



Tribenuron Methyl/DPX-L5300/PC Code 128887/E.I. du Pont de Nemours and Company
DACO 7.4.5/OPPTS 860.1520/OECD IIA 5.5.4 and IIIA 8.5
Processed Food and Feed - Cotton

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This data evaluation record (DER) was originally prepared under contract by Dynamac Corporation (20440 Century Boulevard, Suite 100; Germantown, MD 20874; submitted July 21, 2004). The DER has been reviewed by the Health Effects Division (HED) and revised to reflect current Office of Pesticide Program (OPP) policies.

STUDY REPORT:

45098405. March 21, 2000. Amoo, J.S. Magnitude of Residues of Thifensulfuron Methyl and Tribenuron Methyl in Cotton Following Application of Harmony Extra® Herbicide at Maximum Label Rates. Lab Project Number: AMR 4343-97. Unpublished study prepared by E.I. du Pont de Nemours and Company. 247 pages.

EXECUTIVE SUMMARY:

To fulfill the requirements for a cotton processing study, DuPont has submitted a study depicting the magnitude of the residue of tribenuron methyl in cotton seed following a pre-emergence defoliant application of a 25% tribenuron methyl dry flowable (DF) formulation at an exaggerated rate. We note that the test substance used in this study also contained 50% thifensulfuron methyl; refer to the other processed food and feed DER for the results concerning thifensulfuron methyl residues in/on cotton (S. Ary, D301488, MRID No.: 45098405, 8/10/04).

In one trial conducted in MS, cotton seed was harvested at normal maturity (133 days after treatment) following a single defoliant broadcast application of the 25% tribenuron methyl DF at 0.047 lb ai/A (approximately 5x the field trial application rate) made one day after planting of cotton. Application was made using ground equipment with an adjuvant (surfactant) added to the spray mixture. Residues of tribenuron methyl were nondetectable (less than 0.006 ppm) in cotton seed; therefore, the cotton seed was not further processed. The maximum theoretical concentration factor for cotton is 6x (Office of Prevention, Pesticides, and Toxic Substances [OPPTS] Guideline 860.1520, Table 1).



Samples of cotton seed were analyzed for residues of tribenuron methyl using LC/MS (DuPont Method 1381). The validated limit of quantitation (LOQ) was 0.020 ppm, and the estimated limit of detection (LOD) was 0.006 ppm in/on cotton seed. This method is adequate for data collection based on acceptable concurrent method recovery data. The maximum storage interval of cotton seed (raw agricultural commodity [RAC]) samples, from harvest to analysis, was 335 days (eleven months). Adequate storage stability data are available to support the storage conditions and intervals of cotton seed samples from the exaggerated rate study.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the data from the exaggerated rate cotton field trial are classified as scientifically acceptable. HED concludes that additional data are not required pertaining to cotton processed commodities because residues of tribenuron methyl were nondetectable in/on undelinted cotton seed following a pre-emergence defoliant treatment at an exaggerated rate (approximately 5x the crop field trial rate).

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

Tribenuron methyl is an herbicide registered for food/feed uses on barley, oat, and wheat, and grass grown for seed. Tribenuron methyl is one of the active ingredients in DuPont Harmony® Extra (75% dry flowable formulation containing 50% thifensulfuron methyl and 25% tribenuron methyl) for post-emergence use on barley, oat, and wheat, and as a pre-plant defoliant.

Table A.1. Tribenuron Methyl Nomenclature.	
Chemical structure	
Common name	Tribenuron methyl
Molecular formula	C ₁₅ H ₁₇ N ₃ O ₆ S
Molecular weight	395.39 g/mole



IUPAC name	methyl-2-[4-methoxy-6-methyl-1,3,5-triazin-2-yl(methyl)carbamoyl-sulfamoyl]benzoate
CAS name	methyl-2-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)methylamino]carbonyl]amino]sulfonyl]benzoate
CAS registry number	101200-48-0
PC Code	128887
Current food/feed site registrations	Barley, oat, wheat, and grass grown for seed

Parameter	Value	Reference
Melting point/range	141 °C	R. Cook, Memo, 4/26/88
pH	4.27 (slurry in water)	R. Cook, Memo, 4/26/88
Density	1.54 g/mL	R. Cook, Memo, 4/26/88
Water solubility	28 mg/L at 25 °C, pH 4.0 50mg/L at 25 °C, pH 5.0 280 mg/L at 25 °C, pH 6.0	R. Cook, Memo, 4/26/88
Solvent solubility	43.8 g/L at 25 °C in acetone 54.2 g/L at 25 °C in acetonitrile 3.12 g/L at 25 °C in carbon tetrachloride 17.5 g/L at 25 °C in ethyl acetate 0.028 g/L at 25 °C in hexane 3.39 g/L at 25 °C in methanol	R. Cook, Memo, 4/26/88
Vapor pressure	3.9 x 10 ⁻¹⁰ mm Hg at 25 °C (Knudsen) 2.7 x 10 ⁻⁷ mm Hg at 25 °C (gas saturation) <8.3 x 10 ⁻⁷ mm Hg at 70 °C (gas saturation)	R. Cook, Memo, 4/26/88
Dissociation constant	pK _a = 5.0	R. Cook, Memo, 4/26/88
Octanol/water partition coefficient	Log(K _{ow}) = 15, pH 5 (calculated) Log(K _{ow}) = 0.3, pH 7 Log(K _{ow}) = 0.003, pH 9 (calculated)	R. Cook, Memo, 4/26/88
UV/visible absorption spectrum	Not available	

B. EXPERIMENTAL DESIGN

B.1. Application and Crop Information

Location (City, State; Year)	EP ¹	Application					Tank Mix Adjuvants	Harvest Procedures
		Method; Timing	Vol (GPA ²)	Rate ³ (lb ai/A)	RTI ⁴ (days)	Total Rate ³ (lb ai/A)		
Greenville, MS; 1997	25% DF	Broadcast; 1 day after planting	14.2	0.047	N/A	0.047	Surf Aid (0.25% ai v/v)	Mechanical picker



1. EP = End-use Product; the 75% DF contains 50% thifensulfuron methyl and 25% tribenuron methyl.
2. GPA = Gallons per acre.
3. Calculated by the study reviewer from a target rate of 2.25 oz ai/A, which is equivalent to 1.501 oz ai/A (0.094 lb ai/A) of thifensulfuron methyl and 0.749 oz ai/A (0.047 lb ai/A) of tribenuron methyl.
4. RTI = Retreatment Interval; not applicable because a single application was made.

B.2. Sample Handling and Processing Procedures

A single untreated and duplicate treated samples of mature cotton seed were harvested and frozen at the field site within one hour of collection. Samples were shipped frozen four days after harvest to DuPont Experimental Station (Wilmington, DE), where samples were stored frozen (ca. -20 °C) until residue analysis.

Because residues in/on the RAC were nondetectable, the processed commodities were not generated.

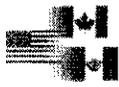
B.3. Analytical Methodology

Samples of cotton seed were analyzed by DuPont for residues of tribenuron methyl using LC/MS DuPont Method 1381, entitled "Analytical Enforcement Method for the Quantitation of Thifensulfuron Methyl and Tribenuron Methyl in Cotton Seed and Cotton Gin Trash by Liquid Chromatography/Electrospray Mass Spectrometry (LC/ESI-MS)". A description of the method was included in the submission; for a complete description of the method, refer to the residue analytical method DER (S. Ary, D305958, MRID No.: 45098401, 8/10/04). This method is proposed for enforcement purposes in cotton.

Briefly, homogenized samples of cotton seed were extracted with acetonitrile:0.1 M ammonium carbonate (80:20, v:v), concentrated to dryness, and reconstituted in methanol for analysis. Analysis was performed by column-switching liquid chromatography. Extracts in methanol were applied to a size exclusion chromatography (SEC) column, and the eluent was diverted onto a reversed phase XDB-C8 column where matrix components were further separated from the residues during analysis. Residue levels were quantitated from a calibration curve of tribenuron methyl standards. The validated LOQ was 0.020 ppm for cotton seed, and the estimated LOD (one-third the LOQ) was 0.006 ppm.

C. RESULTS AND DISCUSSION

In one trial conducted in MS, cotton seed was harvested at normal maturity (133 days after treatment) following a single defoliant broadcast application of the 25% tribenuron methyl DF at 0.047 lb ai/A (approximately 5x the field trial application rate) made one day after planting of cotton. Application was made using ground equipment with an adjuvant (surfactant) added to the spray mixture. We note that the test substance used in this study also contained 50% thifensulfuron methyl; refer to the other processed food and feed DER for the results concerning thifensulfuron methyl residues (S. Ary, D301488, MRID No.: 45098405, 8/10/04).



Concurrent method recovery data are presented in Table C.1. Samples of cotton seed were analyzed by DuPont for residues of tribenuron methyl using LC/MS DuPont Method 1381. The validated LOQ was 0.020 ppm for cotton seed, and the estimated LOD was 0.006 ppm. This method is adequate for data collection based on acceptable concurrent method recovery data. Apparent residues of tribenuron methyl were nondetectable (less than 0.006 ppm) in/on one sample of untreated cotton seed.

Sample storage conditions and intervals are summarized in Table C.2. The maximum storage interval of cotton seed (RAC) samples, from harvest to analysis, was 335 days (eleven months). In conjunction with cotton field trials, the registrant has submitted storage stability data which demonstrate that residues of tribenuron methyl are relatively stable in cotton seed stored frozen for up to 14 months (refer to the storage stability DER, S. Ary, D305598, MRID No.: 45098405, 8/10/04). These data adequately support the storage conditions and intervals of samples from the exaggerated rate study.

Residue data from the study are presented in Table C.3. Residues of tribenuron methyl were nondetectable (less than 0.006 ppm) in/on treated cotton seed; therefore, the cotton seed was not further processed. The maximum theoretical concentration factor for cotton is 6x (OPPTS Guideline 860.1520, Table 1).

Matrix	Spike level (ppm)	Sample size (n)	Recoveries (%)	Mean
Cotton seed	0.02	1	75	75
	0.2	1	75	

Matrix (RAC or Extract)	Storage Temp. (°C)	Actual Storage Duration ¹	Interval of Demonstrated Storage Stability
Cotton seed	ca. -20	335 days (eleven months)	Tribenuron methyl was demonstrated to be relatively stable in/on cotton seed stored frozen for up to fourteen months. ²

1. Samples were analyzed on the day of extraction.

2. Refer to the storage stability DER (S. Ary, D305598, MRID No.: 45098405, 8/10/04).

RAC	Processed Commodity	Total Rate (lb ai/A)	PHI (days)	Residues (ppm)	Processing Factor
Cotton seed, undelinted	None ¹	0.047	133	ND, ND ²	Not applicable

1. Cottonseed was not processed because residues were nondetectable.

2. ND = Not detected; residues were reported as ND by the registrant. The validated LOQ was 0.02 ppm and the estimated LOD was 0.006 ppm.

D. CONCLUSION

Residues of tribenuron methyl were nondetectable (less than 0.006 ppm) in/on undelinted



cottonseed (RAC) harvested at normal maturity (133 days after treatment) following a single defoliant broadcast application of the 25% tribenuron methyl DF at 0.047 lb ai/A (approximately 5x the field trial application rate) made one day after planting of cotton. Cotton seed was not processed because residues were nondetectable in/on the RAC. An acceptable method was used for quantitation of residues in cotton seed.

E. REFERENCES

Cook, R.W. New Chemical Product Chemistry Review DPX-L5300 (Technical). RCB Nos.: 2516 and 2517. Accession No.: 073786. MRID Nos.: 40245503, 40245505, and 40245509. April 26, 1988.

Ary, Samuel. DER: Tribenuron Methyl Residue Analytical Method - Cotton. DP Barcode: 305958. MRID No.: 45098401. August 10, 2004.

Ary, Samuel. DER: Tribenuron Methyl Storage Stability - Cotton. DP Barcode: 305958. MRID No.: 45098405. August 10, 2004.

Ary, Samuel. DER: Thifensulfuron Methyl Processed Food and Feed - Cotton. DP Barcode: 301488. MRID No.: 45098405, August 10, 2004.

F. DOCUMENT TRACKING

RDI: S. Ary (8/5/04); T. Morton (8/9/04); A. Nielsen (8/10/04)

Petition Number: 0F6135

DP Barcode: D305958

PC Code: 128887



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R130307

Chemical: Tribenuron

PC Code:
12887

HED File Code: 11100 Other Chemistry Documents
Memo Date: 8/10/2004
File ID: 0000000
Accession #: 000-00-0108

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