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## **STUDY REPORT**

45830720 Graper, L. (2002) A Confined Rotational Crop Study With (Carbon 14) XDE-638 Using Three Different Crops: Lab Project Number: 000266. Unpublished study prepared by Dow AgroSciences LLC and Research For Hire. 134 p.

Addendum to 45830720 Graper, L.; Dow AgroSciences LLC and Plant Sciences Inc., 5/4/04 (Attachment).

## **EXECUTIVE SUMMARY**

Dow AgroSciences LLC has submitted a confined rotational crop study with [triazolopyrimidine-2-<sup>14</sup>C]penoxsulam and uniformly ring-labeled [phenyl-U-<sup>14</sup>C]penoxsulam. The radiolabeled test substances were applied directly to sandy loam soil in pots maintained outdoors at 0.045-0.046 lb ai/A (low treatment rate) or 0.090-0.093 lb ai/A (high treatment rate), and rotational kale, potato, and wheat were planted directly into that soil 90 days after treatment (DAT). The in-life phase of the study was conducted by Research for Hire (Porterville, CA), and the analytical phase was conducted by Dow AgroSciences (Indianapolis, IN).

Total radioactive residues (TRR) accumulated at  $\geq 0.01$  ppm in certain rotated crops planted 90 days following a single soil application of [triazolopyrimidine-2-<sup>14</sup>C]penoxsulam (TP) at 0.045 and 0.093 lb ai/A, or [phenyl-U-<sup>14</sup>C]penoxsulam (PH) at 0.046 and 0.090 lb ai/A. At the low treatment rate, TRR were  $>0.01$  ppm in potato foliage (0.024 ppm from the TP plot and 0.047 ppm from the PH plot), wheat hay (0.021 ppm, PH plot only), and wheat straw (0.011 ppm, TP plot; 0.024 ppm, PH plot). At the higher treatment rate, TRR were  $>0.01$  ppm in kale (0.014 ppm, TP plot only), potato foliage (0.038 ppm, TP plot; 0.062 ppm, PH plot), wheat hay (0.022 ppm, TP plot; 0.032 ppm, PH plot), and wheat straw (0.030 ppm, TP plot; 0.028 ppm, PH plot). TRR were  $<0.01$  ppm in the following matrices: potato tuber ( $\leq 0.003$  ppm), wheat forage ( $\leq 0.007$  ppm), and wheat grain ( $<0.005$  ppm) from both labels at both treatment rates; TP-treated kale (0.003 ppm) at the low treatment rate and PH-treated kale ( $\leq 0.008$  ppm) at both treatment rates; and TP-treated wheat hay (0.009 ppm) at the low treatment rate. TRR were generally higher in PH-treated commodities than in TP-treated commodities.



Only crop samples with TRR  $\geq 0.008$  ppm were extracted. Solvent extraction released 62-97% TRR from rotational crop matrices. The majority of the extractable residues remained in the aqueous phase after partitioning with dichloromethane, and only the organic phase of potato foliage was subjected to further partitioning. Nonextractable residues in rotational crop commodities ranged 4-31% TRR ( $< 0.001$ - $0.009$  ppm). The extraction procedures released sufficient residues from rotational crop matrices; material balances were 84-107%. Samples were analyzed within 5 months of harvest. All samples were stored frozen. No further storage stability data are required to support this study.

Only the extracts of PH-treated potato foliage were analyzed by HPLC for metabolite identification. In all other samples, because the aqueous extracts contained  $< 0.03$  ppm and the organic extracts contained  $< 0.01$  ppm, no further characterization was attempted. Total identified residues were 8.4% and 38.9% TRR in PH-label potato foliage at the low and high treatment rates, respectively. The 5-OH metabolite was identified at 8.4-13.9% TRR ( $0.004$ - $0.009$  ppm), and a single unknown was characterized at approximately the same level (8.9-11.8% TRR,  $0.004$ - $0.007$  ppm) in both the low and high treatment rate potato foliage. In addition, the 3-[[[2-(2,2-difluoroethoxy)-6-(trifluoromethyl) phenyl]sulfonyl]amino]-1H-1,2,4-triazole-5-carboxylic acid (BSTCA) metabolite was identified in potato foliage from the high treatment rate, at 25.0% TRR ( $0.015$  ppm). Three unknowns were also characterized, each present at  $\leq 18.0\%$  TRR ( $\leq 0.011$  ppm). Residues were characterized/identified in potato foliage by HPLC analysis, and, because no confirmatory method was used, the identifications are considered tentative. However, because no parent was detected and no single component was present at  $> 0.015$  ppm in potato foliage, RAB2 concludes that these methods successfully identified the predominant residues in rotational crop matrices.

The petitioner proposed that the penoxsulam is metabolized in rotated potato foliage to 5-OH XDE-638 and BSTCA, and that both 5-OH XDE-638 and BSTCA are then metabolized to unknowns and nonextractable residues.

### **STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS**

Under the conditions and parameters used in the study, the confined rotational crop residue data are classified as scientifically acceptable.

The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Summary Document, DP Barcode D288152.

### **COMPLIANCE**

Signed and dated GLP, Quality Assurance and Data Confidentiality statements were provided. No deviations from regulatory requirements were reported which would impact the validity of the study.

#### **A. BACKGROUND INFORMATION**

Penoxsulam (company code XDE-638; PC Code 119031) is an herbicide intended for the control of *Echinochloa* grasses, broadleaf weeds, and sedge weeds in both water-injected (transplanted paddy) and postemergence (direct-seeded) rice. A single postemergence application of penoxsulam is to be

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made to rice from the one-leaf growth stage (7-12 days after seeding) to 60 days prior to rice harvest. The application is to be made by aerial or ground equipment once per growing season at a maximum rate of 0.045 lb ai/A (50 g ai/ha). Penoxsulam is to be formulated as a granular (for water-seeded rice) or suspension concentrate (for direct-seeded rice) formulation.

TABLE A.1. Penoxsulam Nomenclature.	
Compound	
Common name (proposed)	Penoxsulam
Company experimental name	XDE-638
IUPAC name	6-(2,2-Difluoroethoxy)-N-(5,8-dimethoxy-s-triazolo[1,5-c]pyrimidin-2-yl)- $\alpha,\alpha,\alpha$ -trifluoro- <i>o</i> -toluenesulfonamide
CAS name	2-(2,2-difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c] pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide
CAS #	219714-96-2
End-use product/EP	GF-443 SC SF (File Symbol 62719-LNN); GF-947 Granule SF (File Symbol 62719-LNG); GF-947 Granule CA (File Symbol 62719-LNR).

TABLE A.2. Physicochemical Properties of Technical Grade Penoxsulam.			
Parameter	Value		Reference
Melting point/range	Not available		
pH	Not available		
Density	Not available		
Water solubility	pH	Solubility (mg/L)	45830720
	(unbuffered)	4.91	
	5	5.66	
	7	408	
	9	1460	
Solvent solubility	Solvent	Solubility (g/L)	45830720
	DMSO	78.4	
	NMP	40.3	
	DMF	39.8	
	acetone	20.3	
	acetonitrile	15.3	
	ethyl acetate	3.23	
	methanol	1.48	
	octanol	0.035	
xylene	0.017		
heptane	<1 $\mu$ g/mL		
Vapor pressure	7.16 x 10 <sup>-16</sup> mm Hg at 25 °C		45830720

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Parameter	Value		Reference
Dissociation constant, pK <sub>a</sub>	5.1		45830720
Octanol/water partition coefficient, Log(K <sub>ow</sub> )	pH	Log(K <sub>ow</sub> )	45830720
	(unbuffered)	-0.354	
	5	1.137	
	7	-0.602	
	9	-1.418	

## B. EXPERIMENTAL DESIGN

### B.1. Test Site and Crop Information

Testing Environment and location	Soil characteristics						
	Type	% Sand	% Silt	% Clay	%OM	pH	CEC (meq/100 g)
Plastic-lined boxes in outdoor plots at Research For Hire (Porterville, CA)	Sandy loam	74	20	6	0.7	7.2	6.9

The monthly minimum and maximum temperatures and precipitation were provided for the study period (not available electronically). Historical weather data were not provided; however, no unusual circumstances were noted. During the period between application of the test substance and planting of the rotational crops, the soil received 0.75 inches/A of water weekly. Irrigation of the rotated plants was conducted by hand as needed.

Crop; crop group	Variety	Plantback intervals (days)	Growth stage at harvest	Harvested RAC	Harvesting procedure
Kale; Vegetable, <i>brassica</i> , leafy, group 5	Dwarf Blue Curled	90	Mature, 148-201 DAP	Leaves	Harvest methods were not specified.
Potato; Vegetable, root and tuber, group 1	White	90	Mature, 208 DAP	Tops (foliage)	
			Mature, 215 DAP	Tubers	
Wheat; Grain, cereal, group 15, and Grain, cereal, forage, fodder, and straw, group 16	Yecora Rojo	90	Immature, 97 DAP	Forage	Harvest method was not specified.
			Immature, 162 DAP	Hay	Hay was dried in a greenhouse for 11 days prior to collection.
			Mature, 204 DAP	Straw and grain	Grain was separated from the chaff, and the chaff was added to the straw sample.

<sup>1</sup> DAP = Days after planting.

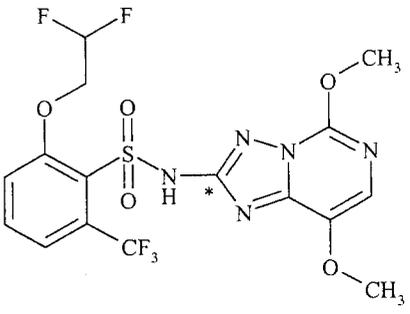
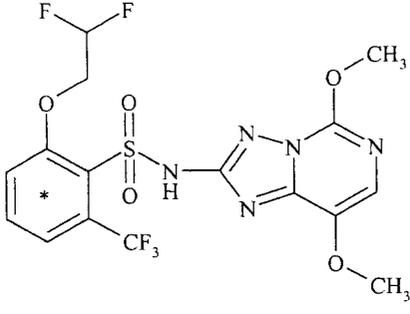
We note that Swiss chard was grown with kale as an alternative leafy vegetable but was not allowed to mature once kale was chosen (77 days following planting) as the leafy vegetable to be grown to maturity. Phytotoxicity was observed in treated kale and potatoes (and Swiss chard) within two to

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three months of planting; however, six months after planting, treated kale and potatoes were growing and maturing normally.

## B.2. Test Materials

Chemical structure		
Radiolabel position	2-triazolopyrimidine labeled (TP)	Uniformly labeled on the phenyl ring (PH)
Lot No.	INV1456	INV1475
Purity	99%	98.4%
Specific activity	28.9 mCi/mmole; 40,700 dpm/μg (test substance)	24.6 mCi/mmole; 40,900 dpm/μg (test substance)
Code	N/A	N/A

The radiolabeled materials were dissolved in ACN and diluted with nonlabeled penoxsulam prior to shipment to Research For Hire.

## B.3. Study Use Pattern

Chemical name	[triazolopyrimidine-2- <sup>14</sup> C]penoxsulam and [phenyl-U- <sup>14</sup> C]penoxsulam
Application method	Each test substance was dissolved in ACN and diluted to volume with ACN for direct application to the soil surface using a backpack pressurized sprayer.
Application rate	A low-dose treatment was made to three boxes for each radiolabel, and a high-dose treatment was made to three other boxes for each radiolabel. TP-label: 0.045 lb ai/A (50.9 g ai/ha); 0.093 lb ai/A (104.1 g ai/ha) PH-label: 0.046 lb ai/A (51.1 g ai/ha); 0.090 lb ai/A (101.3 g ai/ha)
Number of applications	One
Timing of applications	90 days prior to the first planting rotation
PHI	N/A; application to bare soil

## B.4. Identification/Characterization of Residues

### B.4.1. Sample Preparation



Potato tuber samples were washed with water and gentle rubbing to remove adhering soil. Wheat grain was separated from the chaff, and the chaff was added to the straw sample. Except for wheat grain, all rotated crop samples were cut into smaller pieces prior to milling in the presence of dry ice or liquid nitrogen. Because of low TRR levels (<0.010 ppm) in potato tubers and wheat forage and grain from both labels at both treatment levels, and in kale from both labels at the low treatment level, only wheat hay and straw and potato foliage from both labels at both treatment levels, and kale from both labels at the higher treatment level were extracted.

In general, samples were extracted (3x) with acetonitrile:water (80:20, v:v) and vacuum filtered. The combined filtrates were concentrated and partitioned (3x) with dichloromethane, and the dichloromethane phases were combined.

The combined dichloromethane phases of PH-treated potato foliage (both treatment levels) were concentrated and partitioned between hexane and acetonitrile (ACN). The resulting ACN phase was concentrated, diluted with ACN and water, and filtered for HPLC analysis. A subsample of the aqueous phase of PH-treated potato foliage (high treatment level) remaining following dichloromethane partitioning was also concentrated to near dryness, diluted with water, and filtered for HPLC analysis.

#### B.4.2. Analytical Methodology

Total radioactive residues (TRR) in replicate (3-9) milled samples were determined by combustion/LSC. The combustion/LSC limit of quantitation (LOQ) was reported as 0.002 ppm for wheat forage, kale, and potato tubers, and 0.005 ppm for wheat hay, straw, and grain, and potato foliage; the limit of detection (LOD) was reported as 0.001 ppm for all plant matrices. Radioactivity in the extracts was determined by LSC, and nonextractable radioactivity was determined by combustion/LSC.

Only the ACN and aqueous phases of the extracts of PH-treated potato foliage were analyzed by HPLC. In all other samples, because the aqueous extracts contained <0.03 ppm and the organic extracts contained <0.01 ppm, no further characterization was attempted. HPLC analyses were performed using a system equipped with a C-18 column; UV detector (254 nm), flow-through radioactivity detector, and fraction collector. A gradient mobile phase of water and ACN, each with 0.1% acetic acid, was used. Reference standards of unlabeled penoxsulam; 2-(2,2-difluoroethoxy)-N-(5,6-dihydro-8-methoxy-5-oxo[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide (5-OH XDE-638); 3-[[[2-(2,2-difluoroethoxy)-6-(trifluoromethyl)phenyl]sulfonyl]amino]-1H-1,2,4-triazole-5-carboxylic acid (BSTCA); BSTCA, triethylammonium salt; and 2-(2,2-difluoroethoxy)-N-1H-1,2,4-triazol-3-yl-6-(trifluoromethyl)benzenesulfonamide (BST) were used. No confirmatory methods were used; therefore, all identifications were considered to be tentative. Fraction collection/LSC was used for quantitation of metabolites.

### C. RESULTS AND DISCUSSION

TRR in rotational crops are reported in Table C.2.1. TRR accumulated at  $\geq 0.01$  ppm in certain rotated crops planted 90 days following a single soil application of [triazolopyrimidine-2-<sup>14</sup>C]penoxsulam (TP) at 0.045 or 0.093 lb ai/A, or [phenyl-U-<sup>14</sup>C]penoxsulam (PH) at 0.046 or 0.090

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lb ai/A. At the low treatment rate, TRR were  $>0.01$  ppm in potato foliage (0.024 ppm from the TP plot and 0.047 ppm from the PH plot), wheat hay (0.021 ppm, PH plot only), and wheat straw (0.011 ppm, TP plot; 0.024 ppm, PH plot). At the higher treatment rate, TRR were  $>0.01$  ppm in kale (0.014 ppm, TP plot only), potato foliage (0.038 ppm, TP plot; 0.062 ppm, PH plot), wheat hay (0.022 ppm, TP plot; 0.032 ppm, PH plot), and wheat straw (0.030 ppm, TP plot; 0.028 ppm, PH plot). TRR were  $<0.01$  ppm in the following matrices: potato tuber ( $\leq 0.003$  ppm), wheat forage ( $\leq 0.007$  ppm), and wheat grain ( $<0.005$  ppm) from both labels at both treatment rates; TP-treated kale (0.003 ppm) at the low treatment rate and PH-treated kale ( $\leq 0.008$  ppm) at both treatment rates; and TP-treated wheat hay (0.009 ppm) at the low treatment rate. TRR were generally higher in PH-treated commodities than in TP-treated commodities.

Only crops with TRR  $\geq 0.008$  ppm were subjected to extraction procedures. The distribution of radioactivity in the rotated crops, kale (high treatment rate only), potato foliage, and wheat hay and straw, is presented in Tables C.2.2.1 through C.2.2.4. Solvent extraction (ACN/water) released 62-97% TRR from rotational crop matrices. The majority of the extractable residues remained in the aqueous phase after partitioning with dichloromethane, and only the organic phase of potato foliage was subjected to further partitioning. Nonextractable residues in rotational crop commodities ranged 4-31% TRR ( $<0.001$ -0.009 ppm). These procedures extracted sufficient residues from rotational crop matrices.

Only the extracts of PH-treated potato foliage were analyzed by HPLC for metabolite identification; the characterization and identification of residues in PH-label potato foliage is summarized in Table C.2.3.1. Total identified residues were 8.4% and 38.9% TRR in PH-label potato foliage at the low and high treatment rates, respectively. The 5-OH metabolite was identified at 8.4-13.9% TRR (0.004-0.009 ppm), and a single unknown was characterized at approximately the same level (8.9-11.8% TRR, 0.004-0.007 ppm) in both the low and high treatment rate potato foliage. In addition, the BSTCA metabolite was identified in potato foliage from the high treatment rate, at 25.0% TRR (0.015 ppm). Three unknowns were also characterized, each present at  $\leq 18.0\%$  TRR ( $\leq 0.011$  ppm).

Residues were characterized/identified in potato foliage by HPLC analysis, and, because no confirmatory method was used, the identifications are considered tentative. However, because no parent was detected and no single component was present at  $>0.015$  ppm in potato foliage, RAB2 concludes that these methods successfully identified the predominant residues in rotational crop matrices. No storage stability data were submitted in conjunction with the confined rotational crop study. Samples for this confined rotational crop study were analyzed within 5 months of harvest. As samples were analyzed within an acceptable time frame, no further storage stability data are necessary.

We note that data concerning the radioactivity and characterization/identification of metabolites in treated soil were included with this submission; however, these data are not presented herein.

### C.1. Storage Stability

Samples of rotated crops were stored frozen ( $-20$  °C) at the field site within 2 hours of collection, except for wheat hay samples, which were dried in the greenhouse for 11 days after collection, and potato tuber samples, which were placed on "blue" ice for immediate shipment to the analytical

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laboratory. Samples of kale, potato foliage, and wheat hay and straw were extracted within 34-173 days (<6 months) of harvest; samples of potato tubers and wheat forage and grain were not extracted, but TRR determinations were conducted within 1 month of harvest. Actual analysis dates were not provided for potato foliage extracts (the only rotational crop matrix analyzed by HPLC). Samples for this confined rotational crop study were analyzed within 5 months of harvest. As samples were analyzed within an acceptable time frame, no further storage stability data are necessary.

Matrix	Plantback interval	Storage Temp.	Actual Storage Duration	Limit of Demonstrated Storage Stability
Kale	90 days	Frozen; storage temperature at laboratory not specified	36-89 days (harvest to extraction)	Within 4-6 month guideline
Potato, foliage			127 days (harvest to final extraction)	
Potato, tubers			16 days (harvest to combustion/LSC)	
Wheat, forage			23 days (harvest to combustion/LSC)	
Wheat, hay			173 days (harvest to final extraction)	
Wheat, straw			34 days (harvest to extraction)	
Wheat grain			28 days (harvest to combustion/LSC)	

## C.2. Identification, Characterization, and Distribution of Residues

Matrix	Plantback interval (days)	TP-label (ppm) <sup>1</sup>		PH-label (ppm) <sup>1</sup>	
		Low Treatment: 0.045 lb ai/A	High Treatment: 0.093 lb ai/A	Low Treatment: 0.046 lb ai/A	High Treatment: 0.090 lb ai/A
Kale	90	0.003	0.014	0.005	0.008
Potato, foliage	90	0.024	0.038	0.047	0.062
Potato, tuber	90	<LOQ	0.003	<LOQ	0.003
Wheat, forage	90	<LOQ	0.005	0.004	0.007
Wheat, hay	90	0.009	0.022	0.021	0.032
Wheat, straw	90	0.011	0.030	0.024	0.028
Wheat, grain	90	<LOD	<LOQ	<LOD	<LOQ

<sup>1</sup> The LOQ was 0.002 ppm for wheat forage, kale, and potato tubers, and 0.005 ppm for wheat hay, straw, and grain, and potato foliage; the LOD was 0.001 ppm for all plant matrices.

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**TABLE C.2.2.1. Distribution of the Parent and the Metabolites in Rotational Crop Matrices Planted 90 Days Following Application of [Triazolopyrimidine-2-<sup>14</sup>C]Penoxsulam to the Soil at 0.045 lb ai/A.**

Metabolite Fraction	Potato Foliage		Wheat Hay		Wheat Straw	
	TRR = 0.024 ppm		TRR = 0.009 ppm		TRR = 0.011 ppm	
	%TRR	ppm	%TRR	ppm	%TRR	ppm
ACN/water extract	70.1	0.017	73.3	0.006	62.7	0.007
Aqueous phase	47.3	0.011	53.4	0.005	53.1	0.006
Dichloromethane phase	17.0	0.004	26.2	0.002	13.0	0.001
Total extractable	64.3	0.015	79.6	0.007	66.1	0.007
Total identified	0	0	0	0	0	0
Total unidentified	64.3	0.015	79.6	0.007	66.1	0.007
Total bound residues	21.0	0.005	27.5	0.002	25.6	0.003
% Accountability	85.3		107.1		91.7	

**TABLE C.2.2.2. Distribution of the Parent and the Metabolites in Rotational Crop Matrices Planted 90 Days Following Application of [Phenyl-U-<sup>14</sup>C]Penoxsulam to the Soil at 0.046 lb ai/A.<sup>1</sup>**

Metabolite Fraction <sup>2</sup>	Potato Foliage		Wheat Hay		Wheat Straw	
	TRR = 0.047 ppm		TRR = 0.021 ppm		TRR = 0.024 ppm	
	%TRR	ppm	%TRR	ppm	%TRR	ppm
ACN/water extract	79.8	0.037	88.1	0.019	90.8	0.022
Aqueous phase	60.8	0.029	65.1	0.014	69.2	0.017
Dichloromethane phase	19.5	0.009	12.7	0.003	14.6	0.004
Hexane phase	0.5	<0.001				
ACN phase	19.7	0.009				
5-OH XDE-638	8.4	0.004				
Unknown Rt=27-28 min	8.9	0.004				
Total extractable	81.0	0.038	77.8	0.017	83.8	0.021
Total identified	8.4	0.004	0	0	0	0
Total unidentified	70.2	0.033	77.8	0.017	83.8	0.021
Total bound residues	13.4	0.006	23.6	0.005	11.5	0.003
% Accountability	94.4		101.4		95.3	

<sup>1</sup> Shading indicates that the extraction step and/or characterization analysis was not conducted for the matrix in question.

<sup>2</sup> The 5-OH XDE-638 metabolite was tentatively identified by HPLC.

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**TABLE C.2.2.3. Distribution of the Parent and the Metabolites in Rotational Crop Matrices Planted 90 Days Following Application of [Triazolopyrimidine-2-<sup>14</sup>C]Penoxsulam to the Soil at 0.093 lb ai/A.**

Metabolite Fraction	Kale		Potato Foliage		Wheat Hay		Wheat Straw	
	TRR = 0.014 ppm		TRR = 0.038 ppm		TRR = 0.022 ppm		TRR = 0.030 ppm	
	%TRR	ppm	%TRR	ppm	%TRR	ppm	%TRR	ppm
ACN/water extract	96.5	0.013	74.6	0.028	81.1	0.018	61.6	0.018
Aqueous phase	54.1	0.007	53.5	0.020	51.5	0.011	51.2	0.015
Dichloromethane phase	31.5	0.004	16.9	0.006	23.0	0.005	13.6	0.004
Total extractable	85.6	0.011	70.4	0.026	74.5	0.016	64.8	0.019
Total identified	0	0	0	0	0	0	0	0
Total unidentified	85.6	0.011	70.4	0.026	74.5	0.016	64.8	0.019
Total bound residues	13.4	0.002	22.1	0.008	27.8	0.006	31.2	0.009
% Accountability	99.0		92.5		102.3		96.0	

**TABLE C.2.2.4. Distribution of the Parent and the Metabolites in Rotational Crop Matrices Planted 90 Days Following Application of [Phenyl-U-<sup>14</sup>C]Penoxsulam to the Soil at 0.090 lb ai/A.<sup>1</sup>**

Metabolite Fraction <sup>2</sup>	Kale		Potato Foliage		Wheat Hay		Wheat Straw	
	TRR = 0.008 ppm		TRR = 0.062 ppm		TRR = 0.032 ppm		TRR = 0.028 ppm	
	%TRR	ppm	%TRR	ppm	%TRR	ppm	%TRR	ppm
ACN/water extract	80.9	0.007	95.6	0.060	93.8	0.030	86.2	0.024
Aqueous phase	63.0	0.005	68.1	0.042	76.7	0.025	69.3	0.019
BSTCA			25.0	0.015				
Unknown Rt=7 min			18.0	0.011				
Unknown Rt=20 min			16.5	0.010				
Unknown Rt=27-28 min			11.1	0.007				
Dichloromethane phase	16.5	0.001	27.1	0.017	16.6	0.005	17.6	0.005
Hexane phase			0.6	<0.001				
ACN phase			25.3	0.016				
5-OH XDE-638			13.9	0.009				
Unknown Rt=27-28 min			11.8	0.007				
Total extractable	79.5	0.006	94.0	0.058	93.3	0.030	86.9	0.024
Total identified	0	0	38.9	0.024	0	0	0	0
Total unidentified	79.5	0.006	58.0	0.035	93.3	0.030	86.9	0.024
Total bound residues	4.1	<0.001	6.8	0.004	7.8	0.003	15.7	0.004
% Accountability	83.6		100.8		101.1		102.6	

<sup>1</sup> Shading indicates that the extraction step and/or characterization analysis was not conducted for the matrix in question.

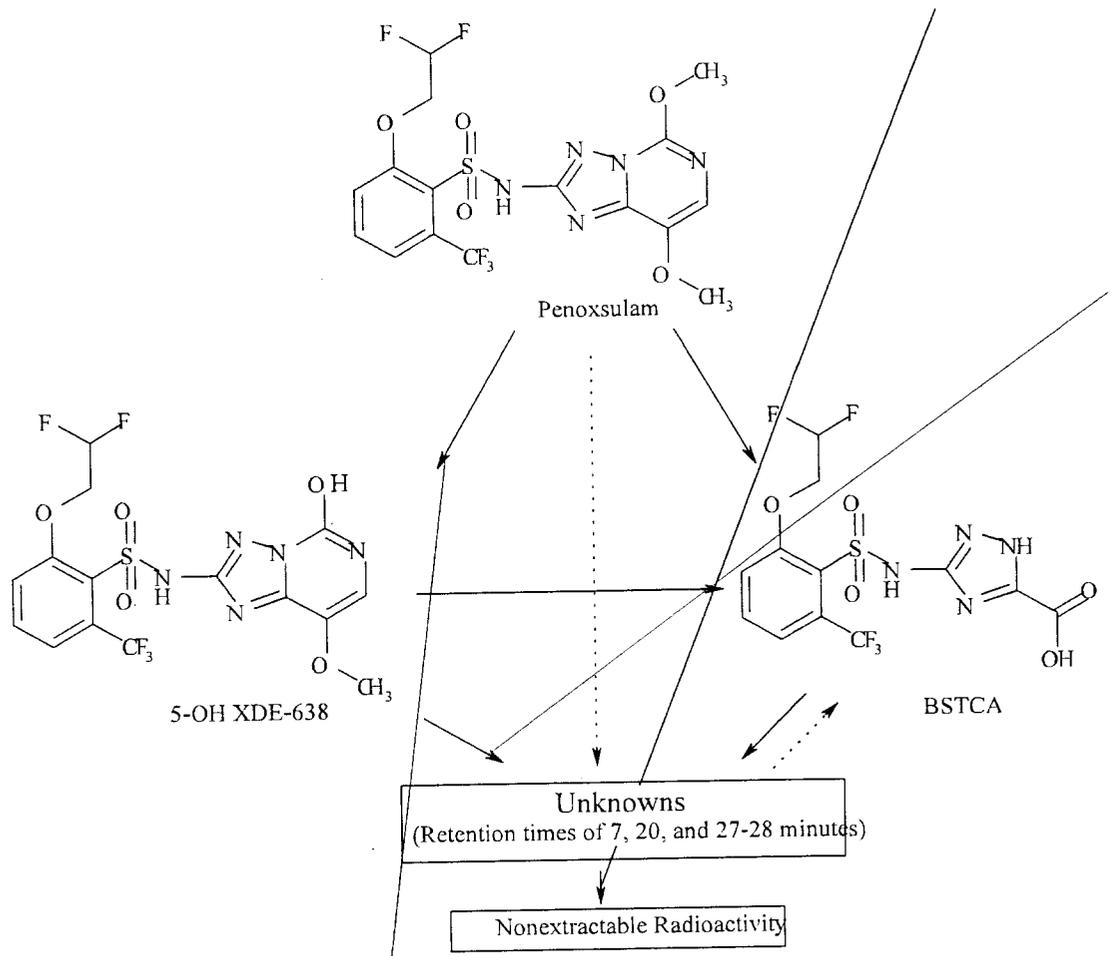
<sup>2</sup> The 5-OH XDE-638 and BSTCA metabolites were tentatively identified by HPLC.



Compound	Potato Foliage; Low Treatment		Potato Foliage; High Treatment	
	TRR = 0.047 ppm		TRR = 0.062 ppm	
	% TRR	ppm	% TRR	ppm
5-OH XDE-638	8.4	0.004	13.9	0.009
BSTCA	--	--	25.0	0.015
Unknown Rt=7 min	--	--	18.0	0.011
Unknown Rt=20 min	--	--	16.5	0.010
Unknown Rt=27-28 min	8.9	0.004	22.9	0.014
Aqueous phase	60.8	0.029	--	--
Hexane phase	0.5	<0.001	0.6	<0.001
Total identified	8.4	0.004	38.9	0.024
Total characterized	70.2	0.033	58.0	0.035
Total extractable	81.0	0.038	94.0	0.058
Total bound	13.4	0.006	6.8	0.004

### C.3. Proposed Metabolic Profile

**FIGURE C.3.1. Proposed Metabolic Profile of Penoxsulam in Rotational Potato Foliage.**



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Common name/code Figure C.3.1 ID No.	Chemical name	Chemical structure
5-OH XDE-638	2-(2,2-difluoroethoxy)-N-(5,6-dihydro-8-methoxy-5-oxo[1,2,4]triazolo[1,5-c]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide	
BSTCA	3-[[[2-(2,2-difluoroethoxy)-6-(trifluoromethyl)phenyl]sulfonyl]amino]-1H-1,2,4-triazole-5-carboxylic acid	

#### D. CONCLUSIONS

TRR accumulated at  $\geq 0.01$  ppm in certain rotated crops planted 90 days following a single soil application of [triazolopyrimidine-2- $^{14}\text{C}$ ]penoxsulam at 0.045 and 0.093 lb ai/A, or [phenyl- $^{14}\text{C}$ ]penoxsulam at 0.046 and 0.090 lb ai/A. At the low treatment rate, TRR were 0.024-0.047 ppm in potato foliage (both labels), 0.021 ppm in PH-treated wheat hay, and 0.011-0.024 ppm in wheat straw (both labels). At the higher treatment rate, TRR were 0.014 ppm in TP-treated kale, 0.038-0.062 ppm in potato foliage (both labels), 0.022-0.032 ppm in wheat hay (both labels), and 0.028-0.030 ppm in wheat straw (both labels). TRR were  $< 0.01$  ppm in potato tuber, wheat forage, and wheat grain from both labels at both treatment rates, in TP-treated kale at the low treatment rate, in PH-treated kale at both treatment rates, and in TP-treated wheat hay at the low treatment rate. TRR were generally higher in PH-treated commodities than in TP-treated commodities.

Only the extracts of PH-treated potato foliage were analyzed by HPLC for metabolite identification. Total identified residues were 8.4% and 38.9% TRR in PH-treated potato foliage at the low and high treatment rates, respectively, and the parent, penoxsulam, was not detected in either sample. The 5-OH XDE-638 and BSTCA metabolites ( $\leq 0.015$  ppm each) were tentatively identified in potato foliage.

Based on data from the confined rotational crop study, no quantifiable residues of penoxsulam or 5-OH XDE-638 are expected to be present in the raw agricultural commodities of small grains, leafy vegetables, and root crops planted 90 days following treatment with penoxsulam at 0.045 or 0.090 lb ai/A. The data also indicate that residues of BSTCA could be present at  $\geq 0.01$  ppm in the foliage of root crops planted 90 days following treatment at 0.090 lb ai/A.

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The petitioner proposed that the penoxsulam is metabolized in rotated potato foliage to 5-OH XDE-638 and BSTCA, and that both 5-OH XDE-638 and BSTCA are then metabolized to unknowns and nonextractable residues.

#### **E. REFERENCES**

None.

#### **F. DOCUMENT TRACKING**

RDI: R. Loranger (7/9/04)  
Petition Number(s): 3F06542  
DP Barcode(s): D288152  
PC Code: 119031

Attachment: L. Graper, Dow AgroSciences, 5/4/04

Template Version April 2003



IN RESPONSE TO: Errico.Philip@epamail.epa.gov  
SENT: Tuesday, May 04, 2004 2:20 PM

SUBJECT: Response to Additional Information Requested by Residue Chemistry for Penoxsulam

STUDY IDENTIFICATION: 45830720 Graper, L. (2002) A Confined Rotational Crop Study With (Carbon-14) XDE-638 Using Three Different Crops: Lab Project Number: 000266.

REQUEST OF RESIDUE CHEMISTRY: Residue Chemistry commented that actual analysis dates were not provided for potato foliage extracts (the only rotational crop matrix analyzed by HPLC) and requested that the petitioner submit the actual analysis dates for potato foliage extracts to allow RAB2 to determine whether storage stability data are required to support the confined rotational crop studies.

RESPONSE OF PETITIONER: The requested dates have been added to an excerpt of Table 3 (page 46, report 000266) shown below and are indicated in **bold**.

Event	Date	Days after Treatment (DAT)	Days after Planting (DAP)	Days after Harvest/Sampling (DAH)
Mature Potato Foliage Harvest	14-May-01	298	208	0
Milling Completed	30-May-01	N/A	N/A	16
Combustion Analysis	01-Jun-01	N/A	N/A	18
Initial Extraction	19-Jun-01	N/A	N/A	36
Final Extraction	18-Sep-01	N/A	N/A	127
<b>Initial HPLC Analysis</b>	<b>6-Sep-01</b>	<b>N/A</b>	<b>N/A</b>	<b>115</b>
<b>Final HPLC Analysis</b>	<b>10-Oct-01</b>	<b>N/A</b>	<b>N/A</b>	<b>149</b>

The additional dates refer to the HPLC analyses for the potato foliage found in Figures 12, 13 and 14 which are located on pages 76, 77 and 78, respectively, in the final report.

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These analysis dates demonstrate that the HPLC analyses were done within approximately 4 to 5 months of harvest of the potato foliage.

The Residue Chemistry Guidelines (OPPTS 860.1300 Nature of the Residue – Plants, Livestock, section (d)(7)) recommend that storage stability data should not normally be required for samples analyzed within 4 to 6 months of collection, provided evidence is given that attempts were made to limit degradation of residues by appropriate storage of matrices and extracts during the analytical portion of the study. Since the 4 to 5 month analysis intervals for potato foliage fall within the recommended 4 to 6 month period and the final report (section 3.7.4, page 28, report 000266) indicates that samples were stored in the freezer, it is the opinion of the petitioner that the supplied data adequately address any concerns of storage stability for residues in this study.

Additional questions concerning the storage or any other aspect of this study may be directed to the study director:

L. Kurt Graper  
Dow AgroSciences  
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