



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OPP OFFICIAL RECORD
HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

July 27, 2001

MEMORANDUM

SUBJECT: **Mepiquat Chloride** (109101), Magnitude of the Residue on Cotton Gin Byproducts; DP Barcode D239446; (MRID No. 44379701); Case No. 2375; Submission No. S530801.

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Executive Summary

The Residue Chemistry Chapter for the Mepiquat Chloride Reregistration Eligibility Decision (RED, DP Barcode D222340, F. Fort, 5/6/96) document required that field residue data must be submitted for cotton gin byproducts. In response, BASF Corporation submitted data (1997; MRID 44379701) depicting the magnitude of mepiquat chloride residues in/on cotton gin byproducts.

The submission was reviewed under contract by Dynamac Corporation. The review has undergone secondary review by HED and the following recommendations and conclusions were made:

1. The analytical method used for determining residues of mepiquat chloride in/on

cotton gin byproducts was an ion chromatography method with conductivity detection (BASF Draft Method D9610). The reported limit of quantification (LOQ) was 0.10 ppm for cotton gin byproducts. Based on acceptable concurrent method recoveries, the ion chromatography method is adequate for data collection purposes.

- 2a. A minimum of three cotton gin byproducts field trials for each type of harvesting (stripper and mechanical picker) are required, for a total of six field trials. Eight trials depicting the magnitude of mepiquat chloride residues in/on cotton gin byproducts were submitted and found to be adequate. Samples were harvested from each test location using either mechanical picker (4 trials) or stripper (4 trials). Residues of mepiquat chloride *per se* ranged from 0.5451 to 5.9198 ppm in/on cotton gin byproducts harvested 29-30 days (label PHI) following the last of six foliar spray applications of a representative SC/L formulation of mepiquat chloride at 0.132 lb ai/A (1x the maximum label seasonal rate). Applications were made using ground equipment in 2-5 GPA of water in six trials, and in two trials ULV applications were made using aerial equipment in 0.24-0.26 GPA of oil concentrate.
- 2b. HED recommends the establishment of a tolerance for mepiquat chloride on cotton gin byproducts at 6 ppm.

cc: RF; List B File; D.Drew , CP Moran (RD); C. Giles-Parker (RD)
RDI: C. Eiden (7/27/01)
D.Drew:CM-2, tm 821E, 305-6028

MEPIQUAT CHLORIDE
PC Code 109101; Case 2375
(DP Barcode D239446)

Registrant's Response to Residue Chemistry Data Requirements

December 10, 1997

Contract No. 68-D4-0010

Submitted to:
U.S. Environmental Protection Agency
Arlington, VA

Submitted by:
Dynamac Corporation
The Dynamac Building
2275 Research Boulevard
Rockville, MD 20850-3268

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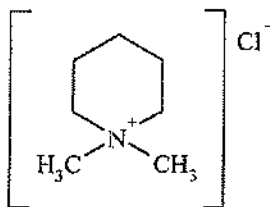
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ADDENDUM TO THE RESIDUE CHEMISTRY CHAPTER OF THE REREGISTRATION ELIGIBILITY DECISION DOCUMENT

BACKGROUND

The Residue Chemistry Chapter for the Mepiquat Chloride Reregistration Eligibility Decision (RED, DP Barcode D222340, F. Fort, 5/6/96) document required that field residue data must be submitted for cotton gin byproducts and a tolerance must be proposed for this commodity when adequate residue data have been submitted. In response, BASF Corporation submitted data (1997; MRID 44379701) depicting the magnitude of mepiquat chloride residues in/on cotton gin byproducts. These data are evaluated in this document for adequacy in fulfilling residue chemistry data requirements under OPPTS GLN 860.1500.

The qualitative nature of the residue in plants and animals is adequately understood based on acceptable metabolism studies with cotton, ruminants, and poultry. The parent, mepiquat chloride, is the residue of concern in plant and animal commodities.

Tolerances are currently established under 40 CFR §180.384 for residues of mepiquat chloride [N,N-dimethylpiperidinium chloride] in/on cotton forage (3.0 ppm), cottonseed (2.0 ppm), eggs and milk (each at 0.05 ppm), and for the fat, meat, and meat byproducts of cattle, goats, hogs, horses, poultry, and sheep (each at 0.1 ppm). A feed additive tolerance is established under 40 CFR §186.2275 for cottonseed meal (3.0 ppm); however, the Residue Chemistry Chapter for the Mepiquat Chloride RED recommended that this tolerance be revoked because any residue that may result in this commodity as a result of processing will be covered by the reassessed RAC tolerance (2.0 ppm).

Adequate methods are available for tolerance enforcement and data collection. The Pesticide Analytical Manual (PAM Volume II) lists Method I as available for the determination of residues of mepiquat chloride *per se* in/on plant and animal commodities. This GLC method, with nitrogen detection, has undergone successful Agency method tryout using plant (cottonseed, cotton forage, and cottonseed processed fractions) and animal (milk, eggs, and meat of chicken and beef) matrices. The stated limit of quantitation is 0.1 ppm for cotton and 0.05 ppm for animal products.

No maximum residue limits (MRLs) for mepiquat chloride have been established by Codex for any agricultural commodity. Therefore no compatibility issues exist with respect to U.S. tolerances.

CONCLUSIONS AND RECOMMENDATIONS

1. The analytical method, used for determining residues of mepiquat chloride in/on cotton gin byproducts was an ion chromatography method with conductivity detection (BASF Draft Method D9610). The reported limit of quantification (LOQ) was 0.10 ppm for cotton gin byproducts. Based on acceptable concurrent method recoveries, the ion chromatography method is adequate for data collection purposes.
- 2a. Eight trials depicting the magnitude of mepiquat chloride residues in/on cotton gin byproducts were submitted and found to be adequate. Residues of mepiquat chloride *per se* ranged from 0.5451 to 5.9198 ppm in/on cotton gin byproducts harvested 29-30 days following the last of six foliar spray applications of a representative SC/L formulation of mepiquat chloride at 0.132 lb ai/A (1x the maximum label seasonal rate).
- 2b. HED recommends the establishment of a tolerance for mepiquat chloride on cotton gin byproducts at 6 ppm.

DETAILED CONSIDERATIONS

Residue Analytical Methods

Samples of cotton gin byproducts from the submitted studies were analyzed for residues of mepiquat chloride by Center Analytical Laboratories, Inc. (State College, PA) using an ion pair chromatography method with conductivity detection and a cation self regenerating suppressor system (BASF Draft Method D9610). Briefly, samples were extracted twice with acidic methanol (MeOH):water (25% 0.5 N HCl in MeOH) and vacuum filtered. The filter cakes were rinsed with MeOH:water (2:1, v:v). The extracts were combined, diluted with MeOH:water (2:1, v:v), and cleaned up by cation exchange chromatography. Mepiquat

chloride residues were eluted with 2 N HCl. The pH of the acidic eluate was adjusted to 10 with 12 N NaOH. Residues were then partitioned with sodium tetraphenyl borate solution. The basic aqueous phase was extracted twice with dichloromethane, and the aqueous phase was discarded. The organic phase was filtered, and the vessels were rinsed with 2 N HCl. The HCl rinse was combined with the organic phase, and the residues were partitioned into the acidic aqueous phase. Additional 2 N HCl was added to the remaining organic fraction, and residues were re-partitioned into the acidic aqueous phase. The acidic fractions were combined and evaporated to dryness. Residues were further purified by alumina column separation. Mepiquat chloride residues were eluted with acetonitrile:MeOH (95:5, v:v). The eluate was evaporated to dryness and redissolved in "ultrapure" water for ion chromatography analysis. Mepiquat chloride was paired with hexane sulfonic acid in the ion pair chromatography method using a C-18 polymer based reversed phase column with an aqueous acetonitrile mobile phase. The reported limit of quantification (LOQ) was 0.10 ppm for cotton gin byproducts.

Concurrent recovery data were submitted for cotton gin byproducts. Untreated samples from the submitted field trials were fortified at 0.10-7.0 ppm and analyzed using the ion chromatography method described above. The results of concurrent method analyses are presented in Table 1. Based on acceptable concurrent method recoveries, the ion chromatography method (BASF Draft Method D9610) is adequate for determining residues of mepiquat chloride *per se* in/on cotton gin byproducts.

Table 1. Concurrent method recoveries of mepiquat chloride from fortified untreated samples of cotton gin byproducts.

| Crop Substrate | Fortification Levels (ppm) | Number of Samples | % Recovery |
|-----------------------|----------------------------|-------------------|------------|
| Cotton gin byproducts | 0.10 | 5 | 71-97 |
| | 2.0 | 1 | 73 |
| | 5.0 | 3 | 88-110 |
| | 7.0 | 1 | 89 |

Storage Stability Data

The harvested samples of cotton from the field trials were ginned at the field. Ginned cotton and gin byproducts samples were frozen and then shipped via freezer truck to BASF (RTP, NC) within 5-87 days of harvest. Samples were homogenized and frozen at BASF, and shipped frozen to the analytical laboratory (Center Analytical Laboratories, State College, PA) for analysis. Samples were stored frozen at the analytical laboratory until analysis. The total storage interval between harvest and analysis was 259-337 days (~9-11 months) for cotton gin byproducts.

The registrant cited previously submitted data (CBRS Nos. 14230, 14703, 13220, and 14891, DP Barcodes D206656, D209020, D199513, and D210500, 6/28/95, F. Fort) depicting the storage stability of mepiquat chloride residues in/on cottonseed and its processed commodities during frozen storage. The data indicate that residues of mepiquat chloride *per se* are stable under frozen conditions for up to 25 months in/on cottonseed and for at least 28.5 months in cottonseed hulls, meal, crude oil, refined oil, and soapstock.

OPPTS GLN 860.1500: Magnitude of the Residue in Plants

Cotton

Established tolerances: Tolerances have been established for residues of mepiquat chloride *per se* in/on cottonseed at 2 ppm and cotton forage at 3 ppm [40 CFR §180.384]; no tolerance is established for cotton gin byproducts. The Residue Chemistry Chapter for the Mepiquat Chloride RED concluded that the tolerance for cottonseed is supported by adequate residue data and should remain at the established level; however, the Chapter recommended that the established tolerance for cotton forage should be revoked because this commodity is not considered a significant livestock feed item and has been deleted from Table 1 of OPPTS GLN 860.1000.

Registered use patterns: The 0.35 and 2.01 lb/gal SC/L (EPA Reg. Nos. 7969-52 and 7969-97, respectively) and 35% DF (EPA Reg. No. 7969-107) formulations are registered for multiple foliar spray applications to cotton plants. The maximum single application rate is 0.066 lb ai/A/application. Up to five low-rate applications, with 7- to 14-day retreatment intervals, may be made provided the maximum seasonal rate of 0.132 lb ai/A is not exceeded. Applications may be made using ground or aerial equipment in a minimum of 2 gal of water per acre (GPA) except when application is made in CA. In CA, the minimum spray volume for ground and aerial equipment is 5 GPA. Ultra low volume (ULV) aerial applications, using oil as the diluent (minimum spray volume of 1 quart oil/A), are permitted for the SC/L formulations in AL, AR, FL, GA, LA, MO, MS, NC, OK, SC, TN, and TX. The established pregrazing/feeding and preharvest interval is 30 days. No plantback intervals have been established for rotational crops.

Discussion of data: The data requirements for cotton gin byproducts, as specified in the Residue Chemistry Chapter for the Mepiquat Chloride RED, are repeated below.

"The Agency currently recognizes cotton gin byproducts (commonly called gin trash which include the plant residues from ginning cotton consisting of burrs, leaves, stems, lint, immature seeds, and sand and/or dirt) as a RAC. Data are required on residues of mepiquat chloride in/on cotton gin byproducts harvested at normal maturity from plants treated at the maximum seasonal application rate. Cotton must be harvested by commercial equipment (stripper and mechanical picker) to provide an adequate

representation of plant residue for the ginning process. A minimum of three field trials for each type of harvesting (stripper and mechanical picker) are required, for a total of six field trials. An appropriate tolerance for this RAC should be proposed once acceptable data have been submitted and evaluated."

BASF has now submitted data (1997; MRID 44379701) from a total of eight trials depicting the magnitude of mepiquat chloride residues in/on cotton gin byproducts. The trials were conducted in the states of CA(1), GA(1), MS(2), and TX(4). Mature cotton was harvested 29 or 30 days following the last of six foliar spray applications, with 5- to 10-day retreatment intervals, of the 0.35 lb/gal SC/L formulation (EPA Reg. No. 7969-52). The treatment schedule consisted of: four applications at 0.011 lb ai/A/application + one application at 0.022 lb ai/A + one application at 0.066 lb ai/A. The total application rate was 0.132 lb ai/A (1x the maximum seasonal rate). Applications were made using ground equipment in 2-5 GPA of water in all trials, except for the two trials conducted in TX and MS, where ULV applications were made using aerial equipment in 0.24-0.26 GPA of oil concentrate.

One control and two treated samples were harvested from each test location using either mechanical picker or stripper. Samples were collected by mechanical picker at the four TX sites, and by stripper at the remaining four sites. The harvested cotton samples were ginned to produce cottonseed and cotton gin byproducts (including large materials like sticks, twigs, leaves, and other fibrous materials). Residues were determined using the ion chromatography method previously discussed. Apparent residues were less than the LOQ (<0.10 ppm) in/on eight samples of untreated cotton gin byproducts. Residues in/on treated samples are presented in Table 2.

Table 2. Residues of mepiquat chloride in/on cotton gin byproducts harvested 29-30 days following the last of six foliar applications of the 0.35 lb/gal SC/L formulation for a total of 0.132 lb ai/A (1x the maximum seasonal rate).

| Test Location County, State (Region) | Type of Application; Type of Harvest ^a | Uncorrected Mepiquat Chloride Residues (ppm) ^b |
|-----------------------------------------|------------------------------------------------------|--------------------------------------------------------------|
| Tulare, CA (Region 10) | Ground; Picker | 0.5451, 0.5487 |
| Tift, GA (Region 2) | Ground; Picker | 1.7405, 2.4982 |
| Washington, MS (Region 4) | Ground; Picker | 1.3506, 1.5528 |
| Washington, MS (Region 4) | ULV Aerial; Picker | 1.0929, 1.5896 |
| Hockley, TX (Region 8) | Ground; Stripper | 0.9249, 1.7677 |
| Lubbock, TX (Region 8) | Ground; Stripper | 3.0994, 4.2037 |
| Uvalde, TX (Region 6) | Ground; Stripper | 1.4427, 1.7934 |
| Uvalde, TX (Region 6) | ULV Aerial; Stripper | 5.5414, 5.9198 |

^a Applications were made with either ground equipment (2-5 GPA) or ULV aerial equipment (0.24-0.26 GPA); cotton was harvested by mechanical picker or stripper.

^b Residue values represent individual samples.

Geographic representation of data: Geographic representation of the submitted residue data for cotton gin byproducts is adequate. The Residue Chemistry Chapter of the Mepiquat Chloride RED required that at least six trials should be conducted for cotton gin byproducts. In the current study, eight trials were conducted in Regions 2 (1 trial), 4 (2 trials), 6 (2 trials), 8 (2 trials), and 10 (1 trial). According to the current guidance (OPPTS 860.1500, Table 6) Regions 2, 4, 6, 8, and 10 account for 97% of the cotton production.

Study summary: The submitted residue data for cotton gin byproducts are adequate. Residues of mepiquat chloride *per se* ranged from 0.5451 to 5.9198 ppm in/on cotton gin byproducts harvested 29-30 days following the last of six foliar spray applications of a representative SC/L formulation of mepiquat chloride at 0.132 lb ai/A (1x the maximum seasonal rate).

Based on the results of the current submission, the registrant needs to propose a tolerance for cotton gin byproducts. Toxicological considerations permitting, the available data will support a tolerance level of 6 ppm.

EPA MEMORANDA CITED IN THIS REVIEW

CBRS No.: 14230, 14703, 13220, and 14891
DP Barcode: D206656, D209020, D199513, and D210500
Subject: Mepiquat Chloride. List B Case No. 2375. Chemical I.D. No. 109101.
Registrant's Response to Data Requirements.
From: F. Fort
To: R. Whitters/K. Davis
Dated: 6/28/95
MRID(s): 43290401-43290404, 43379501, 43024701, and 43440301

CBRS No.: 16830
DP Barcode: D222340
Subject: Mepiquat Chloride. List B Case No. 2375. Chemical No. 109101.
Product and Residue Chemistry Chapters for the Reregistration
Eligibility Decision Document (RED).
From: F. Fort
To: M. Clock/P. Deschamp
Dated: 5/6/96

MASTER RECORD IDENTIFICATION NUMBERS

The citation for the MRID document referred to in this review are presented below.

44379701 Malinsky, D.; Wofford, J. (1997) Magnitude of Mepiquat Chloride Residues in Cotton Gin By-Products: Lab Project Number: 96041: 97/5327: ER97014. Unpublished study prepared by Centre Analytical Laboratories, Inc. 206 p.



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030316

Chemical: N,N-Dimethylpiperidinium chloride

PC Code: 109101

HED File Code 11000 Chemistry Reviews

Memo Date: 07/27/2001

File ID: DPD244052

Accession Number: 412-02-0010

HED Records Reference Center
01/23/2002

