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Approved by: R. Loranger
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In the absence of signatures, this document is considered to be a draft with deliberative material for internal use only.

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:

45322109 Veal, P. and Spillner, C. (1997) Residue Levels on Processed Sugar Beet Fractions from Trials Carried Out in the United States of America During 1995-1996 for Sugar Beets Rotated into Corn: Lab Project Number: ACET-95-PR-01: RJ2264B. Unpublished study prepared by Monsanto Co. and American Agricultural Services, Inc. 53 p.

EXECUTIVE SUMMARY:

In two field trials conducted during 1995-1996 in CA, acetochlor (6.4 lb/gal EC) was applied to a primary crop of sweet corn at 15 lb ai/A. The corn was grown and harvested following common agricultural practices. At each site, a rotational crop of sugar beets was planted 154 or 335 days after treatment (DAT). Single bulk control and treated samples of sugar beet roots were harvested at commercial maturity, 335 and 174 days after planting (489 and 509 DAT). Samples were analyzed from only one of the field sites, as the second site served as a backup in case of crop failure at the primary site. Root samples from the primary field site were stored frozen for up to 98 days prior to analysis, an interval supported by available storage stability data.

A GC/mass selective detector (MSD) method (RAM 280) was used to determine residues acetochlor (converted to EMA) and its metabolites convertible to ethyl methyl aniline (EMA) and hydroxyethyl methyl aniline (HEMA) in sugar beet roots. The LOQ is 0.01 ppm for both EMA and HEMA, or 0.02 ppm when expressed as acetochlor equivalents. 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.

Following a 15 lb ai/A application of acetochlor (EC) to a primary corn crop, residues in mature roots from sugar beets planted 335 DAT were <LOQ (<0.02 ppm acetochlor equivalents) each for EMA and HEMA. Combined residues were <0.04 ppm (EMA plus HEMA, expressed in acetochlor equivalents). As residues were non-quantifiable in roots (RAC) from a rate which is equivalent to a 5x treatment rate based on currently registered uses of acetochlor, no analysis of processed fractions was conducted. The data indicate that acetochlor residues are unlikely to be detectable in sugar beet processed fractions.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in this study, the sugar beet processing 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 EMA or HEMA, to be analyzed as acetochlor, and 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).

TABLE A.1. Acetochlor Nomenclature

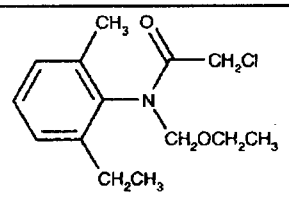
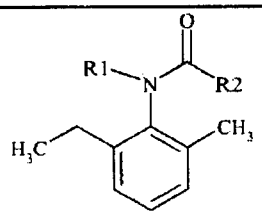
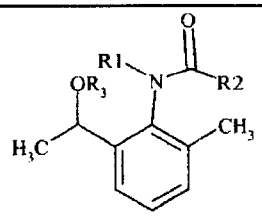
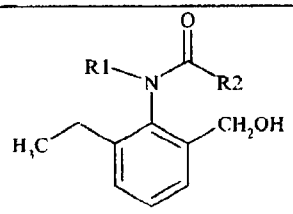
Chemical structure	
Common name	Acetochlor
Molecular Formula	$C_{14}H_{20}ClNO_2$
Molecular Weight	269.8
IUPAC name	2-chloro-N-ethoxymethyl-6'-ethylacet-o-toluidide
CAS name	2-chloro-N-(ethoxymethyl)-N-(2-ethyl-6-methylphenyl)acetamide
CAS #	34256-82-1
PC Code	121601
End-use Product	6.4 lb/gal EC

TABLE A.2. Physicochemical Properties of Acetochlor.		
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 HED Chapter of the TRED, 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	

Table A.3. Acetochlor Metabolite Structures	
Metabolite Type	Structure
EMA-type metabolites	
HEMA-type metabolites	
HMEA-type metabolites	

B. EXPERIMENTAL DESIGN

B.1. Study Site Information

TABLE B.1.1. Trial Site Conditions.

Trial Identification (City, State, Year)	Soil characteristics			
	Type	%OM	pH	CEC (meq/g)
Visalia, CA 1995	Sandy Loam	NR	NR	NR
Hickman, CA 1995 [1]	Sandy Loam	NR	NR	NR

NR = Not reported.
[1] The second field trial was conducted as a backup in case of crop failure in the first trial.

TABLE B.1.2. Study Use Pattern to Primary Corn Crop.

Location (County, State) Year, Trial ID	End-use Product	Application				Rotational Crop
		Method [1]; Timing	Vol. (GPA)	Application Rate (lb ai/A)	PBI [2] (days)	
Visalia, CA 1995 02-CA-95-741	6.4 lb/gal EC	Broadcast Soil; preplant-incorporated	26.8	15.0	335	Sugar beets
Hickmann, CA 1995 [3] 18-CA-95-742	6.4 lb/gal EC	Broadcast Soil: preplant-incorporated	35	15.0	154	Sugar beets

[1] Applications were made using ground equipment.
[2] Plantback Interval.
[3] Backup field trial.

B.2. Sample Handling and Preparation

Single bulk samples of control and treated sugar beet roots (~300 lbs) were harvested at commercial maturity, 335 and 174 days after planting (489 and 509 DAT). Samples were frozen within 4 hours of sampling, 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 from collection to analysis for up to 98 days.

B.3. Analytical Methodology

Samples of sugar beet roots were analyzed for residues of acetochlor *per* se using a GC/NPD Method RAM 244 (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; therefore, not appropriate for use in risk assessment or for tolerance setting purposes. Further, since this data generated from analytical method RAM 244 are not of utility for regulatory purposes, they are not included in this document.

Additionally, samples of sugar beet roots 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 heptafluorobutryic 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 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

Prior to analysis, samples were stored frozen for a maximum of 98 days (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. 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 was adequately validated in conjunction with the field sample analyses (Table C.2). All concurrent recoveries of EMA and HEMA were within the acceptable range of 70% to 120% from sugar beet roots fortified with each analyte at 0.02 and 0.05 ppm. Adequate samples calculations were provided along with example chromatograms. Apparent residues of both analytes were <LOQ in all control samples.

Following an application of acetochlor to a primary corn crop at 15 lb ai/A (5x rate), residues of EMA and HEMA were each <LOQ (0.02 ppm acetochlor equivalents) in a single bulk treated sample. Combined residues were <0.04 ppm (EMA plus HEMA, expressed in acetochlor equivalents). As residues were non-quantifiable in roots (RAC) from a 5x-treatment, no analysis of processed fractions was conducted.

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.

TABLE C.1. Summary of Storage Conditions			
Matrix	Storage Temp. (°C)	Actual Storage Duration [1] (days)	Limit of Demonstrated Storage Stability (months) [2]
Sugar Beet Root	-18	98	9

[1] Samples extracts were analyzed within 2 days of extraction.
[2] D. Davis, 45483301.der.

TABLE C.2. Summary of Concurrent Method Recoveries of HEMA and EMA from Sugar Beet Root Samples. [1]					
Matrix	Analyte	Spike level (mg/kg)	Sample size (n)	Recoveries (%)	Mean ± std dev
Sugar Beet	EMA	0.02	4	116, 89, 77, 81	91 ± 18
		0.10	4	104, 85, 84, 113	96 ± 14
	HEMA	0.02	4	75, 80, 89, 100	86 ± 11
		0.10	4	89, 111, 87, 91	94 ± 11

[1] Residues containing the EMA or HEMA moieties were determined using GC/MSD Method RAM 280.

TABLE C.3. Residue Data from Sugar Beet Processing Study using Roots Grown from Sugar Beets Rotated with Field Corn Treated with Acetochlor (6.4 lb/gal EC).							
RAC	Processed Commodity	Total Rate (lb ai/A)	DALA[1] (days)	Residues [2] (ppm)			Processing Factor
				EMA	HEMA	Combined [3]	
Sugar Beets	NA	15.0	509	<0.02	<0.02	<0.04	NA

[1] DALA= Days After Last Application. The sugar beet rotational crop was planted 335 DAT and mature roots were harvested 174 days after planting.
[2] The LOQ is <0.02 ppm for EMA and HEMA (acetochlor equivalents). The LOD was not reported.
[3] 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.
NA = not applicable; as residues were <LOQ in the RAC from a 5x application, the processed fractions were not analyzed.

D. CONCLUSION

The processing study on rotational sugar beets is adequately supported by field documentation and storage stability data and the residue data were generated using a validated analytical method.

Acetochlor residues are unlikely to be detectable in sugar beet root processed commodities, as residues of acetochlor (converted to EMA), EMA and HEMA were all <LOQ in/on roots harvested from sugar beets planted 335 days after a 15 lb ai/A treatment (equivalent to a 5x-treatment rate based on currently registered uses) to the primary corn crop. No data are provided on residues of HEMA-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 (3/23/06); M. Doherty (4/18/06).
Petition Number(s): 1F6263
DP Barcode(s): D230310 and D275019
PC Code: 121601