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STUDY REPORT

45830717 Lindsay, D.; Miller, A.; Thomas, A. (2002) Frozen Storage Stability of XDE-638 in Rice (Raw Agricultural Commodities: Grain, Straw, Immature Forage) and its Processed Products (Bran, Hulls, Polished Rice)--Interim Report: Lab Project Number: 010100. Unpublished study prepared by Dow AgroSciences LLC. 78 p.

EXECUTIVE SUMMARY

Dow AgroSciences LLC has submitted the interim results of a 24-month storage stability study with penoxsulam. Untreated samples of homogenized rice grain, straw, immature forage, bran, hulls, and polished rice were fortified with penoxsulam at 0.10 ppm. The fortified samples were stored at -20 °C for up to 197 days (processed rice commodities) or 210 days (rice forage, grain, and straw). Under these conditions, residues of penoxsulam were relatively stable in rice grain, straw, immature forage, and processed rice commodities (bran, hulls, and polished rice).

Samples of rice matrices were analyzed for residues of penoxsulam using Dow AgroSciences Method GRM 01.25, an LC/MS/MS method. A complete description of the method is provided in the DER for MRID 45830714.

The interim storage stability data indicate that residues of penoxsulam are stable under frozen storage conditions in rice grain, straw, immature forage, bran, hulls, and polished rice for up to ~7 months. The petitioner stated that the full study will include storage intervals of up to 24 months for rice commodities.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS

Under the conditions and parameters used in the study, the storage stability 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

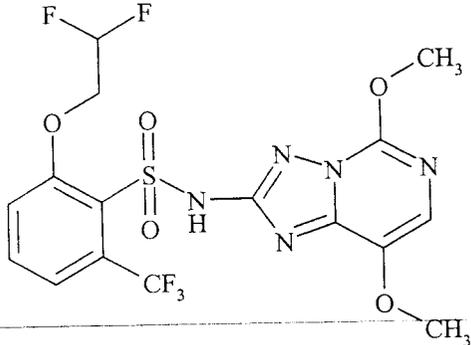
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Signed and dated GLP and Data Confidentiality statements were provided. No deviations from regulatory requirements were reported. An unsigned Quality Assurance statement was included, which noted that the data in the report are interim data and have not yet been audited.

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 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.

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)- α,α,α -trifluoro-o-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).

Parameter	Value		Reference
Melting point/range	Not available		
pH	Not available		
Density	Not available		
Water solubility	pH	Solubility (mg/L)	MRID 45830720
	(unbuffered)	4.91	
	5	5.66	
	7	408	
	9	1460	



Parameter	Value		Reference
	Solvent	Solubility (g/L)	
Solvent solubility	DMSO	78.4	MRID 45830720
	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 µg/mL	
Vapor pressure	7.16 x 10 ⁻¹⁶ mm Hg at 25 °C		MRID 45830720
Dissociation constant, pK _a	5.1		MRID 45830720
Octanol/water partition coefficient, Log(K _{ow})	pH	Log(K _{ow})	MRID 45830720
	(unbuffered)	-0.354	
	5	1.137	
	7	-0.602	
	9	-1.418	

B. EXPERIMENTAL DESIGN

B.1. Sample Preparation

Samples of untreated homogenized rice grain, straw, immature forage, bran, hulls, and polished rice were placed into polypropylene containers and fortified with penoxsulam at 0.10 ppm. Fortified samples were then stored frozen at -20 °C. The penoxsulam fortification standard was prepared in acetonitrile (ACN). Samples were fortified on a staggered schedule so that some samples were analyzed together and shared fresh fortification recoveries.

B.2. Analytical Methodology

Samples of fortified and unfortified rice matrices were analyzed for residues of penoxsulam using Dow AgroSciences Method GRM 01.25 following 0 and 210 days (RAC) or 0, 42, 83, and 197 days (processed commodities) of frozen storage. Fresh fortification samples of each rice matrix were analyzed for concurrent method recoveries. A complete description of the method is provided in the DER for MRID 45830714.

Briefly, homogenized samples of rice matrices were extracted with ACN:water (8:2; v:v) and centrifuged. An aliquot of the supernatant was diluted with water and purified by solid-phase extraction (SPE). Residues were eluted with ACN:formic acid (100:0.1, v:v), evaporated to dryness, and redissolved in ACN:methanol:water (15:15:70, v:v:v, each containing 0.1% acetic acid) for analysis by LC/MS/MS. The validated limit of quantitation (LOQ) was 0.01 ppm for all rice matrices. Although the petitioner made reference to “nondetectable” residues, no limit of detection was defined in the study. In the method submission (MRID 45830714), the LOD was defined as 0.002 ppm.



C. RESULTS AND DISCUSSION

The concurrent method validation data included in the study indicate that the LC/MS/MS method GRM 01.25 is adequate for the determination of residues of penoxsulam in/on rice grain, straw, immature forage, bran, hulls, and polished rice. Apparent residues were nondetectable (presumably <0.002 ppm) in all control (unfortified) samples (two samples each of rice grain, straw, and immature forage, and four samples each of bran, hulls, and polished rice).

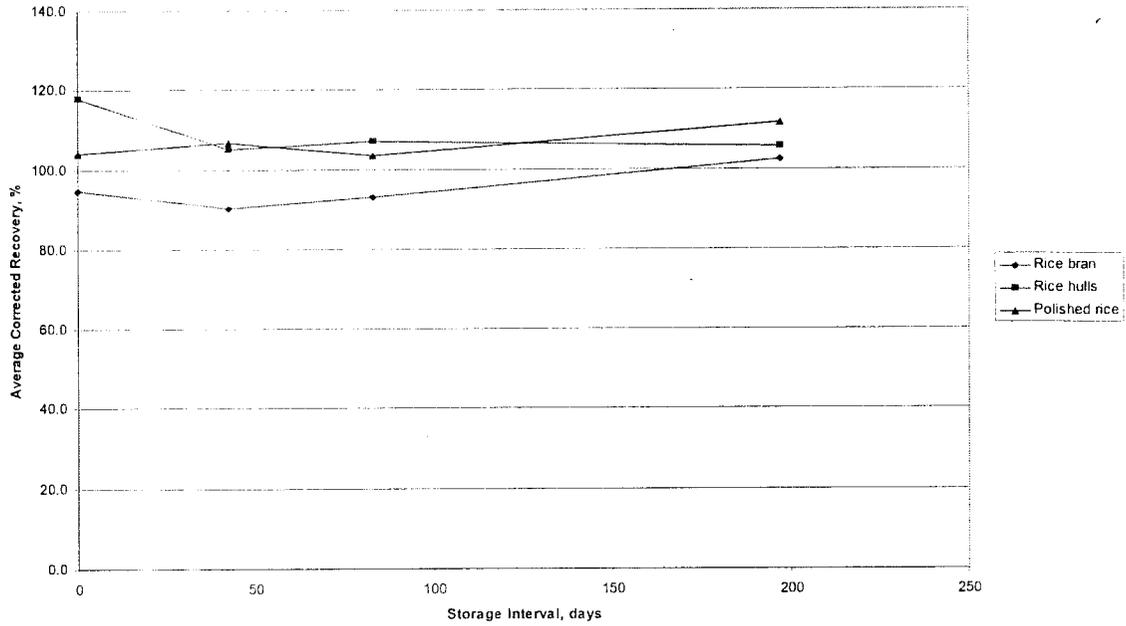
Residues of penoxsulam appear to be stable in rice grain, straw, and immature forage stored frozen for up to 210 days (6.9 months) and in processed rice commodities (bran, hulls, and polished rice) stored frozen for up to 197 days (6.5 months).

Matrix	Analyte	Spike level (ppm)	Storage Interval (days)	Sample size (n)	Recoveries (%)	Mean \pm std dev
Rice grain	Penoxsulam	0.10	0	3	98, 100, 101	100 \pm 1.5
			210	3	89, 96, 98	94 \pm 4.7
Rice straw	Penoxsulam	0.10	0	3	100, 102, 104	102 \pm 2.0
			210	3	92, 94, 97	94 \pm 2.5
Rice forage	Penoxsulam	0.10	0	3	99, 102, 102	101 \pm 1.7
			210	3	91, 97, 101	96 \pm 5.0
Rice bran	Penoxsulam	0.10	0/42	3	83, 93, 132 ¹	88 \pm 7.1
			83/197	3	95, 97, 100	97 \pm 2.5
Rice hull	Penoxsulam	0.10	0/42	3	86, 88, 90	88 \pm 2.0
			83/197	3	92, 95, 101	96 \pm 4.6
Polished rice	Penoxsulam	0.10	0/42	3	90, 92, 94	92 \pm 2.0
			83/197	3	97, 98, 101	99 \pm 2.1

¹ This recovery value was not used in the mean and standard deviation calculation because it was considered by the petitioner to be an outlier.



FIGURE C.1. Graph of Residue Stability in Rice Processed Commodities.



Because only two time points were included in this interim report for rice grain, straw, and forage, a graph of residue stability was not created for these commodities.



TABLE C.2. Stability of Penoxsulam Residues in Rice Matrices Following Storage at 20 °C.				
Commodity	Spike level (ppm)	Storage interval (days)	Recovered residues (ppm)	Corrected % recovery ¹
Rice grain	0.10	0	0.088, 0.089, 0.092	88, 89, 92
		210	0.095, 0.097, 0.101	101, 103, 108
Rice straw	0.10	0	0.097, 0.099, 0.103	95, 97, 101
		210	0.113, 0.114, 0.115	120, 121, 122
Rice forage	0.10	0	0.098, 0.099, 0.104	97, 98, 103
		210	0.115, 0.116, 0.123	120, 121, 128
Rice bran	0.10	0	0.078, 0.078, 0.093	89, 89, 106
		42	0.073, 0.078, 0.085	83, 90, 98
		83	0.087, 0.091, 0.092	90, 94, 95
		197	0.097, 0.100, 0.101	100, 103, 104
Rice hull	0.10	0	0.101, 0.103, 0.108	114, 117, 123
		42	0.078, 0.100, 0.100	89, 113, 113
		83	0.101, 0.104, 0.104	105, 108, 108
		197	0.097, 0.100, 0.107	101, 104, 112
Polished rice	0.10	0	0.094, 0.094, 0.098	102, 103, 107
		42	0.096, 0.097, 0.100	105, 106, 109
		83	0.097, 0.100, 0.107	99, 102, 109
		197	0.107, 0.109, 0.113	109, 111, 115

¹ Corrected for average concurrent method recoveries.

D. CONCLUSIONS

The submitted interim storage stability study adequately demonstrates the stability of penoxsulam residues in rice matrices stored frozen for ~7 months. The data indicate that residues of penoxsulam are relatively stable under frozen storage conditions in rice grain, straw, and immature forage for up to 210 days and in processed rice commodities (bran, hulls, and polished rice) for up to 197 days. The petitioner stated that the full study will include storage intervals of up to 24 months for rice commodities.

E. REFERENCES

None.

F. DOCUMENT TRACKING

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

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