

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539



Data Requirement: PMRA DATA CODE:8.2.4.3.1
 EPA DP Barcode: DP 303488
 OECD Data Point: IIIA 9.3.1
 US EPA Guideline: OPPTS 835.1240;
 OPP§163-1

Test material:

Common name: Prothioconazole
 IUPAC name: 2-[(2RS)-2-(1-chlorocyclopropyl)-3-(2-chlorophenyl)-2-hydroxypropyl]-2H-1,2,4-triazole-3(4H)-thione
 CAS name: 2-[2-(1-Chlorocyclopropyl)-3-(2-chlorophenyl)-2-hydroxypropyl]-1,2-dihydro-3H-1,2,4-triazole-3-thione
 CAS No: 178928-70-6
 synonyms: JAU 6476 Technical

Primary Reviewer (officer number): Émilie Larivière (#1269)
 HC, PMRA, EAD **Date:** May 17, 2005 *Emilie Lariviere* 5/17/05

Secondary Reviewer (officer number): Janine Glaser (#1009)
 HC, PMRA, EAD **Date:** June 28, 2005 *Janine Glaser*

Secondary Reviewer(s) (officer number): Dirk Young, PhD. **Date:** Aug. 22, 2005
 EPA/OPP/EFED/ERB4 *Dirk Young*

Company Code BCZ
Active Code PRB
Use Site Category 7, 13, 14 (Industrial Oil Seed Crops and Fibre Crops, Terrestrial Feed Crops, Terrestrial Food Crops)
EPA PC Code 113961

CITATION: Riegner, K. 1999. Leaching Behavior of JAU6476 Formulated as 250 EC in Soil (parent leaching). Performing Laboratory: Bayer AG Crop Protection Business Group, Germany. Bayer CropScience, North Carolina. Unpublished. Report No. MR-098/99. April 20, 1999.

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

EXECUTIVE SUMMARY

The leaching behaviour of [phenyl-UL-¹⁴C] 2-[2-(1-Chlorocyclopropyl)-3-(2-chlorophenyl)-2-hydroxypropyl]-1,2-dihydro-3H-1,2,4-triazole-3-thione (prothioconazole; JAU6476), formulated as 250 EC (25% prothioconazole), was investigated on two German and two American soils in an unaged soil leaching column experiment: a sandy loam (Laacher Hof AXXa), a silt (Höfchen am Hohenseh 4a), a silty clay loam (Stanley) and a loamy sand (Byromville). The soil characteristics were the following: *sandy loam*: pH 7.2, organic carbon: 2.02%, sand: 72.4%, silt: 22.6%, clay: 5%; *silt*: pH 7.1, organic carbon: 2.14%, sand: 8.5%, silt: 81.3%, clay: 10.2%; *silty clay loam*: pH 5.9, organic carbon: 1.66%, sand: 12.4%, silt: 48%, clay: 39.6%; *loamy sand*: pH 7.0, organic carbon: 0.39%, sand: 86.8%, silt: 7.6%, clay: 5.6%. The experiment was conducted in accordance with SETAC Europe: Procedures for Assessing the Environmental Fate and Ecotoxicity of Pesticides, March 1995, and BBA Guidelines, and in compliance with German and OECD Good Laboratory Practice Standards. The spray solution was spiked with [phenyl-UL-¹⁴C]prothioconazole and non-radiolabelled prothioconazole such that ¹⁴C-labelled prothioconazole was approximately 25% of the total amount of the active substance in the emulsifiable concentrate formulation.

Single glass columns (5 cm diameter x 45 cm length) were filled with a layer of sand and packed to a depth of 30 cm with soil which had been air dried and passed through a 1 mm sieve. The soil columns were saturated with 0.01 M CaCl₂ solution, then the soil was treated with the application solution at a rate equivalent to 204 g a.i./ha (39.96 µg a.i./19.6 cm² surface area of the soil column). Water equivalent to 200 mm of rainfall (394 mL) was passed through the columns over 48 hours, at an infiltration rate of approximately 0.14 mL/minute and a temperature of approximately 20°C. The leachate was collected in two fractions and analyzed by Liquid Scintillation Counting. The soil columns were frozen and then divided into 5 equal sections, each 6 cm in length. The soil segments were extracted three times with 80-100 mL of each of acetonitrile/water (80/20; v/v) to which was added 1 mg/mL of cysteinhydrochloride to suppress any oxidising reactions of parent compound. For each extraction, the soils were shaken for 45 minutes and subsequently centrifuged for 20 minutes. The supernatants were filtered and combined. The remaining soil was hot extracted under reflux for 1 hour with 80 mL of the same solvent mixture as the cold extractions and subsequently centrifuged for 20 minutes. The supernatants were filtered through the same filter used for the filtration of cold extracts. Thin Layer Chromatography (TLC) analysis was performed on the extracts of soil segments containing more than 1% of the applied radioactivity (AR) (all of the 0-6 cm layers and the 6-12 cm layer of the loamy sand). The extracted soil was air-dried, ground and combusted prior to LSC analysis. The filters were also combusted prior to LSC analysis.

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

The mass balance was good for all soil columns, ranging from 97.2 to 99.5% of the AR. The amount of [^{14}C]residues in the leachate was very low (0.13% of the AR in the loamy sand and $\leq 0.03\%$ of the AR for the other soils) and was not characterized. Between 88.4 and 97.9% of the AR remained in the top 6 cm of the soil columns at the end of the study. The 6-12 cm soil segment of the loamy sand contained 8.7% of the AR, but this segment contained less than 1% of the AR in the other soils. The amount of radioactivity in the soil layers below 12 cm was less than 1% of the AR in all soils.

Prothioconazole was detected only in the top 6 cm of all soil columns, at levels ranging from 14.6 to 40.7% of the AR. The transformation product JAU6476-desthio was also detected in the first segment of all soils, at concentrations ranging from 15.4 to 28.0% of the AR. JAU6476-desthio was detected in the 6-12 cm segment of the loamy sand, at 7.5% of the AR. JAU6476-S-methyl (WAK7861) was only detected in the top 6 cm segment of all soils, at concentrations ranging from 6.8 to 11.2% of the AR. The sum of other unidentified transformation products (up to 5 substances) detected in the upper segment ranged from 3.3 to 6.3% of the AR. Unextractable residues in the complete soil columns ranged from 30.5 to 36.3% of the AR, with $\leq 1.1\%$ of the AR attributed to unextractable residues in the individual segments below 6 cm of all soil columns.

This study acceptability was classified as supplemental. The study conducted using [phenyl-UL- ^{14}C]prothioconazole is scientifically sound, but does not satisfy Subdivision N Guideline §163-1 data requirements for a mobility study using unaged soil because the soil columns were leached with an insufficient volume (20 cm, equivalent to 7.9 in) of 0.01M CaCl_2 solution, rather than the required 50.8 cm (20 in).

Sorption coefficients for prothioconazole could not be determined in batch equilibrium studies due to the instability of the compound in these systems. It is equally difficult to determine sorption coefficients in column studies. Difficulties include the same instabilities that would occur in a batch system as well difficulties in determining the exact depth of prothioconazole movement. Prothioconazole showed very low potential for leaching as very low total radioactive residues were detected in the leachate (0.01-0.13% of the AR) and no unchanged parent compound was found in the second soil segment. These results are in agreement with those of the aged residue column leaching study (Babczinski, 2001). The findings of these studies also indicate that prothioconazole has lower mobility (due to both sorption and degradation) than its transformation products JAU6476-desthio and JAU6476-S-methyl. Batch equilibrium studies submitted for JAU6476-desthio (Fent, 1998) and JAU6476-S-methyl (Hein, 1999) indicate that these two transformation products have low mobility in soil.

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

Study Acceptability: This study acceptability was classified as supplemental. The study conducted using [phenyl-UL-¹⁴C]prothioconazole is scientifically sound, but does not satisfy Subdivision N Guideline §163-1 data requirements for a mobility study using unaged soil because the soil columns were leached with an insufficient volume (20 cm, equivalent to 7.9 in) of 0.01M CaCl₂ solution, rather than the required 50.8 cm (20 in).

Supplemental results provided from this study, based on the lower volume of leachate, suggest that prothioconazole has a relatively high sorption coefficient and/or is readily degraded, and that transformation products JAU6476-desthio and JAU6476-S-methyl may have low mobility in soil. They were detected solely in the upper treated soil layer (0 - 6 cm) with the exception of JAU6476-desthio detected in the 6-12 cm layer in the loamy sand (at 7.5% of the AR), radioactivity in the lower soil layers was less than 1% of the AR and only trace levels of radioactivity were detected in the leachate (0.13% of the AR). This is consistent with other mobility/leaching studies with prothioconazole and its transformation products, which indicate that prothioconazole is relatively immobile and that JAU6476-desthio and JAU6476-S-methyl have low mobility in soil.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: SETAC Europe: Procedures for Assessing the Environmental Fate and Ecotoxicity of Pesticides, March 1995; BBA: Richtlinie für die amtliche Prüfung von Pflanzenschutzmitteln, Teil IV, 4-2, Versickerungsverhalten von Pflanzenschutzmitteln, 1986. No deviations were noted by the study author.

COMPLIANCE: German Chemical Law, dated 25 July, 1994, attachment 1, dated March 14, 1997; OECD-GLP (1997). Signed and dated GLP, Quality Assurance and Data Confidentiality statements were provided.]

A. MATERIALS

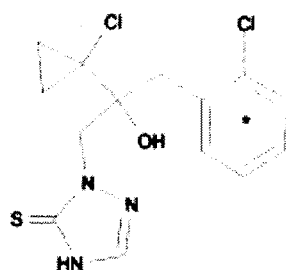
1. Active ingredient Prothioconazole (JAU6476)

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

Chemical Structure:



*: indicates the ¹⁴C-labeling position

Table 1: Physico-chemical properties of prothioconazole.

Parameter	Values		Comments
Water solubility (20°C)	<u>pH</u>	<u>Solubility</u> (mg/L)	low solubility at acidic pH, very soluble at alkaline pHs.
	4	5	
	8	300	
	9	2000	
Vapour pressure/volatility	<u>Temperature (°C)</u>	<u>Vapor pressure(Pa)</u>	relatively non-volatile under field conditions.
	20	<<4 x 10 ⁻⁷	
	25	<<4 x 10 ⁻⁷	
UV absorption	peak maxima at 275 nm. No absorption at > 300 nm.		Phototransformation is not expected to be an important route of transformation
pK _a	pK _a = 6.9		Weak acid, anion at neutral and alkaline pHs
log K _{ow}	<u>pH</u>	<u>log K_{ow}</u>	Potential for bioaccumulation at neutral and acidic pH.
	4	4.16	
	7	3.82	
	9	2.0	
	unbuffered	4.05	

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

Stability of compound at room temperature, if provided	Thermally stable at room temperature under air. Stable to most metals. Colour changes observed in the presence of copper materials.	Thermally stable at room temperature under air.
--	---	---

Data were obtained from Chemistry Review.

2. Formulated product JAU6476 250 EC

Formulation type: emulsifiable concentrate
Guarantee: 24.93% prothioconazole (contains radiolabelled and non-radiolabelled material)
Sample no.: ECW 11582-A
Specific activity: 3.127 MBq/mg
Radiochemical purity of formulation: 98.7 %
Storage conditions: not reported

3. Radiolabelled substance prothioconazole (JAU6476)

Lot no.: 12106/1
Position of ¹⁴C label: [phenyl-UL-¹⁴C]
Specific activity: 3.81 MBq/mg (103µCi/mg)
Radiochemical purity: >99 %
Chemical purity: >99%
Stability: not reported
Storage conditions: As cool as possible to minimise autoradiolysis of the compound

4. Reference substance prothioconazole (JAU6476)

Batch no.: M00175
Chemical purity: 99.9%
Storage conditions: Not reported

5. Soils

Table 2: Description of soil collection and storage.

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

Description	Byromville loamy sand	Laacher Hof AXXa sandy loam	Höfchen am Hohenseh 4a silt	Stanley silty clay loam
Geographic location	USA	Germany	Germany	USA
Pesticide use history at the collection site	not reported (Soil phototransformation of prothioconazole on soil study report (MR-242/00) states no pesticide use	not reported (Biotransformation in aerobic soil study reports (MR-549/99, MR-340/00 and MR-327/00) state no pesticide use since 1988)	not reported (Biotransformation of JAU6476-S-methyl and JAU6476-desthio in aerobic soil study reports (MR-340/00 and MR-327/00) state no pesticide use)	not reported (Biotransformation in aerobic soil study reports (MR-549/99, MR-340/00 and MR-327/00) state no pesticide use)
Collection procedures	Not reported			
Sampling depth (cm)	Not reported			
Storage conditions	Shipped after sampling and stored in a cool place.	Freshly sampled.	Freshly sampled.	Stored moist and under natural vegetation in a container in a greenhouse of the Institute for Metabolism Research and Residue Analysis.
Storage length	June 25, 1998 to July 21, 1998	Freshly sampled.	Freshly sampled.	Not reported
Soil preparation	Air-dried; sieved through 1 mm mesh.			

Data obtained from Appendix 5, p. 21; Appendix 8, p. 24, pp. 8-9.

Table 3: Properties of the soils.

Property	Byromville	Laacher Hof AXXa	Höfchen am Hohenseh 4a	Stanley
Soil Texture (USDA)	loamy sand	sandy loam	silt	silty clay loam
% sand	86.8	72.4	8.5	12.4

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

% silt	7.6	22.6	81.3	48
% clay	5.6	5	10.2	39.6
pH (in H ₂ O)	7	7.2	7.1	5.9
pH (in CaCl ₂ ; 0.01 M)	6	6.6	6.8	5.6
Organic carbon (%)	0.39	2.02	2.14	1.66
CEC (meq/100 g)	5	8	15	18.5
Moisture at 1/3 atm (%)	1.1	3.3	16.1	6.9
Bulk density (g/cm ³)	Not reported	Not reported	Not reported	Not reported
Biomass (mg microbial C/kg soil)	98	395	643	314
Soil taxonomic classification	Not reported	Not reported	Not reported	Not reported
Soil mapping unit (for EPA)	Not reported	Not reported	Not reported	Not reported

Data obtained from Appendix 5, p. 21; Appendix 8, p. 24, pp. 8-9.

C. STUDY DESIGN

1. Soil column study design

Table 4: Experimental design for the unaged soil column leaching study

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

Criteria		Byromville loamy sand	Laacher Hof AXXa sandy loam	Höfchen am Hohenseh 4a silt	Stanley silty clay loam
Have these soils been used for other laboratory studies with soil?		Yes (Bayer Report No. MR-104/01, MR-242/00, FM768, MR-364/00- Aerobic soil biotransformation, phototransformation on soil, batch equilibrium, aged soil leaching)	Yes (Bayer Report Nos. MR-549/99, MR-340/00, MR-327/00, FM768 - Aerobic soil biotransformation, batch equilibrium)	Yes (Bayer Report Nos. MR-104/01, MR-340/00, MR-327/00, FM768 - Aerobic soil biotransformation, batch equilibrium)	Yes (Bayer Report Nos. MR-549/99, MR-340/00, MR-327/00, FM768 - Aerobic soil biotransformation, batch equilibrium)
Dimension of soil column		45-cm long × 5-cm inner diameter; The column was closed with quartz wool at the lower end and the conical part of the column was filled with washed sea sand. The column was then packed to a 30 cm depth and divided into 6-cm long segments			
Soil (aerobic)	Stored or fresh	stored	fresh	fresh	stored
	Amount (g air-dried soil/treatment)	804.6	671.87	704.68	664.3
Controls (Yes/no)		None used.			
Test material concentrations	Nominal application rate	200 g a.i./ha			
	Nominal soil residue	39.2 µg a.i./19.6 cm ² surface area of the soil column (Equivalent to 156.8 ug JAU6476 250 EC/19.6 cm ² surface area of the soil column. Crop coverage was not taken into account.)			
	Measured soil residue	39.96 µg a.i./19.6 cm ² surface area of the soil column (Equivalent to 204 g a.i./ha)			
Co-solvent		None used.			
Replications	Controls	No controls were used.			
	Treatments	1 column per soil, no replication.			

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

Criteria		Byromville loamy sand	Laacher Hof AXXa sandy loam	Höfchen am Hohenseh 4a silt	Stanley silty clay loam
0.01M CaCl ₂	Quantity of CaCl ₂ used for leaching (mL/min)	0.139			
	Total quantity of CaCl ₂ /column (mL)	394			
	Equivalent to rainfall (cm)	20			
	Total duration of leaching (h)	48			
Material used to cover columns and prevent photolysis		Study was conducted in darkness			
Sampling intervals		0-6, 6-12, 12-18, 18-24, 24-30			
Total volume of leachate collected (mL)		390	392	398	392
		Leachate was collected in 2 fractions of about 200 mL each.			
Length of soil column segments sampled for analysis (cm)		6			

Data obtained from Appendix 8, p. 24, p. 10

3. Description of analytical procedures

Extraction/clean up/concentration methods for leachate and soil segments: The pH of all leachate fractions was measured. The radioactivity content of all leachate fractions was determined by LSC under alkaline and acidic conditions in order to obtain indications of the possible presence of H¹⁴CO₃⁻ or ¹⁴CO₃²⁻. The determination of radioactivity content was carried out in duplicates after adding 100 µL HCl (18%) or 50 or 100 µL NaOH (1 N) to 5 mL of leachate (p. 11).

Because of disturbances during measurement of the alkaline samples and because of the very low radioactivity content in the acidic samples, only the results of the acidic samples were used for the calculation of the total balances (Appendices 9-12, top, pp. 25-28).

The frozen soil columns were pushed out of the glass tubes. The complete soil column was cut

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

into 5 segments of about 6 cm each. The sea sand was combined with the 24-30 cm segment. The soil segments were extracted three times with 80-100 mL of each of acetonitrile/water (80/20; v/v) to which was added 1 mg/mL of cysteinhydrochloride to suppress any oxidising reactions of parent compound. For each extraction the soils were shaken for 45 minutes and centrifuged for 20 minutes at about 8000 g. The supernatants were filtered and combined. The remaining soil was hot extracted under reflux for 1 hour with 80 mL of with the same solvent mixture as mentioned previously and centrifuged for 20 minutes. The supernatant was filtered through the same filter used for filtration of cold extracts. The determination of radioactivity of each extract (cold / hot) was carried out in duplicate of 1 mL each.

Total ^{14}C measurement: Total ^{14}C residues were determined by summing the concentrations of residues measured in the leachate fractions, the soil extracts (cold and hot) and the extracted sediment and filters.

Determination of non-extractable residues: The amount of non-extracted radioactivity in soil samples was determined by combusting 3 aliquots (about 1 g each) of air-dried, ground soil from each segment. The radioactivity content in the filters was also determined by combustion.

Identification and quantification of parent and transformation products: Because of low radioactive residues ($<<1\%$ of the AR), the leachate fractions were not investigated by analytical methods. **Only extracts of soil segments containing more than 1% of applied radioactivity (0-12 cm for the loamy sand and 0-6 cm for all other soils) were investigated by analytical methods immediately.**

The separation and quantification of components in the samples was done by TLC analysis using RP 18 plates (F_{254} , Fa. Merck, layer thickness 0.25 mm) and the following solvent system: acetonitrile/water/methanol/acetic acid, 70:25:5:1 (v/v/v/v) with 1 mg/mL ionol.

Sample solutions/extracts were spotted on the plates as bands of 10 mm width using an automatic plate spotter (Linomat IV, Fa. Camag). Different samples were analysed by co-chromatography by spotting relevant reference compounds on the plates overlapping with radioactive bands. To suppress any oxidising reactions of parent compound the zones were underlayered with active ingredient and ionol (1 mg/mL). The agreement of radioactive zone with the reference compound and the enlargement of the radioactive zone in the area of overlap were considered to indicate the identity of the radioactive zone (substance) with the reference substance. Radioactive zones on the TLC plates were measured using a Bio-Imaging Analyser (BAS 2000, Fuji Co.). Radioactive regions on the measured tracks were quantified with the software package TINA (Raytest Co.).

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

Non-labelled reference substances on the plates were visualised under UV light.

Confirmation of the results was also done by TLC using SI 60 plates (F₂₅₄, Fa. Merck, layer thickness 0.25 mm) and the following solvent system: dichloromethane/methanol/ammonia (25%), 90:10:1 (v/v/v) with 1 mg/mL ionol.

Detection limits (LOD, LOQ) for the parent compound and transformation products: The limit of detection (LOD) and the limit of quantification (LOQ) was not reported for LSC analyses. The LOD for a single peak in the soil extracts analyzed by TLC was 0.2% of the AR (p. 12).

II. RESULTS AND DISCUSSION

A. MATERIAL BALANCE: Over the length of the study, total recovery of radiolabelled material ranged from 97.2-99.5% of the AR in the four soils tested (p. 29). The complete mass balance in all soils demonstrated that no relevant radioactivity dissipated from the test system through volatilization.

Table 5: Distribution of total radioactivity in the soil columns and the leachate (raw numbers)

Compartment		% of applied activity			
		Byromville loamy sand	Laacher Hof AXXa sandy loam	Höfchen am Hohenseh 4a silt	Stanley silty clay loam
Leachate ¹		0.13	0.02	0.03	0.01
Extractable (cold and hot)	0-6 cm	53.74	65.06	66.92	68.46
	6-12 cm	7.56	0.75	0.57	0.42
	12-18 cm	0.69	0.07	0.13	0.07
	18-24 cm	0.19	0.04	0.08	0.04
	24-30 cm	0.13	0.03	0.06	0.02
Subtotal extractable (hot and cold)		62.31	65.95	67.76	69.01

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

Non-extractable (+ filter)	0-6 cm	34.67	31.19	30.83	29.46
	6-12 cm	1.10	0.04	0.25	0.21
	12-18 cm	0.24	0.01	0.11	0.17
	18-24 cm	0.28	0.01	0.09	0.11
	24-30 cm	0.02	<0.01	0.07	0.52
Subtotal non-extractable (+ filter)		36.31	31.25	31.35	30.47
Total Soil Column	0-6 cm	88.41	96.25	97.95	97.92
	6-12 cm	8.66	0.79	0.82	0.63
	12-18 cm	0.93	0.08	0.24	0.24
	18-24 cm	0.47	0.05	0.17	0.15
	24-30 cm	0.15	0.03	0.13	0.54
Total Soil Column		98.62	97.20	99.11	99.48
Total recovery		98.74	97.21	99.13	99.48

mean of 2 leachate fractions.

Data obtained from Appendices 9-13, pp. 25-29.

Table 6: Distribution of the parent compound and transformation products in the soil column extracts

Compartment ¹		% of applied activity			
		Byromville loamy sand	Laacher Hof AXXa sandy loam	Höfchen am Hohenseh 4a silt	Stanley silty clay loam
Prothioconazole	0-6 cm	14.63	28.12	25.23	40.68
	6-12 cm	<LOD	n.a.	n.a.	n.a.
JAU 6476-desthio (SXX 0665)	0-6 cm	24.59	28.02	27.29	15.38
	6-12 cm	7.51	n.a.	n.a.	n.a.
JAU 6476-S-methyl (WAK7861)	0-6 cm	11.18	5.47	8.08	6.84
	6-12 cm	<LOD	n.a.	n.a.	n.a.

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

Other minor transformation products ²	0-6 cm	3.33	3.45	6.32	5.55
	6-12 cm	0.05	n.a.	n.a.	n.a.
Extracted	0-6 cm	53.74	65.06	66.92	68.46
	6-12 cm	7.56	0.75	0.57	0.42

n.a. = not analyzed.

¹ Because of low radioactivity residues ($\ll 1\%$ of the AR), the leachate fractions were not investigated by analytical methods. Only extracts of soil segments containing more than 1% of the AR were investigated by analytical methods immediately (0-12 cm for loamy sand and 0-6 cm for all other soils).

² Other minor transformation products = 1-4 substances, including origin for sandy loam; 1-3 substances including origin for loamy sand; 1-5 substances including origin for silt and 1-4 substances including origin for silty clay loam

B. DISTRIBUTION: The majority (88.41% for the loamy sand, 96.25% for the sandy loam, 97.75% for the silt, and 97.92% for the silty clay loam) of the radioactivity in the soil column was recovered from the upper soil segment (0-6 cm). The amount of radioactivity translocated into the 6-12 cm segment was 8.66% of the AR in the loamy sand. The radioactivity in the 6-12 cm segments of the other soils and in all lower segments of the soil columns was below 1% of the AR.

C. PARENT COMPOUND: At the end of the study, prothioconazole was detected only in the 0-6 cm segment of all soil columns, at levels of 14.63, 28.12, 25.23 and 40.68% of the AR in the loamy sand, the sandy loam, the silt and the silty clay loam, respectively.

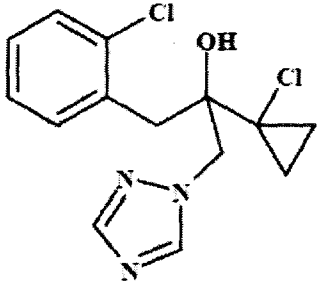
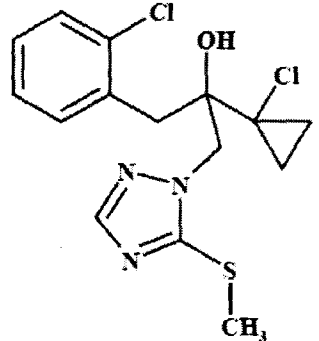
D. TRANSFORMATION PRODUCTS: At study termination, JAU6476-desthio was detected to a depth of 12 cm in the loamy sand at concentrations of 24.59% of the AR in the 0-6 cm segment and 7.51% of the AR in the 6-12 cm segment. JAU6476-desthio was only detected in the first segment in the other three soils (28.02, 27.29, 15.38% of the AR for the sandy loam, the silt and the silty clay loam, respectively). JAU6476-S-methyl (WAK7861) was detected in the upper segment of all soils, at concentrations of 11.18, 5.47, 8.08 and 6.84% of the AR, respectively in the sandy loam, the silt and the silty clay loam. The sum of other unidentified transformation products (up to 5 substances) detected in the upper segment ranged from 3.3 to 6.3% of the AR (of this, not more than 0.05% of the AR was detected in the 6-12 cm segment of the loamy sand).

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

Table 7: Chemical names, CAS number and chemical structure of the transformation products of prothioconazole

Applicant's Code Name	CAS Number	CAS Chemical Name	Chemical structure
JAU6476-desthio (SXX 0665)	120983-64-4	(R,S) 2-(1-chlorocyclopropyl)-1-(2-chlorophenyl)-3-(1,2,4-triazol-1-yl)-propan-2-ol	
JAU6476-S-methyl (WAK 7861)	178928-71-7	Not reported	

E. LEACHING: The amount of radioactivity detected in the leachate was very low, 0.13% of the AR in the loamy sand, 0.02% of the AR in the sandy loam, 0.03% of the AR in the silt and 0.01% of the AR in the silty clay loam. As the radioactivity in the leachate was well below 1% of the AR, the leachate fractions were not investigated by analytical methods to identify components.

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

III. STUDY DEFICIENCIES

The US EPA Guideline 163-1 and the Guidance provided under Trade Memorandum T-1-255 (Agriculture Canada, 1987) recommend that the column be eluted with a volume of water (0.01 M calcium chloride solution) equal to 50.8 cm (20 inches) times the cross-sectional area of the column. This would be equivalent to 996 mL ($50.8 \text{ cm} \times 19.6 \text{ cm}^2$).

This study is considered supplemental. Other mobility/leaching studies have been submitted to characterize the mobility of prothioconazole and its transformation products.

IV. REVIEWER'S COMMENTS

The length of time that the test samples were stored prior to analysis was not reported.

Raw data of TLC Analyses were provided for the loamy sand soil but not for the other three soils.

Single columns were used for each soil, instead of duplicate columns, as recommended by OECD Guideline 312.

This study is classified as supplemental. The study conducted using [phenyl-UL- ^{14}C]prothioconazole is scientifically sound, but does not satisfy Subdivision N Guideline §163-1 data requirements for a mobility study using unaged soil because the soil columns were leached with an insufficient volume (20 cm, equivalent to 7.9 in) of 0.01M CaCl_2 solution, rather than the required 50.8 cm (20 in).

Sorption coefficients for prothioconazole could not be determined in batch equilibrium studies due to the instability of the compound in these systems. It is equally difficult to determine sorption coefficients in column studies. Difficulties include the same instabilities that would occur in a batch system as well as difficulties in determining the exact depth of prothioconazole movement. Prothioconazole showed very low potential for leaching as very low total radioactive residues were detected in the leachate (0.01-0.13% of the AR) and no unchanged parent compound was found in the second soil segment. These results are in agreement with those of the aged residue column leaching study (Babczinski, 2001). The findings of these studies also indicate that prothioconazole has lower mobility than its transformation products JAU6476-desthio and JAU6476-S-methyl. Batch equilibrium studies submitted for JAU6476-desthio (Fent, 1998) and JAU6476-S-methyl (Hein, 1999) indicate that these two transformation products have low mobility in soil.

Data Evaluation Report on the leaching of prothioconazole (JAU6476) Formulated as 250 EC in Soil Columns

PMRA Submission Number 2004-0843

EPA MRID Number 46246539

V. REFERENCES

- Agriculture Canada. 1987. Trade Memorandum T-1-255. Guidelines for Determining Environmental Chemistry and Fate of Pesticides. October 30, 1987.
- Babczinski, P. 2001. Aged Soil Column Leaching of JAU6476. Performing Laboratory: Bayer AG Crop Protection Business Group, Germany. Bayer CropScience, North Carolina. Unpublished. Report No. MR-364/00. June 01, 2001, amended July 4, 2001. Submitted under DACO 8.2.4.3.2.
- Fent, G. 1998. Adsorption/Desorption of [phenyl-UL-¹⁴C]SXX0665 on Four Different Soils. Performing Laboratory: Staatliche Lehr-und Forschungsanstalt für Landwirtschaft, Weinbau und Gartenbau (SLFA). Bayer CropScience, North Carolina. Unpublished. Report No. FM768. August 11, 1998. Submitted under DACO 8.2.4.2.
- Hein, W. 1999. Adsorption/Desorption of S-methyl-JAU6476 on Four Different Soils. Performing Laboratory: Staatliche Lehr-und Forschungsanstalt für Landwirtschaft, Weinbau und Gartenbau (SLFA). Bayer CropScience, North Carolina. Unpublished. Report No. FM774. December 21, 1999. Submitted under DACO 8.2.4.2.
- Sommer, H. 2001. Estimation of the Adsorption Coefficient (K_{oc}) of JAU6476 on Soil Using High Performance Liquid Chromatography (HPLC). Performing Laboratory: Bayer AG Crop Protection Business Group, Germany. Bayer CropScience, North Carolina. Unpublished. Report No. MR-473/00. January 15, 2001. Submitted under DACO 8.2.4.2.