



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

PESTICIDES AND  
SUBSTANCES

OFFICE OF  
PREVENTION,  
TOXIC

August 9, 1999

MEMORANDUM

SUBJECT: Acephate. List A Case No. 0042. Chemical No. 103301. Magnitude of the Residue in/on Cotton treated at Different Growth Stages. DP Barcodes D255614, D254635, and D254638 .

FROM: Felecia A. Fort, Chemist  
Reregistration Branch I  
Health Effects Division (7509C)

THRU: Whang Phang, PhD, Branch Senior Scientist  
Reregistration Branch 1  
Health Effects Division (7509C)

and

Christina Swartz  
Reregistration Branch 1  
Health Effects Division(7509C)

TO: Monica Alvarez, Chemical Review Manager  
Special Review Branch  
Special Review and Reregistration Division (7508W)

Attached is a review of data depicting the magnitude of acephate and methamidophos residues in/on cottonseed and its processed commodities following multiple applications . This information was reviewed by Dynamac Corporation under the supervision of RRB1/HED. The data assessment has undergone secondary review in the Branch and has been revised to reflect Agency policies.

HED makes the following conclusions with respect to the submitted study:

As concluded in the Residue Chemistry Chapter for the Acephate RED, the reregistration

requirements for data pertaining to magnitude of the residue on cottonseed are fulfilled. RRB1, however, notes that the reregistration requirements for residue data on cotton gin byproducts have not been fulfilled and remain outstanding. The cottonseed residue data from the current submissions may be useful to facilitate the development of anticipated residues.

Residues of acephate and methamidophos did not exceed the reassessed tolerances of 0.5 ppm for acephate per se and 0.1 ppm for methamidophos per se in/on cottonseed following applications of representative acephate formulation(s) to cotton plants according to commercial practices and reflecting a combination of registered use patterns.

Cottonseed processing study: As concluded in the Residue Chemistry Chapter for the Acephate RED, the reregistration requirements for cottonseed processing data are fulfilled. The processing data from the current submission (MRID 44777003) are unacceptable because the study was conducted using cottonseed samples treated at non-exaggerated rates and bearing nondetectable residues of acephate and methamidophos (<0.02 and <0.01 ppm, respectively). No detectable residues were found in cotton fractions (hull, meal, crude oil, refined oil and soapstock) processed from cottonseed samples.

cc: Reviewer(F. Fort), L.Phan(SRRD), Reg. Std. File, RF, SF, Circ.

RDI: Expoteam: 7/21/99: WJHazel:8/9/99

7509C:CBRS:CM#2:Rm804S:305-7478:FAFort/FF:7/21/99

Disk8:acephate.

# **ACEPHATE**

**PC Code 103301; Case 0042**

**(DP Barcodes D254635, D255614 and D254638)**

## **REGISTRANT'S RESPONSE TO RESIDUE CHEMISTRY DATA REQUIREMENTS**

### **INTRODUCTION**

Valent U.S.A. Corporation has submitted data depicting the magnitude of acephate and methamidophos residues in/on cottonseed (MRIDs 44777001, 44777004, and 44791202) and its processed commodities (MRID 44777003) following multiple applications of representative acephate formulation(s) reflecting commercial practices. The results of these studies are summarized and presented in this document.

### **BACKGROUND**

The Residue Chemistry Chapter for the Acephate RED (DP Barcode D238151, F. Fort) was issued 5/14/98. The Chapter concluded that based on the available plant and animal metabolism data, the acephate residues of concern in plant commodities are those that are currently regulated, acephate and its cholinesterase-inhibiting metabolite, methamidophos. The Agency will, however, initiate a change in the residue definition of acephate tolerances for plant commodities in order to eliminate redundancy. The Agency is now recommending that all acephate tolerances be expressed in terms of acephate per se under 40 CFR §180.108. Residues of methamidophos resulting from the metabolism of acephate are more appropriately placed under the tolerance regulations for methamidophos as a pesticide [40 CFR §180.315 (c)]. These changes are needed to achieve compatibility with CODEX, if only in terms of residue definition. Such a change in the residue definition would require deletion of paragraph (d) (8) of 40 CFR §180.3 which states that methamidophos residues may not exceed the higher of the two tolerances established for the use of acephate or methamidophos as a pesticide. A statement should be added to 40 CFR §180.108 explaining that residues of the acephate metabolite methamidophos are regulated under 40 CFR §180.315.

The Residue Chapter also concluded that the reregistration requirements for residue data on cottonseed and its processed commodities are satisfied. Following re-evaluation of the available field trial data, the Chapter recommended that the cottonseed tolerance be lowered from 2 ppm to 0.5 ppm for residues of acephate per se (under 40 CFR §180.108) and 0.1 ppm for residues of methamidophos (under 40 CFR §180.315). Following re-evaluation of the available cottonseed

processing study, adjustments in the tolerance levels for cottonseed hulls and meal were required. Based on the Highest Average Field Trial (HAFT) residue of 0.32 ppm (see PP#3F1375, 8/5/74, R. Schmitt) and maximum concentration factors of 2x and 4x for meal and hulls, respectively. Acephate residues are not expected to exceed the reassessed RAC (cottonseed) tolerance of 0.5 ppm. Finally, the Residue Chapter requires data for 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 result of changes in Table 1 (GLN 860.1000). An appropriate tolerance for cotton gin byproducts should be proposed once acceptable data have been submitted and evaluated.

Adequate methods are available for data collection and tolerance enforcement. For tolerance enforcement, the Pesticide Analytical Manual (PAM), Vol. II lists two GLC methods (designated as Methods I and II) with thermionic detection for the determination of acephate (LOD = 0.01 ppm) and methamidophos (LOD = 0.04 ppm) residues in/on plant and animal commodities. The 1/94 FDA PESTDATA database (PAM Volume I, Appendix I) indicates that acephate is recovered (>80%) by using Multiresidue Methods Section 302 (Luke Method; Protocol D); recovery of methamidophos using the same method is variable.

## CONCLUSIONS AND RECOMMENDATIONS

1. Residue analytical methods. Samples of cottonseed and processed commodities from the submitted field and processing studies were analyzed for residues of acephate and methamidophos using a modified version of GC/FPD Method RM-12A-6. The limits of detection (LOD) were 0.02 ppm for acephate and 0.01 ppm for methamidophos. This method is adequate for the purpose of data collection.
2. Storage stability data. Samples of cottonseed collected from the field trials were analyzed within the intervals for which residues of acephate and methamidophos have been demonstrated to be stable under freezer conditions. Storage stability data are not required for the cottonseed processing study because samples were analyzed within one month of harvest.
- 3a. Cottonseed field trials: As concluded in the Residue Chemistry Chapter for the Acephate RED, the reregistration requirements for data pertaining to magnitude of the residue on cottonseed are fulfilled. RRB1, however, notes that the reregistration requirements for residue data on cotton gin byproducts have not been fulfilled and remain outstanding. The cottonseed residue data from the current submissions may be useful to facilitate the development of anticipated residues.
- 3b. Residues of acephate and methamidophos did not exceed the reassessed tolerances of 0.5 ppm for acephate per se and 0.1 ppm for methamidophos per se in/on cottonseed following applications of representative acephate formulation(s) to cotton plants according to commercial practices and reflecting a combination of registered use patterns.

- 3c. Following a treatment schedule consisting of one in-furrow application made at planting of the 15% G acephate formulation at 9 or 12 lb ai/A (9x or 12x) followed by four foliar broadcast applications of the 75% SC/S acephate formulation at 1 lb ai/A/application (1x) with a 7- to 9-day retreatment interval, residues of acephate and methamidophos were 0.09-0.20 ppm and 0.03-0.05 ppm, respectively in/on treated ginned cottonseed samples collected at a 21-day PHI. (MRID 44777001).
- 3d. Following a treatment schedule using the 90% SC/S acephate formulation and consisting of one in-furrow application made at planting at 1 lb ai/A (1x), one sidedress application made either at blooming or squaring stage at 1.8 lb ai/A (1.8x), followed by six foliar applications at 1 lb ai/A/application (1x) with a 7- to 8-day retreatment interval, residues of acephate and methamidophos were 0.18-0.33 ppm and <0.01 ppm, respectively in/on treated ginned cottonseed samples collected at a 21- or 22-day PHI. (MRID 44777004).
- 3e. Following a treatment schedule using the 90% SC/S acephate formulation and consisting of one in-furrow application made at planting at 1 lb ai/A (1x) followed by 2, 4, 6, 10, or 12 foliar applications at 0.18 to 0.9 lb ai/A/application (0.18-0.9x) with a 7-day retreatment interval, residues of acephate and methamidophos were each less than the respective LODs (0.02 ppm for acephate and 0.01 ppm for methamidophos) in/on all treated ginned cottonseed samples collected at PHIs of 33-105 days. (MRID 44791202).
4. Cottonseed processing study: As concluded in the Residue Chemistry Chapter for the Acephate RED, the reregistration requirements for cottonseed processing data are fulfilled. The processing data from the current submission (MRID 44777003) are unacceptable because the study was conducted using cottonseed samples treated at non-exaggerated rates and bearing nondetectable residues of acephate and methamidophos (<0.02 and <0.01 ppm, respectively). No detectable residues were found in cotton fractions (hull, meal, crude oil, refined oil and soapstock) processed from cottonseed **samples**.

## **DETAILED CONSIDERATIONS**

### **OPPTS GLN 860.1340: Residue Analytical Methods**

Samples of cottonseed and its processed commodities were analyzed for residues of acephate and methamidophos by Valent Dublin Laboratory (Dublin, CA) using a modified version of GC/FPD Method RM-12A-6 with detection limits of 0.02 ppm for acephate and 0.01 ppm for methamidophos. Method RM-12A-6 was used previously for collection of residue data, and has been considered by the Agency to be adequate for data-collection purposes. Briefly, samples of ginned cottonseed were extracted with acetonitrile (ACN):hexane (1:1, v:v) with sodium sulfate and filtered. The extract was partitioned with ACN:hexane. The ACN phase was evaporated to dryness, re-dissolved in ether, and cleaned up by silica gel chromatography. Residues were eluted with 10% methanol in ether. Additional clean up steps were required for cottonseed samples. Two additional silica gel column cleanups were added to obtain satisfactory recoveries. Residues were quantitated using a GC system with a flame photometric detector in the phosphorous mode. To enhance the column sensitivity, a DB-1 column was used to quantitate residues in cottonseed instead of the HP-17 column (50% phenyl and 50% methylpolysiloxane).

Concurrent method recovery data were generated by fortifying untreated ginned cottonseed and processed commodities (hulls, crude and refined oil, and soapstock) with acephate and methamidophos. The results of the concurrent method recovery analyses are presented in Table 1. These data indicate that the GC/FPD method including the modifications is adequate for determining residues of acephate and methamidophos in/on ginned cottonseed and its processed commodities. It is noted that no validation of the method was done at the Limit of Detection (LOD). Adequate raw data, sample calculations, and representative chromatograms were submitted.

Table 1. Concurrent method recoveries of acephate and methamidophos from fortified untreated samples of ginned cottonseed and its processed commodities.

Commodity	MRID	Concurrent Method Recovery	
		Fortification Levels (ppm)	Recovery <sup>a</sup>
<i>Acephate</i>			
Ginned Cottonseed	44791202	0.05, 0.25	93.6; 121; 107.3
	44777001	0.05, 0.25	67.8; 75.7-84.2 (3); 76.8
	44777004	0.05, 0.25	89.8, 97.5; 93.7
Ginned Cottonseed (processing)	44777003	0.05, 0.25	66; 77-122 (10); 122-156 (3); 101
-Hulls	44777003	0.05, 0.25	117; 123; 120
-Crude oil	44777003	0.05, 0.25	102, 117; 110
-Refined oil	44777003	0.05, 0.25	71, 99; 85
-Soapstock	44777003	0.05, 0.25	73, 81 <sup>b</sup> ; 125, 130; 102
<i>Methamidophos</i>			
Ginned Cottonseed	44791202	0.02, 0.10	66.9; 70.4; 68.7
	44777001	0.02, 0.10	77.3-98.0 (4); 88.1
	44777004	0.02, 0.10	79.7, 83.4; 81.6
Ginned Cottonseed (processing)	44777003	0.02, 0.10	61-68 (3); 70-107 (11); 81
-Hulls	44777003	0.02, 0.10	71, 72; 72
-Crude oil	44777003	0.02, 0.10	70, 73; 72
-Refined oil	44777003	0.02, 0.10	60; 86; 73
-Soapstock	44777003	0.02, 0.10	115 (2); 115

<sup>a</sup> Recovery values outside the acceptable range of 70-120 are listed separately; each value represents one sample unless otherwise indicated in parentheses. Average recoveries are bolded.

<sup>b</sup> Concurrent methamidophos recoveries were out of range with the 73 and 81 acephate recovery values.

#### OPPTS GLN 860.1380: Storage Stability Data

Samples of cottonseed from the cotton field trials were either maintained at ambient conditions for one day prior to ginning (MS), placed on blue ice in insulated boxes for 4-24 hours prior to ginning (NC and OK), or were ginned the day of harvest (TX). Ginned samples were frozen and shipped on dry ice or by freezer truck to the analytical laboratory (Valent Dublin Laboratories, Dublin, CA). Samples were stored frozen (-20 C) at Valent Laboratories until residue analysis. The total storage intervals between harvest and analysis of cottonseed samples were 108-208

days (~4-7 months). According to the Residue Chemistry Chapter for the Acephate RED, the combined residues of acephate and methamidophos are stable under frozen storage conditions (-20 C) in/on cottonseed for ~10 months, and the overall database supports the reregistration of acephate on supported commodities. No additional storage stability data are required.

Samples of cottonseed from the processing study were ginned the day of harvest except the MS trial samples which were maintained at ambient conditions one day prior to ginning. Ginned cottonseed samples were stored frozen (~-20 C) as cottonseed macerates for 14-153 days (~1-5 months). RAC samples were shipped on dry ice to the analytical laboratory (Valent Dublin Laboratory, Dublin, CA), where the samples were stored frozen (-20 C) until maceration, extraction, and analysis. A subsample of ginned cottonseed from the MS trial was shipped overnight the day of harvest to Engineering Biosciences Research Center, Texas A&M University (Bryan, TX) for processing. At the processing facility, the collected samples of ginned cottonseed, hulls, crude and refined oil, meal, and hulls were frozen and shipped within 6 days by overnight delivery (FedEx) to Valent Dublin Laboratory (Dublin, CA). At Valent samples were frozen (-20 C) until residue analysis. The total storage intervals for samples from the cottonseed processing study were 11-33 days (~1 month) for the processed commodities. Storage stability data are not required in support of the cottonseed processing study because samples were analyzed within one month of harvest.

#### OPPTS GLN 860.1500: Crop Field Trials

##### Cottonseed

*Established tolerance:* A tolerance of 2 ppm has been established for the combined residues of acephate and its cholinesterase-inhibiting metabolite methamidophos in/on cottonseed [40 CFR §180.108]. The Residue Chemistry Chapter of the Acephate RED reassessed the cottonseed tolerance to 0.5 ppm for acephate per se under 40 CFR §180.108 and 0.1 ppm for methamidophos per se under 40 CFR §180.315.

*Use patterns registered to Valent:* There are several acephate end-use products registered to Valent U.S.A. Corporation which are permitted for use on cotton. These formulations may be applied preplant, at-planting, and postemergence (foliar). A summary of acephate uses on cotton eligible for reregistration was previously tabulated in the Residue Chemistry Chapter for the Acephate RED. These uses are reproduced in Table 2.



Table 2. Summary of acephate uses on cotton subject to reregistration.

Application Timing Application Type Application Equipment	Formulation [EPA Reg. No.]	Maximum Single Application Rate (ai)	Max. No. of Applications Per Season	Max. Seasonal Rate (ai)	Preharvest Interval (Days)	Use Limitations
Preplant Seed hopper box treatment	75% SC/S [59639-26] 90% SC/S [59639-33]	3 oz/A or or 0.2 lb/A	1	Not specified	Not applicable (NA)	Use prohibited in AZ, CA, and the Black Lands of TX. The product should be applied evenly to cottonseed in a hopper/planter box as a dry powder. The processing of treated seed for oil or use for food/feed is prohibited.
Preplant Slurry seed treatment	80% SC/S [59639-29] 85% SC/S [59639-85]	6.4 oz/100 lb seed	1	NS	NA	Seed treatment may be made in a slurry tank or in sufficient water for thorough coverage with approved fungicides. The processing of treated seed for oil or use for food/feed is prohibited.
At-planting In-furrow (with soil incorporation) Ground	15% G [59639-75] [OK950002] 75% SC/S [59639-26] [59639-89] 90% SC/S [59639-33]	1.0 lb/A	1	NS	NA	Liquid formulations should be applied with flat fan nozzles using a minimum of 3 gal of water/A. Application may be made as a tank mix with approved fungicides.
Postemergence Sidedress treatment Ground	90% SC/S [TX940001] [TX960003]	1.0 lb/A	NS	NS	NS	Use limited to all areas of TX except the Black Lands. Application should be made beginning at the fourth or fifth node stage, and should cease after peak bloom (90 days after planting).
Postemergence Foliar Ground/aerial	75% SC/S [59639-26] [59639-89] [AR810050] [OK800012] [TX790014] 90% SC/S [59639-33] [59639-86] [AR890005] [AR890008] [OK890002] [TX910003]	1.0 lb/A	NS	NS	21	Initial application should be made when eggs or insects first appear using a minimum of 10 (ground), 1 (aerial), or 3-5 (aerial in AZ, CA) gal of water/A. An unspecified number of repeat applications may be made at 3- to 7-day intervals or as needed. May be tank-mixed with bifenthrin, chlorpyrifos, cyfluthrin, cypermethrin, esfenvalerate, fenpropathrin, lambda-cyhalothrin, permethrin, and tralomethrin. The feeding of treated forage or gin trash to livestock and the grazing of animals on treated areas are prohibited.

*Discussion of data* (1998; MRID 44777001): Two cottonseed field trials were conducted during the 1996 growing season in NC (Region 2) to determine the magnitude of residues following a treatment regimen consisting of: one in-furrow application made at planting of the 15% G acephate formulation at 9 or 12 lb ai/A (9x or 12x) followed by four foliar broadcast applications of the 75% SC/S acephate formulation at 1 lb ai/A/application (1x) with a 7- to 9-day retreatment interval. [Note: All equivalent rates pertain to the specific types of treatment permitted by registered labels]. Applications were made using ground equipment (tractor mounted sprayer for in-furrow application and backpack sprayer for foliar applications) in 14.2-15.4 gal/A. Two subplots were treated at each site; an additional subplot was not treated and served as a control plot. Mature cottonseed was harvested 21 days following the last of the above-described treatment schedule.

One control and duplicate treated samples were collected from each NC test plot. Cotton samples were placed on blue ice in insulated boxes for four hours prior to ginning. Information pertaining to handling and storage procedures of harvested samples is found in the “Storage Stability Data” section of this document. The ginned cottonseed samples were analyzed for residues of acephate and methamidophos using method RM-12A-6 described in the “Residue Analytical Methods” section. Apparent residues of acephate and methamidophos were each less than the respective LODs (0.02 ppm for acephate and 0.01 ppm for methamidophos) in/on one untreated ginned cottonseed sample. Residues of acephate and methamidophos ranged from 0.09-0.20 ppm and 0.03-0.05 ppm, respectively in/on treated ginned cottonseed samples. Individual residue values from the treatment program are presented in Table 3.

Table 3.

NC Trial	Application Rates <sup>a</sup>	Residues, ppm	
		Acephate	Methamidophos
V-11390-A	In-furrow application at 9.3 lb ai/A followed by four foliar applications at 0.99-1.0 lb ai/A/application	0.12, 0.20	0.03, 0.05
	In-furrow application at 11.8 lb ai/A followed by four foliar applications at 0.99-1.0 lb ai/A/application	0.14, 0.18	0.05, 0.05
V-11390-B	In-furrow application at 8.84 lb ai/A followed by four foliar applications at 1.0 lb ai/A/application	0.11, 0.14	0.04, 0.04
	In-furrow application at 12.3 lb ai/A followed by four foliar applications at 1.0-1.1 lb ai/A/application	0.09, 0.12	0.03, 0.04

<sup>a</sup> At planting in-furrow applications were made either at ~9 lb ai/A (low rate) or ~12 lb ai/A (high rate).

*Discussion of data* (1995; MRID 44777004): Two cottonseed field trials were conducted during the 1994 growing season in OK and TX (Region 8) to determine the magnitude of residues following a sequential treatment regimen using the 90% SC/S acephate formulation. The

treatment schedule consisted of: one in-furrow application made at planting at 1 lb ai/A (1x), one sidedress application made either at blooming or squaring stage at 1.8 lb ai/A (1.8x), followed by six foliar applications at 1 lb ai/A/application (1x) with a 7- to 8-day retreatment interval. Applications were made using ground equipment: 5 gal/A for in-furrow applications, 10-15 gal/A for side dress applications, and 10 gal/A for foliar applications. Mature cottonseed was harvested  $21 \pm 1$  days following the last of the above-described treatment schedule.

One control and duplicate treated samples were collected by hand from each test site. Cotton samples from the OK trial were placed on blue ice in insulated boxes for one day prior to ginning; samples from the TX trial were ginned the day of harvest. The ginned cottonseed samples were analyzed for residues of acephate and methamidophos using method RM-12A-6. Apparent residues of acephate and methamidophos were each less than the respective LODs (0.02 ppm for acephate and 0.01 ppm for methamidophos) in/on two untreated ginned cottonseed. Residues of acephate and methamidophos ranged from 0.18-0.33 ppm and <0.01 ppm, respectively in/on treated ginned cottonseed. The registrant contends that adding a sidedress application at either the blooming or squaring stage to the treatment regimen does not result in significantly higher residues. Individual residue values data from the treatment program are presented in Table 4.

Table 4. Residues of acephate and methamidophos in/on ginned cottonseed

Trial Site	Residues, ppm <sup>1</sup>	
	Acephate	Methamidophos
OK	0.18, 0.20	<0.01, <0.01
TX	0.24, 0.33	<0.01, <0.01

1. Cottonseed harvested 20-21 days following a sequential treatment regimen using the 90% SC/S acephate formulation: one in-furrow application made at planting 1 lb ai/A (1x), one sidedress application made either at blooming or squaring stage at 1.8 lb ai/A (1.8x), followed by six foliar applications at 1 lb ai/A/application (1x) with a 7- to 8-day retreatment interval.

*Discussion of data* (1994; MRID 44791202): A single field trial was conducted during the 1993 growing season in MS (Region 4) to determine the magnitude of residues following a sequential treatment regimen using the 90% SC/S acephate formulation. The treatment schedule consisted of: one in-furrow application made at planting at 1 lb ai/A (1x) followed by multiple foliar applications at 0.18 to 0.9 lb ai/A/application (0.18-0.9x) with a 7-day retreatment interval. All treatment applications ended prior to boll opening with PHIs ranging from 33 to 105 days following the last application. Applications were made using ground equipment (tractor mounted boom sprayer) in 5.1 gal/A for in-furrow spray applications and 9.8-10 gal/A for foliar spray applications. The treatment programs and timing of the applications for the MS field trial are outlined in Table 5; an additional subplot was not treated and served as a control plot (Treatment 1).

Table 5. Treatment programs and timing of MS field trial.

Treatment Number	PHI <sup>a</sup> (days)	Application Rate (lb ai/A/application)	No. and Type of Application	Application Timing (growth stage)
2	105	1.0	1 in-furrow	At planting
		0.18	2 foliar	At 2nd and 5th node
3	90	1.0	1 in-furrow	At planting
		0.18	2 foliar	At 2nd and 5th node
		0.50	2 foliar	At pinhead square and 7 days later
4	77	1.0	1 in-furrow	At planting
		0.18	2 foliar	At 2nd and 5th node
		0.50	2 foliar	At pinhead square and 7 days later
		0.9	2 foliar	At 4th week of squaring and first open bloom
5	49	1.0	1 in-furrow	At planting
		0.18	2 foliar	At 2nd and 5th node
		0.50	2 foliar	At pinhead square and 7 days later
		0.9	2 foliar	At 4th week of squaring and first open bloom
		0.45	4 foliar	At 7 day intervals after open bloom application
6	33	1.0	1 in-furrow	At planting
		0.18	2 foliar	At 2nd and 5th node
		0.50	2 foliar	At pinhead square and 7 days later
		0.9	2 foliar	At 4th week of squaring and first open bloom
		0.45	4 foliar	At 7 day intervals after open bloom application
		0.45	1 foliar	At first cracking boll

<sup>a</sup> PHI (preharvest interval) is the number of days following the last foliar application until harvest.

One control and duplicate treated samples were collected by hand from each MS test plot. Cotton samples were maintained at ambient conditions for one day prior to ginning. The ginned cottonseed samples were analyzed for residues of acephate and methamidophos using method RM-12A-6. Apparent residues of acephate and methamidophos were each less than the respective LODs (0.02 ppm for acephate and 0.01 ppm for methamidophos) in/on untreated (n=1) and all treated (n=2 samples for each treatment program) samples of ginned cottonseed.

Summary of field studies: Residues of acephate and methamidophos did not exceed the reassessed tolerances of 0.5 ppm for acephate per se and 0.1 ppm for methamidophos per se

in/on cottonseed following applications of representative acephate formulation(s) to cotton plants according to commercial practices and reflecting a combination of registered use patterns.

Following a treatment schedule consisting of one in-furrow application made at planting of the 15% G acephate formulation at 9 or 12 lb ai/A (9x or 12x) followed by four foliar broadcast applications of the 75% SC/S acephate formulation at 1 lb ai/A/application (1x) with a 7- to 9-day retreatment interval, residues of acephate and methamidophos were 0.09-0.20 ppm and 0.03-0.05 ppm, respectively in/on treated ginned cottonseed samples collected at a 21-day PHI. (MRID 44777001).

Following a treatment schedule using the 90% SC/S acephate formulation and consisting of one in-furrow application made at planting at 1 lb ai/A (1x), one sidedress application made either at blooming or squaring stage at 1.8 lb ai/A (1.8x), followed by six foliar applications at 1 lb ai/A/application (1x) with a 7- to 8-day retreatment interval, residues of acephate and methamidophos ranged 0.18-0.33 ppm and <0.01 ppm, respectively in/on treated ginned cottonseed samples collected at a 21- to 22-day PHI. (MRID 44777004).

Following a treatment schedule using the 90% SC/S acephate formulation and consisting of one in-furrow application made at planting at 1 lb ai/A (1x) followed by 2, 4, 6, 10, or 12 foliar applications at 0.18 to 0.9 lb ai/A/application (0.18-0.9x) with a 7-day retreatment interval, residues of acephate and methamidophos were each less than the respective LODs (0.02 ppm for acephate and 0.01 ppm for methamidophos) in/on all treated ginned cottonseed samples collected at PHIs of 33-105 days. (MRID 44791202).

## **OPPTS GLN 860.1520: Processed Food/Feed**

### Cottonseed Processing Study

Discussion of data (1994; MRID 44777003): Five cottonseed field trials were conducted during the 1993 growing season in AR (Region 4), CA (Region 10), LA (Region 4), MS (Region 4), and TX (Region 6). Mature cottonseed was harvested 88-113 days following a single at planting in-furrow application of the 90% SC/S acephate formulation at 1 lb ai/A followed by two sequential foliar applications at 0.18 lb ai/A/application at the second and fifth node growth stages. A subplot at each trial site received a second treatment regime which included two additional foliar applications at 0.45 or 0.9 lb ai/A/application at the pinhead square growth stage and 7 to 8 days later. Applications were made using ground equipment.

Replicate untreated and treated samples of cottonseed were harvested by hand or by mechanical harvester, and ginned the day of harvest except the MS trial samples which were maintained at ambient conditions one day prior to ginning. Ginned cottonseed samples were stored frozen. RAC samples were shipped on dry ice to the analytical laboratory (Valent Dublin Laboratory, Dublin, CA), where the samples were stored frozen (-20 C) until maceration, extraction, and analysis. A subsample of ginned cottonseed from the MS trial was shipped overnight following ~2 months of frozen storage at the field to Engineering Biosciences Research Center, Texas A&M University (Bryan, TX) for processing. Cottonseed samples were processed to hulls, crude and refined oil, meal, and soapstock using a small-scale processing procedure representative of industrial practices. Briefly, ginned cottonseed samples were saw-delinted to remove a majority of the lint. The delinted seed was cracked mechanically and screened to separate the hull and kernel material. The kernel was heated, flaked, and expanded prior to exposure to hexane to remove the crude oil from the collets. Solvent was evaporated from the spent collets (meal) with warm forced air. The crude oil and hexane extract was adjusted to the proper ratio, and the crude oil then miscella-refined yielding refined oil and soapstock. The registrant submitted adequate descriptions and material balance sheets for the processing procedures.

At the processing facility, the collected samples of ginned cottonseed, hulls, crude and refined oil, meal, and hulls were frozen and shipped within 6 days by overnight delivery (FedEx) to Valent Dublin Laboratory (Dublin, CA). At Valent samples were frozen (-20 C) until residue analysis. Total storage intervals for samples from the cottonseed processing study were 11-33 days (~1 month) for the processed commodities and 14-153 days (~1-5 months) for ginned cottonseed (RAC).

Samples of cottonseed and the processed commodities were analyzed for residues of acephate and methamidophos using method RM-12A-6. Residues of acephate and methamidophos were each less than the respective LODs (0.02 ppm for acephate and 0.01 ppm for methamidophos) in/on five untreated ginned cottonseed samples and twenty treated ginned cottonseed samples from all treatment regimes. Only cottonseed from one field trial was processed. Residues of acephate and methamidophos were less than the respective LODs in/on one sample each of hull, meal, crude oil, refined oil and soapstock processed from cottonseed samples harvested 91 days following the treatment schedule described above.

*Study summary:* This study is not acceptable because no detectable residues of acephate and methamidophos were found on the cottonseed samples treated at non-exaggerated rates. The Residue Chemistry Test Guidelines (OPPTS 860.1000) specifically state that RAC samples used in processing studies should contain field treated quantifiable residues so that the potential for concentration in processed commodities can be determined. This may require field treatment at exaggerated application rates to obtain sufficient residue levels. Residues did not concentrate in cotton fractions (hull, meal, crude oil, refined oil and soapstock) processed from cottonseed samples which were treated at non-exaggerated rates and bearing nondetectable residues of acephate and methamidophos (<0.02 and <0.01 ppm, respectively).

#### AGENCY MEMORANDA CITED IN THIS REVIEW

DP Barcode: D238151  
Subject: Acephate. Product and Residue Chemistry Chapters for the Reregistration Eligibility Decision  
From: F. Fort  
To: A. Chiri and N. Zahedi  
Dated: 5/14/98

#### MASTER RECORD IDENTIFICATION NUMBERS

Citations for the MRID documents referred to in this review are presented below.

44777001 Lai, J. (1998) Magnitude of the Residues of Acephate In/On Cottonseed Following In-Furrow and Foliar Applications of Orthene® Insecticide. Unpublished Study prepared by Valent U.S.A. Corp. 237 p.

44777003 Lai, J. (1995) Magnitude of Acephate Residues In/On Cotton and Processed Cotton Parts Following Treatment with Orthene® 90 S. Unpublished Study prepared by Valent U.S.A. Corp. 511 p.

44777004 Lai, J. (1995) Magnitude of Residues of Acephate In/On Ginned Cottonseed Following In-Furrow, Side Dress and Foliar Applications with Orthene® 90 S. Unpublished Study prepared by Valent U.S.A. Corp. 174 p.

44791202 Lai, J. (1994) Magnitude of Acephate Residues In/On Cotton Treated at Different Growth Stages with Orthene® 90S. Unpublished Study prepared by Valent U.S.A. Corp. 153 p.