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**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**

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OFFICE OF PUBLIC RECORD
HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES**MEMORANDUM**

Date: 18 December 2006

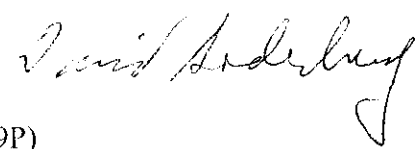
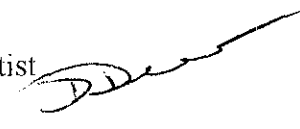
Subject: Penoxsulam. Frozen Storage Stability of XDE-638 (Penoxsulam) in Rice
(Raw Agricultural Commodities: Grain, Straw and Immature Forage) and
its Processed Products (Bran, Hulls, and Polished Rice)

DP Barcode: D328689

MRID No: 46449901

PC Code: 119031

40 CFR 180.605

From: David Soderberg, Chemist 
Reregistration Branch 3
Health Effects Division (7509P)Thru: Danette Drew, Branch Senior Scientist 
Reregistration Branch 3
Health Effects Division (7509P)To: P. Errico/J. Miller PM23
Herbicide Branch
Registration Division (7505P)

The attached document (der) reviews MRID 46449901, which provides the final, 24 month report on storage stability for penoxsulam residues in rice. Interim Storage Stability Reports had been submitted in support of the petition for establishment of tolerances for penoxsulam in rice, as was described in: Penoxsulam: Petition for Establishment of Permanent Tolerances for the Use on Rice. Summary of Analytical Chemistry and Residue Data, DP Barcode D288152, dated 8/11/2004. At the time that document was issued, however, this final report had not yet been submitted and the requirement for a final report addressing the storage stability of penoxsulam in rice was reported as a Residue Chemistry Deficiency.

MRID 46449901 fulfills this regulatory deficiency. It provides acceptable data in support of the rice petition, PP#3F6542. Residues of penoxsulam were tested in/on rice grain, straw and immature forage after frozen storage for up to 732 days (24 months) and in/on the processed rice

commodities: bran, hulls and polished rice for up to 390 days (12.8 months.). Residues were stable in all matrices tested when under frozen storage for either 13 or 24 months – as tested.



Primary Evaluator

David Soderberg
David Soderberg, Chemist, HED, RRB3

Date: 11 Dec 2006

Approved by

Danette Drew
Danette Drew, HED, RRB3

Date: 11 Dec 2006

This DER was originally prepared under contract by Dynamac Corporation (2275 Research Boulevard, Suite 300; Rockville, MD 20850; submitted 08/02/2006). The DER has been reviewed by the Health Effects Division (HED) and has been very minimally revised.

STUDY REPORT:

46449901 Lindsay, D. (2004) Frozen Storage Stability of XDE-638 in Rice (Raw Agricultural Commodities: Grain, Straw, and Immature Forage) and its Processed Products (Bran, Hulls, and Polished Rice). Project Number: 010100/01. Unpublished study prepared by Dow Agrosciences, LLC. 78 p.

EXECUTIVE SUMMARY:

Dow AgroSciences LLC submitted the final report of a storage stability study with penoxsulam on rice grain, straw, forage, and the processed rice commodities bran, hulls, and polished rice. The interim storage stability results (MRID 45830717) were reviewed in conjunction with the initial petition filing for penoxsulam on rice (PP# 3F6542; DP Barcode D288152, 8/11/04, W. Cutchin); the subject report replaces the interim submission. Untreated samples of the homogenized rice commodities were fortified with penoxsulam at 0.10 ppm. Samples were placed in frozen storage at ca. -20 °C and analyzed at storage intervals up to 24 months for rice grain, forage, and straw, and ~13 months for processed bran, hulls, and polished rice.

Samples of rice matrices were analyzed for residues of penoxsulam using Method GRM 01.25, the proposed enforcement method for rice (PP# 3F6542; DP Barcode D288152, 8/11/04, W. Cutchin). The validated limit of quantitation (LOQ) was 0.01 ppm for all rice matrices. The method is adequate based on the concurrent validation recoveries.

The data indicate that residues of penoxsulam are relatively stable in/on rice grain, straw, and immature forage stored frozen for up to 732 days (24 months) and in processed rice commodities (bran, hulls, and polished rice) stored frozen for up to 390 days (12.8 months).

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the storage stability data are scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the accompanying cover memo, D328689.

COMPLIANCE:



Signed and dated Good Laboratory Practice (GLP), Quality Assurance and Data Confidentiality statements were provided. No deviations from regulatory requirements were reported which would have an impact on the validity of the study.

A. BACKGROUND INFORMATION

Penoxsulam is a sulfonamide herbicide conditionally registered on rice for the selective control of grasses, broadleaf, and sedge weeds. The use on rice (PP#3F6542, DP Barcode D288152, 8/11/04, W. Cutchin) represented the first food/feed use of penoxsulam. The herbicide's mode of action at the cellular level involves the inhibition of acetolacetate synthase (ALS).

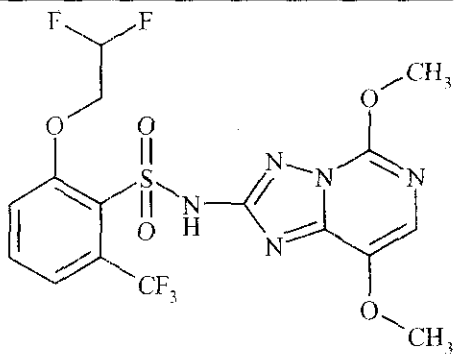
TABLE A.1. Test Compound Nomenclature.	
Compound	
Common name	Penoxsulam
Company experimental name	XDE-638
IUPAC name	6-(2,2-difluoroethoxy)-N-(5,8-dimethoxy-s-triazolo[1,5-c]pyrimidin-2-yl)- α,α,α -trifluoro-o-toluenesulfonamide
CAS name	2-(2,2-difluoroethoxy)-N-(5,8-dimethoxy[1,2,4]triazolo[1,5-c] pyrimidin-2-yl)-6-(trifluoromethyl) benzenesulfonamide
CAS registry number	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 the Technical Grade Test Compound: Penoxsulam.		
Parameter	Value	Reference
Melting point range	Not available	
pH	5.2	MRID 45830707
Density	1.61 g/mL at 20 °C	MRID 45830707
Water solubility at 19 °C	Unbuffered 4.91 mg/L pH 5 5.66 mg/L pH 7 408 mg/L pH 9 1460 mg/L	MRID 45830720



Parameter	Value	Reference	
Solvent solubility at 19 °C	Xylene	0.017 g/L	MRID 45830720
	1-Octanol	0.035 g/L	
	Methanol	1.48 g/L	
	Ethyl acetate	3.23 g/L	
	Acetonitrile	15.3 g/L	
	Acetone	20.3 g/L	
	Dimethylsulfoxide	78.4 g/L	
Vapor pressure	7.16 x 10 ⁻¹⁶ mm Hg at 25 °C	MRID 45830720	
Dissociation constant, pK _a	5.1 (ambient)	MRID 45830720	
Octanol/water partition coefficient, Log(K _{ow})	Unbuffered	-0.354	MRID 45830720
	pH 5	1.137	
	pH 7	-0.602	
	pH 9	-1.418	
UV/visible absorption spectrum	Not available		

B. EXPERIMENTAL DESIGN

B.1. Sample Handling and Preparation

Samples of untreated homogenized rice grain, straw, immature forage, bran, hulls, and polished rice (obtained from field trial and processing studies) were placed into polypropylene containers and fortified with penoxsulam at 0.10 ppm. Fortified samples were then stored frozen at -20 EC. 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.

The interim storage stability results (MRID 45830717) for storage intervals up to ~7 months were reviewed in conjunction with the initial petition (PP# 3F6542) filing for penoxsulam on rice. These data are repeated herein for completeness. Samples of fortified rice grain, forage, and straw were analyzed following approximately 0, 7, 13, 18, and 24 months of frozen storage; samples of fortified processed bran, hulls, and polished rice were analyzed following approximately 0, 1.5, 2.7, 6.5, and 13 months of frozen storage. A control and three fresh fortification samples of the respective rice matrix were analyzed with each sampling point.

B.2. Analytical Methodology

Samples of rice matrices were analyzed for residues of penoxsulam using Dow AgroSciences Method GRM 01.25, the proposed enforcement method for rice (PP# 3F6542; DP Barcode D288152, 8/11/04, W. Cutchin). 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; the method defines the LOD as 0.002 ppm.



C. RESULTS AND DISCUSSION

The concurrent method validation data (Table C.1) 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. No residues were detectable (<0.002 ppm) in/on all control (unfortified) samples (five samples each of rice grain, straw, forage, bran, hulls, and polished rice).

Residues of penoxsulam appear to be stable in/on rice grain, straw, and immature forage stored frozen for up to 732 days (24 months) and in processed rice commodities (bran, hulls, and polished rice) stored frozen for up to 390 days (12.8 months). Graphs of the stability of penoxsulam residues in the rice matrices are presented under Figure 1.

TABLE C.1. Summary of Concurrent Recoveries of Penoxsulam from Rice Matrices.

Matrix	Spike Level (ppm)	Storage Interval (days)	Sample Size (n)	Recoveries (%)	Mean ± SD (%)
Rice, grain	0.10	0	3	98, 100, 101	100 ± 1.5
		210	3	89, 96, 98	94 ± 4.7
		389	3	107, 107, 112	109 ± 2.9
		550/732	3	88, 88, 88	88 ± 0
Rice, straw	0.10	0	3	100, 102, 104	102 ± 2.0
		210	3	92, 94, 97	94 ± 2.5
		389	3	104, 106, 108	106 ± 2.0
		550/732	3	85, 86, 87	86 ± 1.0
Rice, forage	0.10	0	3	99, 102, 102	101 ± 1.7
		210	3	91, 97, 101	96 ± 5.0
		389	3	119, 121, 124	121 ± 2.5
		550/732	3	86, 86, 91	88 ± 2.9
Rice, bran	0.10	0/42	3	83, 93, 132 ¹	88 ± 7.1
		83/197	3	95, 97, 100	97 ± 2.5
		390	3	92, 102, 102	99 ± 5.8
Rice, hull	0.10	0/42	3	86, 88, 90	88 ± 2.0
		83/197	3	92, 95, 101	96 ± 4.6
		390	3	99, 102, 104	102 ± 2.5
Polished rice	0.10	0/42	3	90, 92, 94	92 ± 2.0
		83/197	3	97, 98, 101	99 ± 2.1
		390	3	91, 101, 107	100 ± 8.1

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.



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)	Mean Recovered Residues (ppm)	Mean Recovery (%)	Corrected % Recovery ¹
Rice, grain	0.10	0	0.0881, 0.0887, 0.0922	0.0896	90	90
		210	0.0948, 0.0972, 0.1013	0.0978	98	104
		389	0.1075, 0.1122, 0.1179	0.1125	113	103
		550	0.0821, 0.0854, 0.0854	0.0843	84	96
		732	0.0927, 0.0938, 0.0965	0.0943	94	107
Rice, straw	0.10	0	0.0968, 0.0986, 0.1032	0.0995	100	99
		210	0.1126, 0.1137, 0.1147	0.1137	114	121
		389	0.1141, 0.1170, 0.1236	0.1182	118	112
		550	0.0765, 0.0772, 0.0772	0.0770	77	90
		732	0.0907, 0.0925, 0.0965	0.0932	93	108
Rice, forage	0.10	0	0.0980, 0.0986, 0.1044	0.1003	100	99
		210	0.1147, 0.1158, 0.1179	0.1161	116	121
		389	0.1227, 0.1274, 0.1274	0.1258	126	104
		550	0.0772, 0.0774, 0.0783	0.0776	78	88
		732	0.0896, 0.0956, 0.0958	0.0937	94	106
Rice, bran	0.10	0	0.0779, 0.0781, 0.0926	0.0829	83	95
		42	0.0728, 0.0784, 0.0854	0.0789	79	90
		83	0.0873, 0.0913, 0.0922	0.0903	90	93
		197	0.0966, 0.1002, 0.1010	0.0993	99	102
		390	0.0799, 0.0820, 0.0904	0.0841	84	85
Rice, hull	0.10	0	0.1007, 0.1025, 0.1079	0.1037	104	118
		42	0.0783, 0.0998, 0.0998	0.0926	93	105
		83	0.1010, 0.1037, 0.1037	0.1028	103	107
		197	0.0966, 0.1002, 0.1073	0.1013	101	106
		390	0.1052, 0.1116, 0.1144	0.1104	110	108
Polished rice	0.10	0	0.0935, 0.0944, 0.0980	0.0953	95	104
		42	0.0962, 0.0971, 0.0998	0.0977	98	106
		83	0.0966, 0.1002, 0.1073	0.1013	101	103
		197	0.1073, 0.1090, 0.1126	0.1096	110	112
		390	0.1107, 0.1126, 0.1172	0.1135	114	115

¹ Corrected for mean concurrent recovery (see TABLE C.1.); corrected recoveries were calculated by the petitioner using unrounded values.

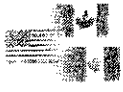
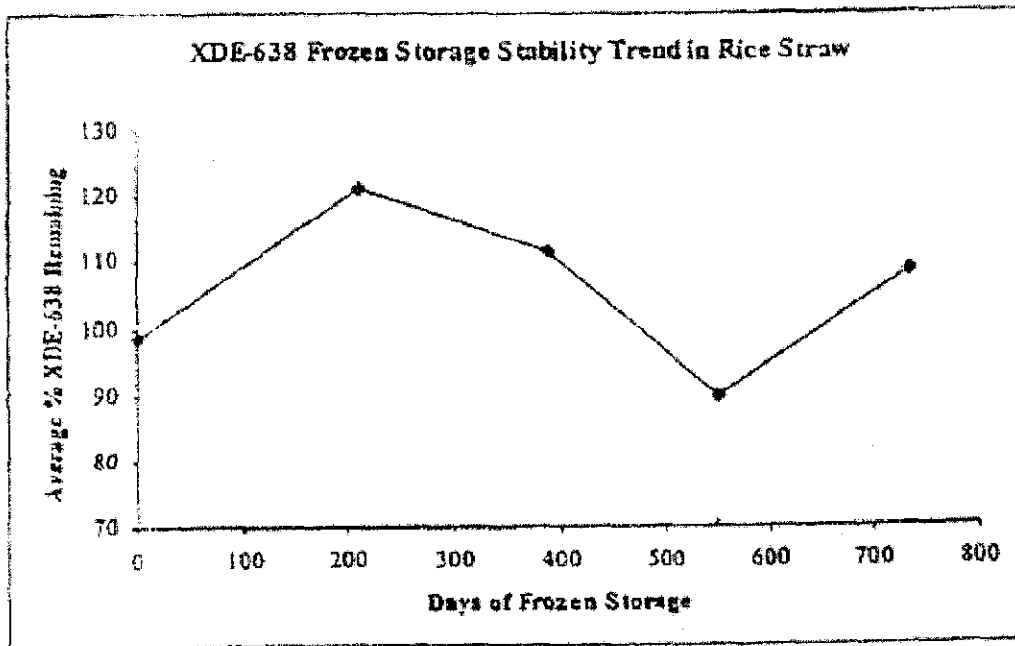
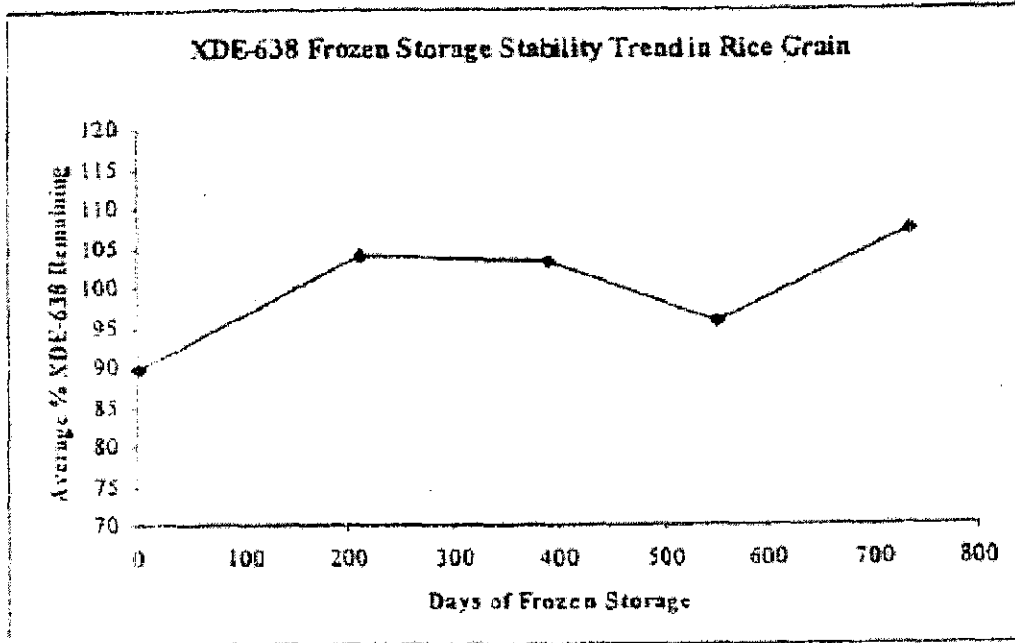
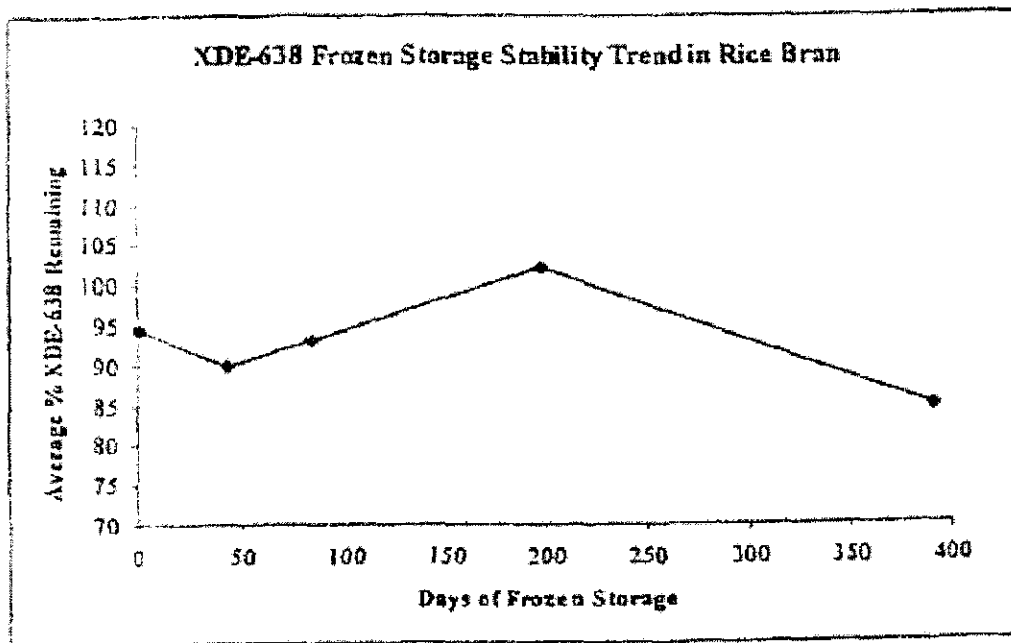
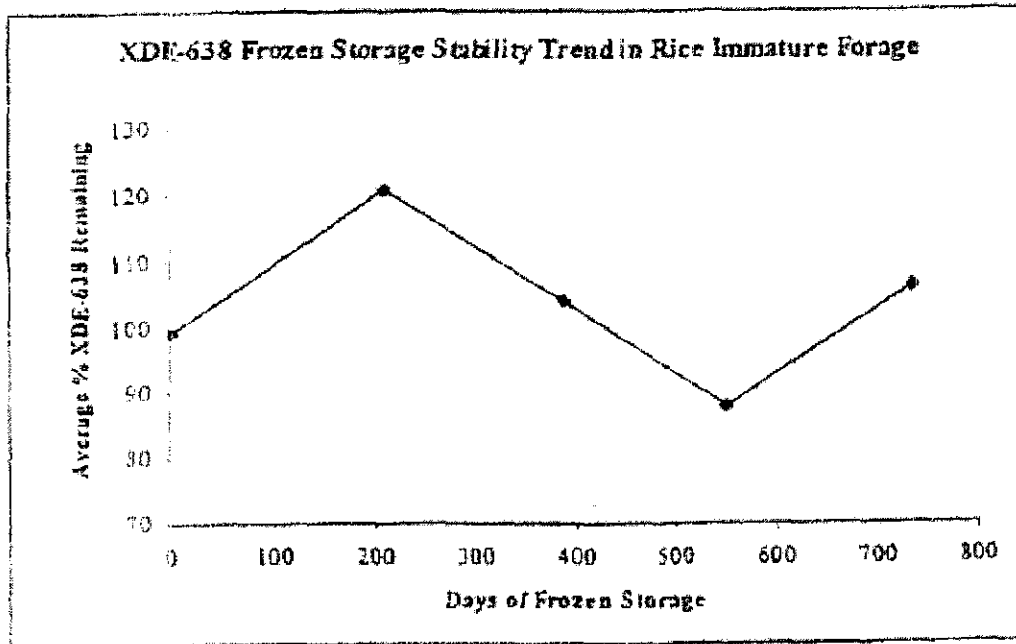
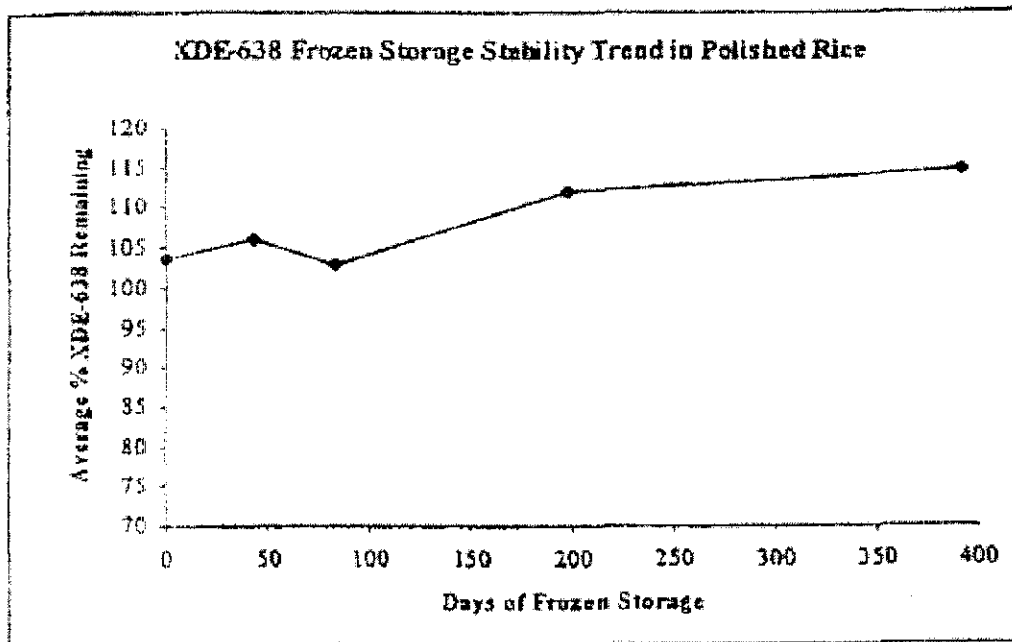
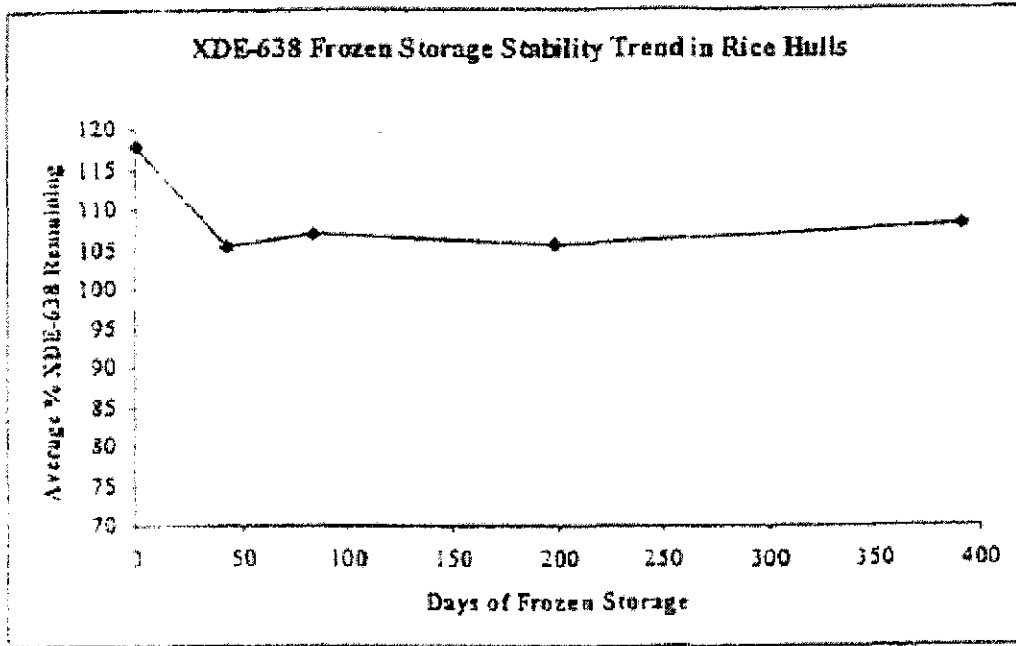
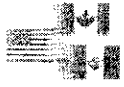


FIGURE C.1. Graphs of Penoxsulam Residue Stability in Rice Matrices.
(Graphs were copied without alteration from MRID 46449901)









D. CONCLUSION

The study results adequately delineate the stability of penoxsulam residues in/on rice matrices stored frozen for up to 24 months (grain, straw, and immature forage) or ~13 months (bran, hulls, and polished rice). Adequate methods were used for quantitation of residues in/on rice matrices.

E. REFERENCES

DP Barcode: D288152
Subject: Penoxsulam. Petition for the Establishment of Permanent Tolerances for the Use on Rice. Summary of Analytical Chemistry and Residue Data. PP#3F6542
From: W. Cutchin
To: P. Errico/J. Miller
Date: 8/11/04
MRIDs: 45830712-17, 45830719-20, 46267601

F. DOCUMENT TRACKING

RDI: D. Soderberg (11 Dec 2006); D. Drew (11 Dec 2006);
Petition Number(s): 3F6542
DP Barcode(s): D328689
PC Code: 119031

Template Version June 2005



13544

R139128

Chemical: Penoxsulam

PC Code:

119031

HED File Code: 11000 Chemistry Reviews

Memo Date: 12/18/2006

File ID: DPD328689

Accession #: 000-00-0115

HED Records Reference Center

1/31/2007