

US EPA ARCHIVE DOCUMENT

PC 108702

Data Evaluation Report on the terrestrial field dissipation of GF-1442

PMRA Submission 2006-4728

EPA MRID Number

Data Requirement:

PMRA Data Code: 8.3.2

OECD Data Point:

IIA 7.3, IIA 7.3.1, IIA 7.3.2, IIA 7.3.3,
IIIA 9.2.1, IIIA 9.2.2, IIIA 9.2.3

US EPA Guideline:

Subdivision N, Section 164-1

Canada PMRA Guideline:

Trade Memorandum T1-255

PMRA#:

OK
~~0.23 (1.4)~~
~~0.23 (1.4)~~

Primary Reviewer:

Amber McCoy

5 March 2007

PMRA

Ecoregion 9.1, 9.2, 9.3

Secondary Reviewer:

Catherine Evans

5 March 2007

SK2 + MB are relevant

no stability confirmed post
 180 d

Test Material:

GF-1442

End Use Product name:

GF-1442

Concentration of a.i.:

XDE-742:

4.7% (wt/wt)

Formulation type: oil dispersion (OD)

Active ingredient

Common name: XDE-742

Chemical name:

IUPAC: N-(5,7-dimethoxy[1,2,4]triazolo[1,5-a]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide

CAS name: N-(5,7-dimethoxy[1,2,4]triazolo[1,5-a]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide

CAS No: 422556-08-9

Synonyms: TSN102482 (Analytical Standard)

SMILES string: c1(c(ccnc1OC)C(F)(F)F)S(Nc2nn3c(n2)nc(cc3OC)OC)(=O)=O

CITATION: Roberts, D.; Coukell, G.; Schelle, G.; McLean, N. 2006, Terrestrial Field Dissipation of XDE-742 Herbicide and Cloquintocet Safener in Canada. Dow AgroSciences, Regulatory Laboratories - Indianapolis Lab. Study ID 040037. Culy, M. August 2006. PMRA# 1283372



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EXECUTIVE SUMMARY:

Field dissipation of GF-1442 under Canadian prairie field conditions was conducted in bare ground plots at 4 sites: Alberta (AB) (Ecoregion 9.1/9.2 ; sandy clay loam); Saskatchewan 1 (SK1) (Ecoregion 9.1/9.2 , clay); Saskatchewan 1 (SK2) (Ecoregion 9.3, loam); and Manitoba (MB) (Ecoregion 9.2, clay loam). The experiment was carried out in accordance with Canada PMRA guideline T1-255, US EPA guideline Subdivision N, Section 164-1, and in compliance with the OECD-GLP standard.

The end-use product, GF-1442, was surface broadcast sprayed to achieve an XDE-742 (a.i.) application rate of 25 g a.i./ha in three replicate 32 m X 6 m plots at each site. Randomly placed application monitors (15 24-cm diameter filter paper circles per site) found that rates were 25.6, 25.6, 24.8 and 25.3 g a.i./ha in AB, SK1, SK2 and MB respectively. Field spiking was not conducted. All sites were irrigated (May-Sept) to a target of 110% of the 30 year precipitation normal. For the duration of the studies, total precipitation was 99%, 136%, 111% and 121% of normal for AB, SK1, SK2 and MB, respectively.

Soil samples were collected at: 0, 5, 7, 15, 22, 29, 68, 91, 120, 370, 403, 433 and 462 days post-application in AB; 0, 7, 14, 20, 35, 59, 93, 136, 370, 398 and 429 days in SK1; 0, 3, 7, 14, 21, 28, 64, 94, 125, 359, 388 and 422 days in SK2; and 0, 3, 8, 15, 21, 28, 62, 92, 126, 371 and 394 days in MB. Samples were taken to a depth of 90 cm, segmented into 15 cm sections and combined to product 3 composite samples per 15 cm segment. Samples were analyzed for XDE-742 and the transformation products 7-OH-XDE-742, 5-OH-XDE-742 and 6-CL-7-OH-XDE-742.

At AB, the initial concentration was 12.8 g a.i./ha. XDE-742 dissipated steadily and rapidly from the maximum concentration. On day 370 the concentration was 1.3 g a.i./ha and at study termination the concentration was 0.8 g a.i./ha. The residues of XDE-742 were primarily detected in the top 30-cm soil profile. The major transformation products detected at AB were 7-OH-XDE-742 and 6-CL-7-OH-XDE-742. The 7-OH-XDE-742 maximum concentration was 5.3 g a.i./ha (parent equivalents) or 41% of initial parent, observed at 68-DAT in the upper 30-cm soil profile. However, there were detections in the 30-45 cm and 45-60 cm depths on day 5 for which there was no discussion by the study author. At the end of the study, 7-OH-XDE-742 was 1.5 g a.i./kg (parent equivalent) or 12% of the initial parent. The residues of 7-OH-XDE-742 were primarily detected in the top 15-cm soil profile. The 6-CL-7-OH-XDE-742 maximum concentration was 0.8 to 0.9 g a.i./kg or 6 to 7% of initial parent, observed at 68-DAT through 462-DAT, primarily observed in the top 15-cm soil profile.

At SK1, the initial concentration was 25.9 g a.i./ha. XDE-742 dissipated steadily and rapidly from the maximum concentration. On day 35 the parent was last detected at a concentration of 0.3 g a.i./ha and at study termination the concentration was 0 g a.i./ha. The residues of XDE-742 were primarily detected in the top 30-cm soil profile. The major transformation products detected at SK1 were 5-OH-XDE-742, 7-OH-XDE-742 and 6-CL-7-OH-XDE-742. The 5-OH-XDE-742 maximum concentration and only day of detection was 0.5 g a.i./ha (parent equivalents) or 2% of initial parent, observed at 7-DAT in the upper 15-cm soil profile. The 7-OH-XDE-742 maximum concentration was 2.1 g a.i./ha (parent equivalents) or 8% of initial parent, observed at 7-DAT in the upper 15-cm soil profile. The last detection of 7-OH-XDE-742 was 0.3 g a.i./kg (parent equivalent) or 1% of the initial parent on day 35. The residues of 7-OH-XDE-742 were primarily detected in the top 15-cm soil profile. The 6-CL-7-

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OH-XDE-742 only concentration measured was 0.2 g a.i./kg or 1% of initial parent, observed on days 14, 35 and 370 in the top 15-cm soil profile.

At SK2, the initial concentration was 21.7 g a.i./ha. XDE-742 dissipated steadily and rapidly from the maximum concentration. On day 28 the parent was last detected at a concentration 0.2 g a.i./ha. The residues of XDE-742 were primarily detected in the top 15-cm soil profile. The major transformation products detected at SK2 were 7-OH-XDE-742 and 6-CL-7-OH-XDE-742. The 7-OH-XDE-742 maximum concentration was 0.8 g a.i./ha (parent equivalents) or 4% of initial parent, observed on day 14 in the upper 15-cm soil profile. The last detection of 7-OH-XDE-742 was on day 21 at 0.4 g a.i./kg (parent equivalent) or 2% of the initial parent. The residues of 7-OH-XDE-742 were primarily detected in the top 15-cm soil profile. The 6-CL-7-OH-XDE-742 maximum concentration was 0.7 g a.i./kg or 3% of initial parent, observed on day 14. The last detection was on day 28 at 0.1 g a.i./ha or 0.5% of the initial parent. All detections were primarily observed in the top 15-cm soil profile.

At MB, the initial concentration was 16.6 g a.i./ha and then rose to 17.0 g a.i./ha on day 3. XDE-742 dissipated steadily and rapidly from the maximum concentration. At study termination, the parent was detected at a concentration of 0.7 g a.i./ha. The residues of XDE-742 were detected primarily in the 30-cm soil profile, however, on day 15 there were detections in each section down to a depth of 60 cm. The major transformation products detected at MB were 7-OH-XDE-742 and 6-CL-7-OH-XDE-742. The 7-OH-XDE-742 maximum concentration was 1.3 g a.i./ha (parent equivalents) or 8% of initial parent, observed on day 28 in the upper 30-cm soil profile. The last detection of 7-OH-XDE-742 was on day 62 at 0.4 g a.i./kg (parent equivalent) or 2% of the initial parent. The residues of 7-OH-XDE-742 were primarily detected in the top 30-cm soil profile. The transformation product 6-CL-7-OH-XDE-742 was not detected at the MB site.

Under field conditions, XDE-742 was found to have a DT50 ranging from 5 - 29 days and a DT90 ranging from 15 - 97 days. The major transformation product 7-OH-XDE-742 was found to have a DT50 ranging from 3 - 97 days and a DT90 10-321 days. A DT50 could only be calculated for 6-CL-7-OH-XDE-742 in the AB site at 84 days and the DT90 was found to be 279 days.

The maximum carry-over to the next growing season occurred at the AB site with 10% of the parent still present the following spring.

Results Synopsis:

SITE	AB	SK1	SK2	MB
XDE-742				
DT50	29 d	5 d	5 d	13 d
DT90	97 d	15 d	15 d	44 d
7-OH-XDE-742				
DT50	97 d	3 d	6 d	21 d
DT90	321 d	10 d	21 d	70 d
6-CL-7-OH-XDE-742				
DT50	84 d	--	--	--
DT90	279 d	--	--	--

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I. MATERIALS AND METHODS

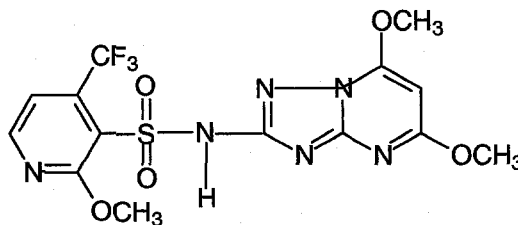
GUIDELINE FOLLOWED: Canada PMRA Trade Memorandum T1-255; U.S. EPA Subdivision N, Sec. 164-1. No deviations from guidelines.

COMPLIANCE: U.S. EPA GLPS and OECD-GLP; signed and dated GLP, Quality Assurance, and Data Confidentiality statements were provided.

A. MATERIALS:

1. Test Material GF-1442

Chemical structure of the active ingredient:



XDE-742 (a.i.)

Description: oil dispersible (OD)

Storage conditions of test chemicals: Test material (GF-1442) was stored under normal ambient conditions and mixed light/dark illumination.

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2. Test site: Description of the location and pesticide use history at each test site.

Table 1: Geographic Location, Site Description and Climatic Data at the Study Sites.

Details		AB Site	SK1 Site	SK2 Site	MB Site
Geographic coordinates	Latitude	53° 75.45'N	52° 16.57'N	51° 49.42'N	49° 57.83'N
	Longitude	113° 19.25'W	106° 18.82'W	106° 26.25'W	98° 16.06'W
	Province	Alberta	Saskatchewan	Saskatchewan	Manitoba
	Country	Canada	Canada	Canada	Canada
	Ecoregion	9.1/9.2 Boreal Plain/Temperate Prairie	9.1/9.2 Boreal Plain/Temperate Prairie	9.3 West-Central semi-arid Prairies	9.2 Temperate Prairies
Slope gradient		<2%	<2%	<2%	≤1%
Depth to groundwater (m)		ca. 97 m	ca. 13.7 m	ca. 13.7 m	ca. 3 m
Distance from weather station used for climatic measurements		Precipitation: 5 m 5 km (Jul-04) Air temp.: 8 km	Precipitation: 5 m 5 km (Jul-04) Air temp.: 20 km	Precipitation: 5 m 7 km (Jul-04) Air temp.: 40 km	Precipitation: 5 m Air temp.: 5 km
Indicate whether the meteorological conditions before starting or during the study were within 30 year normal levels (Yes/No). If no, provide details.		No Jun., Aug-Nov. 2004: very dry Jul., Dec. 2004: 2X normal precip. Overall: 99% of normal precip.	No Sep. 2004: dry Oct. 2004: 3X normal precip. Nov. 2004: dry May, Jun. 2005: dry Aug. 2005: 3X normal precip. Overall: 136% of normal precip.	No Sep., Nov. 2004: very dry Mar. 2005: 2X normal precip. Overall: 111% of normal precip.	No Sep., Nov. 2004: dry Feb.-Apr. 2005: dry Jul. 2005: 2X normal precip. Overall: 99% of normal precip.
Other details, if any		None	None	None	None

Table 2: Site Usage and Management History for the Previous Three Years

Use	Year	AB Site	SK1 Site	SK2 Site	MB Site
Crops grown	Previous year	Potato	Wheat	Canola	Barley
	2 years previous	Barley	Wheat	Wheat	Barley
	3 years previous	Wheat	Wheat	Alfalfa	Fallow
Pesticides used	Previous year	Metribuzin Rimsulfuron Diquat	Lambda-cyhalothrin Bromoxynil MCPA ester	Glyphosate	Fenoxaprop-p-ethyl Bromoxynil MCPA ester
	2 years previous	Tralkoxydim Bromoxynil MCPA ester	Dicamba 2,4-D amine Mecoprop	Clodinafop-propargyl Thifen-sulfuron methyl	Thifen-sulfuron methyl Tribenuron methyl propanil
	3 years previous	Clodinafop-propargyl Thifensulfuron methyl	n/d	n/d	Glyphosate
Fertilizers used	Previous year	n/d	n/d	n/d	n/d
	2 years previous	n/d	n/d	n/d	n/d
	3 years previous	n/d	n/d	n/d	n/d
Cultivation methods, if provided	Previous year	n/d	n/d	n/d	n/d
	2 years previous	n/d	n/d	n/d	n/d
	3 years previous	n/d	n/d	n/d	n/d

n/d – no data available

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Table 3: Properties of the Soil from AB Site.

Depth	Bulk Density ^c	Organic Matter	Soil pH	CEC ^a	Water Content at		Particle Distribution			Texture Class ^b
					1/3 Bar	15 Bar	Sand	Silt	Clay	
(cm)	(g/cm ³)	(%)		(meq/100g)	(%)	(%)	(%)	(%)	(%)	
0-15	0.87	6.1	5.8	20.1	21.5	14.1	55	22	23	SCL
15-30	1.07	3.6	6.3	18.7	17.8	10.9	49	30	21	L
30-45	1.10	0.9	7.6	17.0	15.0	9.1	57	20	23	SCL
45-60	1.08	0.6	8.0	14.8	13.2	8.3	63	18	19	SL
60-75	1.05	0.7	8.0	14.7	14.1	8.7	63	16	21	SCL
75-90	1.06	0.7	7.9	16.5	15.7	10.2	57	19	24	SCL
Taxonomic classification:										
Canadian: chernozemic order, Black great group, Eluviated Black subgroup										
US: Udic Boroll subgroups										

a Cation exchange capacity

b Per USDA. L: loam, S: sand, C: clay, Si: silt

c Undisturbed soil core

Table 4: Properties of the Soil from SK1 Site.

Depth	Bulk Density ^c	Organic Matter	Soil pH	CEC ^a	Water Content at		Particle Distribution			Texture Class ^b
					1/3 Bar	15 Bar	Sand	Silt	Clay	
(cm)	(g/cm ³)	(%)		(meq/100g)	(%)	(%)	(%)	(%)	(%)	
0-15	1.07	3.5	8.0	30.9	33.3	21.9	23	27	50	C
15-30	0.97	2.2	8.2	33.3	32.8	21.8	23	25	52	C
30-45	0.91	1.9	8.4	29.2	30.8	21.4	23	27	50	C
45-60	0.94	1.6	8.4	32.2	30.9	21.1	25	27	48	C
60-75	0.98	1.4	8.2	37.4	29.9	20.1	26	24	50	C
75-90	1.02	0.9	8.2	61.6	30.1	19.7	27	19	54	C
Taxonomic classification:										
Canadian: chernozemic order, Dark Brown great group, Orthic Dark Brown subgroup										
US: Typic Boroll subgroups										

a Cation exchange capacity

b Per USDA. L: loam, S: sand, C: clay, Si: silt

c Undisturbed soil core

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Table 5: Properties of the Soil from SK2 Site.

Depth	Bulk Density ^c	Organic Matter	Soil pH	CEC ^a	Water Content at		Particle Distribution			Texture Class ^b
					1/3 Bar	15 Bar	Sand	Silt	Clay	
(cm)	(g/cm3)	(%)		(meq/100g)	(%)	(%)	(%)	(%)	(%)	
0-15	1.01	3.8	7.2	18.8	23.1	12.4	42	38	20	L
15-30	1.02	1.7	6.9	18.7	20.2	10.9	44	33	23	L
30-45	1.02	1.5	8.1	17.1	21.9	11.6	26	50	24	L
45-60	1.08	1.1	8.6	16.1	21.9	11.4	30	46	24	L
60-75	1.32	0.9	8.7	17.3	20.7	10.7	34	40	26	L
75-90	1.35	0.9	8.8	15.9	17.6	8.6	50	24	26	SCL
Taxonomic classification:										
Canadian: chernozemic order, Dark Brown great group, Orthic Dark Brown Black subgroup										
US: Typic Boroll subgroups										

a Cation exchange capacity

b Per USDA. L: loam, S: sand, C: clay, Si: silt

c Undisturbed soil core

Table 6: Properties of the Soil from MB Site.

Depth	Bulk Density ^c	Organic Matter	Soil pH	CEC ^a	Water Content at		Particle Distribution			Texture Class ^b
					1/3 Bar	15 Bar	Sand	Silt	Clay	
(cm)	(g/cm3)	(%)		(meq/100g)	(%)	(%)	(%)	(%)	(%)	
0-15	0.96	8.0	7.7	36.5	41.7	28.2	26	42	32	CL
15-30	0.79	6.6	8.0	31.2	41.9	28.4	24	48	28	CL
30-45	0.80	5.1	8.3	32.9	41.7	27.7	22	47	31	CL
45-60	0.89	3.2	8.5	29.0	36.1	26.7	22	42	36	CL
60-75	0.96	1.5	8.6	22.1	32.9	21.9	25	41	34	CL
75-90	—	0.8	8.6	19.7	26.5	12.6	26	50	24	L
Taxonomic classification:										
Canadian: chernozemic order, Black great group, Gleyed Carbonated Rego Black subgroup										
US: Udic Boroll subgroups										

a Cation exchange capacity

b Per USDA. L: loam, S: sand, C: clay, Si: silt

c Undisturbed soil core

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B. EXPERIMENTAL DESIGN:

1. Experimental design:

Table 7: Experimental Design.

Details		AB Site	SK1 Site	SK2 Site	MB Site
Duration of study		462 days	370 days	359 days	126 days
Uncropped (bare) or cropped		Bare	Bare	Bare	Bare
Control used (Yes/No)		Yes	Yes	Yes	Yes
No. of replications	Controls	1	1	1	1
	Treatments	3	3	3	3
Plot size (L x W m)	Control	8 x 6 m	8 x 6 m	8 x 6 m	8 x 6 m
	Treatment	(32 x 6 m) x 3	(32 x 6 m) x 3	(32 x 6 m) x 3	(32 x 6 m) x 3
Distance between control plot and treated plot		> 15 m	> 15 m	> 15 m	> 15 m
Distance between treated plots		2 m	2 m	2 m	2 m
Application rate(s) used (g a.i./ha)		25.6 g a.i./ha	25.6 g a.i./ha	25.6 g a.i./ha	25.6 g a.i./ha
Was the maximum label rate per ha used in study? (Yes/No)		Yes	Yes	Yes	Yes
Number of applications		1	1	1	1
Application Date(s) (dd mm yyyy)		27 May 2004	8 June 2004	22 June 2004	29 June 2004
For multiple applications, application rate at Day 0 and at each application time (mg a.i./kg soil)		n/a	n/a	n/a	n/a
Application method (e.g., spraying, broadcast etc.)		Ground broadcast	Ground broadcast	Ground broadcast	Ground broadcast
Type of spray equipment, if used		Tractor boom	Tractor boom	Tractor boom	Tractor boom
Total volume of spray solution applied/plot OR total amount broadcasted/plot		288.09 L/ha	287.02 L/ha	278.02 L/ha	286.62 L/ha
Identification and volume of carrier (e.g., water), if used		Water 24.9 L	Water 24.8 L	Water 24.8 L	Water 27.0 L
Name and concentration of co-solvents, adjuvants and/or surfactants, if used		Assist adjuvant 0.8% (v/v)	Assist adjuvant 0.8% (v/v)	Assist adjuvant 0.8% (v/v)	Assist adjuvant 0.8% (v/v)

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Indicate whether the following monthly reports were submitted: Average minimum and maximum precipitation Average minimum and maximum air temperature Average minimum and maximum soil temperature Average annual frost-free periods		Total precipitation (including snow and irrigation) min., max and ave. air temperature	Total precipitation (including snow and irrigation) min., max and ave. air temperature soil temperature (10 cm)	Total precipitation (including snow and irrigation) min., max and ave. air temperature soil temperature (10 cm)	Total precipitation (including snow and irrigation) min., max and ave. air temperature soil temperature (7.6 cm) (10 June - 31 July 2005)
Indicate whether the Pan evaporation data were submitted		No	No	No	No
Meteorological conditions during application	Cloud cover	No	No	No	No
	Temperature (°C)	16°C	13.5°C	12.8°C	22°C
	Humidity	36%	48%	77%	35%
	Sunlight (hr)	No	No	No	No
Pesticides used during study: name of product/a.i concentration: amount applied: application method:		Reglone or glyphosate, 5 appl.s, ground broadcast	Glyphosate, 4 appl.s, ground broadcast	Reglone or glyphosate, 4 appl.s, ground broadcast	Glyphosate, 3 appl.s, ground broadcast
Supplemental irrigation used (Yes/No) If yes, provide the following details: No. of irrigation: Interval between irrigation: Amount of water added each time: Method of irrigation:		Yes 5 2/month as needed ave. 14 mm semi-permanent (hand-line sprinkler)	Yes 10 2/month as needed ave. 10 mm semi-permanent (hand-line sprinkler)	Yes 4 2/month as needed ave. 15 mm semi-permanent (hand-line sprinkler)	Yes 8 2/month as needed ave. 12 mm semi-permanent (hand-line sprinkler)
Indicate whether water received through rainfall + irrigation equals the 30 year average rainfall (Yes/No)		Yes (99%)	Yes (136%)	Yes (111%)	Yes (121%)
Were the application concentrations verified? (Briefly describe in Section 2*, if used)		Yes	Yes	Yes	Yes
Were field spikes used? (Briefly describe in Section 3†, if used)		No	No	No	No
Good agricultural practices followed (Yes or No)		Yes	Yes	Yes	Yes

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Indicate if any abnormal climatic events occurred during the study (e.g., drought, heavy rainfall, flooding, storm etc.)	Jun., Aug-Nov. 2004: very dry Jul., Dec. 2004: 2X normal precip.	Sep. 2004: dry Oct. 2004: 3X normal precip. Nov. 2004: dry May, Jun. 2005: dry Aug. 2005: 3X normal precip.	Sep., Nov. 2004: very dry Mar. 2005: 2X normal precip.	Sep., Nov. 2004: dry Feb.-Apr. 2005: dry Jul. 2005: 2X normal precip.
If cropped plots are used, provide the following details: Plant - Common name/variety: Details of planting: Crop maintenance (e.g., fertilizers used):	n/a	n/a	n/a	n/a
Volatilization included in the study (Yes/No) (if included, describe in Section 4 ^s)	No	No	No	No
Leaching included in the study (Yes/No) (if included, describe in Section 5 ⁱ)	Yes	Yes	Yes	Yes
Run off included in the study (Yes/No) (if included, describe in Section 6 ^y)	No	No	No	No

2. Application Verification:

For each replicate plot, 5 randomly located 24-cm diameter filter paper disks were used.

3. Field Spiking (not applicable)

4. Volatilization (not applicable)

5. Leaching: Determined for each of three replicate plots by soil residue analysis of one composite sample (each comprised of five soil cores) per 15-cm layer (through 90 cm), per replicate plot.

6. Run-off (not applicable)

7. Supplementary Study: In a 2004 spray tank solution study (3), the test substance was mixed in a water-based spray solution (containing Assist* adjuvant at 1% v/v) at a range of solution concentrations (0.03-0.6 mg/mL XDE-742; 0.09-1.8 mg/mL cloquintocet mexyl) to encompass the concentration used in this study. At 4 and 24 hours after mixing, the test substance was determined to be stable and uniform throughout the spray tank solution.

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Table 8. Sampling:

Details	AB Site	SK1 Site	SK2 Site	MB Site
Method of sampling (random or systematic)	Random	Random	Random	Random
Sampling intervals	0, 5, 7, 15, 22, 29, 68, 91, 120, 370, 403, 433, 462 days	0, 7, 14, 20, 35, 59, 93, 136, 370 days	0, 3, 7, 14, 21, 28, 64, 94, 125, 359 days	0, 3, 8, 15, 21, 28, 62, 92, 126 days
Method of soil collection (e.g., cores)	Cores	Cores	Cores	Cores
Sampling depth	90 cm	90 cm	90 cm	90 cm
Number of cores collected per plot	5	5	5	5
Number of segments per core	6	6	6	6
Length of soil segments	15 cm	15 cm	15 cm	15 cm
Core diameter (Provide details if more than one width)	0-15 cm: 5.5 cm 15-90 cm: 3.0 cm	0-15 cm: 5.5 cm 15-90 cm: 3.0 cm	0-15 cm: 5.5 cm 15-90 cm: 3.0 cm	0-15 cm: 5.5 cm 15-90 cm: 3.0 cm
Method of sample processing, if any	Like depth core segments were combined, producing one composite sample per replicate plot. Ground and mixed frozen.	Like depth core segments were combined, producing one composite sample per replicate plot. Ground and mixed frozen.	Like depth core segments were combined, producing one composite sample per replicate plot. Ground and mixed frozen.	Like depth core segments were combined, producing one composite sample per replicate plot. Ground and mixed frozen.
Storage conditions	Freezer within 15 min. of sampling	Freezer within 15 min. of sampling	Freezer within 15 min. of sampling	Freezer within 15 min. of sampling
Storage length (days)	XDE-742: 588 days CGC: 416 days	XDE-742: 588 days CGC: 416 days	XDE-742: 588 days CGC: 416 days	XDE-742: 588 days CGC: 416 days

*No stability confirmed past 180 d!
(270 d for CGC)*

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8. Analytical Procedures: Soil samples were analyzed for XDE-742 and its metabolites by a validated method using liquid chromatography with tandem mass spectrometry (LC/MS/MS) (1).

Number of soil samples analyzed per treatment or composite sample:

155 samples total (per analyte, per test site)

Number of plant samples analyzed per treatment or composite sample:
not applicable

Extraction, clean up and concentration of soil samples:

Residues of XDE-742 and its metabolites were extracted from the soil by sonicating with 1 N hydrochloric acid and shaking after the addition of methanol to produce a methanol/1N hydrochloric acid solution (90:10). A mixed XDE-742 and metabolite stable isotope internal standard solution was added to the extraction solvent and an aliquot of the extract was evaporated to dryness. The sample was reconstituted in 0.1 N hydrochloric acid and purified using a polymeric 96-well solid phase extraction (SPE) plate. The SPE plate was washed with a water/methanol solution (75:25) and eluted with an acetonitrile/methanol solution (50:50). The eluate was evaporated to dryness and the residues were reconstituted in a water/methanol solution (90:10) containing 2 mM ammonium acetate. The purified extract was analyzed by high performance liquid chromatography with positive-ion electrospray (ESI) tandem mass spectrometry (LC/MS/MS).

GRM 05.05

LOD: 0.3

LOQ: 1.0
(ng/g)

Identification and quantification of parent and transformation compounds:

The samples were analyzed by liquid chromatography with tandem mass spectrometry (LC/MS/MS). Sample analysis was performed using a Phenomenex Synergi, 4 μ , Hydro RP column installed in an Spark Holland Symbiosis Pharma coupled to a MDS Sciex model API 4000 tandem mass spectrometer. During analysis the following MS/MS transitions were monitored:

Compound	Ion m/z		Time, ms	Collision Energy,
	Q1	Q3		V
XDE-742 Quant	435	195	75	35
XDE-742 IS	438	198	75	35
5-OH-XDE-742 Quant	421	181	75	31
5-OH-XDE-742 IS	425	185	75	31
7-OH-XDE-742 Quant	421	181	75	31
7-OH-XDE-742 IS	425	185	75	31

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The efficiency of the analytical method was determined at the time of analysis of each set of samples by fortifying aliquots of the appropriate control matrix with analyte and analyzing according to the method. An unfortified control matrix, reagent blank, and control matrix fortified at the limit of detection (LOD) were included in each set as well. The LD recovery samples were analyzed only to demonstrate observable peaks at the LOD level. Therefore, the percent recovery of analyte was not calculated for these samples. Fortified recoveries were analyzed over a range of 0.001 to 0.1 µg/g.

Detection limits (LOD, LOQ) for the parent XDE-742 and transformation compounds in soil:

LOD: 0.0003 µg/g

LOQ: 0.001 µg/g

II. RESULTS AND DISCUSSION

1. APPLICATION MONITORS:

The average recoveries for parent XDE-742 (a.i.) in the field application monitors were 87.6% (AB), 79.7% (SK1), 77.6% (SK2) and 114% (MB) based on the field application calculations.

2. MASS ACCOUNTING: Because this study was conducted using non-radio-labelled material, calculation of total mass accounting with time was not possible.

3. PARENT XDE-742 (a.i.):

0-DAT at 12.8

At AB, the maximum starting concentration of parent XDE-742 (a.i.) in soil core samples was observed on 5-DAT at 14.0 g a.i./ha, representing 55 % of theoretically applied 25.6 g a.i./ha, or 70% of the proposed maximum seasonal application rate of 20 g a.i./ha. Parent XDE-742 (a.i.) dissipated steadily and rapidly from an initial concentration (0-DAT) of 12.8 g a.i./ha or 50% of applied parent XDE-742 (a.i.), or 64% of the proposed, maximum seasonal use rate of 20 g a.i./ha. The residues of XDE-742 were primarily detected in the top 30-cm soil profile. At the end of the 462-day study period, the total carryover of residues of XDE-742 (a.i.) was 6% of applied a.i.. *should be 540 d*

At SK1, the maximum starting concentration of parent XDE-742 (a.i.) in soil core samples was observed on 0-DAT at 25.9 g a.i./ha, representing 101 % of theoretically applied 25.6 g a.i./ha, or 130% of the proposed maximum seasonal application rate of 20 g a.i./ha. The residues of XDE-742 were primarily detected in the top 30-cm soil profile. At the end of the 370-day study period, the total carryover of residues of XDE-742 was 0.0% of applied a.i..

At SK2, the maximum starting concentration of parent XDE-742 (a.i.) in soil core samples was observed on 0-DAT at 21.7 g a.i./ha, representing 88% of theoretically applied 24.8 g a.i./ha, or 109% of the proposed maximum seasonal application rate of 20 g a.i./ha. The residues of XDE-742 were primarily detected in the top 15-cm soil profile. At the end of the 359-day study period, the total carryover of residues of XDE-742 (a.i.) was 0.0% of applied a.i..

At MB, the maximum starting concentration of parent XDE-742 (a.i.) in soil core samples was observed on 0-DAT at 16.6 g a.i./ha, representing 66% of theoretically applied 25.3 g a.i./ha, or 83%

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of the proposed maximum seasonal application rate of 20 g a.i./ha. The residues of XDE-742 were primarily detected in the top 30-cm soil profile, with isolated, trace detections ($\leq$ LOQ) in one replicate plot at 30-60 cm on 15-DAT. At the end of the 126-day study period, the total carryover of residues of XDE-742 (a.i.) was 4% of applied a.i..

The field half-life or 50% dissipation time (DT_{50}) and DT_{90} of parent XDE-742 (a.i.) in soil under terrestrial field conditions using LC/MS/MS (1) was:

AB site:	XDE-742 (a.i.)	Half-life/ DT_{50} = 29 days	DT_{90} = 97 days
SK1 site:	XDE-742 (a.i.)	Half-life/ DT_{50} = 5 days	DT_{90} = 15 days
SK2 site:	XDE-742 (a.i.)	Half-life/ DT_{50} = 5 days	DT_{90} = 15 days
MB site:	XDE-742 (a.i.)	Half-life/ DT_{50} = 13 days	DT_{90} = 44 days

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Table 9. Concentration of XDE-742 products expressed as g/ha and transformation (percent of a.i. equivalent) at AB.

Compound	Soil depth (cm)	Sampling times (days, hours, or other time period)												
		0*	5	7	15	22	29	68	91	120	370	403	433	462
XDE-742 ^a	0-15	12.8	12.6	10.3	8.7	7.2	6.3	2.9	2.2	1.7	1.3	0.9	0.8	0.8
	15-30		0.2				0.2				0.2			
	% Initial	100%	100%	81%	68%	51%	51%	23%	17%	11%	12%	7%	6%	6%
7-OH-XDE ^a	0-15		1.1	1.5	2.8	3.5	2.1	5.2	3.9	3.1	3.0	2.4	2.0	1.4
	15-30							0.5			0.6			
	% Initial	0%	9%	13%	22%	29%	34%	45%	31%	25%	28%	20%	16%	12%
6-CL-7-OH-XDE ^a	0-15						0.3	0.8	0.8	0.7	1.0	0.9	0.8	0.8
	15-30													
	30-45		0.4											
	45-60		0.5											
	% Initial		7%				3%	6%	6%	5%	6%	7%	6%	6%

^aFor each depth, the mean of three replicates (n=3)

^b As percent of initial parent concentration in upper 15 cm. Transformation products converted to parent equivalents.

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Table 10. Concentration of XDE-742 products expressed as g/ha and transformation (percent of a.i. equivalent) at SK1.

Compound	Soil depth (cm)	Sampling times (days, hours, or other time period)								
		0*	7	14	20	35	59	93	136	370
XDE-742	0-15	25.9	7.5	2.2	1.2	0.3				
	15-30		1.6	0.3						
	% Initial	100%	35%	9%	5%	1%				
5-OH-XDE	0-15		0.5							
	% Initial		2%							
7-OH-XDE	0-15		2.1	1.1	0.5	0.3				
	15-30									
	% Initial		8%	4%	2%	1%				
6-CL-7-OH-XDE	0-15			0.2		0.2				0.2
	% Initial			1%		1%				1%

^a For each depth, the mean of three replicates (n=3)

^b As percent of initial parent concentration in upper 15 cm. Transformation products converted to parent equivalents.

Table 11. Concentration of XDE-742 products expressed as g/ha and transformation (percent of a.i. equivalent) at SK2.

Compound	Soil depth (cm)	Sampling times (days, hours, or other time period)									
		0*	3	7	14	21	28	64	94	125	359
XDE-742	0-15	21.7	13.0	7.9	2.6	1.0	0.2				
	% Initial	100%	60%	36%	12%	5%	1%				
7-OH-XDE	0-15			0.7	0.8	0.4					
	% Initial			3%	4%	2%					
6-CL-7-OH-XDE	0-15				0.7	0.5	0.1				
	% Initial				3%	2%	1%				

^a For each depth, the mean of three replicates (n=3)

^b As percent of initial parent concentration in upper 15 cm. Transformation products converted to parent equivalents.

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Table 12. Concentration of XDE-742 products expressed as g/ha and transformation (percent of a.i. equivalent) at MB.

Compound	Soil depth (cm)	Sampling times (days, hours, or other time period)								
		0*	7	14	20	35	59	93	136	370
XDE-742	0-15	16.6	17.0	13.3	6.3	4.3	4.5	1.3	0.8	0.7
	15-30				0.5	0.2	0.2			
	30-45				0.3					
	45-60				0.2					
	% Initial		100%	78%	43%	26%	27%	8%	5%	4%
7-OH-XDE	0-15			0.2	1.0	1.0	1.3	0.4		
	15-30						0.2			
	% Initial			1%	6%	6%	9%	2%		

^a For each depth, the mean of three replicates (n=3)

^b As percent of initial parent concentration in upper 15 cm. Transformation products converted to parent equivalents

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4. TRANSFORMATION PRODUCTS OF XDE-742 (a.i.):

At AB, the major transformation products detected were 7-OH-XDE-742 and 6-CL-7-OH-XDE-742. The 7-OH-XDE-742 maximum concentration was 41% of initial a.i. (parent XDE-742), observed at 68-DAT in the upper 30-cm soil profile. At the end of the study, 7-OH-XDE-742 was 12% of initial a.i.. The residues of 7-OH-XDE-742 were primarily detected in the top 15-cm soil profile. The 6-CL-7-OH-XDE-742 maximum concentration was 6 to 7% of initial a.i., observed at 68-DAT through 462-DAT, primarily observed in the top 15-cm soil profile. At the end of the study, 6-CL-7-OH-XDE-742 was 6% of initial a.i.. The residues of 6-CL-7-OH-XDE-742 were primarily detected in the top 15-cm soil profile. However, there were detection on day 5 in the 30-45 cm and the 45-60 cm samples at 1-2% of the initial a.i..

At SK1, the major transformation products detected were 7-OH-XDE-742 and 6-CL-7-OH-XDE-742. The 7-OH-XDE-742 maximum concentration was 2% of initial a.i. (parent XDE-742), observed at 7-DAT in the upper 15-cm soil profile. At the end of the study, 7-OH-XDE-742 was 0% of initial a.i.. The residues of 7-OH-XDE-742 were primarily detected in the top 15-cm soil profile. The 6-CL-7-OH-XDE-742 maximum concentration was 1% of initial a.i., observed at 14, 35 and 370-DAT in the top 15-cm soil profile. At the end of the study, 6-CL-7-OH-XDE-742 was 0% of initial a.i.. The residues of 6-CL-7-OH-XDE-742 were primarily detected in the top 15-cm soil profile. The only detection of 5-OH-XDE occurred 7DAT at 2% of the initial a.i. in the top 15-cm of the soil profile.

At SK2, the major transformation products detected were 7-OH-XDE-742 and 6-CL-7-OH-XDE-742. The 7-OH-XDE-742 maximum concentration was 4% of initial a.i. (parent XDE-742), observed at 14-DAT in the upper 15-cm soil profile. At the end of the study, 7-OH-XDE-742 was at 0% of initial a.i.. The residues of 7-OH-XDE-742 were primarily detected in the top 15-cm soil profile. The 6-CL-7-OH-XDE-742 maximum concentration was 3% of initial a.i., observed at 14-DAT in the top 15-cm soil profile. At the end of the study, 6-CL-7-OH-XDE-742 was at 0% of initial a.i.. The residues of 6-CL-7-OH-XDE-742 were primarily detected in the top 15-cm soil profile.

At MB, the major transformation product detected was 7-OH-XDE-742. The 6-CL-7-OH-XDE-742 degradate was not detected in any sample. The 7-OH-XDE-742 maximum concentration was 8% of initial a.i. (parent XDE-742), observed at 28-DAT in the upper 30-cm soil profile. At the end of the study, 7-OH-XDE-742 was at 0% of initial a.i.. The residues of 7-OH-XDE-742 were primarily detected in the top 30-cm soil profile.

The field half-life or 50% dissipation time (DT_{50}) and DT_{90} of 7-OH-XDE-742 (a.i.) in soil under terrestrial field conditions using LC/MS/MS (1) was:

AB site: 7-OH-XDE-742 Half-life/ DT_{50} = 97 days DT_{90} = 321 days

SK1 site: 7-OH-XDE-742 Half-life/ DT_{50} = 3 days DT_{90} = 10 days

SK2 site: 7-OH-XDE-742 Half-life/ DT_{50} = 6 days DT_{90} = 21 days

MB site: 7-OH-XDE-742 Half-life/ DT_{50} = 21 days DT_{90} = 70 days

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The field half-life or 50% dissipation time (DT₅₀) and DT₉₀ of 6-CL-7-OH-XDE-742 (a.i.) in soil under terrestrial field conditions using LC/MS/MS (1) was calculated for the AB site, but not for SK1 and SK2, where detections were isolated and/or trace-level (between LOQ and LOD). At MB, 6-CL-7-OH-XDE-742 detections were not observed.

AB site: 6-CL-7-OH-XDE-742 Half-life/DT₅₀ = 84 days DT₉₀ = 279 days

Table 13: Chemical Names and CAS Numbers for the Transformation Products of XDE-742.

Applicant's Code Name	CAS Number	IUPAC Chemical Name(s)	Chemical Formula	Molecular Weight (g/mol)	SMILES String
XDE-742	422556-08-9	N-(5,7-dimethoxy[1,2,4]triazolo[1,5-a]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide	C ₁₄ H ₁₃ F ₃ N ₆ O ₅ S	434.4	<chem>c1(c(ccnc1OC)C(F)(F)F)S(Nc2nn3c(n2)nc(cc3OC)OC)(=O)=O</chem>
5-OH-XDE-742a	99607-70-2	N-(5-hydroxy-7-methoxy[1,2,4]triazolo[1,5-a]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide	C ₁₃ H ₁₁ F ₃ N ₆ O ₅ S	420.3	<chem>c1(c(ccnc1OC)C(F)(F)F)S(Nc2nn3c(n2)nc(cc3OC)O)(=O)=O</chem>
7-OH-XDE-742	not available	N-(7-hydroxy-5-methoxy[1,2,4]triazolo[1,5-a]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)-3-pyridinesulfonamide	C ₁₃ H ₁₁ F ₃ N ₆ O ₅ S	420.3	<chem>c1(c(ccnc1OC)C(F)(F)F)S(Nc2nn3c(n2)nc(cc3O)OC)(=O)=O</chem>
6-CL-7-OH-XDE-742	not available	N-(6-chloro-7-hydroxy-5-methoxy[1,2,4]triazolo[1,5-a]pyrimidin-2-yl)-2-methoxy-4-(trifluoromethyl)pyridine-3-sulfonamide	C ₁₃ H ₁₀ ClF ₃ N ₆ O ₅ S	454.8	<chem>c1(c(ccnc1OC)C(F)(F)F)S(Nc2nn3c(n2)nc(c(c3O)Cl)OC)(=O)=O</chem>

^a The 5-OH-XDE-742 transformation product was observed at only one test site (SK1) on one sampling event (7-DAT) at the limit of analytical detection (0.0003 µg/g)

5. EXTRACTABLE AND NON-EXTRACTABLE RESIDUES: non-extractable residues were not measured.

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Table 14: Dissipation Routes of XDE-742 (a.i.) under Field Conditions.

Route of dissipation	AB: % of applied amount (to the start of the next growing season (370 days))	SK1: % of applied amount (at the end of study period)	SK2: % of applied amount (at the end of study period)	MB: % of applied amount (at the end of study period)
Accumulation (residues) in soil/ carry over	10%	0%	0%	4%
Transformation (% of transformation products)	29%	1%	0%	0%
Leaching, if measured	Upper 30 cm layer, with detections of the transformation product 6-CL-7- OH-XDE to a depth of 60 cm.	Upper 30 cm layer	Upper 30 cm layer	Upper 30 cm layer, with detections of the parent down to 60 cm
Total	39%	1%	0%	4%

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6. VOLATILIZATION: not determined.

7. PLANT UPTAKE: not determined.

8. LEACHING: At AB, XDE-742 (a.i.) and degradates were not detected below 30 cm at any sampling event except isolated, trace detections (<1% of applied XDE-742 a.i.) of 6-CL-7-OH-XDE-742 in the 30-60-cm layer at 5-DAT. At SK1, detections of XDE-742 and degradates were limited to the upper 30-cm of the soil profile. At SK2, detections of XDE-742 and degradates were limited to the upper 15-cm of the soil profile. At MB, detections of XDE-742 and degradates were limited to the upper 30-cm of the soil profile, except for trace detections at 0.2 to 0.3 g/ha (1 to 2 % of initial XDE-742 a.i.) observed in one of three replicate composite samples at 30-60 cm on 15-DAT.

9. RUN OFF: not determined.

10. RESIDUE CARRYOVER:

At **AB**, the DT_{90} value for XDE-742 (a.i.) was 97 days. After 120 days, 11% of initial XDE-742 (a.i.) was detected with 10% detected at the beginning of the following growing season, 370-DAT. At the beginning of the next growing season (370-DAT), carryover of the transformation products, 7-OH-XDE-742 and 6CL-7-OH-XDE-742 was 23% and 3% of initial XDE-742 (a.i.), respectively.

At **SK1**, the DT_{90} value for XDE-742 (a.i.) was 15 days. After 136 days, 0% of initial XDE-742 (a.i.) was detected with no potential for carryover into the following growing season. At the end of the study (370-DAT), carryover of the transformation products, 7-OH-XDE-742 and 6-CL-7OH-XDE-742 was 0% and 1% of initial XDE-742 (a.i.), respectively.

At **SK2**, the DT_{90} value for XDE-742 (a.i.) was 15 days. After 125 days, 0% of initial XDE-742 (a.i.) was detected with no potential of carryover into the following growing season. At the end of the study (359-DAT), carryover of the transformation products, 7-OH-XDE-742 and 6-CL-7OH-XDE-742 was 0% and 0% of initial XDE-742 (a.i.), respectively.

At **MB**, the DT_{90} value for XDE-742 (a.i.) was 44 days. After 126 days, 4% of initial XDE-742 (a.i.) was detected. At the end of the study (126-DAT), carryover of the transformation products, 7-OH-XDE-742 and 6-CL-7OH-XDE-742 was 0% and 0% of initial XDE-742 (a.i.), respectively.

11. SUPPLEMENTARY STUDY RESULTS: Samples for this field study were analyzed for XDE-742 and degradates after storage in frozen condition for a maximum of 588 days (ca. 20 months). In a separate, ongoing study (4), XDE-742 and degradates were stable in soil under frozen conditions for 6 months.

III. STUDY DEFICIENCIES: No deficiencies were noted.

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IV. REVIEWER'S COMMENTS:

In general, the EAD reviewer agreed with the findings presented by the applicant. A copy of the applicant DER has been edited by the EAD reviewer (comments in yellow) to reflect the information required by the PMRA.

1. Cloquintocet-methyl is registered for use in Canada, the US and is listed as registered in Australia on the PAN site, therefore, this portion of the study was not included in the PMRA review. However, based on the review of the XDE-742 portion of the study, the results are expected to be acceptable.

2. The Ecoregions provided were incorrect and corrections were made.

3. Detections between the LOD and LOQ were changed to $(LOQ+LOD/2)$ for all calculations, rather than using the values presented. LOD and LOQ values were converted from $\mu\text{g/g}$ to g/ha based on a 15 cm soil depth and the reported bulk density for the top 15 cm of soil for calculation purposes.

4. The applicant reported the percent of parent and transformation product in the soil based on nominal applied parent. However, this is not the method used by the PMRA. Rather, the initial (or maximum parent measured) is used to calculate the percent of transformation product in the soil (based on parent equivalent concentrations).

5. Concentrations for each sampling depth were added and then the percentage recovered was determined based on nominal parent. The concentrations can not be added, therefore, percentage recovery values have been re-calculated for each sampling depth separately based on initial parent concentration.

6. Parent equivalents were calculated to be 1.03 for 7-OH-XDE-742 and 0.96 for 6-CL-7-OH-XDE-742. No value was determined for 5-OH-XDE-742 due to the very low detections.

7. The PMRA does not use Model Maker, however, DT50 calculations were recalculated using SygmaPlot and values were similar. Therefore, applicant values were accepted.

8. Carry-over was determined based on the concentrations in the soil at study termination. The PMRA determines carry-over as what is present at the start of the next growing season, therefore, some of these numbers changed. The trigger of 30% carry-over was not met.

9. In the supplementary study, XDE-742 and its transformation products were found to be stable in frozen soil samples for six months. However, study samples were stored as long as 588 days. Average sample storage times were 376, 419, 402 and 428 days for AB, SK1, SK2 and MB, respectively. Therefore, the most conservative value should be used in any risk assessment.

V. REFERENCES

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