (to wheat seed)	30#	-	-	60
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2501 09-2501-04-2B 2009	Lettuce Gisela	Netherlands	FS (100 g/L)	1 (to wheat seed)	30#	-	-	60
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2501 09-2501-04-2C 2009	Winter barley Naomie	Netherlands	FS (100 g/L)	1 (to wheat seed)	30#	-	-	90
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2502 09-2502-04-2A 2009	Turnip Rubin	Southern France	FS (100 g/L)	1 (to wheat seed)	30#	-	-	129
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2502 09-2502-04-2B 2009	Lettuce Belino	Southern France	FS (100 g/L)	1 (to wheat seed)	30#	-	-	122
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2502 09-2502-04-2C 2009	Winter barley Orelie	Southern France	FS (100 g/L)	1 (to wheat seed)	30#	-	-	129
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2503 09-2503-04-2A 2009	Carrot Nantesa 5 Coral	Spain	FS (100 g/L)	1 (to wheat seed)	30#	-	-	95
			EC (250 g/L)	3 (to wheat plant)	200	67	65	
09-2503 09-2503-04-2B 2009	Lettuce Arena tipo Maravilla	Spain	FS (100 g/L)	1 (to wheat seed)	30#	-	-	95
			EC (250 g/L)	3 (to wheat plant)	200	67	65	
09-2503 09-2503-04-2C 2009	Winter barley Graphic	Spain	FS (100 g/L)	1 (to wheat seed)	30#	-	-	200
			EC (250 g/L)	3 (to wheat plant)	200	67	65	

FL: Formulation; GS: BBCH growth stage at last application PBI : plant back interval

Corresponds to an application rate of 15 g as/dt with a seeding rate of 200 kg seed/ha.

* Deviation from study plan. The intended plant-back intervals were 60-150 days for carrot and lettuce and 120-200 days for winter barley.

Table 6.6.2-1 (cont'd): Application summary of residue trials conducted on rotational crops following the use of FS and EC formulations containing 100 g/L and 250 g/L of prothioconazole

Study No. Trial No. Plot No. Year	Crop Variety	Country	Application of prothioconazole					PBI (days)
			FL	No	g a.s./ha	g as/hL	GS	
Plant-back interval of 270-365 days								
09-2500 09-2500-04-3A 2009	Carrot Cestas F1	Germany	FS (100 g/L)	1 (to wheat seed)	30#	-	-	313
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2500 09-2500-04-3B 2009	Lettuce Argentinas	Germany	FS (100 g/L)	1 (to wheat seed)	30#	-	-	315
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2500 09-2500-04-3C 2009	Spring barley Simba	Germany	FS (100 g/L)	1 (to wheat seed)	30#	-	-	293
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2501 09-2501-04-3A 2009	Carrot Nerja	Netherlands	FS (100 g/L)	1 (to wheat seed)	30#	-	-	345
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2501 09-2501-04-3B 2009	Lettuce Gisela	Netherlands	FS (100 g/L)	1 (to wheat seed)	30#	-	-	343
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2501 09-2501-04-3C 2009	Spring barley Tipple	Netherlands	FS (100 g/L)	1 (to wheat seed)	30#	-	-	277
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2502 09-2502-04-3A 2009	Turnip Oceanic	Southern France	FS (100 g/L)	1 (to wheat seed)	30#	-	-	296
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2502 09-2502-04-3B 2009	Lettuce Belhegarde Q110	Southern France	FS (100 g/L)	1 (to wheat seed)	30#	-	-	298
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2502 09-2502-04-3C 2009	Spring barley Prestige	Southern France	FS (100 g/L)	1 (to wheat seed)	30#	-	-	296
			EC (250 g/L)	3 (to wheat plant)	200	67	69	
09-2503 09-2503-04-3A 2009	Carrot Nantesa 5 Coral	Spain	FS (100 g/L)	1 (to wheat seed)	30#	-	-	311
			EC (250 g/L)	3 (to wheat plant)	200	67	65	
09-2503 09-2503-04-3B 2009	Lettuce Murai tipo Lollo Rossa	Spain	FS (100 g/L)	1 (to wheat seed)	30#	-	-	329
			EC (250 g/L)	3 (to wheat plant)	200	67	65	
09-2503 09-2503-04-3C 2009	Winter barley Graphic	Spain	FS (100 g/L)	1 (to wheat seed)	30#	-	-	279
			EC (250 g/L)	3 (to wheat plant)	200	67	65	

FL: Formulation; GS : BBCH growth stage at last application PBI : plant back interval
Corresponds to an application rate of 15 g as/dt with a seeding rate of 200 kg seed/ha.
* Deviation from study plan. The intended plant-back intervals were 60-150 days for carrot and lettuce and 120-200 days for winter barley.

The residues of prothioconazole-desthio were analysed according to method 01013 (cf. Point KCA 4.1.2). The residues were extracted from samples with acetonitrile/water (4/1, v/v) by

high-speed blending. After filtration of the extract, the stable isotopically labeled analyte was added. The solution was made up to volume, diluted and subjected to reversed phase HPLC-MS/MS without a further clean-up step. Prothioconazole-desthio was detected using electrospray ionization in the positive ion mode (ESI+). The limit of quantification (LOQ), defined as the lowest validated fortification level, was 0.010 mg/kg for all matrices.

Findings

- Recoveries:

An overview of the recovery rates for prothioconazole-desthio in the analysed plant matrices is given in Table 6.6.2-2. If a signal in the unfortified sample was present, the recoveries were background corrected by subtracting the signal of the unfortified sample from the signal of the fortified sample. After background correction (if appropriate) the average recoveries per matrix were within the acceptable range of 70-110%. The RSDs were below 20%.

Table 6.6.2-2: Recovery data for prothioconazole-desthio in/on rotational crops

Sample Material	FL [mg/kg]	Single Values [%]	Mean Value [%]	RSD [%]	LOQ [mg/kg]
Carrot leaf	0.01	82 95 101 107 116	100	12.8	0.01
	0.10	94 95 96 100 105 107 107 111	102	6.3	
		Overall Recovery (n = 13)	**101**	**8.8**	
Carrot root	0.01	92 93 109 109 111 116	105	9.5	0.01
	0.10	104 112 112	109	4.2	
		Overall Recovery (n = 9)	**106**	**8.0**	
Turnip leaf	0.01	100 107 115	107	7.0	0.01
	0.10	97 101 102	100	2.6	
	2.5	92 93	93	-	
		Overall Recovery (n = 8)	**101**	**7.4**	
Edible turnip tuber body	0.01	102 103 104	103	1.0	0.01
	0.10	96 105 106	102	5.4	
	2.5	98 104	101	-	
		Overall Recovery (n = 8)	**102**	**3.4**	
Lettuce head	0.01	89 94 100 101 104 105 105 106	101	6.0	0.01
	0.10	99 100 102 105 107	103	3.3	
		Overall Recovery (n = 13)	**101**	**5.1**	
Barley green material	0.01	92 120	106	-	0.01
	0.10	90 91 92	91	1.1	
		Overall Recovery (n = 5)	**97**	**13.3**	
Barley grain	0.01	93 94 97 98	96	2.5	0.01
	0.10	98 98 99 99	99	0.6	
		Overall Recovery (n = 8)	**97**	**2.3**	

FL = fortification level; RSD = relative standard deviation; n = number of tests; LOQ = limit of quantification
Determined as prothioconazole-desthio and calculated as prothioconazole-desthio.

- Storage stability:

The maximum storage periods of deep-frozen samples before determination of prothioconazole-desthio residues are described in the table below:

Table 6.6.2-3 Maximum storage period of samples from rotational crop trials

Crop	Compound	Sample material	Maximum storage period (months)	Duration Covered (months)	Study
Barley	Prothioconazole-desthio	Grain	13.6	36	M-426709-01-1
		Green material	20.3		M-426710-01-1
		Straw	13.6		M-426705-01-1
Carrot	Prothioconazole-desthio	Leaf	25.2	36	M-426705-01-1
		Root			
Lettuce	Prothioconazole-desthio	Head	26.8	36	M-426705-01-1
Turnip	Prothioconazole-desthio	Leaf	12.7	36	M-426710-01-1
		Body			

- Residue results:

No residues above the LOQ were found in the control samples. An overview of the residues measured in the samples of carrot, turnip, lettuce and barley planted as rotational crops is given in the tables below.

Table 6.6.2-4: Results of residue trials conducted on carrot or turnip grown as rotational crop following the use of FS and EC formulations containing 100 g/L and 250 g/L of prothioconazole

Carrot/turnip		Plant-back interval of 20-35 days			Plant-back interval of 60-200 days			Plant-back interval of 270-365 days		
Report No	Country Trial No	DALT (days)	Com-modity	PTZ-desthio[a] (mg/kg)	DALT (days)	Com-modity	PTZ-desthio[a] (mg/kg)	DALT (days)	Com-modity	PTZ-desthio[a] (mg/kg)
09-2500	Germany 09-2500-01 and 04	112	leaf	< 0.01	126	leaf	< 0.01	394	leaf	< 0.01
		112	root	< 0.01	126	root	< 0.01	394	root	< 0.01
		126	leaf	< 0.01	140	leaf	< 0.01	408	leaf	< 0.01
		126	root	< 0.01	140	root	< 0.01	408	root	< 0.01
09-2501	Netherlands 09-2501-01 and 04	130	leaf	< 0.01				402	leaf	< 0.01
		130	root	< 0.01				402	root	< 0.01
		144	leaf	< 0.01				416	leaf	< 0.01
		144	root	< 0.01				416	root	< 0.01
09-2502	France* 09-2502-01 and 04	134	leaf	< 0.01	242	leaf	< 0.01	352	leaf	< 0.01
		134	root	< 0.01	242	root	< 0.01	352	root	< 0.01
		147	leaf	< 0.01	255	leaf	< 0.01	366	leaf	< 0.01
		147	root	< 0.01	255	root	< 0.01	366	root	< 0.01
09-2503	Spain 09-2503-01 and 04	110	leaf	< 0.01	187	leaf	< 0.01	405	leaf	< 0.01
		110	root	< 0.01	187	root	< 0.01	405	root	< 0.01
		124	leaf	< 0.01	201	leaf	< 0.01	419	leaf	< 0.01
		124	root	< 0.01	201	root	< 0.01	419	root	< 0.01

DALT: Days after last treatment
* The carrots were destroyed by wild geese.
** This trial was conducted with turnip instead of carrots.
[a] determined as prothioconazole-desthio and calculated as prothioconazole-desthio.

Table 6.6.3-5: Results of residue trials conducted on lettuce grown as rotational crop following the use of FS and EC formulations containing 100 g/L and 250 g/L of prothioconazole

Lettuce		Plant-back interval of 20-35 days			Plant-back interval of 60-200 days			Plant-back interval of 270-365 days		
Report No	Country Trial No	DALT (days)	Com-modity	PTZ-desthio[a] (mg/kg)	DALT (days)	Com-modity	PTZ-desthio[a] (mg/kg)	DALT (days)	Com-modity	PTZ-desthio[a] (mg/kg)
09-2500	Germany 09-2500-02 and 04	67	head	< 0.01	98	head	< 0.01	362	head	< 0.01
		81	head	< 0.01	112	head	< 0.01	376	head	< 0.01
09-2501	Netherlands 09-2501-02 and 04	49	head	< 0.01	93	head	< 0.01	366	head	< 0.01
		63	head	< 0.01	107	head	< 0.01	380	head	< 0.01
09-2502	France 09-2502-02 and 04	74	head	< 0.01	169	head	< 0.01	346	head	< 0.01
		88	head	< 0.01	183	head	< 0.01	360	head	< 0.01
09-2503	Spain 09-2503-02 and 04	64	head	< 0.01	133	head	< 0.01	371	head	< 0.01
		78	head	< 0.01	148	head	< 0.01	385	head	< 0.01

DALT: Days after last treatment
[a] determined as prothioconazole-desthio and calculated as prothioconazole-desthio.

Table 6.6.3-6: Results of residue trials conducted on barley grown as rotational crop following the use of FS and EC formulations containing 100 g/L and 250 g/L of prothioconazole

Barley		Plant-back interval of 20-35 days			Plant-back interval of 60-200 days			Plant-back interval of 270-365 days		
Report No	Country Trial No	DALT (days)	Com-modity	PTZ-desthio[a] (mg/kg)	DALT (days)	Com-modity	PTZ-desthio[a] (mg/kg)	DALT (days)	Com-modity	PTZ-desthio[a] (mg/kg)
09-2500	Germany 09-2500-03 and 04	219	plant	< 0.01	287	plant	< 0.01	342	plant	< 0.01
		318	straw	< 0.01	386	straw	< 0.01	415	straw	< 0.01
		318	grain	< 0.01	386	grain	< 0.01	415	grain	< 0.01
09-2501	Netherlands 09-2501-03 and 04	230	plant	< 0.01	328	plant	< 0.01	353	plant	< 0.01
		306	straw	< 0.01	394	straw	< 0.01	402	straw	< 0.01
		306	grain	< 0.01	394	grain	< 0.01	402	grain	< 0.01
09-2502	France 09-2502-03 and 04	76	plant	< 0.01	184	plant	< 0.01	340	plant	< 0.01
		292	straw	< 0.01	400	straw	< 0.01	407	straw	< 0.01
		292	grain	< 0.01	400	grain	< 0.01	407	grain	< 0.01
09-2503	Spain 09-2503-03 and 04	126	plant	< 0.01	307	plant	< 0.01	334	plant	< 0.01
		234	straw	< 0.01	406	straw	< 0.01	408	straw	< 0.01
		234	grain	< 0.01	406	grain	< 0.01	408	grain	< 0.01

DALT: Days after last treatment
[a] determined as prothioconazole-desthio and calculated as prothioconazole-desthio.

Conclusion

Supervised field trials to investigate the residues in rotational crops after the use of FS and EC formulations containing 100 g/L and 250 g/L of prothioconazole were conducted at four test sites in Germany, the Netherlands, the southern part of France and Spain. At each test site three ranges of plant-back intervals (20-35 days, 60-200 days and 270-365 days) and three crop groups (root crops represented by turnip and carrot, leafy crops represented by lettuce and cereals represented by barley) were investigated. In the trials simulating a crop failure (emergency rotation) the EC formulation was applied once to bare soil at the rate of 630 g a.s./ha of prothioconazole. The rotational crops were sown or planted 21-34 days after the application. In the trials simulating a normal rotation the FS formulation was used to treat wheat seed at the rate of 15 g/as/dt. The seed was sown at a nominal rate of 200 kg seed/ha and the wheat plants received 3 spray treatments at the rate of 200 g a.s./ha with the EC formulation. The treatments were conducted at the growth stages BBCH 32, BBCH 39 and BBCH 65-69, respectively, with intervals of 7-30 days between subsequent treatments. At harvest the wheat straw was ploughed in and the plot was left bare until rotational crops were sown or planted. The plant-back intervals were variable depending on the crop and ranged between 56 and 200 days for the short crop rotation and between 277 and 345 days for the annual crop rotation. No residues above the LOQ were found in the control samples. At all samplings dates and for all matrices, no residue of prothioconazole-desthio was detected above 0.01 mg/kg (LOQ).

CA 6.7 Proposed residue definitions and maximum residue levels

CA 6.7.1 Proposed residue definitions

The metabolism of prothioconazole in plants, livestock and confined rotational crops was investigated using phenyl- and triazole-labelled parent compound.

An additional goat metabolism study with phenyl-labelled JAU 6476-desthio *(M04)* was conducted since this metabolite was determined as the dominating residue in feed items. Thus, animals are mainly exposed to this metabolite.

In the Annex II dossier of the basic EU Dossier of prothioconazole plant and animal metabolism studies were submitted in March 2002. The metabolism studies of prothioconazole in plant and animal matrices presented in this Annex II dossier were in the majority of cases conducted using the phenyl-labelled parent compound. Based on requests of US EPA additionally plant and livestock metabolism studies using the phenyl- and triazole-labelled prothioconazole were conducted after the dossier submission. For a general survey and a better comparability the corresponding parts of tier 2 summary of Chapter KCA 6.2 were renewed including all available metabolism studies.

The metabolic profile found after foliar application of prothioconazole in wheat, peanuts and sugar beets is very similar to the profile found after seed treatment of wheat and also similar to the one found in rotational crops after soil application. Therefore, the metabolism studies already conducted on wheat (foliar and seed treatment), peanuts (foliar) and sugar beet (foliar) and confined rotational crop studies (soil application followed by planting of wheat, Swiss chard and turnips) adequately describe the metabolism of prothioconazole in crops. These studies cover three different crop groups, i.e. cereals, oilseeds and root vegetables and employ two labelling positions.

The metabolic behaviour of prothioconazole in the farm animal studies with laying hens and lactating goat also corresponds very well, and is also almost identical with the metabolism in the rat.

Prothioconazole is extensively metabolised, with the main metabolites deriving directly from the intact parent compound molecule. Due to the conjugation with glucuronic acid, the sulfur in the triazolinethione moiety of prothioconazole is protected against cleavage and the metabolic routes via JAU 6476-desthio *(M04)* are impeded. Thus, only a less important branch of the metabolic pathway is based on metabolite JAU 6476-desthio *(M04)* as precursor. However, the metabolic mechanisms involved are identical for the parent compound or for metabolite JAU 6476-desthio *(M04)* as precursors. Hydroxylation, oxidative hydroxylation of the phenyl moiety and subsequent conjugation with glucuronic acid or sulfate are observed as major metabolic routes.

The metabolites detected in the goat metabolism study performed with JAU 6476-desthio were identical with the metabolites originating from JAU 6476-desthio *(M04)* in the goat metabolism studies conducted with prothioconazole as test substance. No additional metabolic routes or mechanisms were observed.

Thus, it can be concluded that the studies adequately describe the metabolism of prothioconazole in primary and rotational crops as well as in farm animals.

The results of all reported metabolism studies form the basis for the proposals of the residue definitions as discussed in the subsequent chapters.

The current residue definition for risk assessment and enforcement in plant and animal commodities given in the EFSA Scientific Report (2007) is summarised below.

Matrices	Residue definition		Reference
Food of plant origin	Risk assessment	Sum of prothioconazole-desthio and all metabolites containing the 2-(1-chlorocyclopropyl)-3-(2-chloro-phenyl)-2-hydroxypropyl-2H-1,2,4-triazole moiety) expressed as prothioconazole-desthio.	EFSA Scientific Report (2007) 106, 1-98
	Monitoring	Prothioconazole-desthio	
Food of animal origin	Risk assessment	Sum of prothioconazole-desthio and all metabolites containing the 2-(1-chlorocyclopropyl)-3-(2-chloro-phenyl)-2-hydroxypropyl-2H-1,2,4-triazole moiety) expressed as prothioconazole-desthio.	
	Monitoring	Sum of prothioconazole-desthio and its glucuronide conjugate, expressed as prothioconazole-desthio*	

* in EFSA Journal 2014; 12(5):3689, the enforcement residue definition is proposed as prothioconazole-desthio (sum of isomers) only.

Residue definition in plants for risk assessment

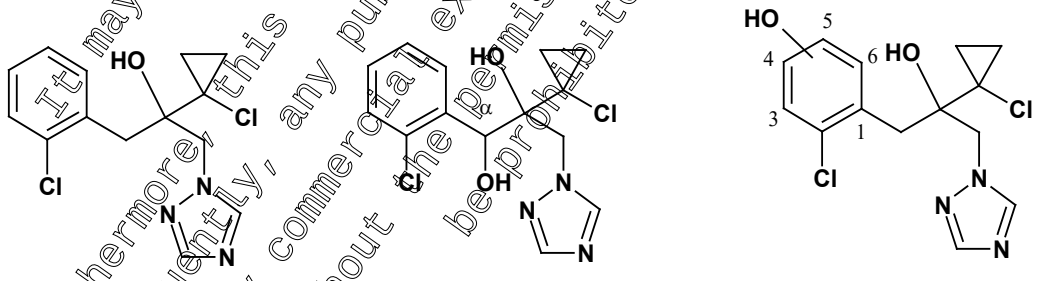
Report: KCA 6.7.1.01 [redacted], 2015; M-533058-01-1
Title: Prothioconazole: Risk assessment residue definition and conversion factors for plant matrices
Report No.: M-533058-01-1
Document No.: M-533058-01-1
Guideline(s): not specified
Guideline deviation(s): not specified
GLP/GEP: **no**

The prothioconazole residue definition for risk assessment in plant is the sum of prothioconazole-desthio and all metabolites containing the 2-(1-chlorocyclopropyl)-3-(2- chlorophenyl)-2-hydroxypropyl-2H-1,2,4-triazole moiety, expressed as prothioconazole-desthio.
Although it is a common moiety definition, it is not possible to develop a common moiety method. Indeed, the chemical structures of the different metabolites vary with the position of the (hydroxy-) substituent on the phenyl ring, or on the benzylic position (please refer to Figure 6.7-1). There is no chemical reaction (or set of reactions) that would allow the selective and complete cleavage of these hydroxy substituents. For this reason, all the analytes have to be determined separately.
As a result, since 2008, in all the residue trials performed with prothioconazole, 6 analytes have been determined: prothioconazole-desthio, 3-hydroxy-prothioconazole-desthio, 4-hydroxy-prothioconazole-desthio, 5-hydroxy-prothioconazole-desthio, 6-hydroxy-prothioconazole-desthio and alpha-hydroxy-prothioconazole-desthio (including all their acid-hydrolysable conjugates). This is because they represented the major portion of the TRR (Total Radioactive Residue) in the plant metabolism studies.

Following CRD's advice provided during the first pre-submission meeting to reduce the definition of residues for risk assessment in plants to a limited number of metabolites, BCS made a statistical analysis based on metabolism studies and residue data from 163 residue trials (where the 6 analytes were determined.

NB: At the time of this analysis, 163 trials (with foliar application) were available. These are trials where prothioconazole-desthio was determined, as well as the hydroxylated derivatives of prothioconazole-desthio (and their acid hydrolysable conjugates). The use patterns cover the current uses/GAPs defended in the EU. These trials were performed during 6 growing seasons (2008 to 2013) and on 7 different crops. Some of the studies presented in section KC 6.3 were not yet available at the time of the analysis so they were not included. They nevertheless would have not changed the conclusion, as their results are in agreement with the proposal.

Based on this statistical analysis, BCS wishes to propose a residue definition for risk assessment for prothioconazole as the sum of prothioconazole-desthio, 3-hydroxy-prothioconazole-desthio, 4-hydroxyprothioconazole- desthio, 5-hydroxy-prothioconazole-desthio, 6-hydroxy-prothioconazoledesthio and alpha-hydroxy-prothioconazole-desthio, expressed as prothioconazole-desthio.

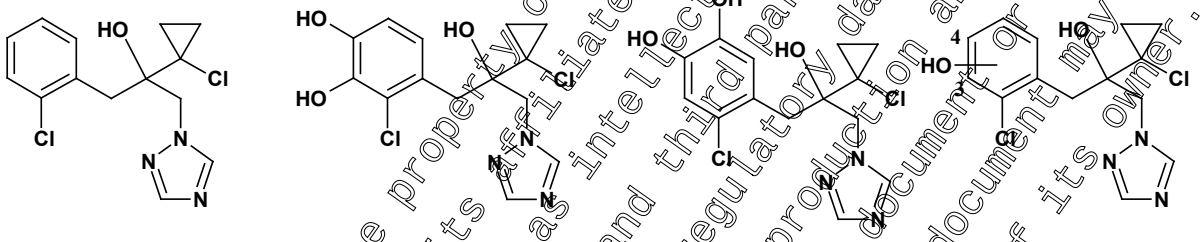


Prothioconazole-desthio Prothioconazole-α-hydroxy-desthio Prothioconazole-3-(or 4-;5-;6-)hydroxy-desthio

Figure 6.7-1

Residue definition in animal for risk assessment

As there is a proposal to reduce the definition of residue for risk assessment in plants (to a fixed number of metabolites), it was appropriate to make a similar proposal for risk assessment in animal commodities. Based on metabolism studies (see section KCA 6.2), the residue definition for risk assessment for prothioconazole in animal matrices could be either the sum of prothioconazole-desthio, 3-hydroxyprothioconazole- desthio, 4-hydroxy-prothioconazole-desthio and 3,4-dihydroxyprothioconazole- desthio and 4,5-dihydroxy-prothioconazole-desthio expressed as prothioconazole-desthio or as proposed by JMPR the sum of prothioconazole-desthio, 3-hydroxy-prothioconazole-desthio-, 4-hydroxy-prothioconazole-desthio-and their conjugates expressed as prothioconazole-desthio.



Prothioconazole-desthio | Prothioconazole-3,4-dihydroxy-desthio | Prothioconazole-4,5-dihydroxy-desthio | Prothioconazole-3-or 4-hydoxy-desthio

Figure 6.7-2

Residue definition in animal for enforcement

The residue definition for monitoring in animal commodities is the metabolite prothioconazole-desthio and its glucuronide conjugate, expressed as prothioconazole-desthio. Due to the fact that all attempts to synthesise prothioconazole-desthio-glucuronide failed it was not possible to develop a method (see section KCA 4.2). Based on the feeding study performed with prothioconazole-desthio and in particular the results in milk, Bayercropscience believes that prothioconazole-desthio is a valid marker for monitoring prothioconazole in all animal matrices including milk. Moreover, the current animal enforcement methods 0655/M002 and 01009 are suitable to monitor the relevant marker in animal commodities. Also, due to a very low transfer in milk, the residue defination for enforcement in animal products is proposed to be reduced to prothioconazole-desthio (sum of isomers) for all livestock matrices. This was supported by EFSA in their recent reasoned Opinion (Reasoned Opinion on the review of the existing MRLs for prothioconazole according to Article 12 of Regulation (EC) N° 396/2005 (EFSA Journal 2014; 2(5):3689).

CA 6.7.2 Proposed MRLs and justification of the acceptability of the levels proposed

According to Article 12 of Regulation (EC) No 396/2005, the European Food Safety Authority (EFSA) has reviewed the Maximum Residue Levels (MRLs) currently established at European level for the pesticide active substance prothioconazole. A reasoned opinion on the review of the existing maximum residue levels (MRLs) for prothioconazole was published in EFSA Journal 2014; 2(5):3689.

The most recent MRLs for prothioconazole are published in Annex III Part A of Regulation (EC) 834/2013. They will be replaced in spring 2016 following the review of the existing MRLs for prothioconazole according to Article 12 of Regulation (EC) N° 396/2005. Please refer to draft Regulation SANCO 11481/2014.
MRLs relevant to this submission are summarised in Table 6.7.2-1.

Table 6.7.2-1: EU MRLs set for the uses of prothioconazole

Crop/animal commodities	EU MRL (mg/kg) Reg. (EU) 834/2013	EU MRL (mg/kg) draft Regulation SANCO 11481/2014	STMR (mg/kg)	HR (mg/kg)
Barley	0.3	0.2	0.01	0.03
Oat	0.05	0.05		
Wheat (Spelt Triticale)	0.1	0.1	0.01	0.02
Rye	0.1	0.05		
Meat, fat of ruminants, swine and sheep	0.05	0.01*		0.01*
kidney, offal of ruminants, swine and sheep	0.5	0.5	-	0.01*
Liver of ruminant, swine	0.5	0.5	-	0.015
Liver of sheep	0.5	0.5	-	0.034
Meat, fat, liver and kidney of poultry	0.05	0.01*	-	0.01*
Milk	0.01*	0.01*	-	0.004*
Eggs	0.05	0.01*	-	0.01*

* indicates that the MRL is set at the LOQ-

As shown with the residue trials reported under KCA 6.3.1 and 6.3.2, when prothioconazole is applied according to the supported GAP, the residue levels of prothioconazole-desthio range from <0.01 to 0.02 mg/kg in wheat grain. In barley grain, the residue levels of prothioconazole-desthio range from <0.01 to 0.03 mg/kg.

Therefore, the existing MRL of 0.2 mg/kg on barley, 0.1 mg/kg on wheat and 0.05 mg/kg in rye and oat covers the new use detended in this dossier. No MRL needs to be changed.

CA 6.7.3 Proposed MRLs and justification of the acceptability of the levels proposed for imported products (import tolerance)

MRL settings based on imported products are not proposed with this dossier.

CA 6.8 Proposed safety intervals

It is not necessary to define a pre-harvest interval for the uses on cereals. Instead, the pre-harvest interval is given by the period between the latest growth stage for the last treatment and harvest. Further safety intervals are not needed.

CA 6.9 Estimation of the potential and actual exposure through diet and other sources

In order to evaluate the potential acute and chronic exposure to prothioconazole residues through the diet, the National Estimated Maximum Dietary Intakes (NEDI) and the National Estimated Short Term Intakes (NESTI) were calculated using the EFSA PRIMo model (revision 2) and the following toxicological endpoints.

Active substance	End-point	Value (mg/kg bw/day)		Study	Safety factor	Reference
Prothioconazole-desthio	Acceptable Daily Intake (ADI)	0.01		Oncogenicity study in rat	100	EFSA Scientific Report (2007) 106, 1-98
	Acute Reference Dose (ARfD)	Women of childbearing age	0.01	Developmental study in rat	100	
		General population	0.022	Rat 90-d tox	100	This dossier (Section 5)

Acceptable Daily Intake (ADI) and Dietary Exposure Calculation

NEDI calculations

Scenario 1

Some of the residue trials were analysed only for prothioconazole-desthio and do not provide data on other metabolites which are included in the risk assessment residue definition (sum of prothioconazole-desthio and all metabolites containing the 2-(1 chlorocyclopropyl)-3-(2-chlorophenyl)-2 hydroxypropyl-2H-1,2,4-triazole moiety). To address this issue, for cereal grain, an appropriate conversion factor of 2 between enforcement and risk assessment was derived by EFSA.

Scenario 2

Nevertheless, as a significant number of trials were determined for all 6 analytes, the "total prothioconazole" values were taken and no conversion factor was used.

Table 6.9-1 Input values for the consumer risk assessment (NEDI) of Prothioconazole

Commodity	Input value (mg/kg)	Comment	Reference
Barley	0.02	STMR x CF(2)	This dossier Scenario 1
Oat	0.02	STMR x CF(2)	
Wheat, Rye, (Triticale)	0.02	STMR x CF(2)	
Barley	0.06	STMR	This dossier Scenario 2
Oat	0.06	STMR	
Wheat, Rye, (Triticale)	0.06	STMR	
Meat	0.01	HR* x CF(1)	This dossier
Fat	0.01	HR* x CF(1)	
Bovine Liver	0.030	HR* x CF(2)	
Sheep liver	0.068	HR* x CF(2)	
Swine liver	0.02	HR* x CF(2)	
Poultry liver	0.01	HR* x CF(1)	
Bovine Kidney	0.04	HR* x CF(4)	
Sheep kidney	0.04	HR* x CF(4)	
Swine kidney	0.04	HR* x CF(4)	
Poultry kidney	0.01	HR* x CF(1)	
Milk	0.004	HR* x CF(1)	
Eggs	0.01	HR* x CF(1)	

* For simplicity reasons, the maximum value (and not the mean) was used (see KCA 6.4).

As shown in Table 6.9-2 and Table 6.9-3 (scenario 1 and 2), the highest NEDI was calculated for the "NL child" diet and represents 2.6% for scenario 1 and 6.2% for scenario 2 of the ADI with the highest contributor being milk and wheat respectively. The NEDI calculations were below the ADI. Therefore, a long-term intake of residues of prothioconazole is unlikely to present a public health concern.

Table 6.9-2: Scenario 1: Prothioconazole - NEDI calculation (EFSA model rev. 2.0)

ADI: 0.01 mg/kg body weight/day Body weight: see individual populations

NEDI in % of ADI	MS Diet	Highest contributor to MS diet	
		% of ADI	Commodity / group of commodities
2,6	NL child	1,2	Milk and milk products: Cattle
2,4	WHO Cluster diet B	1,7	Wheat
2,1	DK child	1,1	Wheat
1,9	ES child	0,9	Wheat
1,9	WHO cluster diet D	1,3	Wheat
1,8	DE child	0,8	Wheat
1,6	WHO cluster diet E	0,8	Wheat
1,5	WHO Cluster diet F	0,7	Wheat
1,4	FR infant	1,0	Milk and milk products: Cattle
1,4	WHO regional European diet	0,6	Wheat
1,3	IT kids/toddler	1,3	Wheat
1,3	SE general population 90th percentile	0,6	Wheat
1,3	IE adult	0,5	Wheat
1,1	ES adult	0,5	Wheat
1,0	NL general	0,4	Wheat
1,0	FR all population	0,7	Wheat
0,9	FR toddler	0,5	Wheat
0,8	IT adult	0,8	Wheat
0,8	PT General population	0,8	Wheat
0,8	LT adult	0,2	Rye
0,8	UK Toddler	0,8	Wheat
0,6	DK adult	0,4	Wheat
0,6	UK Infant	0,5	Wheat
0,4	UK vegetarian	0,4	Wheat
0,4	FI adult	0,2	Wheat
0,4	UK Adult	0,3	Wheat

Table 6.9-3: Scenario 2: Prothioconazole - NEDI calculation (EFSA model rev. 2.0)

ADI: 0.01 mg/kg body weight/day Body weight: see individual populations

NEDI in % of ADI	MS Diet	Highest contributor to MS diet	
		% of ADI	Commodity / group of commodities
6,2	DK child	3,3	Wheat
5,9	WHO Cluster diet B	5,1	Wheat
4,8	WHO cluster diet D	3,9	Wheat
4,7	NL child	2,8	Wheat
4,0	IT kids/toddler	4,0	Wheat
3,9	DE child	2,5	Wheat
3,7	WHO cluster diet E	2,4	Wheat
3,7	ES child	2,7	Wheat
3,6	WHO Cluster diet F	2,2	Wheat
2,8	IE adult	1,4	Wheat
2,7	WHO regional European diet	1,8	Wheat
2,7	SE general population 90th percentile	1,9	Wheat
2,5	IT adult	2,5	Wheat
2,5	PT General population	2,4	Wheat
2,4	UK Toddler	2,4	Wheat
2,3	FR all population	2,0	Wheat
2,2	ES adult	1,4	Wheat
2,0	NL general	1,2	Wheat
2,0	FR toddler	1,6	Wheat
1,8	UK Infant	1,6	Wheat
1,8	DK adult	1,2	Wheat
1,7	LT adult	0,6	Rye
1,7	FR infant	1,0	Milk and milk products: Cattle
1,3	UK vegetarian	1,2	Wheat
1,1	FI adult	0,6	Wheat
1,0	UK Adult	1,0	Wheat

Acute Reference Dose (ARfD) and Dietary Exposure Calculation

Scenario 1

Some of the residue trials were analysed only for prothioconazole-desthio and do not provide data on other metabolites which are included in the risk assessment residue definition (sum of prothioconazole-desthio and all metabolites containing the 2-(1 chlorocyclopropyl)-3-(2-chlorophenyl)-2 hydroxypropyl-2H-1,2,4-triazole moiety). To address this issue, for cereal grain, an appropriate conversion factor of 2 between enforcement and risk assessment was derived by EFSA.

The acute risk assessment was performed with the input value of 0.04 mg/kg for wheat and rye and 0.06 mg/kg for barley and oat *i.e.* highest residue determined in supervised field trials in cereals investigating the representative GAPs multiplied by a conversion factor of 2 proposed by EFSA.

Scenario 2

Nevertheless, as a significant number of trials were determined for all 6 analytes, the "total prothioconazole" values were taken and no conversion factor was used.

The acute risk assessment was performed with the input value of 0.06 mg/kg for wheat and rye and also barley and oat *i.e.* highest residue determined in supervised field trials in cereals investigating the representative GAPs where all 6 analytes were determined.
Calculation details are compiled in Table 6.9-4 (Scenario 1) and Table 6.9-5 (Scenario 2).

Table 6.9-4 Input values for the consumer risk assessment (NEDI) of Prothioconazole

Commodity	Input value (mg/kg)	Comment	Reference
Barley, oat	0.06	HR x CF(2)	This dossier . Scenario 1
Wheat, Rye, (Triticale)	0.04	HR x CF(2)	
Barley, oat	0.06	HR	This dossier Scenario 2
Wheat, Rye, (Triticale)	0.06	HR	

Scenario 1: Using the EFSA PRIMo model, the highest calculated NESTI was found in a maximum exhaustion of the ARfD of 2.6% for wheat consumed by children and of 4.3% of the ARfD for barley consumed by adults.
Scenario 2: Using the EFSA PRIMo model, the highest calculated NESTI was found in a maximum exhaustion of the ARfD of 3.9% for wheat consumed by children and of 4.7% of the ARfD for wheat consumed by adults.

It is concluded that a short term intake of prothioconazole residues via cereals is unlikely to present a public health risk.

Table 6.9-4: Scenario 1: Prothioconazole – NESTI calculation – (EFSA/PRIMo rev. 2)

Body weight: see individual populations

Commodity	Input value (mg/kg)	Maximum food intake reported (g/kg bw/d)	Percentile	MS diet	Body weight (kg)	IESTI (mg/kg bw/d)	% ARfD
Children (ADI 0.022 mg/kg bw/d)							
Barley	0.06	1.77	97.5	UK 4 - 6 year	20.50	0.0001	0.5
Oat	0.06	3.98	97.5	DE	16.15	0.0002	1.1
Rye	0.04	6.32	97.5	UK Infant	8.70	0.0003	1.1
Wheat	0.04	14.45	97.5	UK 4 - 6 year	20.50	0.0006	2.6
Adult (ADI 0.01 mg/kg bw/d)							
Barley	0.06	7.24	97.5	NL	63.00	0.0004	4.3
Oat	0.06	1.43	97.5	LT	70.00	0.0001	0.9
Rye	0.04	4.85	97.5	LT	70.00	0.0002	1.9
Wheat	0.04	7.82	97.5	UK Vegetarian	66.70	0.0003	3.1

Table 6.9-5: Scenario 2: Prothioconazole – NESTI calculation – (EFSA/PRIMo rev. 2)

Body weight: see individual populations

Commodity	Input value (mg/kg)	Maximum food intake reported (g/kg bw/d)	Percentile	MS diet	Body weight (kg)	IESTI (mg/kg bw/d)	% ARfD
Children (ADI 0.022 mg/kg bw/d)							
Barley	0.06	1.77	97.5	UK 4- 6 year	20.50	0.0001	0.5
Oat	0.06	3.98	97.5	DE	16.15	0.0002	1.1
Rye	0.06	6.32	97.5	UK infant	8.7	0.0004	1.7
Wheat	0.06	14.45	97.5	UK 4- 6 year	20.5	0.0009	3.9
Adult (ADI 0.01 mg/kg bw/d)							
Barley	0.06	7.24	97.5	NL	63.00	0.0004	4.3
Oat	0.06	1.43	97.5	LT	70.00	0.0001	0.9
Rye	0.06	4.85	97.5	LT	70.0	0.0003	2.9
Wheat	0.06	7.82	97.5	UK vegetarian	66.70	0.0005	4.7

CA 6.10 Other studies

CA 6.10.1 Effect on the residue level in pollen and bee products

In the absence of a test guideline about how to investigate the residues in pollen and bee products this point was not addressed experimentally. Moreover, also, it is understood that in the EFSA GD referred to (not applicable for AIR III substances), in Appendix D cereals "can be visited for pollen (not nectar), but are not attractive to bees".

A general consideration is that for the evaluation of residues in pollen and bee products for human consumption, the relevant question regarding bee attractiveness is not whether or not the crop is occasionally visited by bees, but if the crop is foraged by honey bees to an extent of economic relevance. Only if a significant portion of pollen and nectar is collected from a cereal field not only by single bees, but from the whole colony, a relevant residue level in stored pollen and bee products could occur. The guidance Document clearly indicates this is not the case.

Appendix 1 Tier 1 summaries

Barley EU foliar spray residue trials – northern EU

Fluoxastrobin & prothioconazole EC 200 in/on winter barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address) : Bayer CropScience AG, Monheim
Country : Germany
Content of active substance (g/kg or g/L) : 100 g/L
Formulation (e.g. WP) : 200 EC

Commercial product (name) : Fluoxastrobin & Prothioconazole EC 200
Producer of commercial product : Bayer CropScience AG

Active substance : prothioconazole
Crop/Crop Group : Cereals
Page : 1
Indoor/outdoor : Outdoor
Other a.s. in formulation (common name and content) : fluoxastrobin 100 g/L
Residues determined as : JAU 6476-desthio
Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2013/00 R 2000 0152/5 0152-00 Sweden S-[redacted]	Barley, winter Jura	1) 03.09.1999 2) 31.05.2000 - 08.06.2000 3) 18.07.2000	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	10.05.2000/0 01.06.2000/22	61	ear rest of plant straw grain	0.01 2.5 0.01 0.45 1.4 0.14 0.07 0.01	0* 0 35 0* 0 35 47 47	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *before last application (h) 0.01 mg/kg

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Page**	:	**2**
Formulation (e.g. WP)	:	**200 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**fluoxastrobin 100 g/L**
Commercial product (name)	:	**Fluoxastrobin & Prothioconazole EC 200**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment	Application rate per treatment			Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT; PHI (days) (f)	Remarks
			(e)	kg a.s./ha	Water (L/ha)	kg a.s./hL						
RA-2013/00 R 2000 0153/3 0153-00 France, north F-[redacted]	Barley, winter Nickel	1) 12.10.1999 2) 11.05.2000 - 23.05.2000 3) 05.07.2000	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	02.05.2000/0 12.05.2000/10	61	ear rest of plant straw grain	0.01 2.0 <0.01 0.44 0.96 0.06 0.06 <0.01	0* 0 35 0* 0 35 54 54	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *before last application (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole
Country	:	Germany	Crop/Crop Group	:	Cereals
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	3
Formulation (e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 200	Residues determined as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues calculated as	:	JAU 6476-desthio

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT; PHI (days) (f)	Remarks
RA-2013/00 R 2000 0154/1 0154-00 United Kingdom GB-[redacted]	Barley, winter Regina	1) 10.11.1999 2) 23.05.2000 - 30.05.2000 3) 25.07.2000	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	09.05.2000/0 30.05.2000/21	69	Ear	<0.01 2.4	0* 0	(c) SPI:Spraying (g) 00647
										<0.01	36	(h) 0.01 mg/kg day 0*: c=0.01 mg/kg
									rest of plant	0.11 1.2	0* 0	(h) 0.05 mg/kg
										0.05	36	*before last application
									straw	0.08	56	
									grain	<0.01	56	(h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Page**	:	**4**
Formulation (e.g. WP)	:	**200 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**fluoxastrobin 100 g/L**
Commercial product (name)	:	**Fluoxastrobin & Prothioconazole EC 200**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) (f)	DALT; PHI (days)	Remarks
RA-2013/00 R 2000 0156/8 0156-00 Germany D-[redacted]	Barley, winter Theresa	1) 17.09.1999 2) 09.05.2000 - 14.05.2000 3) 15.07.2000	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	26.04.2000/0 10.05.2000/14	61	ear	0.01 1.6	0* 0	(c) SPI:Spraying (g) 00647
										<0.01	35	(h) 0.01 mg/kg
									rest of plant	0.32 1.6	0* 0	(h) 0.05 mg/kg
										0.13	35	*before last application
									straw	<0.05	71	
									grain	<0.01	71	(h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Tebuconazole & prothioconazole EC 250 in/on spring barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**: Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**: Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**: 125 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**: 250 EC**	**Indoor/outdoor**	**:**	**Outdoor**
		Other a.s. in formulation (common name and content)	**:**	**tebuconazole 125 g/L**
Commercial product (name)	**: JAU 6476 & HWG 1608 EC 250**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**: Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hl						
RA-2103/02 R 2002 0529/5 0529-02 France, north F-[redacted]	Barley, spring Nevada	1) 01.03.2002 2) 17.06.2002 - 25.06.2002 3) 30.07.2002	SPI SPI	0.1250 0.1250	300 300	0.04163 0.04163	30.05.2002/0 25.06.2002/26	61	ear rest of plant grain straw	0.94 1.2 0.02 0.29	0 0 35<< 35<<	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	: **Bayer CropScience AG, Monheim**		**Active substance**	:	**prothioconazole**
Country	: **Germany**		**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	: **125 g/L**		**Page**	:	**2**
Formulation (e.g. WP)	: **250 EC**		**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**tebuconazole 125 g/L**
Commercial product (name)	: **JAU 6476 & HWG 1608 EC 250**		**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	: **Bayer CropScience AG**		**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			1 Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) (f)	DALT; PHI (days)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						
RA-2103/02 R 2002 0539/2 0539-02 Germany D-[redacted]	Barley, spring Jacinta	1) 20.03.2002 2) 17.06.2002 - 20.06.2002 3) 16.08.2002	SPI SPI	0.1250 0.1250	300 300	0.04163 0.04163	05.06.2002/0 17.06.2002/12	61	ear	1.0 <0.01	0 35<<	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg
									rest of plant	1.7 0.10	0 35<<	(h) 0.05 mg/kg
									grain	<0.01	56	(h) 0.01 mg/kg
									straw	0.05	56	(h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**125 g/L**	**Page**	:	**3**
Formulation (e.g. WP)	:	**250 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**tebuconazole 125 g/L**
Commercial product (name)	:	**JAU 6476 & HWG 1608 EC 250**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT; PHI (days) (f)	Remarks
RA-2103/02 R 2002 0540/6 0540-02 Germany D-[redacted]	Barley, spring Extract	1) 26.03.2002 2) 10.06.2002 - 13.06.2002 3) 15.08.2002	SPI SPI	0.1250 0.1250	300 300	0.04163 0.04163	03.06.2002/0 10.06.2002/7	61	ear	1.4 <0.01	0 35<<	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg
									rest of plant	2.8 0.16	0 35<<	(h) 0.05 mg/kg
									grain	<0.01	57	(h) 0.01 mg/kg
									straw	0.05	57	(h) 0.05 mg/kg
RA-2103/02 R 2002 0541/4 0541-02 United Kingdom GB-[redacted]	Barley, spring Prisma	1) 26.03.2002 2) 15.06.2002 - 20.06.2002 3) 11.08.2002	SPI SPI	0.1250 0.1250	300 300	0.04163 0.04163	07.06.2002/0 07.07.2002/30	77	ear	0.86	0 0	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg
									rest of plant	2.2	0	(h) 0.05 mg/kg
									grain	0.01	35<<	(h) 0.01 mg/kg
									straw	0.21	35<<	(h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Fluoxastrobin & prothioconazole & trifloxystrobin EC 300 in/on barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance	**(g/kg or g/L)**	:	**150 g/L**	**Page**	:	**1**
Formulation	**(e.g. WP)**	:	**300 EC**	**Indoor/outdoor**	:	**Outdoor**
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 75 g/L trifloxystrobin 75 g/L**
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole & Trifloxystrobin EC 300**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2016/03 R 2003 0131/6 0131-03 Germany D-[redacted] 2003	Barley Theresa	1) 01.09.2002 2) 21.05.2003 - 27.05.2003 3) 16.07.2003	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	05.05.2003/0 21.05.2003/16	61	first of plant ear grain straw	0.30 0.96 <0.01 1.2 <0.01 <0.01 <0.05 0.05	0* 0 0* 0 35< 51 35<< 51	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg *before last application (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**150 g/L**	**Page**	**:**	**2**
Formulation (e.g. WP)	**:**	**300 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 75 g/L trifloxystrobin 75 g/L**
Commercial product (name)	**:**	**Fluoxastrobin & Prothioconazole & Trifloxystrobin EC 300**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg) (f)	DALT/ PHI (days)	Remarks
RA-2016/03 R 2003 0250/9 0250-03 France, north F-[redacted] [redacted]2003	Barley Esterel	1) 10.10.2002 2) 15.05.2003 - 30.05.2003 3) 10.07.2003	SPI SPI	0.1665 0.1665	300 300	0.05550 0.05550	28.04.2003/0 23.05.2003/25	65	rest of plant ear grain straw	0.11 0.53 0.03 2.8 <0.01 <0.01 0.05 <0.05	0* 0 0* 0 35<< 48 35<< 48	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg *before last application (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	: **Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	: **Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	: **150 g/L**	**Page**	:	**3**
Formulation (e.g. WP)	: **300 EC**	**Indoor/outdoor**	:	**Outdoor**
		Other a.s. in formulation (common name and content)	:	**fluoxastrobin 75 g/L, trifloxystrobin 75 g/L**
Commercial product (name)	: **Fluoxastrobin & Prothioconazole & Trifloxystrobin EC 300**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	: **Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2016/03 R 2003 0251/7 0251-03 United Kingdom GB-[redacted] [redacted]2003	Barley Caret	1) 04.10.2002 2) 22.05.2003 - 01.06.2003 3) 09.07.2003	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	08.05.2003/0 27.05.2003/19	61	rest of plant ear grain straw	0.37 1.1 0.06 0.01 3.0 0.02 <0.01 0.09	0* 0 35< 0* 0 35<< 43 43	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg day 0*: c=0.01 mg/kg *before last application (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**150 g/L**	**Page**	**:**	**4**
Formulation (e.g. WP)	**:**	**300 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 75 g/L trifloxystrobin 75 g/L**
Commercial product (name)	**:**	**Fluoxastrobin & Prothioconazole & Trifloxystrobin EC 300**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2016/03 R 2003 0252/5 0252-03 Sweden S-[redacted] 2003	Barley Pasadena	1) 28.03.2003 2) 17.06.2003 - 27.06.2003 3) 09.08.2003	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	05.06.2003/0 18.06.2003/13	61	rest of plant ear grain straw	1.2 2.3 0.16 <0.01 1.4 <0.01 <0.01 0.19	0* 0 35< 0* 0 35< 52 52	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg *before last application (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Prothioconazole & trifloxystrobin SC 325 in/on barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**175 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**325 SC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**trifloxystrobin 150 g/L**
Commercial product (name)	**:**	**JAU 6476 & CGA 279202 SC 325**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha (d)	Water (L/ha) (d)	kg a.s./hl (d)	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2106/03 R 2003 0939/2 0939-03 France, north F-[redacted] [redacted]2003	Barley Esterel	1) 10.10.2002 2) 20.05.2003 - 30.05.2003 3) 10.07.2003	SPI SPI	0.1965 0.1965	337.2 337.2	0.05828 0.05828	28.04.2003/0 23.05.2003/25	61	green material ear grain straw	0.57 2.3 <0.01 <0.01 <0.05 <0.05	0 0 35<< 48 35<< 48	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	: **Bayer CropScience AG, Monheim**		**Active substance**	:	**prothioconazole**
Country	: **Germany**		**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	: **175 g/L**		**Page**	:	**2**
Formulation (e.g. WP)	: **325 SC**		**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**trifloxystrobin 150 g/L**
Commercial product (name)	: **JAU 6476 & CGA 279202 SC 325**		**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	: **Bayer CropScience AG**		**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT; PHI (days) (f)	Remarks
RA-2106/03 R 2003 0974/0 0974-03 Germany D-[redacted] [redacted]2003	Barley Tafeno	1) 16.09.2002 2) 17.05.2003 - 26.05.2003 3) 01.07.2003 - 10.07.2003	SPI SPI	0.1750 0.1750	300 300	0.05828 0.05828	07.05.2003/0 19.05.2003/12	61	green material	0.87	0	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg
									ear	1.8	0	(h) 0.01 mg/kg
									grain	<0.01 <0.01	35<< 42	
									straw	0.06 0.08	35<< 42	(h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**175 g/L**	**Page**	:	**3**
Formulation (e.g. WP)	:	**325 SC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**trifloxystrobin 150 g/L**
Commercial product (name)	:	**JAU 6476 & CGA 279202 SC 325**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT, PHI (days) (f)	Remarks
RA-2106/03 R 2003 0975/9 0975-03 United Kingdom GB-[redacted] [redacted]2003	Barley Caret	1) 04.10.2002 2) 22.05.2003 - 01.06.2003 3) 09.07.2003	SPI SPI	0.1750 0.1750	300 300	0.05828 0.05828	08.05.2003/0 27.05.2003/19	61	ear rest of plant grain straw	2.5 0.02 1.1 0.12 <0.01 0.11	0 35< 0 35<< 43 43	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg
RA-2106/03 R 2003 0976/7 0976-03 France, north F-[redacted] [redacted]2003	Barley Nickel	1) 09.10.2002 2) 09.05.2003 - 20.05.2003 3) 27.06.2003	SPI SPI	0.1750 0.1750	300 300	0.05828 0.05828	29.04.2003/0 09.05.2003/10	61	ear rest of plant grain straw	2.9 <0.01 1.1 0.09 <0.01 0.10	0 35<< 0 35<< 49 49	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

JAU 6476 (prothioconazole) & HWG 1608 (tebuconazole) & KWG 4168 (spiroxamine) EC450 in/on spring barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**100 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**450 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**spiroxamine 250 g/L tebuconazole 100 g/L**
Commercial product (name)	**:**	**JAU 6476 & KWG 4168 & HWG 1608 EC 450**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2571/05 R 2005 0030/0 0030-05 France, north F-[redacted] [redacted]2005	Barley, spring CARAFE	1) 14.03.2005 2) 08.06.2005 - 20.06.2005 3) 16.07.2005 - 26.07.2005	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	31.05.2005/0 10.06.2005/10	61	green material grain straw	0.48 1.5 0.28 0.19 <0.01 0.75	0* 0 28 35 39 39	(c) SPI:Spraying (g) 00598/M001 (h) 0.05 mg/kg *before last application (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Page**	:	**2**
Formulation (e.g. WP)	:	**450 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**spiroxamine 250 g/L, cproconazole 100 g/L**
Commercial product (name)	:	**JAU 6476 & KWG 4168 & HWG 1608 EC 450**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment	Application rate per treatment (e) kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2571/05 R 2005 0031/9 0031-05 United Kingdom GB-[redacted] [redacted]2005	Barley, spring Optic	1) 05.04.2005 2) 22.06.2005 - 10.07.2005 3) 05.08.2005 - 15.08.2005	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	14.06.2005/0 22.06.2005/8	61	green material grain straw	1.8 0.06 <0.01 0.10	0 35 47 47	(c) SPI:Spraying (g) 00598/M001 (h) 0.05 mg/kg day 0: c=0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**100 g/L**	**Page**	**:**	**3**
Formulation (e.g. WP)	**:**	**450 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**spiroxamine 250 g/L tebuconazole 100 g/L**
Commercial product (name)	**:**	**JAU 6476 & KWG 4168 & HWG 1608 EC 450**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2571/05 R 2005 0801/8 0801-05 Germany D-[redacted] 2005	Barley, spring Adonis	1) 12.04.2005 2) 28.06.2005 - 04.07.2005 3) 14.08.2005 - 16.08.2005	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	22.06.2005/0 28.06.2005/6	61	green material grain straw	1.6 <0.05 <0.01 <0.05	0 38 49 49	(c) SPI:Spraying (g) 00598/M001 (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Page**	:	**4**
Formulation (e.g. WP)	:	**450 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**spiroxamine 250 g/L, cproconazole 100 g/L**
Commercial product (name)	:	**JAU 6476 & KWG 4168 & HWG 1608 EC 450**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			1) Dates of treatment(s) 2) Application interval 3) or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT; PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						
RA-2571/05 R 2005 0802/6 0802-05 Sweden SE-[redacted] [redacted]2005	Barley, spring Pasadena	1) 08.04.2005 2) 26.06.2005 - 03.07.2005 3) 10.08.2005 - 20.08.2005	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	14.06.2005/0 26.06.2005/12	61	green material	1.6 0.16	0 35	(c) SPI:Spraying (g) 00598/M001 (h) 0.05 mg/kg
									grain	<0.01	51	(h) 0.01 mg/kg
									straw	0.22	51	(h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

BYF 00587 (bixafen) & JAU 6476 (prothioconazole) EC225 in/on spring and winter barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**150 g/L**	**Page**	**:**	**1- A**
Formulation (e.g. WP)	**:**	**225 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**BYF 00587 75 g/L**
Commercial product (name)	**:**	**Bixafen & Prothioconazole EC 225**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days)	Remarks (f)
RA-2328/06 R 2006 0457/2 0457-06 France, north F-[redacted] 2006	Barley, spring Carafe	1) 18.03.2006 2) 18.06.2006 - 24.06.2006 3) 20.07.2006 - 10.08.2006	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	24.05.2006/0 20.06.2006/27	61	green material straw grain	0.06 0.84 0.36 <0.01	0* 0 34 34	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *before last application day 0*: c=0.04 mg/kg
RA-2328/06 R 2006 0458/0 0458-06 Sweden S-[redacted] [redacted]2006	Barley, spring Prestige	1) 28.04.2006 2) 29.06.2006 - 05.07.2006 3) 08.08.2006 - 09.08.2006	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	15.06.2006/0 29.06.2006/14	61	green material straw grain	1.8 1.5 0.59 0.53 0.56 <0.01	0 7 14 28 40 40	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**150 g/L**	**Page**	:	**2- A**
Formulation (e.g. WP)	:	**225 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**BYF 00587 75 g/L**
Commercial product (name)	:	**Bixafen & Prothioconazole EC 225**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT; PHI (days) (f)	Remarks
RA-2039/07 R 2007 0426/7 0426-07 France, north F-[redacted] [redacted]2007	Barley, spring Heinley	1) 12.03.2007 2) 28.05.2007 - 07.06.2007 3) 25.07.2007 - 04.08.2007	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	10.05.2007/0 29.05.2007/19	61	green material ear rest of plant straw grain	0.27 1.3 <0.01 0.03 0.03 <0.01	0* 0 35 35 58 58	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *before last application
RA-2039/07 R 2007 0427/5 0427-07 Germany D-[redacted] [redacted]2007	Barley, spring Annabell	1) 10.04.2007 2) 18.06.2007 - 24.06.2007 3) 06.08.2007	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	31.05.2007/0 18.06.2007/8	61	green material straw grain	0.21 0.82 0.11 0.04 <0.01 <0.01	0* 0 35 49 35 49	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *before last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**150 g/L**	**Page**	:	**1- B**
Formulation (e.g. WP)	:	**225 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**BYF 00587 75 g/L**
Commercial product (name)	:	**Bixafen & Prothioconazole EC 225**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) / Application interval / or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT; PHI (days) (f)	Remarks
RA-2328/06 R 2006 0459/9 0459-06 United Kingdom GB-[redacted] 2006	Barley, winter Sequel	1) 29.09.2005 2) 14.05.2006 - 25.05.2006 3) 18.07.2006 - 19.07.2006	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	05.05.2006/0 17.05.2006/12	61	green material straw grain	0.21 0.71 0.12 <0.01	0* 0 62 62	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *before last application day 62: c=0.01 mg/kg
RA-2328/06 R 2006 0460/2 0460-06 Germany D-[redacted] 2006	Barley, winter Duet	1) 20.09.2005 2) 13.05.2006 - 23.05.2006 3) 05.07.2006 - 15.07.2006	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	28.04.2006/0 15.05.2006/17	61	green material straw grain	0.15 0.81 0.38 0.17 0.06 0.04 <0.01	0* 0 7 14 28 51 51	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *before last application day 0*: c=0.02 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Bixafen & Prothioconazole & Spiroxamine EC 400 in/on spring and winter barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Crop/Crop Group**	:	**Cereals**
Country	:	**Germany**	**Page**	:	**1- A**
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Indoor/outdoor**	:	**Outdoor**
Formulation (e.g. WP)	:	**400 EC**	**Other a.s. in formulation (common name and content)**	:	**BYF 00587 50 g/L spiroxamine 250 g/L**
Commercial product (name)	:	**Bixafen & Prothioconazole & Spiroxamine EC 400**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**
			Active substance	:	**prothioconazole**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2042/07 R 2007 0448/8 0448-07 France, north F-[redacted] [redacted]2007	Barley, spring Heinley	1) 12.03.2007 2) 28.05.2007 - 07.06.2007 3) 25.07.2007 - 04.08.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	10.05.2007/0 29.05.2007/19	61	green material ear rest of plant straw grain	0.13 1.3 <0.01 0.02 0.02 <0.01	0* 0 35 35 58 58	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *before last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**100 g/L**	**Page**	**:**	**2- A**
Formulation (e.g. WP)	**:**	**400 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**BYF 00587 50 g/L spiroxamine 250 g/L**
Commercial product (name)	**:**	**Bixafen & Prothioconazole & Spiroxamine EC 400**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg) (h)	DALT/ PHI (days) (f)	Remarks
RA-2042/07 R 2007 0527/1 0527-07 United Kingdom GB-[redacted] 2007	Barley, spring Cocktail	1) 24.01.2007 2) 25.05.2007 - 09.06.2007 3) 15.08.2007 - 31.08.2007	SPI SPI	0.1500 0.1590	300 318	0.05000 0.05000	09.05.2007/0 25.05.2007/16	61	green material rest of plant ear straw grain	0.07 1.3 0.02 <0.01 0.02 <0.01	0* 0 34 34 96 96	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *before last application 2nd application 6% overdosed

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals
Country	:	Germany	Page	:	3- A
Content of active substance (g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor
Formulation (e.g. WP)	:	400 EC	Other a.s. in formulation (common name and content)	:	BYF 00587 50 g/L spiroxamine 250 g/L
Commercial product (name)	:	Bixafen & Prothioconazole & Spiroxamine EC 400	Residues determined as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues calculated as	:	JAU 6476-desthio
			Active substance	:	prothioconazole

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2042/07 R 2007 0529/8 0529-07 Netherlands NL-[redacted] [redacted]2007	Barley, spring Prestige	1) 06.04.2007 2) 12.06.2007 - 24.06.2007 3) 20.08.2007 - 02.09.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	30.05.2007/0 13.06.2007/14	61	green material straw grain	0.21 1.2 0.02 0.02 <0.01 <0.01	0* 0 35 71 35 71	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *before last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**100 g/L**	**Page**	**:**	**1- R**
Formulation (e.g. WP)	**:**	**400 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**BYF 00587 50 g/L spiroxamine 250 g/L**
Commercial product (name)	**:**	**Bixafen & Prothioconazole & Spiroxamine EC 400**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropSciences AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2042/07 R 2007 0449/6 0449-07 Germany D-[redacted] 2007	Barley, winter Naomi	1) 25.09.2006 2) 03.05.2007 - 14.05.2007 3) 20.06.2007 - 28.06.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	18.04.2007/0 04.05.2007/16	61	green material ear rest of plant straw grain	0.31 1.4 0.01 0.16 0.06 <0.01	0* 0 35 35 55 55	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Bixafen & Fluoxastrobin & Prothioconazole EC 190 in/on spring barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	1 - A	
Formulation	(e.g. WP)	:	190 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2204 10-2204-01 Netherlands [redacted] 2010	Barley, spring Tipple	1) 14.04.2010 3) 01.08.2010 - 20.08.2010	SPI SPI	0.150 0.150	300 300	0.0500 0.0500	15.06.2010/0 29.06.2010/14	61	green material grain straw	0.36 0.51 <0.01 <0.01 0.11 0.05	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.05 0.05 <0.01 <0.01 0.10 0.04	0.07 0.08 <0.01 <0.01 0.12 0.04	0* 0 35 43 35 43	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	2- A	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	190 EC	Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2204 10-2204-02 Germany [redacted] 2010	Barley, spring Quench	1) 06.04.2010 2) 21.06.2010 - 28.06.2010 3) 01.08.2010 - 15.08.2010	SPI SPI	0.150 0.150	300 300	0.0500 0.0500	07.06.2010/0 21.06.2010/14	61	green material	0.53 1.5	<0.01 <0.01	0.09 0.10	0.09 0.09	0* 0	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment
									grain	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	34 46	
									straw	0.54 0.17	<0.01 <0.01	0.32 0.17	0.25 0.12	34 46	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1 - 8**	
Formulation	**(e.g. WP)**	:	**190 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**BYF 00587 40 g/L fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Bixafen & Fluoxastrobin & Prothioconazole EC 190**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as : JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as : JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
10-2204 10-2204-01 Netherlands [redacted] 2010	Barley, spring Tipple	1) 14.04.2010 3) 09.08.2010 - 20.08.2010	SPI SPI	0.150 0.150	300 300	0.0500 0.0500	15.06.2010/0 29.06.2010/14	61	green material grain straw	0.03 0.03 <0.01 <0.01 0.06 0.02	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.53 0.69 <0.06 <0.06 0.41 0.17	0* 0 35 43 35 43	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**2- B**	
Formulation	**(e.g. WP)**	:	**190 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**BYF 00587 40 g/L fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Bixafen & Fluoxastrobin & Prothioconazole EC 190**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
10-2204 10-2204-02 Germany [redacted] 2010	Barley, spring Quench	1) 06.04.2010 2) 21.06.2010 - 28.06.2010 3) 01.08.2010 - 15.08.2010	SPI SPI	0.150 0.150	300 300	0.0500 0.0500	07.06.2010/0 21.06.2010/1#	61	green material grain straw	0.05 0.06 <0.01 <0.01 0.10 0.08	<0.01 <0.01 <0.01 <0.01 0.01 <0.01	0.78 1.8 <0.06 <0.06 1.2 0.56	0* 0 34 46 34 46	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

Fluoxastrobin & Prothioconazole EC 200 in/on barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1 - 1**	
Formulation	**(e.g. WP)**	:	**200 EC**	**Indoor/outdoor**	:	**Outdoor**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 200**	**Other a.s. in formulation (common name and content)**	:	**fluoxastrobin 100 g/L**	
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-alpha-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-3-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-4-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
13-2137 13-2137-01 Germany [redacted] 2013	Barley, spring Conchita	1) 28.05.2013 2) 14.06.2013 - 21.06.2013 3) 15.08.2013 - 31.08.2013	SPI SPI	0.125 0.125	300 300	0.0417 0.0417	31.05.2013/0 14.06.2013/14	61	green material	0.27 0.94 0.88 0.42 0.15 0.067 0.049	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.023 0.032 0.062 0.093 0.084 0.074 0.079	0.032 0.044 0.075 0.091 0.080 0.066 0.064	0* 0 7 14 21 28 35	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment
									grain	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	35 69	
									straw	0.087 0.036	<0.01 <0.01	0.14 0.029	0.12 0.030	35 69	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		**:**	**Bayer CropScience AG, Monheim**	**Crop/Crop Group**	**:**	**Cereals**	
Country		**:**	**Germany**	**Page**	**:**	**2- A**	
Content of active substance	**(g/kg or g/L)**	**:**	**100 g/L**	**Indoor/outdoor**	**:**	**Outdoor**	
Formulation	**(e.g. WP)**	**:**	**200 EC**	**Other a.s. in formulation (common name and content)**	**:**	**fluoxastrobin 100 g/L**	
Commercial product	**(name)**	**:**	**Fluoxastrobin & Prothioconazole EC 200**	**Residues determined as**	**:**	**JAU 6476-desthio**	**Residues calculated as : JAU 6476-desthio**
Producer of commercial product		**:**	**Bayer CropScience AG**	**Residues determined as**	**:**	**JAU 6476-alpha-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	**:**	**JAU 6476-3-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	**:**	**JAU 6476-4-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg) **JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	DALT/ PHI (days) (f)	Remarks
13-2137 13-2137-02 Germany [redacted] 2013	Barley, spring Grace	1) 09.04.2013 2) 17.06.2013 - 22.06.2013 3) 15.08.2013 - 25.08.2013	SPI SPI	0.125 0.125	300 400	0.0417 0.0313	1)2.06.2013/0 2)18.06.2013/6	61	green material	0.50 1.1 0.47 0.22 0.098 0.067 0.041	<0.01 <0.01 0.010 0.013 0.013 0.011 <0.01	0.023 0.026 0.070 0.080 0.092 0.078 0.038	0.010 0.012 0.025 0.027 0.028 0.025 0.012	0* 0 7 14 21 28 42	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment
									grain	0.011	<0.01	<0.01	<0.01	68	
									straw	0.044	<0.01	0.020	<0.01	68	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**3- A**	
Formulation	**(e.g. WP)**	:	**200 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 100 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 200**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as : JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-alpha-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-3-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-4-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	DALT/ PHI (days) (f)	Remarks
13-2158 13-2158-01 France, north [redacted] 2013	Barley Esterel	1) 02.09.2012 3) 09.07.2013	SPI SPI	0.125 0.125	200 200	0.0625 0.0625	07.05.2013/0 27.05.2013/20	61	green material grain straw	0.17 1.5 0.46 0.14 0.078 0.067 0.079 <0.01 <0.01 0.039 0.15	<0.01 <0.01 0.012 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.051 0.056 0.076 0.080 0.088 0.057 0.067 <0.01 <0.01 0.051 0.11	0.047 0.049 0.068 0.065 0.074 0.046 0.057 <0.01 <0.01 0.036 0.083	0* 0 7 14 21 28 31 35 43 35 43	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	4-A	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	200 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Fluoxastrobin & Prothioconazole EC 200	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (l/ha)	kg a.s./hL	Dates of treatment(s) Application interval/ or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
13-2158 13-2158-02 France, north [redacted] 2013	Barley, spring Sébastian	1) 21.02.2013 3) 23.07.2013	SPI SPI	0.133 0.135	212.7 216.7	0.0625 0.0623	03.06.2013/0 18.06.2013/15	61	green material	0.33 0.89 0.72 0.49 0.40 0.44	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.11 0.13 0.17 0.16 0.15 0.16	0.15 0.16 0.21 0.20 0.16 0.17	0* 0 7 14 21 17	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *before last treatment
									grain	<0.01	<0.01	<0.01	<0.01	35	
									straw	0.81	0.023	0.55	0.55	35	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1- B**	
Formulation	**(e.g. WP)**	:	**200 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 100 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 200**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (l/ha)	kg a.s./hL	Dates of treatment(s) Application interval; or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
13-2137 13-2137-01 Germany [redacted] 2013	Barley, spring Conchita	1) 28.03.2013 2) 14.06.2013 - 21.06.2013 3) 15.08.2013 - 31.08.2013	SPI SPI	0.125 0.125	300 300	0.0417 0.0417	31.05.2013/0 14.06.2013/14	61	green material grain straw	0.018 0.024 0.044 0.059 0.053 0.039 0.038 <0.01 <0.01 0.067 0.020	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.36 1.1 1.1 0.68 0.39 0.27 0.25 <0.06 <0.06 0.43 0.14	0* 0 7 14 21 28 35 35 69 35 69	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	2- B	
Formulation	(e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Fluoxastrobin & Prothioconazole EC 200	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-8-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
13-2137 13-2137-02 Germany [redacted] 2013	Barley, spring Grace	1) 09.04.2013 2) 17.06.2013 - 22.06.2013 3) 15.08.2013 - 25.08.2013	SPI SPI	0.125 0.125	300 400	0.0417 0.0313	12.06.2013/0 18.06.2013/6	61	green material grain straw	0.013 0.013 0.030 0.032 0.029 0.024 0.013 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.57 1.2 0.62 0.38 0.27 0.22 0.12 0.061 0.10	0* 0 7 14 21 28 42 68 68	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address) : **Bayer CropScience AG, Monheim**
Country : **Germany**
Content of active substance (g/kg or g/L) : **100 g/L**
Formulation (e.g. WP) : **200 EC**
Commercial product (name) : **Fluoxastrobin & Prothioconazole EC 200**
Producer of commercial product : **Bayer CropScience AG**

Active substance : **prothioconazole**
Crop/Crop Group : **Cereals**
Page : **3-B**
Indoor/outdoor : **Outdoor**
Other a.s. in formulation (common name and content) : **fluoxastrobin 100 g/L**
Residues determined as : **JAU 6476-desthio** **Residues calculated as** : **JAU 6476-desthio**
Residues determined as : **JAU 6476-5-hydroxy-desthio** **Residues calculated as** : **JAU 6476-desthio**
Residues determined as : **JAU 6476-6-hydroxy-desthio** **Residues calculated as** : **JAU 6476-desthio**
Residues determined as : **Total residues calc.** **Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (l/ha)	kg a.s./hL	Dates of treatment(s) Application interval/ or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
13-2158 13-2158-01 France, north [redacted] 2013	Barley Esterel	1) 02.10.2012 3) 09.07.2013	SPI SPI	0.125 0.125	200 200	0.0625 0.0625	07.05.2013/0 27.05.2013/20	61	green material grain straw	0.044 0.044 0.065 0.059 0.058 0.039 0.048 <0.01 <0.01 0.034 0.092	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.33 1.7 0.69 0.36 0.32 0.23 0.27 <0.06 <0.06 0.18 0.46	0* 0 7 14 21 28 31 35 43 35 43	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	4- B	
Formulation	(e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Fluoxastrobin & Prothioconazole EC 200	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-8-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
13-2158 13-2158-02 France, north [redacted] 2013	Barley, spring Sébastian	1) 21.02.2013 3) 23.07.2013	SPI SPI	0.133 0.133	212.7 216.7	0.0625 0.0623	03.06.2013/0 18.06.2013/15		green material grain straw	0.078 0.091 0.10 0.082 0.065 0.077 <0.01 0.23	<0.01 <0.01 <0.01 0.010 0.010 <0.01 <0.01 0.042	0.69 1.3 1.2 0.95 0.80 0.87 <0.06 2.2	0* 0 7 14 21 17 35 35	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

Barley EU foliar spray residue trials – southern EU

Fluoxastrobin & prothioconazole EC 200 in/on barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**100 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**200 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 100 g/L**
Commercial product (name)	**:**	**Fluoxastrobin & Prothioconazole EC 200**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropSciences AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) (g)	DALT/ PHI (days) (f)	Remarks
RA-2014/00 R 2000 0157/6 0157-00 Italy I-[redacted] 2000	Barley Ganora	1) 08.11.1999 2) 04.05.2000 - 08.05.2000 3) 24.06.2000	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	21.04.2000(i) 04.05.2000/3	61	ear rest of plant straw grain	0.02 2.0 0.56 1.7 0.14 0.11 <0.01 0.02	0* 0 0* 0 35 55 35 55	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *before last application (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals
Country	:	Germany	Page	:	2
Content of active substance (g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor
Formulation (e.g. WP)	:	200 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 200	Residues determined as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues calculated as	:	JAU 6476-desthio
			Active substance	:	prothioconazole

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) (a)	DALT/ PHI (days) (f)	Remarks
RA-2014/00 R 2000 0158/4 0158-00 France, south F-[redacted] [redacted]2000	Barley, winter Univer	1) 20.10.1999 2) 01.05.2000 - 07.05.2000 3) 07.06.2000	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	19.04.2000/0 03.05.2000/14	61	ear rest of plant straw grain	<0.01 2.9 0.55 1.4 0.14 0.01	0* 0 0* 0 35 35	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *before last application (h) 0.01 mg/kg
RA-2014/00 R 2000 0159/2 0159-00 France, south F-[redacted] [redacted]2000	Barley, winter Pastoral	1) 04.11.1999 2) 03.05.2000 - 12.05.2000 3) 23.06.2000	SPI SPI	0.1250 0.1250	280 300	0.04460 0.04170	17.04.2000/0 05.05.2000/18	61	ear rest of plant straw grain	<0.01 3.4 0.01 0.24 1.0 0.10 0.05 <0.01	0* 0 35 0* 0 35 49 49	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *before last application (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Tebuconazole & prothioconazole EC 250 in/on spring barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**125 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**250 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**tebuconazole 125 g/L**
Commercial product (name)	**:**	**JAU 6476 & HWG 1608 EC 250**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						
RA-2104/02 R 2002 0530/9 0530-02 Italy I-[redacted] 2002	Barley, spring Pandas	1) 26.02.2002 2) 29.05.2002 - 06.06.2002 3) 30.06.2002	SPI SPI	0.1250 0.1250	300 300	0.04163 0.04163	29.04.2002/0 21.05.2002/22	59	ear rest of plant grain straw	1.5 0.85 <0.01 <0.01 0.08 0.08	0 0 35<< 41 35<< 41	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**125 g/L**	**Page**	**:**	**2**
Formulation (e.g. WP)	**:**	**250 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**tebuconazole 125 g/L**
Commercial product (name)	**:**	**JAU 6476 & HWG 1608 EC 250**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2104/02 R 2002 0542/2 0542-02 Italy I-[redacted] [redacted]2002	Barley, spring Klaxon	1) 03.03.2002 2) 30.05.2002 - 06.06.2002 3) 18.07.2002	SPI SPI	0.1250 0.1250	300 300	0.04163 0.04163	20.05.2002/0 10.06.2002/21	75	ear rest of plant grain straw	0.86 0.03 1.1 0.13 0.01 0.24	0 35< 0 35<< 42 42	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg
RA-2104/02 R 2002 0544/9 0544-02 Spain E-[redacted] [redacted]2002	Barley, spring Grafit	1) 04.11.2001 3) 15.06.2002	SPI SPI	0.1250 0.1250	300 300	0.04163 0.04163	18.04.2002/0 02.05.2002/14	55	ear rest of plant grain straw	1.5 2.2 0.01 <0.01 1.2 1.2	0 0 35<< 42 35<< 42	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Fluoxastrobin & prothioconazole & trifloxystrobin EC 300 in/on barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**150 g/L**	**Page**	:	**1**
Formulation (e.g. WP)	:	**300 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**fluoxastrobin 75 g/L** **trifloxystrobin 75 g/L**
Commercial product (name)	:	**Fluoxastrobin & Prothioconazole & Trifloxystrobin EC 300**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2017/03 R 2003 0132/4 0132-03 France, south F-[redacted] [redacted]2003	Barley Print	1) 25.02.2003 2) 23.05.2003 - 02.06.2003 3) 30.06.2003	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	13.05.2003/0 26.05.2003/13	55-65	rest of plant ear grain straw	0.67 2.1 0.05 1.3 <0.01 0.62	0* 0 0* 0 35<< 35<<	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg *before last application (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**150 g/L**	**Page**	:	**2**
Formulation (e.g. WP)	:	**300 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**fluoxastrobin 75 g/L** **trifloxystrobin 75 g/L**
Commercial product (name)	:	**Fluoxastrobin & Prothioconazole & Trifloxystrobin EC 300**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (d)	Residues (mg/kg) (g)	DALT/ PHI (days) (f)	Remarks
RA-2017/03 R 2003 0253/3 0253-03 Italy I-[redacted] [redacted]2003	Barley Aliseo	1) 30.10.2002 2) 05.05.2003 - 15.05.2003 3) 09.06.2003 - 20.06.2003	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	17.04.2003/0 05.05.2003/18	61	rest of plant ear grain straw	0.19 0.74 <0.01 2.4 <0.01 <0.01 <0.05 0.05	0* 0 0* 0 35<< 45 35<< 45	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg *before last application (h) 0.05 mg/kg
RA-2017/03 R 2003 0254/1 0254-03 Spain E-[redacted] 2002	Barley Hispanic	1) 20.11.2002 2) 23.04.2003 - 10.05.2003 3) 01.07.2003	SPI SPI	0.1500 0.1598	300 320	0.04995 0.04995	08.04.2003/0 23.04.2003/15	61	rest of plant ear grain straw	0.30 1.5 0.21 0.02 2.6 0.03 <0.01 0.31	0* 0 35<< 0* 0 35<< 69 69	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg *before last application (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**150 g/L**	**Page**	**:**	**3**
Formulation (e.g. WP)	**:**	**300 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 75 g/L, trifloxystrobin 75 g/L**
Commercial product (name)	**:**	**Fluoxastrobin & Prothioconazole & Trifloxystrobin EC 300**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2017/03 R 2003 0256/8 0256-03 Italy I-[redacted] 2003	Barley Sonora	1) 18.12.2002 2) 15.05.2003 - 20.05.2003 3) 20.06.2003 - 25.06.2003	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	30.04.2003/0 16.05.2003/16	61	rest of plant ear grain straw	0.20 4.2 <0.01 3.5 <0.01 <0.01 0.10 0.14	0* 0 0* 0 35<< 40 35<< 40	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg *before last application (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Prothioconazole & trifloxystrobin SC325 in/on barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address) : **Bayer CropScience AG, Monheim**
Country : **Germany**
Content of active substance (g/kg or g/L) : **175 g/L**
Formulation (e.g. WP) : **325 SC**

Commercial product (name) : **JAU 6476 & CGA 279202 SC 325**
Producer of commercial product : **Bayer CropScience AG**

Active substance : **prothioconazole**
Crop/Crop Group : **Cereals**
Page : **1**
Indoor/outdoor : **Outdoor**
Other a.s. in formulation (common name and content) : **trifloxystrobin 150 g/L**
Residues determined as : **JAU 6476-desthio**
Residues calculated as : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						
RA-2107/03 R 2003 0940/6 0940-03 France, south F-[redacted] 2003	Barley Esterel	1) 13.11.2002 2) 12.05.2003 - 19.05.2003 3) 27.06.2003	SPI SPI	0.1750 0.1750	300 300	0.05828 0.05828	29.04.2003/0 12.05.2003/13	61	ear rest of plant grain straw	3.6 0.02 0.94 0.09 <0.01 0.10	0 35< 0 35<< 44 44	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg
RA-2107/03 R 2003 0977/5 0977-03 Italy I-[redacted] 2003	Barley Aliseo	1) 30.10.2002 2) 05.05.2003 - 15.05.2003 3) 09.06.2003 - 20.06.2003	SPI SPI	0.1750 0.1750	300 300	0.05828 0.05828	24.04.2003/0 06.05.2003/12	61	ear rest of plant grain straw	3.0 0.02 0.96 0.08 <0.01 0.06	0 35< 0 35<< 44 44	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address) : **Bayer CropScience AG, Monheim**
Country : **Germany**
Content of active substance (g/kg or g/L) : **175 g/L**
Formulation (e.g. WP) : **325 SC**

Commercial product (name) : **JAU 6476 & CGA 279202 SC 325**
Producer of commercial product : **Bayer CropScience AG**

Active substance : **prothioconazole**
Crop/Crop Group : **Cereals**
Page : **2**
Indoor/outdoor : **Outdoor**
Other a.s. in formulation (common name and content) : **trifloxystrobin 150 g/L**
Residues determined as : **JAU 6476-desthio**
Residues calculated as : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2107/03 R 2003 0978/3 0978-03 Spain E-[redacted] [redacted]2003	Barley Hispania	1) 25.10.2002 2) 24.04.2003 - 02.05.2003 3) 15.06.2003	SPI SPI	0.1750 0.1750	300 300	0.05828 0.05828	22.04.2003/0 29.04.2003/7	69	ear	2.3	0	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg
									rest of plant	4.3	0	(h) 0.05 mg/kg
									grain	0.03	34	(h) 0.01 mg/kg
									straw	2.5 1.3	34 42	(h) 0.05 mg/kg
RA-2107/03 R 2003 0979/1 0979-03 Greece GR-[redacted] [redacted] 2003	Barley Scarlet	1) 22.11.2002 2) 15.05.2003 - 22.05.2003 3) 21.06.2003	SPI SPI	0.1750 0.1750	300 300	0.05828 0.05828	02.05.2003/0 15.05.2003/13	61	ear	1.3	0	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg
									rest of plant	2.8	0	(h) 0.05 mg/kg
									grain	<0.01	35<<	(h) 0.01 mg/kg
									straw	1.1	35<<	(h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

JAU 6476 (prothioconazole) & HWG 1608 (tebuconazole) & KWG 4168 (spiroxamine) EC450 in/on winter barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Crop/Crop Group**	**:**	**Cereals**
Country	**:**	**Germany**	**Page**	**:**	**1**
Content of active substance (g/kg or g/L)	**:**	**100 g/L**	**Indoor/outdoor**	**:**	**Outdoor**
Formulation (e.g. WP)	**:**	**450 EC**	**Other a.s. in formulation (common name and content)**	**:**	**spiroxamine 250 g/L tebuconazole 100 g/L**
Commercial product (name)	**:**	**JAU 6476 & KWG 4168 & HWG 1608 EC 450**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**
			Active substance	**:**	**prothioconazole**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						
RA-2572/05 R 2005 0468/3 0468-05 France, south F-[redacted] [redacted] 2005	Barley, winter BARAKA	1) 25.10.2004 2) 22.04.2005 - 04.05.2005 3) 22.06.2005 - 23.06.2005	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	14.04.2005/0 22.04.2005/8	61	green material grain straw	0.06 2.1 0.06 0.05 <0.01 0.19	0* 0 28 35 61 61	(c) SPI:Spraying (g) 00598/M001 (h) 0.05 mg/kg *before last treatment (h) 0.01 mg/kg (h) 0.05 mg/kg
RA-2572/05 R 2005 0469/1 0469-05 Spain E-[redacted] 2005	Barley, winter Graphic	1) 20.01.2005 2) 10.05.2005 - 25.05.2005 3) 01.07.2005 - 10.07.2005	SPI SPI	0.1250 0.1250	300 300	0.04170 0.04170	02.05.2005/0 11.05.2005/9	59	green material grain straw	2.4 <0.01 <0.01 1.6 1.2	0 35 49 35 49	(c) SPI:Spraying (g) 00598/M001 (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

BYF 00587 (bixafen) & JAU 6476 (prothioconazole) EC225 in/on spring and winter barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**150 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**225 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**BYF 00587 75 g/L**
Commercial product (name)	**:**	**Bixafen & Prothioconazole EC 225**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) 	DALT/ PHI (days) (f)	Remarks
RA-2329/06 R 2006 0461/0 0461-06 France, south F-[redacted] [redacted]2006	Barley, spring Scarlett	1) 03.02.2006 2) 03.06.2006 - 07.06.2006 3) 01.07.2006 - 07.07.2006	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	11.05.2006/0 03.06.2006/23	61	green material straw grain	0.38 1.3 1.0 0.01	0* 0 34 34	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application
RA-2329/06 R 2006 0462/9 0462-06 Italy I-[redacted] [redacted]2006	Barley, winter Marigold	1) 10.11.2005 2) 06.05.2006 - 16.05.2006 3) 15.06.2006 - 30.06.2006	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	24.04.2006/0 03.05.2006/9	61	green material straw grain	0.53 1.7 1.2 0.57 0.36 0.64 0.56 0.01 0.02	0* 0 7 14 28 35 43 35 43	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole
Country	:	Germany	Crop/Crop Group	:	Cereals
Content of active substance (g/kg or g/L)	:	150 g/L	Page	:	2
Formulation (e.g. WP)	:	225 EC	Indoor/outdoor	:	Outdoor
			Other a.s. in formulation (common name and content)	:	BYF 00587 75 g/L
Commercial product (name)	:	Bixafen & Prothioconazole EC 225	Residues determined as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues calculated as	:	JAU 6476-desthio

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) (f)	DALT/ PHI (days) (f)	Remarks
RA-2329/06 R 2006 0463/7 0463-06 Spain E-[redacted] 2006	Barley, winter GRAPHIC	1) 20.12.2005 2) 20.04.2006 - 30.04.2006 3) 20.06.2006 - 30.06.2006	SPI SPI	0.1605 0.1500	321 300	0.04995 0.04995	05.04.2006/0 26.04.2006/21	61	green material straw grain	0.38 1.3 0.67 0.79 <0.01 <0.01	0* 0 35 48 35 48	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application 1st application 7% overdosed
RA-2329/06 R 2006 0464/5 0464-06 Portugal P-[redacted] 2006	Barley, winter Scarlet	1) 19.12.2005 2) 25.04.2006 - 07.05.2006 3) 06.06.2006 - 10.06.2006	SPI SPI	0.1605 0.1500	321 300	0.04995 0.04995	30.03.2006/0 26.04.2006/27	61	green material straw grain	0.02 0.88 0.26 0.12 0.15 0.32 0.33 0.01 0.01	0* 0 7 14 28 35 43 35 43	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

Bixafen & prothioconazole & spiroxamine EC 400 in/on spring and winter barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting	(name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole
Country		:	Germany	Crop/Crop Group	:	Cereals
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	1
Formulation	(e.g. WP)	:	400 EC	Indoor/outdoor	:	Outdoor
				Other a.s. in formulation (common name and content)	:	BYF 00587 50 g/L spiroxamine 250 g/L
Commercial product	(name)	:	Bixafen & Prothioconazole & Spiroxamine EC 400	Residues determined as	:	JAU 6476-desthio
Producer of commercial product		:	Bayer CropSciences AG	Residues calculated as	:	JAU 6476-desthio

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT* PHI (days) (f)	Remarks
RA-2043/07 R 2007 0530/1 0530-07 Italy I-[redacted] 2007	Barley, spring Tunica	1) 28.05.2007 2) 15.07.2007 - 25.07.2007 3) 10.09.2007 - 20.09.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	12.07.2007/0 20.07.2007/8	61	green material straw grain	0.38 2.0 0.14 0.04 <0.01 <0.01	0* 0 39 55 39 55	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last applicatopn
RA-2043/07 R 2007 0451/8 0451-07 France, south F-[redacted] 2007	Barley, winter Esterel	1) 12.10.2006 2) 24.04.2007 - 02.05.2007 3) 13.06.2007 - 26.06.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	10.04.2007/0 24.04.2007/14	61	green material ear rest of plant straw grain	0.34 0.97 0.01 0.33 0.09 <0.01	0* 0 35 35 58 58	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		**: Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country		**: Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance	**(g/kg or g/L)**	**: 100 g/L**	**Page**	**:**	**2**
Formulation	**(e.g. WP)**	**: 400 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**BYF 00587 50 g/L spiroxamine 250 g/L**
Commercial product	**(name)**	**: Bixafen & Prothioconazole & Spiroxamine EC 400**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product		**: Bayer CropScience AG**	**Residues calculated as**		**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2043/07 R 2007 0452/6 0452-07 Spain E-[redacted] [redacted]2007	Barley, winter County R2	1) 01.12.2006 2) 23.04.2007 - 30.04.2007 3) 01.06.2007 - 10.07.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	08.04.2007/0 23.04.2007/20	61	green material straw grain	0.15 1.8 0.65 0.69 0.01 0.01	0* 0 36 42 36 42	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application
RA-2043/07 R 2007 0532/8 0532-07 Portugal P-[redacted] [redacted]2007	Barley, winter Scarlet	1) 09.02.2007 2) 05.05.2007 - 15.05.2007 3) 10.06.2007 - 15.06.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	19.04.2007/0 07.05.2007/20	61	green material straw grain	0.12 0.95 0.67 <0.01	0* 0 35 35	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

Bixafen & fluoxastrobin & prothioconazole EC 190 in/on barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	1-A	
Formulation	(e.g. WP)	:	190 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (l/ha)	kg a.s./hL	Dates of treatment(s) Application interval/ or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2206 10-2206-01 France, south [redacted] 2010	Barley Ketos	1) 05.20.2009 2) 11.05.2010 - 20.05.2010 3) 01.07.2010 - 27.07.2010	SPI SPI	0.15 0.15	300 300	0.050 0.050	27.04.2010/0 12.05.2010/15	61	green material grain straw 	0.45 1.7 <0.01 <0.01 0.25 0.33	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.06 0.05 <0.01 <0.01 0.16 <0.01	0.10 0.09 <0.01 <0.01 0.22 0.02	0* 0 34 47 34 47	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	2- A	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	190 EC	Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2206 10-2206-02 Italy [redacted] 2010	Barley Ketos	1) 05.10.2009 2) 02.05.2010 - 10.05.2010 3) 10.06.2010 - 30.06.2010	SPI SPI	0.15 0.15	400 400	0.038 0.038	19.04.2010/0 03.05.2010/14	61	green material grain straw	0.15 1.5 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	0.03 0.04 <0.01 0.12	0.05 0.06 <0.01 0.21	0* 0 52 52	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1- B**	
Formulation	**(e.g. WP)**	:	**190 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**BYF 00587 40 g/L fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Bixafen & Fluoxastrobin & Prothioconazole EC 190**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
10-2206 10-2206-01 France, south [redacted] 2010	Barley Ketos	1) 05.10.2009 2) 11.05.2010 - 20.05.2010 3) 01.07.2010 - 27.07.2010	SPI SPI	0.15 0.15	300 300	0.050 0.050	27.04.2010/0 12.05.2010/15	61	green material grain straw	0.05 0.05 <0.01 <0.01 0.10 0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.68 1.9 <0.06 <0.06 0.75 0.39	0* 0 34 47 34 47	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	2- B	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	190 EC	Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

Active substance : prothioconazole

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2206 10-2206-02 Italy [redacted] 2010	Barley Ketos	1) 05.10.2009 2) 02.05.2010 - 10.05.2010 3) 10.06.2010 - 30.06.2010	SPI SPI	0.15 0.15	400 400	0.038 0.038	19.04.2010/0 03.05.2010/14	61	green material grain straw	0.03 0.03 <0.01 0.10	<0.01 <0.01 <0.01 <0.01	0.28 1.7 <0.06 0.46	0* 0 52 52	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Fluoxastrobin & prothioconazole EC 150 in/on winter barley

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Crop/Crop Group**	:	**Cereals**		
Country	:	**Germany**	**Page**	:	**1- A**		
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Indoor/outdoor**	:	**Outdoor**		
Formulation (e.g. WP)	:	**150 EC**	**Other a.s. in formulation (common name and content)**	:	**fluoxastrobin 50 g/L**		
Commercial product (name)	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-alpha-hydroxy-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
			Residues determined as	:	**JAU 6476-3-hydroxy-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
			Residues determined as	:	**JAU 6476-4-hydroxy-desthio**	**Residues calculated as**	: **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	DALT/ PHI (days) (f)	Remarks
11-2111 11-2111-01 France, south [redacted] 2011	Barley, winter Ketos	1) 04.10.2010 2) 26.04.2011 - 05.05.2011 3) 17.06.2011 - 27.06.2011	SPI SPI	0.175 0.175	300 300	0.0583 0.0583	13.04.2011/0 27.04.2011/14	61	green material	0.79 2.0 1.4 0.97 0.49	<0.01 0.052 <0.01 <0.01 <0.01	0.057 0.086 0.10 0.14 0.15	0.096 0.14 0.21 0.18	0* 0 7 14 28	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg 0*: c=0.051 mg/kg for JAU 6476-desthio *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	1- A (continued)	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	150 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L	
Commercial product	(name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
11-2111 11-2111-01 (continued)	Barley, winter Ketos	1) 14.10.2010 2) 26.04.2011 - 05.05.2011 3) 17.06.2011 - 27.06.2011	SPI SPI	0.175 0.175	300 300	0.0583 0.0583	13.04.2011/0 27.04.2011/14	61	grain**	0.014	<0.01	<0.01	<0.01	51	**samples have been analysed twice (examination and reserve sample). The mean value of both measurements is reported.
									straw**	1.7	0.016	0.45	0.68	51	day 51: c=0.021 mg/kg for JAU 6476-desthio

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	2- A	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	150 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L	
Commercial product	(name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
11-2111 11-2111-02 France, south [redacted] 2011	Barley, winter Campanil	1) 20.10.2010 2) 26.04.2011 - 05.05.2011 3) 15.06.2011 - 30.06.2011	SPI SPI	0.175 0.175	300 300	0.0583 0.0583	14.04.2011/0 27.04.2011/13	61	green material	1.9 0.36	<0.01 <0.01	0.051 0.14	0.079 0.19	0 22	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg day 0: c=0.013 mg/kg for JAU 6476-desthio

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	2- A (continued)		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
11-2111 11-2111-02 (continued)	Barley, winter Campanil	1) 20.10.2010 2) 26.04.2011 - 05.05.2011 3) 15.06.2011 - 30.06.2011	SPI SPI	0.175 0.175	300 300	0.0583 0.0583	14.04.2011/0 27.04.2011/13	61	grain*	0.01	<0.01	<0.01	<0.01	55	*samples have been analysed twice (examination and reserve sample). The mean value of both measurements is reported.
									straw*	0.54	0.015	0.16	0.32	55	day 55: c=0.014 mg/kg for JAU 6476-desthio

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	3- A		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s) / Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL				JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio		
	(a)	(b)	(c)				(d)	(e)	(a)					(f)	
11-2111 11-2111-03 Italy [redacted] 2011	Barley, winter Lutece	1) 15.10.2010 2) 19.04.2011 - 20.04.2011 3) 20.06.2011 - 30.06.2011	SPI SPI	0.175 0.175	400 400	0.0438 0.0438	08.04.2011/0 21.04.2011/13	65	green material	0.82 1.8 1.6 0.74 0.51	<0.01 <0.01 <0.01 <0.01 <0.01	0.044 0.050 0.085 0.12 0.16	0.060 0.063 0.11 0.15 0.19	0* 0 7 14 28	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg
									grain**	<0.01	<0.01	<0.01	<0.01	63	**samples have been analysed twice (examination and reserve sample). The mean value of both measurements is reported.
									straw**	0.34	<0.01	0.15	0.21	63	*day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	4- A	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	150 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L	
Commercial product	(name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
11-2111 11-2111-04 Italy [redacted]2011	Barley, winter Ketos	1) 18.10.2010 2) 23.03.2011 - 30.03.2011 3) 25.06.2011 - 30.06.2011	SPI SPI	0.175 0.175	300 300	0.0583 0.0583	14.04.2011/0 04.05.2011/20	61	green material grain* straw*	3.0 1.1 0.01 0.93	<0.01 <0.01 <0.01 0.025	0.096 0.23 <0.01 0.33	0.13 0.23 <0.01 0.47	0 13 43 43	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *samples have been analysed twice (examination and reserve sample). The mean value of both measurements is reported.

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals		
Country	:	Germany	Page	:	5- A		
Content of active substance (g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor		
Formulation (e.g. WP)	:	150 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
11-2111 11-2111-05 Spain [redacted]2011	Barley, winter Graphic	1) 15.12.2010 2) 28.04.2011 - 03.05.2011 3) 15.06.2011 - 15.07.2011	SPI	0.175	300	0.0583	18.04.2011/0	69	green material	2.4	0.015	0.052	0.060	0	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *samples have been analysed twice (examination and reserve sample). The mean value of both measurements is reported.
			SPI	0.175	300	0.0583	03.05.2011/15			1.1	0.022	0.10	0.11	10	
									grain*	<0.01	<0.01	<0.01	<0.01	49	
									straw*	0.79	0.014	0.18	0.17	49	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1- B**	
Formulation	**(e.g. WP)**	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)			DALT/ PHI (days)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL				**JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**		
	(a)	(b)	(c)				(d)	(e)	(a)				(f)	
11-2111 11-2111-01 France, south [redacted]2011	Barley, winter Ketos	1) 14.10.2010 2) 26.04.2011 - 05.05.2011 3) 17.06.2011 - 27.06.2011	SPI SPI	0.175 0.175	300 300	0.0583 0.0583	13.04.2011/0 27.04.2011/14	61	green material	0.050 0.046 0.088 0.11 0.072	<0.01 <0.01 <0.01 <0.01 0.01	1.0 2.2 1.7 1.5 0.91	0* 0 7 14 28	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc.
									grain**	<0.01	<0.01	0.064	51	
									straw**	0.20	0.042	3.1	51	**samples have been analysed twice (examination and reserve sample). The mean value of both measurements is reported. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Crop/Crop Group**	:	**Cereals**	
Country		:	**Germany**	**Page**	:	**1- B**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Indoor/outdoor**	:	**Outdoor**	
Formulation	**(e.g. WP)**	:	**150 EC**	**Other a.s. in formulation (common name and content)**	:	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Crop/Crop Group**	:	**Cereals**	
Country		:	**Germany**	**Page**	:	**2- B**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Indoor/outdoor**	:	**Outdoor**	
Formulation	**(e.g. WP)**	:	**150 EC**	**Other a.s. in formulation (common name and content)**	:	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	1- B		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	Total residues calc.	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)			DALT/ PHI (days)	Remarks

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1- B**	
Formulation	**(e.g. WP)**	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
Year of Trial	(a)	(b)	(c)	kg a.s./ha	Water (L/ha)	kg a.s./hL	(d)	(e)	(a)	**JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	(f)	
	Barley, winter Campanil		SPI	0.175	300	0.0583	14.04.2011/0	61	green material	0.038	<0.01	2.1	0	
			SPI	0.175	300	0.0583	27.04.2011/13			0.074	<0.01	0.78	22	
									grain*	<0.01	<0.01	0.06	55	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1- B**	
Formulation	**(e.g. WP)**	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date	Growth stage at last treatment	Portion analysed	Residues (mg/kg)			DALT/ PHI (days)	Remarks
	(a)	(b)	(c)	kg a.s./ha	Water (L/ha)	kg a.s./hL	(d)	(e)	(a)	**JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	(f)	
11-2111 11-2111-02 France, south 31290 Gardouch 2011		1) 20.10.2010 2) 26.04.2011 - 05.05.2011 3) 15.06.2011 - 30.06.2011							straw*	0.11	<0.01	1.2	55	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *samples have been analysed twice (examination and reserve sample). The mean value of both measurements is reported. **day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**		
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**		
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Page**	:	**1- B**		
Formulation (e.g. WP)	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**		
			Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**		
Commercial product (name)	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
			Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
			Residues determined as	:	**Total residues calc.**	**Residues calculated as**	: **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**		
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**		
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Page**	:	**3- B**		
Formulation (e.g. WP)	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**		
			Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**		
Commercial product (name)	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
			Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
			Residues determined as	:	**Total residues calc.**	**Residues calculated as**	: **JAU 6476-desthio**

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	1- B		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	Total residues calc.	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)			DALT/ PHI (days)	Remarks

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**	
Country		**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**	
Content of active substance	**(g/kg or g/L)**	**:**	**100 g/L**	**Page**	**:**	**1- B**	
Formulation	**(e.g. WP)**	**:**	**150 EC**	**Indoor/outdoor**	**:**	**Outdoor**	
				Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	**:**	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	**:**	**JAU 6476-desthio**	**Residues calculated as : JAU 6476-desthio**
Producer of commercial product		**:**	**Bayer CropScience AG**	**Residues determined as**	**:**	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	**:**	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	**:**	**Total residues calc.**	**Residues calculated as : JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
Year of Trial	(a)	(b)	(c)	kg a.s./ha	Water (L/ha)	kg a.s./hL	(d)	(e)	(a)	**JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	(f)	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1- B**	
Formulation	**(e.g. WP)**	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
11-2111 11-2111-03 Italy [redacted] 2011	Barley, winter Lutece	1) 15.10.2010 2) 19.04.2011 - 26.04.2011 3) 20.06.2011 - 30.06.2011	SPI	0.175	400	0.0438	08.04.2011/0	65	green material	0.047	<0.01	0.99	0*	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. **samples have been analysed twice (examination and reserve sample). The mean value of both measurements is reported. *day before last treatment
			SPI	0.175	400	0.0438	21.04.2011/13			0.056	<0.01	2.0	0	
										0.077	<0.01	1.9	7	
										0.10	0.011	1.1	14	
										0.12	0.011	1.0	28	
									grain**	<0.01	<0.01	<0.06	63	
									straw**	0.15	<0.01	0.87	63	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole			
Country		:	Germany	Crop/Crop Group	:	Cereals			
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	1- B			
Formulation	(e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor			
				Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L			
Commercial product	(name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	:	JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as	:	JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole			
Country		:	Germany	Crop/Crop Group	:	Cereals			
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	4- B			
Formulation	(e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor			
				Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L			
Commercial product	(name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	:	JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as	:	JAU 6476-desthio

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	1- B		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	Total residues calc.	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)			DALT/ PHI (days)	Remarks

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
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Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1- B**	
Formulation	**(e.g. WP)**	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
Year of Trial	(a)	(b)	(c)	kg a.s./ha	Water (L/ha)	kg a.s./hL	(d)	(e)	(a)	**JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	(f)	
	Barley, winter Ketos		SPI	0.175	300	0.0583	14.06.2011/0	61	green material	0.072	<0.01	3.3	0	
			SPI	0.175	300	0.0583	04.05.2011/20			0.13	<0.01	1.7	13	
									grain*	<0.01	<0.01	0.06	43	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**	
Country		**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**	
Content of active substance	**(g/kg or g/L)**	**:**	**100 g/L**	**Page**	**:**	**1- B**	
Formulation	**(e.g. WP)**	**:**	**150 EC**	**Indoor/outdoor**	**:**	**Outdoor**	
				Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	**:**	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	**:**	**JAU 6476-desthio**	**Residues calculated as : JAU 6476-desthio**
Producer of commercial product		**:**	**Bayer CropScience AG**	**Residues determined as**	**:**	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	**:**	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	**:**	**Total residues calc.**	**Residues calculated as : JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha (d)	Water (L/ha) (d)	kg a.s./hL (d)	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
11-2111 11-2111-04 Italy [redacted] 2011		1) 18.10.2010 2) 23.05.2011 - 30.05.2011 3) 25.06.2011 - 30.06.2011							straw*	0.19	0.018	2.0	43	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *samples have been analysed twice (examination and reserve sample). The mean value of both measurements is reported.

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	5- B		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	Total residues calc.	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
11-2111 11-2111-05 Spain [redacted] 2011	Barley, winter Graphic	1) 15.12.2010 2) 28.04.2011 - 03.05.2011 3) 15.06.2011 - 15.07.2011	SPI SPI	0.175 0.175	300 300	0.0583 0.0583	18.04.2011/0 03.05.2011/15	69	green material grain* straw*	0.040 0.069 <0.01 0.079	<0.01 <0.01 <0.01 <0.01	2.6 1.4 <0.06 1.2	0 10 49 49	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *samples have been analysed twice (examination and reserve sample). The mean value of both measurements is reported.

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Wheat EU foliar spray residue trials – northern EU

Fluoxastrobin & prothioconazole EC 200 in/on winter wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**: Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**: Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**: 100 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**: 200 EC**	**Indoor/outdoor**	**:**	**Outdoor**
		Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 100 g/L**
Commercial product (name)	**: Fluoxastrobin & Prothioconazole EC 200**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**: Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						
RA-2011/00 R 2000 0144/4 0144-00 Sweden S-[redacted] 2000	Wheat, winter Tarso	1) 25.09.1999 2) 09.06.2000 - 17.06.2000 3) 14.08.2000	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	26.05.2000/0 14.06.2000/19	65-69	ear rest of plant straw grain	0.09 1.3 0.04 0.10 0.74 0.08 0.07 <0.01	-1 0 35 -1 0 35 61 61	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	: **Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	: **Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	: **100 g/L**	**Page**	:	**2**
Formulation (e.g. WP)	: **200 EC**	**Indoor/outdoor**	:	**Outdoor**
		Other a.s. in formulation (common name and content)	:	**fluoxastrobin 100 g/L**
Commercial product (name)	: **Fluoxastrobin & Prothioconazole EC 200**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	: **Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2011/00 R 2000 0145/2 0145-00 France, north F-[redacted] 2000	Wheat, winter Shango	1) 06.10.1999 2) 30.05.2000 - 14.06.2000 3) 01.08.2000	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	26.05.2000/0 14.06.2000/19	69	ear rest of plant straw grain	0.04 0.54 0.02 <0.05 0.67 0.10 0.09 <0.01	0* 0 35 0* 0 35 48 48	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *days before last treatment (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	: **prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	: **Cereals**
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Page**	: **3**
Formulation (e.g. WP)	:	**200 EC**	**Indoor/outdoor**	: **Outdoor**
			Other a.s. in formulation (common name and content)	: **fluoxastrobin 100 g/L**
Commercial product (name)	:	**Fluoxastrobin & Prothioconazole EC 200**	**Residues determined as**	: **JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	: **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (e)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2011/00 R 2000 0146/0 0146-00 United Kingdom GB-[redacted] 2000	Wheat, winter Abbot	1) 18.10.1999 2) 06.06.2000 - 13.06.2000 3) 11.08.2000	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	26.05.2000/0 14.06.2000/19	69	ear rest of plant straw grain	0.01 0.60 0.02 <0.05 0.93 0.07 0.11 <0.01	0* 0 35 0* 0 35 58 58	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *days before last treatment (h) 0.01 mg/kg
RA-2011/00 R 2000 0147/9 0147-00 Germany D-[redacted] 2000	Wheat, winter Flair	1) 20.10.1999 2) 02.06.2000 - 06.06.2000 3) 02.08.2000	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	15.05.2000/0 21.06.2000/37	81	ear rest of plant straw grain	<0.01 0.70 <0.05 1.1 <0.05 0.05 <0.01 <0.01	0* 0 0* 0 35 42 35 42	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *days before last treatment (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Prothioconazole & spiroxamine EC 460 in/on spring wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Crop/Crop Group**	**:**	**Cereals**
Country	**:**	**Germany**	**Page**	**:**	**1**
Content of active substance (g/kg or g/L)	**:**	**160 g/L**	**Indoor/outdoor**	**:**	**Outdoor**
Formulation (e.g. WP)	**:**	**460 EC**	**Other a.s. in formulation (common name and content)**	**:**	**spiroxamine 300 g/L**
Commercial product (name)	**:**	**JAU 6476 & KWG 4168 EC 460**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

Active substance **prothioconazole**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2092/00 R 2000 0081/2 0081-00 United Kingdom GB-[redacted] 2000	Wheat, spring Chablis	1) 23.03.2000 2) 18.06.2000 - 28.06.2000 3) 23.08.2000	SPI SPI	0.2000 0.2000	300 300	0.06672 0.06672	22.05.2000/0 28.06.2000/37	69	ear rest of plant grain straw	1.1 0.03 0.02 2.2 0.08 0.06 <0.01 <0.01 0.06 0.06	0 28 35 0 28 35 42 56 42 56	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**: Bayer CropScience AG, Monheim**		**Active substance**	**: prothioconazole**
Country	**: Germany**		**Crop/Crop Group**	**: Cereals**
Content of active substance (g/kg or g/L)	**: 160 g/L**		**Page**	**: 2**
Formulation (e.g. WP)	**: 460 EC**		**Indoor/outdoor**	**: Outdoor**
			Other a.s. in formulation (common name and content)	**: spiroxamine 300 g/L**
Commercial product (name)	**: JAU 6476 & KWG 4168 EC 460**		**Residues determined as**	**: JAU 6476-desthio**
Producer of commercial product	**: Bayer CropScience AG**		**Residues calculated as**	**: JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) / Application interval / or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2092/00 R 2000 0430/3 0430-00 Germany D-[redacted] [redacted]2000	Wheat, spring Lavett	1) 23.03.2000 2) 15.06.2000 - 18.06.2000 3) 16.08.2000	SPI SPI	0.2000 0.2000	300 300	0.06672 0.06672	26.05.2000/0 19.06.2000/24	69	ear	1.7 0.06 0.04 0.04	0 28 35 42	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg
									rest of plant	2.4 0.05 0.06 <0.05	0 28 35 42	(h) 0.05 mg/kg
									grain	<0.01	58	(h) 0.01 mg/kg
									straw	0.07	58	(h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	: **Bayer CropScience AG, Monheim**		**Active substance**	:	**prothioconazole**
Country	: **Germany**		**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	: **160 g/L**		**Page**	:	**3**
Formulation (e.g. WP)	: **460 EC**		**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**spiroxamine 300 g/L**
Commercial product (name)	: **JAU 6476 & KWG 4168 EC 460**		**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	: **Bayer CropScience AG**		**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2092/00 R 2000 0431/1 0431-00 Germany D-[redacted] [redacted]2000	Wheat, spring Lavett	1) 23.03.2000 2) 10.06.2000 - 15.06.2000 3) 31.07.2000	SPI SPI	0.2000 0.2000	300 300	0.06672 0.06672	16.05.2000/0 15.06.2000/30	69	ear rest of plant grain straw	1.5 0.10 0.07 2.9 0.39 0.36 <0.01 <0.01 0.28 0.14	0 28 35 0 28 35 41 49 41 49	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg
RA-2092/00 R 2000 0433/8 0433-00 France, north F-[redacted] [redacted]2000	Wheat, spring Furio	1) 10.03.2000 2) 23.06.2000 - 06.07.2000 3) 17.08.2000	SPI SPI	0.2000 0.2000	300 300	0.06672 0.06672	16.05.2000/0 06.07.2000/51	69	ear rest of plant grain straw	0.92 0.04 1.9 0.06 <0.01 <0.01 0.06 0.06	0 28 0 28 35 42 35 42	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Fluoxastrobin & prothioconazole & trifloxystrobin EC 300 in/on wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**150 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**300 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 75 g/L trifloxystrobin 75 g/L**
Commercial product (name)	**:**	**Fluoxastrobin & Prothioconazole & Trifloxystrobin EC 300**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropSciences AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT* PHI (days) (f)	Remarks
RA-2018/03 R 2003 0133/2 0133-03 Germany D-[redacted] 2003	Wheat Magnus	1) 23.10.2002 2) 05.06.2003 - 16.06.2003 3) 15.08.2003	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	26.05.2003/0 12.06.2003/17	69	rest of plant	<0.05 2.2	0* 0	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg
									ear	<0.01 1.0	0* 0	(h) 0.01 mg/kg *: before last application
									grain	<0.01	35<<	
									straw	0.06	35<<	(h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**150 g/L**	**Page**	**:**	**2**
Formulation (e.g. WP)	**:**	**300 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 75 g/L trifloxystrobin 75 g/L**
Commercial product (name)	**:**	**Fluoxastrobin & Prothioconazole & Trifloxystrobin EC 300**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2018/03 R 2003 0249/5 0249-03 Germany D-[redacted] 2003	Wheat Winnetou	1) 10.10.2002 2) 29.05.2003 - 08.06.2003 3) 22.07.2003	SPI SPI	0.1500 0.1500	300 300	0.04995 0.04995	28.05.2003/0 04.06.2003/12	69	rest of plant ear grain straw	0.06 0.65 0.04 0.62 <0.01 <0.01 0.05 0.05	0* 0 0* 0 35<< 46 35<< 46	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg *: before last application (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Prothioconazole & trifloxystrobin SC325 in/on soft wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**175 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**325 SC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**trifloxystrobin 150 g/L**
Commercial product (name)	**:**	**JAU 6476 & CGA 279202 SC 325**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2108/03 R 2003 0941/4 0941-03 France, north F-[redacted] 2003	Wheat, soft Tremie	1) 30.10.2002 2) 30.05.2003 - 11.06.2003 3) 18.07.2003	SPI SPI	0.1750 0.1750	300 300	0.05828 0.05828	14.05.2003/0 11.06.2003/28	69	ear rest of plant grain straw	0.32 0.70 <0.01 0.09	0 0 37 37	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg
RA-2108/03 R 2003 0980/5 [redacted] 2003	Wheat, soft Claire	1) 05.10.2002 2) 16.06.2003 - 29.06.2003 3) 11.08.2003	SPI SPI	0.1750 0.1750	300 300	0.05828 0.05828	05.06.2003/0 04.07.2003/29	71	ear rest of plant grain straw	0.25 1.0 0.02 0.79	0 0 34 34	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Prothioconazole & trifloxystrobin SC 263 in/on winter wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**175 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**263 SC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**trifloxystrobin 88 g/L**
Commercial product (name)	**:**	**JAU 6476 + CGA 279202 SC 263**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2575/05 R 2005 0028/9 0028-05 France, north F-[redacted] [redacted](Centre) 2005	Wheat, winter APACHE	1) 14.10.2004 2) 20.05.2005 - 30.05.2005 3) 13.07.2005	SPI SPI	0.1995 0.1995	300 300	0.06650 0.06650	12.05.2005/0 30.05.2005/18	69	green material grain straw	0.06 0.62 0.28 0.10 <0.01 0.20	0* 0 28 35 42 42	(c) SPI:Spraying (g) 00598/M001 (h) 0.05 mg/kg *: before last application (h) 0.01 mg/kg (h) 0.05 mg/kg
RA-2575/05 R 2005 0029/7 0029-05 Sweden S-[redacted] [redacted]2005	Wheat, winter Ritmo	1) 08.09.2004 2) 16.06.2005 - 23.06.2005 3) 16.08.2005 - 17.08.2005	SPI SPI	0.1995 0.1995	300 300	0.06650 0.06650	08.06.2005/0 23.06.2005/15	69	green material grain straw	1.3 0.85 <0.01 0.65	0 35 54 54	(c) SPI:Spraying (g) 00598/M001 (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**175 g/L**	**Page**	:	**2**
Formulation (e.g. WP)	:	**263 SC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**trifloxystrobin 88 g/L**
Commercial product (name)	:	**JAU 6476 + CGA 279202 SC 263**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2575/05 R 2005 0895/6 0895-05 United Kingdom GB-[redacted] [redacted] 2005	Wheat, winter Einstein	1) 09.10.2004 2) 10.06.2005 - 21.06.2005 3) 08.08.2005 - 20.08.2005	SPI SPI	0.1995 0.1995	300 300	0.06650 0.06650	27.05.2005/0 21.06.2005/25	69	green material grain straw	0.17 0.61 0.14 0.11 <0.01 0.22	0* 0 50 50	(c) SPI:Spraying (g) 00598/M001 (h) 0.05 mg/kg *: before last application (h) 0.01 mg/kg (h) 0.05 mg/kg
RA-2575/05 R 2005 0896/4 0896-05 Germany D-[redacted] [redacted] 2005	Wheat, winter Winnetou	1) 10.10.2004 2) 05.06.2005 - 15.06.2005 3) 01.08.2005 - 25.08.2005	SPI SPI	0.1995 0.1995	300 300	0.06650 0.06650	27.05.2005/0 08.06.2005/12	69	green material grain straw	0.46 0.08 <0.01 0.12	0 35 54 54	(c) SPI:Spraying (g) 00598/M001 (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Bixafen & Prothioconazole EC 225 in/on spring wheat and winter wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address) : **Bayer CropScience AG, Monheim**
Country : **Germany**
Content of active substance (g/kg or g/L) : **150 g/L**
Formulation (e.g. WP) : **225 EC**

Commercial product (name) : **Bixafen & Prothioconazole EC 225**
Producer of commercial product : **Bayer CropScience AG**

Active substance : **prothioconazole**
Crop/Crop Group : **Cereals**
Page : **1**
Indoor/outdoor : **Outdoor**
Other a.s. in formulation (common name and content) : **BYF 00587 75 g/L**
Residues determined as : **JAU 6476-desthio**
Residues calculated as : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2037/07 R 2007 0418/6 0418-07 Netherlands NL-[redacted] 2007	Wheat, spring Baldus	1) 06.04.2007 2) 15.06.2007 - 27.06.2007 3) 24.08.2007 - 03.09.2007	SPI SPI	0.1875 0.1875	300 300	0.06255 0.06255	11.06.2007/0 27.06.2007/16	69	green material straw grain 	0.07 0.86 0.09 0.04 <0.01 0.02 0.03 <0.01 <0.01	0* 0 7 14 28 35 61 35 61	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *: days before last application
RA-2037/07 R 2007 0420/8 0420-07 United Kingdom GB-[redacted] 2007	Wheat, spring Belvoir	1) 18.03.2007 2) 13.06.2007 - 20.06.2007 3) 31.08.2007 - 01.09.2007	SPI SPI	0.1875 0.1875	300 300	0.06255 0.06255	01.06.2007/0 19.06.2007/18	69	green material ear rest of plant straw grain	0.14 1.1 <0.01 0.02 0.03 <0.01	0* 0 35 35 73 73	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *: days before last application

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address) : **Bayer CropScience AG, Monheim**

Active substance : **prothioconazole**
Crop/Crop Group : **Cereals**

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**150 g/L**	**Page**	:	**1**
Formulation (e.g. WP)	:	**225 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**BYF 00587 75 g/L**
Commercial product (name)	:	**Bixafen & Prothioconazole EC 225**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			Dates of treatment(s)/ Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						
Country		: **Germany**					**Page**	: **2**				
Content of active substance (g/kg or g/L)		: **150 g/L**					**Indoor/outdoor**	: **Outdoor**				
Formulation (e.g. WP)		: **225 EC**					**Other a.s. in formulation (common name and content)**	: **BYF 00587 75 g/L**				
Commercial product (name)		: **Bixafen & Prothioconazole EC 225**					**Residues determined as**	: **JAU 6476-desthio**				
Producer of commercial product		: **Bayer CropScience AG**					**Residues calculated as**	: **JAU 6476-desthio**				

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	: **prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	: **Cereals**
Content of active substance (g/kg or g/L)	:	**150 g/L**	**Page**	: **1**
Formulation (e.g. WP)	:	**225 EC**	**Indoor/outdoor**	: **Outdoor**
			Other a.s. in formulation (common name and content)	: **BYF 00587 75 g/L**
Commercial product (name)	:	**Bixafen & Prothioconazole EC 225**	**Residues determined as**	: **JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	: **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2037/07 R 2007 0421/6 0421-07 Germany D-[redacted] 2007	Wheat, spring Thasos	1) 05.04.2007 2) 21.06.2007 - 05.07.2007 3) 27.08.2007 - 28.08.2007	SPI SPI	0.1875 0.1875	300 300	0.06255 0.06255	14.06.2007/0 05.07.2007/21	69	green material ear rest of plant straw grain	0.02 1.2 0.01 0.03 0.04 <0.01	0* 0 35 35 53 53	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *: days before last application
RA-2037/07 R 2007 0419/4 0419-07 France, north F-[redacted] 2007	Wheat, winter Mercato	1) 09.10.2006 2) 09.05.2007 - 18.05.2007 3) 13.07.2007	SPI SPI	0.1875 0.1875	300 300	0.06255 0.06255	25.04.2007/0 18.05.2007/23	69	green material ear rest of plant straw grain	0.08 1.7 0.32 0.12 0.06 0.01 0.07 0.10 <0.01	0* 0 7 14 28 35 35 55 55	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *: days before last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

Bixafen & prothioconazole & spiroxamine EC 400 in/on spring and winter wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1**
Formulation	**(e.g. WP)**	:	**400 EC**	**Indoor/outdoor**	:	**Outdoor**
				Other a.s. in formulation (common name and content)	:	**BYF 00587 50 g/L spiroxamine 250 g/L**
Commercial product	**(name)**	:	**Bixafen & Prothioconazole & Spiroxamine EC 400**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT* PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						
RA-2040/07 R 2007 0443/7 0443-07 France, north F-[redacted] 2007	Wheat, winter Mendel	1) 16.10.2006 2) 15.05.2007 - 25.05.2007 3) 04.07.2007 - 14.07.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	30.04.2007/0 22.05.2007/22	69	green material ear rest of plant straw grain	0.04 0.84 0.02 0.08 0.08 <0.01	0* 0 35 35 44 44	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *: days before last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	: **Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	: **Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	: **100 g/L**	**Page**	:	**2**
Formulation (e.g. WP)	: **400 EC**	**Indoor/outdoor**	:	**Outdoor**
		Other a.s. in formulation (common name and content)	:	**BYF 00587 50 g/L spiroxamine 250 g/L**
Commercial product (name)	: **Bixafen & Prothioconazole & Spiroxamine EC 400**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	: **Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) (f)	DALT/ PHI (days) (f)	Remarks
RA-2040/07 R 2007 0444/5 0444-07 Netherlands NL-[redacted] 2007	Wheat, spring Baldus	1) 06.04.2007 2) 15.06.2007 - 27.06.2007 3) 24.08.2007 - 03.09.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	11.06.2007/0 27.06.2007/16	69	green material straw grain	0.03 0.92 0.02 0.02 <0.01 <0.01	0* 0 35 61 35 61	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *: days before last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Page**	:	**3**
Formulation (e.g. WP)	:	**400 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**BYF 00587 50 g/L spiroxamine 250 g/L**
Commercial product (name)	:	**Bixafen & Prothioconazole & Spiroxamine EC 400**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT* PHI (days) (f)	Remarks
RA-2040/07 R 2007 0523/9 0523-07 United Kingdom GB-[redacted] [redacted]2007	Wheat, spring Belvoir	1) 18.03.2007 2) 13.06.2007 - 20.06.2007 3) 31.08.2007 - 01.09.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	01.06.2007/0 19.06.2007/18	69	green material ear rest of plant straw grain	0.04 0.92 <0.01 0.01 0.03 <0.01	0* 0 35 35 73 73	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *: days before last application
RA-2040/07 R 2007 0524/7 0524-07 Germany D-[redacted] 2007	Wheat, spring Thasos	1) 04.04.2007 2) 06.06.2007 - 15.06.2007 3) 14.08.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	06.06.2007/0 19.06.2007/13	69	green material straw grain	0.16 1.5 0.04 0.04 <0.01 <0.01	0* 0 35 56 35 56	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *: days before last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Bixafen & Prothioconazole EC 260 in/on winter wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**: Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**: Germany**	**Crop / Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**: 200 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**: 260 EC**	**Indoor/outdoor**	**:**	**Outdoor**
		Other a.s. in formulation (common name and content)	**:**	**BYF 00587 60 g/L**
Commercial product (name)	**: Bixafen & Prothioconazole EC 260**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**: Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT* PHI (days) (f)	Remarks
09-2057 09-2057-01 Germany [redacted] 2009	Wheat, winter Dekan	1) 31.10.2008 2) 09.06.2009 - 18.06.2009 3) 30.07.2009	SPI SPI	0.20 0.20	300 300	0.067 0.067	28.05.2009/0 18.06.2009/21	69	green material grain straw	0.07 0.76 <0.01 <0.01 0.04 0.05	0* 0 35 42 35 42	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *days before last treatment
09-2057 09-2057-02 Netherlands [redacted] 2009	Wheat, winter Tataros	1) 15.10.2008 2) 01.06.2009 - 19.06.2009 3) 22.07.2009 - 28.07.2009	SPI SPI	0.20 0.20	300 300	0.067 0.067	28.05.2009/0 19.06.2009/22	69	green material grain straw	0.18 0.98 <0.01 0.06	0* 0 35 35	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg *days before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Bixafen & Fluoxastrobin & Prothioconazole EC 190 in/on spring and winter wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	1-A		
Formulation (e.g. WP)	:	190 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L		
Commercial product (name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (l/ha)	kg a.s./hL	Dates of treatment(s) Application interval; or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2205 10-2205-01 United Kingdom [redacted] 2010	Wheat, winter Robigus	1) 09.10.2009 2) 10.06.2010 - 25.06.2010 3) 04.08.2010 - 13.08.2010	SPI SPI	0.175 0.175	200 200	0.0875 0.0875	11.06.2010/0 25.06.2010/14	69	green material grain straw	0.41 1.2 <0.01 0.29	0.10 0.11 <0.01 0.10	0.07 0.08 <0.01 0.18	0.08 0.09 <0.01 0.15	0* 0 35 35	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	2-A	
Formulation	(e.g. WP)	:	190 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (l/ha)	kg a.s./hL	Dates of treatment(s) Application interval; or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2205 10-2205-02 Germany [redacted] 2010	Wheat, spring Kadrilj	1) 08.04.2010 2) 01.07.2010 - 07.07.2010 3) 11.08.2010 - 13.08.2010	SPI SPI	0.175 0.175	400 400	0.0438 0.0438	23.06.2010/0 07.07.2010/14	69	green material grain straw	0.38 1.4 <0.01 0.19	0.14 0.13 <0.01 0.30	0.16 0.15 <0.01 0.34	0.12 0.12 <0.01 0.27	0* 0 35 35	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	1- B	
Formulation	(e.g. WP)	:	190 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (l/ha)	kg a.s./hL	Dates of treatment(s) Application interval/ or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2205 10-2205-01 United Kingdom [redacted] 2010	Wheat, winter Robigus	1) 09.10.2009 2) 10.06.2010 - 25.06.2010 3) 04.08.2010 - 13.08.2010	SPI SPI	0.175 0.175	200 200	0.0875 0.0875	11.06.2010/0 25.06.2010/14	69	green material grain straw	0.08 0.10 <0.01 0.15	0.02 0.02 <0.01 0.04	0.76 1.6 <0.06 0.91	0* 0 35 35	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	2- B	
Formulation	(e.g. WP)	:	190 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-8-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2205 10-2205-02 Germany [redacted] [redacted]2010	Wheat, spring Kadrilj	1) 08.04.2010 2) 01.07.2010 - 07.07.2010 3) 11.08.2010 - 13.08.2010	SPI SPI	0.175 0.175	400 400	0.0438 0.0438	23.06.2010/0 07.07.2010/14	69	green material grain straw	0.13 0.13 <0.01 0.28	0.02 0.02 <0.01 0.04	0.95 2.0 <0.06 1.4	0* 0 35 35	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Fluoxastrobin & Prothioconazole EC 200 in/on wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	1- A		
Formulation (e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor		
Commercial product (name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L		
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
13-2138 13-2138-01 Germany [redacted] 2013	Wheat, spring Taifun	1) 03.03.2013 2) 21.06.2013 - 26.06.2013 3) 15.08.2013 - 31.08.2013	SPI SPI	0.15 0.15	300 300	0.050 0.050	11.06.2013/0 26.06.2013/15	69	green material grain straw	0.063 0.90 0.18 0.055 0.042 0.023 0.025 <0.01 <0.01 0.027 0.038	0.081 0.094 0.088 0.075 0.064 0.051 0.052 <0.01 <0.01 0.044 0.020	0.068 0.079 0.087 0.094 0.085 0.086 0.079 <0.01 <0.01 0.099 0.035	0.072 0.089 0.10 0.10 0.088 0.091 0.086 <0.01 <0.01 0.086 0.035	0* 0 7 14 21 28 28 35 57 35 57	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole			
Country	:	Germany	Crop/Crop Group	:	Cereals			
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	1- A			
Formulation (e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor			
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L			
Commercial product (name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s) Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)	Remarks
Year of Trial	(a)	(b)	(c)	kg a.s./ha	Water (L/ha)	kg a.s./hL	(d)	(e)	(a)	JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	(f)	

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole			
Country	:	Germany	Crop/Crop Group	:	Cereals			
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	2- A			
Formulation (e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor			
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L			
Commercial product (name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	1- A		
Formulation (e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L		
Commercial product (name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)				DALT/ PHI (days) (f)	Remarks
										JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio		
1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)	Remarks

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	1- A	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	200 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s) Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)	Remarks
Year of Trial	(a)	(b)	(c)	kg a.s./ha	Water (L/ha)	kg a.s./hL	(d)	(e)	(a)	**JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	(f)	
Year of Trial	(a)	(b)	(c)	kg a.s./ha	Water (L/ha)	kg a.s./hL	(d)	(e)	(a)	**JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	(f)	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	1- A	
Formulation	(e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
13-2138 13-2138-02 [redacted] 2013	Wheat, spring Kadrilj	1) 09.04.2013 2) 01.07.2013 - 01.07.2013 3) 20.08.2013 - 30.08.2013	SPI SPI	0.15 0.15	400 400	0.038 0.038	18.06.2013/0 02.07.2013/14	69	green material grain straw	0.25 0.92 0.51 0.33 0.19 0.074 0.11 <0.01 0.11	0.13 0.12 0.24 0.26 0.32 0.18 0.21 <0.01 0.17	0.11 0.11 0.14 0.18 0.21 0.16 0.15 <0.01 0.11	0.096 0.093 0.13 0.15 0.17 0.13 0.13 <0.01 0.12	0* 0 7 14 21 28 35 54 54	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	3- A	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	200 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
13-2138 13-2138-03 United Kingdom [redacted] 2013	Wheat, spring Alderon; (feed)	1) 03.04.2013 2) 24.06.2013 - 08.07.2013 3) 12.08.2013 - 30.08.2013	SPI SPI	0.158 0.15	211 200	0.0749 0.075	19.06.2013/0 08.07.2013/19	69	green material	0.45 1.8 1.1 0.64 0.37 0.50 0.44	0.061 0.065 0.13 0.17 0.14 0.14 0.18	0.15 0.14 0.21 0.25 0.22 0.24 0.27	0.083 0.069 0.14 0.17 0.14 0.16 0.18	0* 0 7 14 21 28 24	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment
									grain	<0.01	<0.01	<0.01	<0.01	35	
									straw	0.92	0.31	0.64	0.42	35	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	4- A	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	200 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) / Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
13-2159 13-2159-01 France, north [redacted] 2013	Wheat Siala	1) 09.10.2012 3) 23.07.2013	SPI SPI	0.15 0.15	200 200	0.075 0.075	14.05.2013/0 04.06.2013/21	69	green material	0.027 1.4 0.71 0.25 0.13 0.075 0.074	0.014 0.018 0.13 0.13 0.078 0.062 0.055	0.021 0.025 0.11 0.12 0.097 0.096 0.088	0.015 0.019 0.10 0.10 0.078 0.086 0.084	0* 0 7 14 21 28 34	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg *day before last treatment
									grain	<0.01	<0.01	<0.01	<0.01	49	
									straw	0.14	0.052	0.12	0.091	49	day 49: c=0.014 mg/kg for JAU 6476-desthio

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole			
Country	:	Germany	Crop/Crop Group	:	Cereals			
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	4- A			
Formulation (e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor			
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L			
Commercial product (name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s); Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL				JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio		
Year of Trial	(a)	(b)	(c)				(d)	(e)	(a)					(f)	

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole			
Country	:	Germany	Crop/Crop Group	:	Cereals			
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	5- A			
Formulation (e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor			
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L			
Commercial product (name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	:	JAU 6476-desthio

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	4- A	
Formulation	(e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)	Remarks

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**4- A**	
Formulation	**(e.g. WP)**	:	**200 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 100 g/L**	
Commercial product	**(name)**	:	**Bixafen & Fluoxastrobin & Prothioconazole EC 190**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-alpha-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-3-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-4-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s) Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)	Remarks
Year of Trial	(a)	(b)	(c)	kg a.s./ha	Water (L/ha)	kg a.s./hL	(d)	(e)	(a)	**JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	(f)	
Year of Trial	(a)	(b)	(c)	kg a.s./ha	Water (L/ha)	kg a.s./hL	(d)	(e)	(a)	**JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	(f)	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	4- A	
Formulation	(e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
13-2159 13-2159-02 France, north [redacted] [redacted]2013	Wheat, spring Valbona	1) 12.03.2013 3) 30.07.2013	SPI SPI	0.136 0.150	181 212	0.0750 0.0750	06.06.2013/0 25.06.2013/19	69	green material grain straw	1.1 0.46 <0.01 0.61	<0.01 <0.01 <0.01 0.014	0.052 0.081 <0.01 0.22	0.045 0.076 <0.01 0.15	0 15 35 35	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	1- B	
Formulation	(e.g. WP)	:	200 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (l/ha)	kg a.s./hL	Dates of treatment(s) Application interval/ or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
13-2138 13-2138-01 Germany [redacted] 2013	Wheat, spring Taifun	1) 25.03.2013 2) 21.06.2013 - 26.06.2013 3) 15.08.2013 - 31.08.2013	SPI SPI	0.15 0.15	300 300	0.050 0.050	1) 06.2013/0 26.06.2013/15	69	green material	0.053 0.073 0.072 0.070 0.059 0.063 0.059	0.011 0.015 0.014 0.012 0.012 0.012 0.012	0.35 1.3 0.54 0.41 0.35 0.33 0.31	0* 0 7 14 21 28 28	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc.
									grain	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06	35 57	*day before last treatment
									straw	0.070 0.031	0.016 <0.01	0.34 0.17	35 57	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	2- B	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	200 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-8-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

Active substance : prothioconazole

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
13-2138 13-2138-02 Germany [redacted] 2013	Wheat, spring Kadrilj	1) 09.04.2013 2) 01.07.2013 - 01.07.2013 3) 20.08.2013 - 30.08.2013	SPI SPI	0.15 0.15	400 400	0.038 0.038	18.06.2013/0 02.07.2013/14	69	green material grain straw	0.11 0.11 0.13 0.18 0.19 0.13 0.12 <0.01 0.13	0.018 0.018 0.023 0.038 0.046 0.028 0.031 <0.01 0.017	0.71 1.4 1.2 1.1 1.1 0.70 0.75 <0.06 0.66	0* 0 7 14 21 28 35 54 54	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	3- B	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	200 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-8-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
13-2138 13-2138-03 United Kingdom [redacted] 2013	Wheat, spring Alderon; (feed)	1) 03.04.2013 2) 24.06.2013 - 08.07.2013 3) 12.08.2013 - 30.08.2013	SPI SPI	0.158 0.15	211 200	0.0749 0.075	19.06.2013/0 08.07.2013/19	69	green material grain straw	0.089 0.070 0.14 0.14 0.12 0.12 0.14 <0.01 0.30	<0.01 <0.01 0.018 0.023 0.021 0.023 0.028 <0.01 0.055	0.84 2.2 1.7 1.4 1.0 1.2 1.2 <0.06 2.6	0* 0 7 14 21 28 24 35 35	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	4- B	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	200 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-8-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
13-2159 13-2159-01 France, north [redacted] 2013	Wheat Siala	1) 09.10.2012 3) 23.07.2013	SPI SPI	0.15 0.15	200 200	0.075 0.075	14.05.2013/0 04.06.2013/21	69	green material grain straw	0.020 0.023 0.11 0.10 0.086 0.086 0.078 <0.01 0.11	<0.01 <0.01 0.027 0.025 0.027 0.023 0.022 <0.01 0.029	0.11 1.5 1.2 0.73 0.50 0.43 0.40 <0.01 0.54	0* 0 7 14 21 28 34 49 49	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. *day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Crop/Crop Group**	:	**Cereals**	
Country		:	**Germany**	**Page**	:	**5- B**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Indoor/outdoor**	:	**Outdoor**	
Formulation	**(e.g. WP)**	:	**200 EC**	**Other a.s. in formulation (common name and content)**	:	**fluoxastrobin 100 g/L**	
Commercial product	**(name)**	:	**Bixafen & Fluoxastrobin & Prothioconazole EC 190**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as : JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as : JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as : JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
13-2159 13-2159-02 France, north [redacted] [redacted]2013	Wheat, spring Valbona	1) 03.03.2013 3) 30.07.2013	SPI SPI	0.136 0.159	181 212	0.0750 0.0750	06.06.2013/0 25.06.2013/19	69	green material grain straw	0.11 0.13 <0.01 0.26	0.012 0.021 <0.01 0.053	1.3 0.78 <0.06 1.3	0 15 35 35	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc.

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Wheat EU foliar spray residue trials – southern EU

Fluoxastrobin & prothioconazole EC 200 in/on winter and durum wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)
(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**: Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**: Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**: 100 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**: 200 EC**	**Indoor/outdoor**	**:**	**Outdoor**
		Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 100 g/L**
Commercial product (name)	**: Fluoxastrobin & Prothioconazole EC 200**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**: Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2012/00 R 2000 0148/7 0148-00 [redacted] 2000	Wheat, durum Soisson	1) 01.12.1999 3) 06.07.2000	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	10.05.2000(-) 16.05.2000(-)	71	ear rest of plant straw grain	0.10 1.9 0.08 2.7 0.29 0.22 <0.01 <0.01	0* 0 0* 0 34 51 34 51	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *: days before last treatment (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals
Country	:	Germany	Page	:	2
Content of active substance (g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor
Formulation (e.g. WP)	:	200 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 200	Residues determined as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues calculated as	:	JAU 6476-desthio
			Active substance	:	prothioconazole

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2012/00 R 2000 0149/5 0149-00 Italy I-[redacted] 2000	Wheat, durum Creso	1) 12.11.1999 2) 19.04.2000 - 03.05.2000 3) 19.06.2000	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	07.04.2000/0 03.05.2000/26	69	ear rest of plant straw grain	<0.01 2.3 <0.05 1.0 0.23 0.36 <0.01 <0.01	0* 0 0* 0 35 47 35 47	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *: days before last treatment (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals
Country	:	Germany	Page	:	3
Content of active substance (g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor
Formulation (e.g. WP)	:	200 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 100 g/L
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 200	Residues determined as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues calculated as	:	JAU 6476-desthio
			Active substance	:	prothioconazole

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2012/00 R 2000 0150/9 0150-00 France, south F-[redacted]2000	Wheat, winter Tremie	1) 28.10.1999 2) 10.05.2000 - 17.05.2000 3) 13.07.2000	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	05.05.2000/0 17.05.2000/12	69	ear rest of plant straw grain	0.04 1.6 0.05 0.11 1.4 0.13 0.30 <0.01	0* 0 35 0* 0 35 57 57	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *: days before last treatment (h) 0.01 mg/kg
RA-2012/00 R 2000 0151/7 0151-00 France, south F-[redacted]2000	Wheat, winter Sideral	1) 04.11.1999 2) 08.05.2000 - 18.05.2000 3) 03.07.2000	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	02.05.2000/0 15.05.2000/13	69	ear rest of plant straw grain	0.12 1.2 0.23 0.86 0.10 0.22 <0.01 <0.01	0* 0 0* 0 35 49 35 49	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg *: days before last treatment (h) 0.01 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Prothioconazole & spiroxamine EC 460 in/on spring wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**160 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**460 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**spiroxamine 300 g/L**
Commercial product (name)	**:**	**JAU 6476 & KWG 4168 EC 460**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						
RA-2092/00 R 2000 0082/0 0082-00 France, south F-[redacted] [redacted]2000	Wheat, spring Furio	1) 10.03.2000 2) 05.06.2000 - 14.06.2000 3) 01.08.2000	SPI SPI	0.2000 0.2000	300 300	0.06672 0.06672	12.05.2000/0 13.06.2000/32	69	ear rest of plant grain straw	1.5 0.06 2.9 0.63 <0.01 <0.01 <0.01 1.1 0.90 1.2	0 28 0 28 35 42 49 35 42 49	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	: **Bayer CropScience AG, Monheim**		**Active substance**	:	**prothioconazole**
Country	: **Germany**		**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	: **160 g/L**		**Page**	:	**2**
Formulation (e.g. WP)	: **460 EC**		**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**spiroxamine 300 g/L**
Commercial product (name)	: **JAU 6476 & KWG 4168 EC 460**		**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	: **Bayer CropScience AG**		**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval, or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2092/00 R 2000 0434/6 0434-00 Italy [redacted] 2000	Wheat, spring Centauro	1) 23.12.1999 2) 10.05.2000 - 15.05.2000 3) 29.06.2000 - 07.07.2000	SPI SPI	0.2000 0.2000	300 300	0.06672 0.06672	02.05.2000/0 15.05.2000/13	69	green material	2.7	0	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg
									ear	0.05 0.06	28 35	(h) 0.01 mg/kg
									rest of plant	0.15 0.18	28 35	(h) 0.05 mg/kg
									grain	<0.01 <0.01	42 49	(h) 0.01 mg/kg
									straw	0.18 0.17	42 49	(h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Fluoxastrobin & prothioconazole & trifloxystrobin EC 300 in/on wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**150 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**300 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**fluoxastrobin 75 g/L trifloxystrobin 75 g/L**
Commercial product (name)	**:**	**Fluoxastrobin & Prothioconazole & Trifloxystrobin EC 300**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT* PHI (days) (f)	Remarks
RA-2019/03 R 2003 0134/0A 0134-03A France, south F-[redacted] 2003	Wheat Frelon	1) 20.10.2002 2) 11.05.2003 - 17.05.2003 3) 25.06.2003	SPI SPI	0.15 0.15	300 300	0.050 0.050	05.05.2003/0 15.05.2003/10	69	rest of plant ear grain straw	0.24 1.7 0.21 2.1 <0.01 <0.01 <0.05 0.27	0* 0 0* 0 35<< 41 35<< 41	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg (h) 0.01 mg/kg *: before last the application (h) 0.05 mg/kg
RA-2019/03 R 2003 0257/6 0257-03 Spain E-[redacted] 2003	Wheat Yecora	1) 16.01.2003 2) 23.04.2003 - 08.05.2003 3) 18.06.2003	SPI SPI	0.1422 0.1422	300 300	0.04740 0.04740	23.04.2003/0 08.05.2003/15	69	grain straw	<0.01 <0.01 0.40 0.62	34 46 34 46	(c) SPI:Spraying (g) 00647 (h) 0.01 mg/kg (h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Prothioconazole &Trifloxystrobin SC325 in/on soft wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**175 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**325 SC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**trifloxystrobin 150 g/L**
Commercial product (name)	**:**	**JAU 6476 & CGA 279202 SC 325**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2109/03 R 2003 0942/2 0942-03 Italy I-[redacted] 2003	Wheat, soft Centauro	1) 15.10.2002 2) 08.05.2003 - 19.05.2003 3) 26.06.2003	SPI SPI	0.1638 0.1750	281 300	0.05828 0.05828	04.05.2003/0 13.05.2003/9	69	green material	0.75	-9	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg
									ear	0.11	-9	(h) 0.01 mg/kg
									grain	<0.01 <0.01	35<< 44	
									straw	0.64 0.75	35<< 44	(h) 0.05 mg/kg
RA-2109/03 R 2003 0981/3 0981-03 Portugal P-[redacted] 2003	Wheat, soft Bancal	1) 04.02.2003 3) 15.07.2003	SPI SPI	0.1750 0.1614	300 277	0.05828 0.05828	15.05.2003/0 30.05.2003/15	69	green material	0.99	0	(c) SPI:Spraying (g) 00647 (h) 0.05 mg/kg
									ear	1.4	0	(h) 0.01 mg/kg
									grain	<0.01	35<<<	
									straw	1.9	35<<<	(h) 0.05 mg/kg

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Bixafen & Prothioconazole EC 225 in/on winter and durum wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**150 g/L**	**Page**	:	**1**
Formulation (e.g. WP)	:	**225 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**BYF 00587 75 g/L**
Commercial product (name)	:	**Bixafen & Prothioconazole EC 225**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) 	DALT/ PHI (days) (f)	Remarks
RA-2038/07 R 2007 0422/4 0422-07 France, south F-[redacted] 2007	Wheat, winter Andalou	1) 23.10.2006 2) 02.05.2007 - 09.05.2007 3) 06.07.2007 - 17.07.2007	SPI SPI	0.1875 0.1875	300 300	0.06255 0.06255	25.04.2007/0 09.05.2007/14	69	green material ear rest of plant straw grain	0.33 0.92 0.70 0.24 0.05 <0.01 0.07 0.10 <0.01	0* 0 7 14 28 35 35 63 63	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**: prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**: Cereals**
Content of active substance (g/kg or g/L)	**:**	**150 g/L**	**Page**	**: 2**
Formulation (e.g. WP)	**:**	**225 EC**	**Indoor/outdoor**	**: Outdoor**
			Other a.s. in formulation (common name and content)	**: BYF 00587 75 g/L**
Commercial product (name)	**:**	**Bixafen & Prothioconazole EC 225**	**Residues determined as**	**: JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**: JAU 6476-desthio**

1	2	3	4	5			6	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment			Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
				kg a.s./ha	Water (L/ha)	kg a.s./hL						
RA-2038/07 R 2007 0423/2 0423-07 Spain E-[redacted] 2007	Wheat, winter Bolido R1	1) 13.12.2006 2) 11.04.2007 - 18.04.2007 3) 01.06.2007 - 30.06.2007	SPI SPI	0.1875 0.1875	300 300	0.06255 0.06255	30.03.2007/0 18.04.2007/19	69	green material rest of plant ear straw grain	0.27 0.31 0.20 0.02 0.27 <0.01	0* 0 36 36 54 54	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals
Country	:	Germany	Page	:	3
Content of active substance (g/kg or g/L)	:	150 g/L	Indoor/outdoor	:	Outdoor
Formulation (e.g. WP)	:	225 EC	Other a.s. in formulation (common name and content)	:	BYF 00587 75 g/L
Commercial product (name)	:	Bixafen & Prothioconazole EC 225	Residues determined as	:	JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues calculated as	:	JAU 6476-desthio
			Active substance	:	prothioconazole

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2038/07 R 2007 0424/0 0424-07 Portugal P-[redacted] [redacted] 2007	Wheat, winter Galeira	1) 03.03.2007 2) 05.05.2007 - 15.05.2007 3) 10.07.2007 - 15.07.2007	SPI SPI	0.1875 0.1875	300 300	0.06255 0.06255	23.04.2007/0 14.05.2007/21	69	green material rest of plant ear straw grain	0.07 1.4 0.60 0.08 0.67 <0.01	0* 0 35 35 53 53	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application
RA-2038/07 R 2007 0425/9 0425-07 Italy I-[redacted] [redacted] 2007	Wheat, durum Simeto	1) 08.01.2007 2) 15.04.2007 - 30.04.2007 3) 05.06.2007 - 25.06.2007	SPI SPI	0.1875 0.1875	300 300	0.06255 0.06255	12.04.2007/0 28.04.2007/14	69	green material straw grain	0.78 2.7 2.2 1.4 1.4 1.5 1.6 0.02 <0.01	0* 0 7 14 28 35 53 35 53	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Bixafen & prothioconazole & spiroxamine EC 400 in/on winter and durum wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**100 g/L**	**Page**	**:**	**1**
Formulation (e.g. WP)	**:**	**400 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**BYF 00587 50 g/L spiroxamine 250 g/L**
Commercial product (name)	**:**	**Bixafen & Prothioconazole & Spiroxamine EC 400**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT* PHI (days) (f)	Remarks
RA-2041/07 R 2007 0445/3 0445-07 France, south F-[redacted] [redacted]2007	Wheat, winter Autan	1) 13.10.2006 2) 08.05.2007 - 15.05.2007 3) 28.06.2007 - 10.07.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	28.04.2007/0 15.05.2007/17	69	green material ear rest of plant straw grain	0.17 0.99 0.04 0.05 0.05 <0.01	0* 0 35 35 44 44	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	**:**	**Bayer CropScience AG, Monheim**	**Active substance**	**:**	**prothioconazole**
Country	**:**	**Germany**	**Crop/Crop Group**	**:**	**Cereals**
Content of active substance (g/kg or g/L)	**:**	**100 g/L**	**Page**	**:**	**2**
Formulation (e.g. WP)	**:**	**400 EC**	**Indoor/outdoor**	**:**	**Outdoor**
			Other a.s. in formulation (common name and content)	**:**	**BYF 00587 50 g/L spiroxamine 250 g/L**
Commercial product (name)	**:**	**Bixafen & Prothioconazole & Spiroxamine EC 400**	**Residues determined as**	**:**	**JAU 6476-desthio**
Producer of commercial product	**:**	**Bayer CropScience AG**	**Residues calculated as**	**:**	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2041/07 R 2007 0446/1 0446-07 Italy I-[redacted] 2007	Wheat, durum Simeto	1) 10.11.2006 2) 25.04.2007 - 01.05.2007 3) 01.06.2007 - 30.06.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	18.04.2007/0 09.05.2007/26	69	green material ear rest of plant straw grain	0.13 1.4 0.08 0.63 0.73 <0.01	0* 0 35 35 44 44	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Bayer CropScience

Document MCA: Section 6 Residues in or on treated products, food and feed
Prothioconazole

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Page**	:	**3**
Formulation (e.g. WP)	:	**400 EC**	**Indoor/outdoor**	:	**Outdoor**
			Other a.s. in formulation (common name and content)	:	**BYF 00587 50 g/L spiroxamine 250 g/L**
Commercial product (name)	:	**Bixafen & Prothioconazole & Spiroxamine EC 400**	**Residues determined as**	:	**JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues calculated as**	:	**JAU 6476-desthio**

1	2	3	4	5			6	7	8	9	10	11
Study Trial No.; Plot Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hl	Dates of treatment(s)/ Application interval or nos. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg)	DALT/ PHI (days) (f)	Remarks
RA-2041/07 R 2007 0525/5 0525-07 Spain E-[redacted] 2007	Wheat, winter Bolido R1	1) 13.12.2006 2) 11.04.2007 - 18.04.2007 3) 01.06.2007 - 30.06.200	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	30.03.2007/0 18.04.2007/19	69	green material rest of plant ear straw grain	0.19 1.1 0.35 0.09 0.72 <0.01	0* 0 36 36 55 55	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application
RA-2041/07 R 2007 0526/3 0526-07 Greece GR-[redacted] 2007	Wheat, winter Moro (durum)	1) 28.11.2006 2) 21.04.2007 - 27.04.2007 3) 05.06.2007 - 06.06.2007	SPI SPI	0.1500 0.1500	300 300	0.05000 0.05000	19.04.2007/0 27.04.2007/8	69	green material straw grain	1.1 2.7 1.2 0.01	0* 0 35 35	(c) SPI:Spraying (g) 01013 (h) 0.01 mg/kg * prior to last application

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Fluoxastrobin & prothioconazole EC 150 in/on wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	1 - A	
Formulation	(e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor	
Commercial product	(name)	:	Fluoxastrobin & Prothioconazole EC 150	Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L	
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-01 France, south [redacted] 2010	Wheat Pescadou	1) 02.03.2010 2) 01.06.2010 - 09.06.2010 3) 19.07.2010 - 25.07.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	25.05.2010/0 08.06.2010/14	69	green material	0.48 2.0 0.97 0.43 0.23	0.02 0.02 0.01 0.02 0.01	0.08 0.08 0.06 0.10 0.08	0.06 0.07 0.05 0.09 0.08	0* 0 7 14 27	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg day 0*: c=0.10 mg/kg for JAU 6476-desthio and c=0.02 mg/kg for JAU 6476-3-hydroxy-desthio and JAU 6476-4-hydroxy-desthio * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	1- A (continued)		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha (d)	Water (L/ha) (d)	kg a.s./hL (d)	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT, PHI (days) (f)	Remarks
10-2156 10-2156-01 (continued)	Wheat Pescadou	1) 02.03.2010 2) 01.06.2010 - 09.06.2010 3) 19.07.2010 - 25.07.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	25.05.2010/0 08.06.2010/14	69	grain	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	34 41	
									straw	1.0 1.6	0.07 0.08	0.38 0.37	0.32 0.32	34 41	day 34: c=0.10 mg/kg and day 41: c=0.12 mg/kg for JAU 6476-desthio day 34 and day 41: c=0.07 mg/kg for JAU 6476-3-hydroxy-desthio day 34 and day 41: c=0.04 mg/kg for JAU 6476-4-hydroxy-desthio

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	2-A		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (l/ha)	kg a.s./hL	Dates of treatment(s) Application interval; or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-02 Spain [redacted]2010	Wheat Nogal	1) 2012-2009 2) 13.05.2010 - 26.05.2010 3) 24.06.2010 - 30.07.2010	SPI SPI	0.20 0.20	300 400	0.067 0.050	07.05.2010/0 26.05.2010/19	71	green material grain straw	0.12 1.9 <0.01 <0.01 0.85 0.84	0.05 0.05 <0.01 <0.01 0.37 0.35	0.03 0.03 <0.01 <0.01 0.29 0.27	0.03 0.04 <0.01 <0.01 0.26 0.24	0* 0 35 43 35 43	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	3- A		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-03 Italy [redacted](BT) 2010	Wheat, durum Perseo	1) 02.12.2009 2) 01.03.2010 - 20.03.2010 3) 15.06.2010 - 30.06.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	06.05.2010/0 20.05.2010/14	69	green material grain straw	0.60 2.6 1.9 1.1 0.99 <0.01 <0.01 1.6 2.3	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 0.02 0.03	0.06 0.06 0.10 0.13 0.14 <0.01 <0.01 0.34 0.35	0.04 0.03 0.06 0.07 0.07 <0.01 <0.01 0.19 0.19	0* 0 7 14 28 35 42 35 42	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	4- A		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s); Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-04 Italy [redacted]2010	Wheat, durum Saragolla	1) 26.11.2009 2) 30.04.2010 - 20.05.2010 3) 20.06.2010 - 30.06.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	03.05.2010/0 17.05.2010/14	69	green material grain straw	0.58 1.7 0.01 <0.01 1.8 1.2	<0.01 <0.01 <0.01 <0.01 0.03 0.02	0.05 0.06 <0.01 <0.01 0.32 0.22	0.03 0.04 <0.01 <0.01 0.18 0.12	0* 0 35 44 35 44	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	5- A		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-05 France, [redacted] 2010	Wheat, winter Aubusson	1) 15.10.2009 2) 13.05.2010 - 24.05.2010 3) 10.07.2010 - 15.07.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	03.05.2010/0 20.05.2010/1	69	green material grain straw	0.09 0.93 <0.01 <0.01 0.10 0.22	0.03 0.03 <0.01 <0.01 0.08 0.08	0.05 0.07 <0.01 <0.01 0.35 0.32	0.03 0.05 <0.01 <0.01 0.22 0.21	0* 0 34 53 34 53	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

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RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**6- A**	
Formulation	**(e.g. WP)**	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-alpha-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-3-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-4-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-07 Greece [redacted]2010	Wheat, winter Yecora	1) 12.11.2009 2) 02.05.2010 - 03.05.2010 3) 11.06.2010 - 12.06.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	22.04.2010/0 06.05.2010/14	69	green material grain straw	0.86 1.7 1.4 1.3 0.86 0.01 1.1	0.35 0.28 0.69 0.43 0.39 <0.01 0.66	0.09 0.08 0.18 0.12 0.10 <0.01 0.29	0.14 0.13 0.38 0.18 0.29 <0.01 0.77	0* 0 7 14 28 35 35	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	7- A		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-06 Portugal [redacted] 2010	Wheat, winter Poison	1) 20.01.2010 2) 18.03.2010 - 25.03.2010 3) 01.07.2010 - 10.07.2010	SPI SPI	0.20 0.20	400 400	0.050 0.050	30.04.2010/0 14.05.2010/14	69	green material	0.62 2.9 1.3 1.0 0.96	0.10 0.14 0.33 0.28 0.43	0.08 0.12 0.18 0.19 0.25	0.06 0.10 0.16 0.15 0.20	0* 0 7 14 21	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg * day before last treatment
									ear	0.23	0.12	0.06	0.04	28	
									rest of plant	1.0	0.13	0.23	0.16	28	
									grain	<0.01	<0.01	<0.01	<0.01	48	
									straw	1.7	0.33	0.43	0.37	48	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals		
Country	:	Germany	Page	:	1- B		
Content of active substance (g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor		
Formulation (e.g. WP)	:	150 EC	Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	Total residues calc.	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-01 France, south [redacted]2010	Wheat Pescadou	1) 02.03.2010 2) 01.06.2010 - 09.06.2010 3) 19.07.2010 - 25.07.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	25.05.2010/0 08.06.2010/14	69	green material	0.11 0.11 0.08 0.12 0.08	0.01 0.01 0.01 0.02 0.01	0.76 2.3 1.2 0.78 0.49	0* 0 7 14 27	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. day 0*: c=0.03 mg/kg for JAU 6476-5-hydroxy-desthio * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	1- B (continued)		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	Total residues calc.	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-01 (continued)	Wheat Pescadou	1) 02.03.2010 2) 01.06.2010 - 09.06.2010 3) 19.07.2010 - 25.07.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	25.05.2010/0 08.06.2010/14	69	grain	<0.01 <0.01	<0.01 <0.01	<0.06 <0.06	34 41	
									straw	0.36 0.33	0.09 0.08	2.2 2.8	34 41	day 34: c=0.06 mg/kg and day 41: c=0.07 mg/kg for JAU 6476-5-hydroxy-desthio day 34 and day 41: c=0.01 mg/kg for JAU 6476-6-hydroxy-desthio

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	2- B		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-8-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	Total residues calc.	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-02 Spain [redacted] 2010	Wheat Nogal	1) 20.12.2009 2) 13.05.2010 - 26.05.2010 3) 24.06.2010 - 30.07.2010	SPI SPI	0.20 0.20	300 400	0.067 0.050	02.05.2010/0 26.05.2010/19	71	green material grain straw	0.02 0.02 <0.01 <0.01 0.15 0.14	<0.01 <0.01 <0.01 <0.01 0.03 0.03	0.26 2.1 <0.06 <0.06 2.0 1.9	0* 0 35 43 35 43	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	3- B		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-8-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	Total residues calc.	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-03 Italy [redacted] (BT) 2010	Wheat, durum Perseo	1) 02.12.2009 2) 01.03.2010 - 20.03.2010 3) 15.06.2010 - 30.06.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	06.05.2010/0 20.05.2010/14	69	green material grain straw	0.09 0.08 0.12 0.14 0.12 <0.01 <0.01 0.41 0.38	<0.01 <0.01 0.01 0.02 0.03 <0.01 <0.01 0.09 0.10	0.81 2.8 2.2 1.5 1.4 <0.06 <0.06 2.7 3.4	0* 0 7 14 28 35 42 35 42	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**		
Country	:	**Germany**	**Crop/Crop Group**	:	**Cereals**		
Content of active substance (g/kg or g/L)	:	**100 g/L**	**Page**	:	**4- B**		
Formulation (e.g. WP)	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**		
			Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**		
Commercial product (name)	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
Producer of commercial product	:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
			Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as**	: **JAU 6476-desthio**
			Residues determined as	:	**Total residues calc.**	**Residues calculated as**	: **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-04 Italy [redacted]2010	Wheat, durum Saragolla	1) 26.11.2009 2) 30.04.2010 - 20.05.2010 3) 20.06.2010 - 30.06.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	03.05.2010/0 17.05.2010/14	69	green material grain straw	0.05 0.06 <0.01 <0.01 0.29 0.23	<0.01 <0.01 <0.01 <0.01 0.07 0.05	0.73 1.9 0.06 <0.06 2.7 1.8	0* 0 35 44 35 44	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	5- B		
Formulation (e.g. WP)	:	150 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	fluoxastrobin 50 g/L		
Commercial product (name)	:	Fluoxastrobin & Prothioconazole EC 150	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	Total residues calc.	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-05 France, south [redacted] 2010	Wheat, winter Aubusson	1) 15.10.2009 2) 13.05.2010 - 24.05.2010 3) 10.07.2010 - 15.07.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	03.05.2010/0 20.05.2010/17	69	green material grain straw	0.03 0.05 <0.01 <0.01 0.22 0.19	0.01 0.02 <0.01 <0.01 0.03 <0.01	0.24 1.2 <0.06 <0.06 1.0 1.0	0* 0 34 53 34 53	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**6- B**	
Formulation	**(e.g. WP)**	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-07 Greece [redacted]2010	Wheat, winter Yecora	1) 12.11.2009 2) 02.05.2010 - 03.05.2010 3) 11.06.2010 - 12.06.2010	SPI SPI	0.20 0.20	300 300	0.067 0.067	22.04.2010/0 06.05.2010/14	69	green material grain straw	0.05 0.04 0.11 0.07 0.07 <0.01 0.15	<0.01 <0.01 0.03 0.01 0.02 <0.01 0.03	1.5 2.2 2.8 2.1 1.7 0.06 3.0	0* 0 7 14 28 35 35	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**7- B**	
Formulation	**(e.g. WP)**	:	**150 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Fluoxastrobin & Prothioconazole EC 150**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-5-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-6-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**Total residues calc.**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-5-hydroxy-desthio**	**JAU 6476-6-hydroxy-desthio**	**Total residues calculated**	DALT/ PHI (days) (f)	Remarks
10-2156 10-2156-06 Portugal [redacted] 2010	Wheat, winter Poison	1) 20.01.2010 2) 18.03.2010 - 25.03.2010 3) 01.07.2010 - 10.07.2010	SPI SPI	0.20 0.20	400 400	0.050 0.050	30.04.2010/0 14.05.2010/14	69	green material	0.05 0.10 0.16 0.14 0.19	<0.01 0.02 0.02 0.02 0.03	0.92 3.4 2.2 1.8 2.1	0* 0 7 14 21	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. * day before last treatment
									ear	0.03	<0.01	0.49	28	
									rest of plant	0.14	0.02	1.7	28	
									grain	<0.01	<0.01	<0.06	48	
									straw	0.27	0.07	3.2	48	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Bixafen & Fluoxastrobin & Prothioconazole EC 190 in/on wheat

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	1 - A	
Formulation	(e.g. WP)	:	190 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s) / Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (g)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks
10-2207 10-2207-01 France, south [redacted] 2010	Wheat Cezanne	1) 26.10.2009 2) 24.05.2010 - 31.05.2010 3) 06.07.2010 - 16.07.2010	SPI SPI	0.113 0.175	300 300	0.0583 0.0583	17.05.2010/0 31.05.2010/14	69	green material	0.80 2.0	0.07 0.07	0.12 0.12	0.10 0.11	0* 0	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg day 0*: c=0.01 mg/kg for JAU 6476-desthio * day before last treatment
									grain	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	35 38	
									straw	0.41 0.40	0.25 0.18	0.45 0.40	0.34 0.30	35 38	

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	1- A		
Formulation (e.g. WP)	:	190 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L		
Commercial product (name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha (d)	Water (L/ha) (d)	kg a.s./hL (d)	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-desthio	JAU 6476-alpha-hydroxy-desthio	JAU 6476-3-hydroxy-desthio	JAU 6476-4-hydroxy-desthio	DALT/ PHI (days) (f)	Remarks

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)	:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole		
Country	:	Germany	Crop/Crop Group	:	Cereals		
Content of active substance (g/kg or g/L)	:	100 g/L	Page	:	2- A		
Formulation (e.g. WP)	:	190 EC	Indoor/outdoor	:	Outdoor		
			Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L		
Commercial product (name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as	: JAU 6476-desthio
Producer of commercial product	:	Bayer CropScience AG	Residues determined as	:	JAU 6476-alpha-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-3-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio
			Residues determined as	:	JAU 6476-4-hydroxy-desthio	Residues calculated as	: JAU 6476-desthio

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1- A**	
Formulation	**(e.g. WP)**	:	**190 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**BYF 00587 40 g/L fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Bixafen & Fluoxastrobin & Prothioconazole EC 190**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-alpha-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-3-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-4-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	DALT/ PHI (days) (f)	Remarks
1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code	Commodity / Variety	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting	Method of treatment	Application rate per treatment			Dates of treatment(s)/ Application interval or no. of treatments and last date/	Growth stage at last treatment	Portion analysed	Residues (mg/kg)				DALT/ PHI (days)	Remarks

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	**Bayer CropScience AG, Monheim**	**Active substance**	:	**prothioconazole**	
Country		:	**Germany**	**Crop/Crop Group**	:	**Cereals**	
Content of active substance	**(g/kg or g/L)**	:	**100 g/L**	**Page**	:	**1- A**	
Formulation	**(e.g. WP)**	:	**190 EC**	**Indoor/outdoor**	:	**Outdoor**	
				Other a.s. in formulation (common name and content)	:	**BYF 00587 40 g/L fluoxastrobin 50 g/L**	
Commercial product	**(name)**	:	**Bixafen & Fluoxastrobin & Prothioconazole EC 190**	**Residues determined as**	:	**JAU 6476-desthio**	**Residues calculated as** : **JAU 6476-desthio**
Producer of commercial product		:	**Bayer CropScience AG**	**Residues determined as**	:	**JAU 6476-alpha-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-3-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**
				Residues determined as	:	**JAU 6476-4-hydroxy-desthio**	**Residues calculated as** : **JAU 6476-desthio**

1	2	3	4	5			6	7	8	9				10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha (d)	Water (L/ha) (d)	kg a.s./hL (d)	Dates of treatment(s)/ Application interval or no. of treatments and last date (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) **JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	DALT/ PHI (days) (f)	Remarks
Year of Trial	(a)	(b)	(c)	kg a.s./ha	Water (L/ha)	kg a.s./hL	(d)	(e)	(a)	**JAU 6476-desthio**	**JAU 6476-alpha-hydroxy-desthio**	**JAU 6476-3-hydroxy-desthio**	**JAU 6476-4-hydroxy-desthio**	(f)	
10-2207 10-2207-02 Portugal [redacted]2010	Wheat Jordao	1) 04.12.2009 2) 08.04.2010 - 20.04.2010 3) 15.06.2010 - 15.07.2010	SPI SPI	0.175 0.175	300 300	0.0583 0.0583	29.03.2010/0 19.04.2010/21	69	green material grain straw	0.02 1.2 <0.01 0.37	0.01 0.02 <0.01 0.21	0.04 0.04 <0.01 0.25	0.03 0.04 <0.01 0.26	0* 0 63 63	(c) SPI:Spraying (g) 01013 for JAU 6476-desthio and 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy

(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

Note: All entries to be filled in as appropriate. Date format dd.mm.yy

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Crop/Crop Group	:	Cereals	
Country		:	Germany	Page	:	1- B	
Content of active substance	(g/kg or g/L)	:	100 g/L	Indoor/outdoor	:	Outdoor	
Formulation	(e.g. WP)	:	190 EC	Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-8-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

Active substance : prothioconazole

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2207 10-2207-01 France, south [redacted] [redacted] 2010	Wheat Cezanne	1) 26.10.2009 2) 24.03.2010 - 31.03.2010 3) 06.07.2010 - 16.07.2010	SPI SPI	0.175 0.175	300 300	0.0583 0.0583	12.05.2010/0 31.05.2010/14	69	green material grain straw	0.09 0.09 <0.01 <0.01 0.25 0.20	0.02 0.02 <0.01 <0.01 0.06 0.05	1.2 2.41 <0.06 <0.06 1.8 1.5	0* 0 35 38 35 38	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report

RESIDUE DATA FROM SUPERVISED TRIALS (SUMMARY)

(Application on agricultural and horticultural crops)

Responsible body for reporting (name and address)		:	Bayer CropScience AG, Monheim	Active substance	:	prothioconazole	
Country		:	Germany	Crop/Crop Group	:	Cereals	
Content of active substance	(g/kg or g/L)	:	100 g/L	Page	:	2- B	
Formulation	(e.g. WP)	:	190 EC	Indoor/outdoor	:	Outdoor	
				Other a.s. in formulation (common name and content)	:	BYF 00587 40 g/L fluoxastrobin 50 g/L	
Commercial product	(name)	:	Bixafen & Fluoxastrobin & Prothioconazole EC 190	Residues determined as	:	JAU 6476-desthio	Residues calculated as : JAU 6476-desthio
Producer of commercial product		:	Bayer CropScience AG	Residues determined as	:	JAU 6476-5-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	JAU 6476-6-hydroxy-desthio	Residues calculated as : JAU 6476-desthio
				Residues determined as	:	Total residues calc.	Residues calculated as : JAU 6476-desthio

1	2	3	4	5			6	7	8	9			10	11
Study Trial No.; Location incl. postal code Year of Trial	Commodity / Variety (a)	Date of 1) Sowing or planting 2) Flowering 3) Harvest 4) Transplanting (b)	Method of treatment (c)	Application rate per treatment kg a.s./ha	Water (L/ha)	kg a.s./hL	Dates of treatment(s)/ Application interval or no. of treatments and last date/ (d)	Growth stage at last treatment (e)	Portion analysed (a)	Residues (mg/kg) JAU 6476-5-hydroxy-desthio	JAU 6476-6-hydroxy-desthio	Total residues calculated	DALT/ PHI (days) (f)	Remarks
10-2207 10-2207-02 Portugal [redacted]2010	Wheat Jordao	1) 04.12.2009 2) 08.04.2010 - 20.04.2010 3) 15.06.2010 - 15.07.2010	SPI SPI	0.175 0.175	300 300	0.0583 0.0583	29.03.2010/0 19.04.2010/21	69	green material grain straw	0.02 0.03 <0.01 0.15	<0.01 <0.01 <0.01 0.03	0.13 1.3 <0.06 1.3	0* 0 63 63	(c) SPI:Spraying (g) 00979/M001 for JAU 6476-desthio-hydroxy metabolites (h) 0.01 mg/kg for JAU 6476-desthio-hydroxy metabolites and 0.06 mg/kg for total residues calc. * day before last treatment

(a) According to Codex (or other e.g. EU) Classification/Guide
(b) Only if relevant
(c) High or low volume spraying, spreading, dusting etc. overall broadcast
(d) Year must be indicated
(e) BBCH Monograph, Growth Stages of Plants, 1997, (Blackwell, ISBN 3-8263-3152-4)
Note: All entries to be filled in as appropriate. Date format dd.mm.yy
(f) Minimum no. of days after last treatm. (DALT, Label pre-harvest interval, PHI = '<<')
(g) Reference to analytical method
(h) Limit of determination/quantitation
(i) Dosage of a.s. or water given as...
(-) Missing data in the above columns occurs where the information is not available in the original report