

US EPA ARCHIVE DOCUMENT



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This DER was originally prepared under contract by Dynamac Corporation (1910 Sedwick Rd., Building 100, Suite B; Durham, NC 27713; submitted 2/20/2005). This DER has been reviewed by the HED and revised to reflect current OPP policies.

STUDY REPORT:

45322103 Veal, P. and Spillner, C. (1997) Residue Levels on Sugar Beets Planted as a Rotational Crop Following Corn From Trials Carried Out in the United States of America During 1995-1996: Lab Project Number: ACET-95-CR-03: RJ2263B. Unpublished study prepared by Monsanto Co. and American Agricultural Services, Inc. 80 p.

EXECUTIVE SUMMARY:

Twelve rotational crop field trials on sugar beets were conducted throughout the U.S. during 1995. At each site, acetochlor (6.4 lb/gal EC) was applied to a primary crop of corn (field or sweet) as a preplant incorporated or preemergence broadcast application at 3.0 lb ai/A. The corn was grown and harvested following common agricultural practices. A rotational crop of sugar beets was planted at 296-366 days after treatment (DAT) at eleven sites and at 154 DAT at one site. Single control and duplicate treated samples of sugar beet roots and tops were harvested at commercial maturity, 104-335 days after planting (460-522 DAT). Samples were stored frozen for up to 5.3 months prior to analysis, an interval supported by available storage stability data.

A GC/nitrogen-phosphorus detection (GC/NPD) method (RAM 244/02) was used to determine residues of acetochlor *per se*. The registrant has not demonstrated that this method can extract field weathered residues. Therefore data on residues of acetochlor *per se* from field samples are not considered supported by adequate validation data and are not appropriate for use in risk assessment or for tolerance setting purposes. Further, since the data generated from analytical method RAM 244/02 is not of utility for regulatory purposes, they are not included in this document.

Additionally, samples were analyzed using a GC/MSD method (RAM 280). The LOQ is 0.01 ppm for both EMA and HEMA, or 0.02 ppm each when expressed as acetochlor equivalents.

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The LOD was not reported. The extraction procedure in this method is substantially similar to the extraction scheme employed in the current enforcement method; therefore, HED concludes that this method has been adequately demonstrated to extract weathered residues and has been adequately validated for data collection purposes.

Residues of EMA and HEMA were each <0.02 ppm in all root and top samples. Combined residues were <0.04 ppm (EMA plus HEMA, expressed in acetochlor equivalents) in all root and top samples.

No data were provided on residues of the hydroxymethyl ethyl aniline (HMEA) metabolites.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in this study, the sugar beet field rotational crop data are classified as scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the forthcoming U. S. EPA document entitled *Acetochlor: Petitions for Tolerances on Sweet Corn and Rotational Crops of Nongrass Animal Feeds (Group 18), Sugar Beets, Dried Shelled Beans and Peas (Subgroup 6C), Sunflowers, Potatoes, Cereal Grains (Group 15), and Forage, Fodder, and Straw of Cereal Grains (Group 16). Summary of Analytical Chemistry and Residue Data* (D. Davis, D230310).

COMPLIANCE:

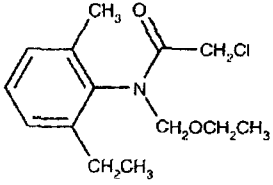
Signed and dated GLP, quality assurance, and data confidentiality statements were provided. No deviations from regulatory requirements were noted that would impact the study results or their interpretation.



A. BACKGROUND INFORMATION

Acetochlor is a chloroacetanilide herbicide used for preemergence control of weeds in corn. In the United States, acetochlor is conditionally registered for use on corn to the Acetochlor Registration Partnership (ARP), which is comprised of Monsanto and Dow AgroSciences. Acetochlor is formulated as a variety of emulsifiable concentrate (EC), emulsion in water (EW), microencapsulated (Mcap), or granular (G) formulations that can be applied to corn as a preplant, preemergence, or early postemergence application using only ground equipment. Tolerances are established for the combined residues of acetochlor and its metabolites convertible to ethyl methyl aniline (EMA) or hydroxyethyl methyl aniline (HEMA), expressed as acetochlor equivalents [40 CFR §180.470]. Tolerances range from 0.05 to 1.5 ppm in/on corn commodities resulting from the direct use of acetochlor and from 0.02 to 1.0 ppm in commodities from rotational crops of sorghum, soybean, or wheat.

The ARP has submitted a petition (PP#1F6263) proposing tolerances for inadvertent residues of acetochlor in rotated dried peas and beans (subgroup 6C), sugar beets, sunflowers, potatoes, cereal grains (group 15, except corn and rice), and the forage, fodder, and straw of cereal grains (group 16, except corn and rice).

Chemical structure	
Common name	Acetochlor
Molecular Formula	C ₁₄ H ₂₀ ClNO ₂
Molecular Weight	269.8
IUPAC name	2-chloro-N-ethoxymethyl-6'-ethylacet-o-toluidide
CAS name	2-chloro-N-(ethoxymethyl)-N-(2-ethyl-6-methylphenyl)acetamide
CAS #	34256-82-1
PC Code	121601
End-use Product	6.4 lb/gal EC



Parameter	Value	Reference
Boiling point/range	163 °C at 10 mm Hg; decomposition occurs before the boiling point at atmospheric pressure; (calculated by extrapolation of vapor pressure at lower temperature)	Acetochlor TRED, Revised HED Chapter, 3/1/06
pH	4.41, 1% solution in acetone:water (1:1, v:v)	
Density at 20 °C	1.123 g/mL	
Water solubility at 25 °C	223 mg/L	
Solvent solubility at 25 °C	Infinitely soluble in acetone, benzene, carbon tetrachloride, ethanol, chloroform, and toluene	
Vapor pressure at 25 °C	0.045 μ Hg (4.5×10^{-5} mm Hg)	
Dissociation constant, pK_a	Not applicable because acetochlor is neither an acid nor a base.	
Octanol/water partition coefficient	970 or 1082	
UV/visible absorption spectrum	Not available	

Metabolite Type	Structure
EMA-type metabolites	
HEMA-type metabolites	
HMEA-type metabolites	



B. EXPERIMENTAL DESIGN

B.1. Study Site Information

Twelve rotational sugar beet field trials were conducted at field sites throughout the U.S. during 1995 (Table B.1.1). At each site, corn (sweet or field) was planted and treated once with acetochlor (6.4 lb/gal EC) as a broadcast application at a target rate of 3 lb ai/A using ground equipment (Table B.1.2). A rotational crop of sugar beets was planted 296-366 DAT (10-12 months) at eleven sites and at 154 DAT (5 months) at one site.

Detailed soil characteristics and meteorological data were not provided, but maintenance pesticides and detailed plot history were provided. In addition, the study authors noted that weather patterns at all trial sites were typical of the growing regions. Rainfall was supplemented with irrigation as needed.

Location (County, State) Year, Trial ID	End-Use Product	Application Information				Rotational Crop
		Method ¹ ; Timing	Vol. (GPA)	Application Rate (lb ai/A)	PBI ² (days)	
Boone, IA 1995 63-IA-95-725	6.4 lb/gal EC	Broadcast Soil: preplant incorporated	15	3	358	Sugar Beets
Champaign, IL 1995 60-IL-95-726	6.4 lb/gal EC	Broadcast Soil: preemergence	20	3	352	Sugar Beets
Conklin, MI 1995 04-MI-95-727	6.4 lb/gal EC	Broadcast Soil: preplant incorporated	22.8	3	357	Sugar Beets
Riga, MI 1995 67-MI-95-728	6.4 lb/gal EC	Broadcast Soil: preemergence	14.8	3	366	Sugar Beets
Mooreton, ND 1995 34-ND-95-729	6.4 lb/gal EC	Broadcast Soil: preemergence	14.5	3	360	Sugar Beets
Scottsbluff, NE 1995 48-NE-95-730	6.4 lb/gal EC	Broadcast Soil: preplant incorporated	20.8	3	349	Sugar Beets
Ault, CO 1995 48-CO-95-731	6.4 lb/gal EC	Broadcast Soil: preplant incorporated	14.8	3	354	Sugar Beets
Austin, CO 1995 14-CO-95-732	6.4 lb/gal EC	Broadcast Soil: preplant incorporated	24.5	3	311	Sugar Beets
Visalia, CA 1995 02-CA-95-733	6.4 lb/gal EC	Broadcast Soil: preplant incorporated	27.6	3	348	Sugar Beets
Hickman, CA 1995 18-CA-95-734	6.4 lb/gal EC	Broadcast Soil: preplant incorporated	34.9	3	154	Sugar Beets
Jerome, ID 1995 16-ID-95-735	6.4 lb/gal EC	Broadcast Soil: preplant incorporated	20	3	348	Sugar Beets
Hermiston, OR 1995 15-OR-95-737	6.4 lb/gal EC	Broadcast Soil: preemergence	10.8	3	296	Sugar Beets

¹ All applications were made using ground equipment.

² Plant-back Interval.



TABLE B.1.2. Trial Numbers and Geographical Locations.

NAFTA Growing Zones ¹	Sugar Beets		
	Submitted	Requested	
		Canada	US
1	--	NA	-
2	--	NA	
3	--	NA	
4	--	NA	--
5	5	NA	5
6	--	NA	--
7	1	NA	1
8	1	NA	1
9	1	NA	1
10	2	NA	2
11	2	NA	2
12	--	NA	--
Total	12	NA	12

¹ Regions 13-2 and 1A, 5A, 5B, and 7A were not included as the use is restricted to the US.

B.2. Sample Handling and Preparation

Single control and duplicate treated samples of sugar beet roots and tops (≥ 12 plants) were harvested at commercial maturity, 104-335 days after planting (460-522 DAT). After collection, samples were placed in frozen storage at the test facility within 5 hours of collection, then shipped frozen to the analytical laboratory, Jealott's Hill Research Station, Berkshire, UK and stored frozen (~ -18 °C) until analysis. Samples were stored frozen for up to 5.3 months prior to EMA/HEMA analysis.

B.3. Analytical Methodology

Samples of sugar beet roots and tops were analyzed for residues of acetochlor *per se* using a GC/NPD Method RAM 244/02 (D. Davis, 44107102.der). The registrant has not demonstrated that this method can extract field weathered residues. Therefore data on residues of acetochlor *per se* from field samples are not considered supported by adequate validation data and are not appropriate for use in risk assessment or for tolerance setting purposes. Further, since the data generated from analytical method RAM 244/02 are not of utility for regulatory purposes, they are not included in this document.

Additionally, samples of sugar beet roots and tops were analyzed for residues of acetochlor (converted to EMA) and its metabolites convertible to ethyl methyl aniline (EMA) and hydroxyethyl methyl aniline (HEMA) using GC/MSD Method RAM 280 (D. Davis, 44107103.der).



For Method RAM 280, residues are extracted with acetonitrile:water (80:20, v/v), concentrated, and base hydrolyzed by refluxing with saturated potassium hydroxide and methanol to yield EMA and HEMA. The resulting hydrolysate is diluted with water and saturated sodium chloride, and residues of EMA and HEMA are partitioned into toluene. Residues are acylated with heptafluorobutyric acid anhydride, and partitioned against a sodium bicarbonate solution to remove the derivatizing agent. Residues are then analyzed by GC/MSD operating in the selective ion monitoring (SIM) mode, and using the 162 and 314 ions for quantifying EMA and HEMA, respectively. Residues are quantified by comparison to external standards. The LOQ is 0.01 ppm for both EMA and HEMA, or 0.02 ppm each when expressed as acetochlor equivalents. The LOD was not reported.

Method RAM 280 employs an extraction scheme substantially similar to that used in the current enforcement method; therefore, HED considers that this method is adequate to recover weathered residues from field samples. Additionally, the method has been adequately validated as a data collection method based on the results of concurrent fortification sample spiked with HEMA- or EMA-type compounds.

C. RESULTS AND DISCUSSION

Samples were stored frozen for a maximum of 5.3 months (Table C.1). Adequate storage stability data are available (D. Davis, 45483301.der) indicating that acetochlor and metabolites of EMA and HEMA are stable up to 9 in months in potato tubers and sugar beet tops. The potato tuber data can be translated to sugar beet roots; therefore, these data will support the frozen storage intervals in this trial.

The method used to determine the combined residues of acetochlor (converted to EMA) and its EMA- and HEMA-type metabolites in sugar beet roots and tops was adequately validated in conjunction with the field sample analyses (Table C.2). Concurrent recovery sugar beet and root samples were fortified with both EMA and HEMA at 0.02-0.10 ppm acetochlor equivalents. Recoveries of EMA from eight top and eight root samples were all within the 70% - 120% acceptable recovery range. Recoveries of HEMA from eight top and eight root samples were all within the 70% - 120% acceptable range with the exception of one sugar beet top sample fortified with HEMA at 0.02 ppm acetochlor equivalents with a recovery of 61%. Individual sample recoveries are shown in the table below. Adequate sample calculations were provided along with example chromatograms. Apparent residues of EMA and HEMA were <LOQ in all control samples.

Residues of EMA and HEMA were each <LOQ (<0.02 ppm acetochlor equivalents) in all root and top samples, for combined residues of <0.04 ppm expressed as acetochlor equivalents.

No data are provided on the HMEA-type metabolites.



Common cultural practices were used to maintain plants, and the weather conditions and the maintenance chemicals and fertilizer used in the study did not have a notable impact on the residue data.

Matrix	Analyte	Storage Temp. (°C)	Actual Storage Duration (days) ¹	Limit of Demonstrated Storage Stability (months) ²
Sugar beet roots and tops	EMA/HEMA	-18	64-161	9

¹ Samples extracts were analyzed within 1-6 days of extraction, with the exception of samples from one field trial for which extracts were stored for 22 days.

² 45483301.doc.

Matrix	Analyte	Spike level (mg/kg) ¹	Sample size (n)	Recoveries (%)	Mean ± std dev
Roots	EMA	0.02	4	116, 89, 77, 81	91 ± 18
		0.1	4	104, 85, 84, 113	96 ± 14
	HEMA	0.02	4	75, 80, 89, 100	86 ± 11
		0.1	4	89, 111, 87, 91	94 ± 11
Tops	EMA	0.02	4	87, 88, 71, 99	86 ± 12
		0.1	3	92, 83, 118	98 ± 18
		0.2	1	107	n/a
	HEMA	0.02	4	61, 71, 79, 96	77 ± 15
		0.1	3	72, 81, 109	87 ± 19
		0.2	1	90	n/a

¹ Spiking level expressed as acetochlor equivalents.



TABLE C.3. Residues of EMA and HEMA Residues in Rotational Sugar Beets.

Location (County, State, Year)	EPA Region	Variety	Total Rate (lb ai/A)	PBI ¹ (days)	Harvest DALA ²	RAC	Residues (ppm) ³		
							EMA	HEMA	Combined ⁴
Boone, IA 1995 63-IA-95-725	5	Kwi800	3	358	462	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Champaign, IL 1995 60-IL-95-726	5	Ace 192	3	352	460	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Conklin, MI 1995 04-MI-95-727	5	Monitor Sugar co. E-4	3	357	505	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Riga, MI 1995 67-MI-95-728	5	Beta 5931	3	366	505	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Moreton, ND 1995 34-ND-95-729	5	Beta 3843	3	360	511	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Scottsbluff, NE 1995 48-NE-95-730	7	Kw 2398	3	349	516	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Ault, CO 1995 48-CO-95-731	8	Seedex sx-1	3	354	498	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Austin, CO 1995 14-CO-95-732	9	Ace 177	3	311	494	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Visalia, CA 1995 02-CA-95-733	10	ss-L53R	3	348	522	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Hickman, CA 1995 18-CA-95-734	10	SS NB3	3	154	489	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Jerome, ID 1995 16-ID-95-735	11	PM-9	3	348	503	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
Hermiston, OR 1995 15-OR-95-737	11	WS PM9	3	296	483	Root	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04
						Top	<0.02, <0.02	<0.02, <0.02	<0.04, <0.04

¹ PBI = Plant Back Interval; the PBI was 296-366 days (10-12 months) at eleven sites and 154 days (5 months) at one site.
² DALA = Days After Last Application.
³ The LOQ is 0.02 ppm for EMA and HEMA. The LOD was not reported.
⁴ As acetochlor is converted to EMA by the GC/MSD method, the combined total residues are the sum of EMA and HEMA residues, expressed in acetochlor equivalents.



TABLE C.4. Summary of Residue Data in Rotational Sugar Beets									
Commodity	Total Rate (lb ai/A)	PBI ¹ (days)	Residue Levels (ppm) ²						
			n	Min.	Max.	HAFT ³	Median (STMdR) ⁴	Mean (STMR) ⁴	Std. Dev.
EMA									
Root	3.0	154-366	24	<0.02	<0.02	<0.02	0.01	0.01	NA
Top			24	<0.02	<0.02	<0.02	0.01	0.01	NA
HEMA									
Root	3.0	154-366	24	<0.02	<0.02	<0.02	0.01	0.01	NA
Top			24	<0.02	<0.02	<0.02	0.01	0.01	NA
Combined⁵									
Root	3.0	154-366	24	<0.04	<0.04	<0.04	0.02	0.02	NA
Top			24	<0.04	<0.04	<0.04	0.02	0.02	NA

¹ The PBI was 296-366 days (10-12 months) at eleven sites and 154 days (5 months) at one site.
² LOQ 0.02 ppm for EMA and HEMA. The LOD was not reported.
³ HAFT = Highest Average Field Trial.
⁴ STMdR = Supervised Trial Median Residue; STMR = Supervised Trial Mean Residue. For calculation of the median, mean and standard deviation, 1/2 the LOQ (0.01 ppm) was used for residues reported at <LOQ.
⁵ As acetochlor is converted to EMA by the GC/MSD method, the combined total residues are the sum of EMA and HEMA residues, expressed in acetochlor equivalents

D. CONCLUSION

The submitted field rotational crop data on sugar beets are adequately supported by field documentation and storage stability data. The residue data were generated using a validated analytical method.

Residues of EMA and HEMA in sugar beets planted 5 months (1 site) or 10 – 12 months (11 sites) after application of acetochlor to a primary crop of corn at 3 lbs ai/A were each <LOQ (<0.02 ppm acetochlor equivalents) in all root and top samples. Combined residues were <0.04 ppm (EMA plus HEMA, expressed in acetochlor equivalents) in all root and top samples. No data were provided on HMEA-type metabolites.

E. REFERENCES

DP Barcode: D292336
 Subject: **ACETOCHLOR**. Revised HED Chapter of the Tolerance Reassessment Eligibility Decision (TRED) Document.
 From: A. Protzel
 To: F. Fort
 Dated: 3/1/06
 MRID(s): None



F. DOCUMENT TRACKING

RDI: D. Davis (3/16/06), T. Goodlow (3/27/07).

Petition Number(s): 1F6263

DP Barcode(s): D230310 and D275019

PC Code: 121601