



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

197

OCT 12 1993

OFFICE OF  
PREVENTION, PESTICIDES AND  
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Mepiquat chloride on Cotton. Proposed Increases in the Maximum Seasonal Rate. MRID #'s 424268-00, -01, -02, and -03, CBTS #'s 10671, 10689, and 10690, DP Barcode #'s 183140, 183187, and 183197.

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TO: Cynthia Giles-Parker/Dolphine Wilson, PM-22  
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and

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RD has requested CBTS to comment on proposed label increases for the maximum seasonal rate of mepiquat chloride [N,N-dimethyl-piperidinium chloride] on cotton (See Proposed Use, this memo). The registrant, BASF Corporation, has also submitted 3 new studies (MRID #'s 424268-01, -02, and -03) to satisfy reregistration data gaps [additional field trials and a processing study previously requested (See DEB Transmittal Sheet Phase 4 Review, 1/15/91, S. Frank)] for mepiquat chloride. These studies will be reviewed in this memo to determine their adequacy to fill data gaps and to determine if sufficient residue data have been submitted, both from



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previous submissions and these new submissions, to support the proposed label increases. A data evaluation report of these studies (MRID #'s 424268-01, -02, and -03) has been performed by Dynamac Corporation under the supervision of CBTS, HED. These reports have undergone secondary review in the branch and have been incorporated into this memo. Conclusions are solely those of CBTS.

Tolerances are established for cotton forage (3.0 ppm) and cottonseed (2.0 ppm), livestock meat, fat, and meat byproducts (0.1 ppm), milk (0.05 ppm), and eggs (0.05 ppm) (40 CFR §180.384). A 3.0 ppm feed additive tolerance is established on cottonseed meal (40 CFR §186.2275).

#### Recommendations:

TOX considerations permitting, CBTS can recommend for either of the proposed label increases in the maximum seasonal rate of mepiquat chloride [N,N-dimethyl-piperidinium chloride] on cotton, i.e., from the present label maximum of 0.066 lb a.i./A to the proposed label maximum of 0.088 lb a.i./A or 0.132 lb a.i./A.

CBTS has also determined that no additional field or processing residue data is required to support the registered use of mepiquat chloride on cotton. However, a feed additive petition needs to be submitted proposing an increase in the feed additive tolerance for cottonseed meal to 4 ppm.

#### Conclusions:

1. Analytical Method No. A9106 is adequate for the determination of mepiquat chloride residues in/on cottonseed. This method will be sent to FDA to be included as a letter method in PAM II. However, if the petitioner wants this method to replace the current PAM II method, then he must submit a laboratory validation of this method from an independent laboratory for CBTS review. The method must also be validated in the Agency laboratory in Beltsville, MD before the adequacy of this method for enforcement purposes can be determined by CBTS.
2. The proposed label restriction: "Do not graze or feed forage to livestock", is acceptable based upon the present agricultural cotton growing cultural practices.
- 3a. Adequate field residue data have been submitted to support the proposed label increases of the maximum rate of mepiquat chloride (to include ULV aerial applications with oil as diluent) from the present label maximum of 0.066 lb a.i./A to the proposed label maximum of 0.088 lb a.i./A (1.3X) or 0.132 lb a.i./A (2X). The established 2.0 ppm tolerance will cover any mepiquat chloride residues from the proposed label increases.

- 3b. Adequate storage stability data for the field trials have been submitted to support the proposed label increases of mepiquat chloride on cotton.
- 3c. No additional field trial residue data are necessary to support the registered use of mepiquat chloride on cotton.
- 4a. Processing residue data indicate that mepiquat chloride residues concentrated approximately 2x in cottonseed meal, and about 1.5X in undelinted seed. Based upon this data, the established feed additive tolerance of 3.0 ppm for cottonseed meal must be increased to 4.0 ppm. A feed additive petition must be submitted.
- 4b. Adequate storage stability data for the processing study have been submitted to support the proposed label increases of mepiquat chloride on cotton.
- 4c. No additional processing residue data are necessary to support the registered use of mepiquat chloride on cotton.
5. Any secondary mepiquat chloride residues that might occur from the proposed label increases will be adequately covered by the established tolerances for livestock, meat, fat, and meat byproducts (0.1 ppm), milk (0.05 ppm), and eggs (0.05 ppm).

Detailed Considerations:

Current Use:

**PIX® Formulation (4.2% a.i.)**

Up to four low-rate multiple applications should be made each season, generally using 1/8 - 1/4 pt./A (0.0055 - 0.011 lb a.i./A) each time. After the first application, the rate and timing of subsequent application(s) will depend partially on growing conditions; under good growing conditions additional treatments (1/8 - 1/4 pt/A) should be made at 7-14 day intervals. If new growth at any time is excessive, 1/4 - 1/2 pt/A (0.011 - 0.022 lb a.i./A) can be used. Total use per season should not exceed 1-1/2 pt/A (0.066 lb a.i./A).

**Water diluent:** When applying with ground equipment, in all states except CA, use a minimum of 10 gallons of water/A; in CA use 20 gallons/A. For aerial application, in all states but CA use a minimum of 3 gallons of water/A; in CA use a minimum of 3 gallons/A. **Oil diluent:** Ultra low volume (ULV) aerial applications are allowed only in AL, AR, FL, GA, LA, MO, MS, NC, OK, SC, TN, and TX. The minimum total final spray volume is 2 pt/A.

Label restrictions: "Do not graze or feed forage to livestock within 30 days of application, or after applying in oil as a ULV application by air. Do not apply within 30 days of harvest."

**PIX® Concentrate (22.5% a.i.) (EUP permit: 769-EUP-EI)**

Up to four low-rate multiple applications should be made each season, generally using 0.35 - 0.70 fl. oz./A (0.0055 - 0.011 lb a.i./A) each time. After the first application, the rate and timing of subsequent application(s) will depend partially on growing conditions; under good growing conditions additional treatments (0.35 - 0.70 fl. oz./A) should be made at 7-14 day intervals. If new growth at any time is excessive, 0.70 - 1.40 fl. oz./A (0.011 - 0.022 lb a.i./A) can be used. Total use per season should not exceed 4.2 fl. oz./A (0.066 lb a.i./A).

Water diluent: When applying with ground equipment, in all states except CA, use a minimum of 10 gallons of water/A; in CA use 20 gallons/A. For aerial application, in all states but CA use a minimum of 3 gallons of water/A; in CA use a minimum of 3 gallons/A. Oil diluent: Ultra low volume (ULV) aerial applications are allowed only in MS, NC, and TX for the EUP. The minimum total final spray volume is 2 pt/A.

Label restrictions: "Do not graze or feed forage to livestock within 30 days of application, or after applying in oil as a ULV application by air. Do not apply within 30 days of harvest."

Proposed Use:

**PIX® Formulation (4.2% a.i.)**

**DP Barcode #183140:** Single application rates are the same as above, but the total use per season would increase by 1/2 pt/A/season. The proposed maximum total use per season should not exceed 2 pt/A (0.088 lb a.i./A).

Label restrictions: Same as current label.

**DP Barcode #183187:** Single application rates of 1/8 to 1/2 pt/A using up to five low-rate applications are allowed, and the total use per season would increase by 1-1/2 pt/A (0.066 lb a.i.)/season. If new growth at any time is excessive, 1/4 - 3/4 pt/A (0.011 - 0.033 lb a.i./A) can be used. Late season application rates range from 1/2 pt/A (0.022 lb a.i.) to 1-1/2 pt/A (0.066 lb a.i.). The proposed maximum total use per season should not exceed 3 pt/A (0.132 lb a.i./A).

Label restrictions: "Do not graze or feed forage to livestock. Do not apply within 30 days of harvest."

**PIX® Concentrate (22.5% a.i.)**

**DP Barcode #183197:** Up to five low-rate multiple applications can be made each season, using 0.35 - 1.4 fl. oz./A (0.0055 - 0.022 lb a.i./A) each time. After the first application, the rate and timing of subsequent application(s) will depend partially on growing conditions; under good growing conditions additional treatments [0.35 - 0.70 fl. oz./A (0.0055 - 0.011 lb a.i.)] should be made at 7-14 day intervals. If new growth at any time is excessive, 0.70 - 2.1 fl. oz./A (0.011 - 0.033 lb a.i./A) can be used. Late season applications rate range from 1.4 - 4.2 fl. oz./A (0.022 - 0.066 lb a.i./A). Total use per season (early plus late applications) must not exceed 8.4 fl. oz./A (0.132 lb a.i./A).

**Water diluent:** When applying with ground equipment, in all states except CA, use a minimum of 2 gallons of water/A; in CA use 5 gallons/A. For aerial application, in all states but CA use a minimum of 2 gallons/A of water/A; in CA use a minimum of 5 gallons/A. **Oil diluent:** Ultra low volume (ULV) aerial applications are allowed only in AL, AR, FL, GA, LA, MO, MS, NC, OK, SC, TN, and TX. The minimum total final spray volume is 2 pt/A.

Label restrictions: "Do not graze or feed forage to livestock. Do not apply within 30 days of harvest."

The proposed label restriction: "Do not graze or feed forage to livestock", is acceptable based upon the present agricultural cotton growing cultural practices (See Magnitude of the Residue, this memo).

#### Analytical Methodology:

BASF Corporation submitted (MRID #42426801) an analytical method (BASF Method No. A9106) for the determination of mepiquat chloride residues in/on cottonseed by ion chromatography (IC).

Cottonseed samples are manually homogenized prior to subsampling and extraneous materials are removed. The petitioner cautions that mechanical milling of seed samples should be avoided because it causes separation of the seed and lint. After homogenization, a 25-gram subsample of seed is extracted with a 25% solution of 5 N hydrochloric acid in methanol in a high speed blender with added celite. The homogenate is vacuum-filtered through a 1- to 2-cm pad of celite on filter paper. The filtrate is collected and the extracted solids are rinsed once with methanol:distilled water (2:1, v:v) and filtered. The extracted solids, celite, and filter paper are placed in the blender and extracted with a 25% solution

of 5 N hydrochloric acid in methanol, filtered, and rinsed twice with methanol:distilled water (2:1, v:v). The filtrates are combined and adjusted to pH 10-12 with 12 N sodium hydroxide. The solution is permitted to stand for 15-30 minutes to allow a precipitate to form, and then is vacuum-filtered through a layer of celite. The remaining solids are rinsed twice with methanol:distilled water as described above and filtered. The filtrates are combined and the solvents are removed by rotary evaporation at 50-60 C.

Water is added to the extract which is then extracted sequentially with hexane and dichloromethane; the organic phases are discarded after partitioning, but any emulsion remaining after dichloromethane extraction is retained with the aqueous fraction. The aqueous phase is extracted three times with a dipicrylamine solution. After each of the first two extractions, the organic phase is collected and any emulsion is left with the aqueous phase. After the third extraction, the organic phase is collected and any emulsion layer is passed through phase separation paper. The emulsion filtrate is combined with the collected organic layers and the alkaline water phase is discarded. Organosoluble residues are extracted with 2 M hydrochloric acid and the organosoluble fraction is discarded and any emulsion layer is retained with the aqueous phase. The aqueous phase is then rinsed with dichloromethane and any emulsion layer is retained with the aqueous phase.

The aqueous-soluble residues are rotary evaporated to dryness at 55-65 C and then mixed with alumina (activity level IV). Acetone:methanol (95:5, v:v) is added and the mixture is sonicated until no particles remain adhered to the inside of the mixing flask. After the alumina residues have settled, the acetone:methanol solution is decanted. The alumina is rinsed again with the same solvent and the rinsates combined. The alumina residue is dried under a gentle stream of nitrogen or air. Dried alumina residues are applied to a column containing a slurry consisting of alumina (activity level II) in acetone:methanol (95:5, v:v). The solvent level is 8-10 cm above the alumina level in the column. The decanted acetone:methanol rinses are added to the column and eluates are collected until the solvent level is within 0.5-1 cm of the alumina. The flask that contained the dried alumina residues is rinsed with acetone:methanol and the rinse is applied to the column and the eluates are collected as previously described. A third volume of acetone:methanol is added to the column and eluates are collected until the column runs dry. All collected eluates are combined and rotary evaporated to dryness at 40-50 C. The dried residues are dissolved in methanol and evaporated to dryness under a gentle stream of nitrogen at approximately 50 C.

The dried residues are redissolved in deionized water and analyzed by ion chromatography (IC) using an isocratic mobile phase consisting of 2 mM hexanesulfonic acid with 5% acetonitrile in water. Residues are separated on an NG-1 Ionpac precolumn and NS-1

Ionpac main column equipped with a cation suppressor. Mepiquat chloride residues are detected by conductivity detection. The column is regenerated with ca. 25 mM tetrabutylammonium hydroxide in deionized water. The quantitation limit for mepiquat chloride residues in cottonseed is 0.1 ppm.

Mepiquat chloride residues are quantified by comparison of the analyte peak height against a standard curve of peak heights generated using a mepiquat chloride reference standard. Four different standard concentrations (e.g. 0.25, 0.5 ppm, 1.0 ppm, and 2.0 ppm) are used to generate the standard curve. The petitioner has provided representative chromatograms for the standard curve and representative calculations.

For the method validation, subsamples of untreated cottonseed are fortified with mepiquat chloride and analyzed by the above method. Recoveries of mepiquat chloride from fortified cottonseed samples are presented in following table:

| Recoveries of Mepiquat Chloride from Fortified Cottonseed Samples |                |                               |
|---|----------------|-------------------------------|
| Fortified Level (ppm) <sup>a</sup>                                | No. of Samples | Percent Recovery <sup>b</sup> |
| 0.10  | 9              | 75-96                         |
| 0.50  | 5 (1)          | 72-87 (48)                    |
| 2.0   | 9              | 65-83                         |

<sup>a</sup> Fortifications are added prior to extraction and are run concurrently with control samples; six control samples gave <0.1 ppm.

<sup>b</sup> The petitioner stated that recoveries are corrected for any residues found in the untreated samples.

Analytical Method No. A9106 is adequate for the determination of mepiquat chloride residues in/on cottonseed. This method will be sent to FDA to be included as a letter method in PAM II. If the petitioner wants this method to replace the current PAM II method as the enforcement method, then he must submit a laboratory validation of this method from an independent lab. After submission of this independent validation, the method must be validated in the Agency laboratory in Beltsville, MD before the adequacy of this method for enforcement purposes can be determined by CBTS.

Magnitude of the Residue:

BASF Corporation has submitted data (MRID # 424268-02) from 13 field trials conducted in AL(1), AR(1), AZ(1), CA(2), GA(1), LA(1), MS(1), NC(1), TN(1), and TX(3). Geographic representation is adequate since the test states of AL(2%), AR(7%), AZ(6%), CA(18%), GA(2%), LA(7%), MS(12%), NC(2%), TN(3%), and TX(33%) accounted for approximately 90% of the 1990 U.S. cottonseed production (Agricultural Statistics 1991, USDA). Cotton plants were treated with three foliar applications of the 0.35 lb/gal FlC formulation (PIX<sup>®</sup> Plant Regulator; EPA Reg. No. 7969-52) according to the following schedule: (1) 0.011 lb a.i./A at the match-head square stage; (2) 0.055 lb a.i./A at the early bloom stage; and (3) 0.066 lb a.i./A at 30-33 days prior to harvest. The total of these applications was equivalent to 1X the maximum proposed seasonal application rate (0.132 lb a.i./A) for the 0.35 lb/gal FlC formulation. Applications were made using ground equipment (CO<sub>2</sub> backpack sprayer) in 15-20 gal/A of finished spray. No tests were reported for ULV or aerial applications in this residue data submission. Samples of cottonseed were collected at maturity, 30-33 days following the final application.

According to Table II of the Subdivision O Guidelines, the raw agricultural commodities of cotton include seed and forage. However, the petitioner has stated that cotton forage was not analyzed because the proposed label will bear a feeding/grazing restriction. The current label specifies a 30-day pregrazing/feeding interval. This restriction is not in conflict with current agricultural practices in cotton farming. According to Dr. William F. Lalor (Cotton, Inc., 919-782-6330, private communication, J. Stokes, 6/17/93) most cotton-growing states require that the material remaining after boll harvest must be plowed under for insect control. In the state of CA, although the cotton plant is twice the size and has a lot more foliage, the state "plow-down" law (with high fines for offenders) prevents livestock grazing or feeding of the postharvest plant. He knows of only some Texas cotton-growing areas that do not have this "plow-down" requirement, but the postharvest plant in this area is basically only stems, and not useful as forage.

Therefore, CBTS has determined that the proposed label restriction "Do not graze or feed forage to livestock", is acceptable based upon the present agricultural cotton growing cultural practices.

The apparent residues of mepiquat chloride were <0.1 ppm (nondetectable) in/on 12 untreated control samples. One additional control sample from NC showed detectable residues of mepiquat chloride of 0.9 ppm. The petitioner indicated that control and treated samples from the NC test site may have been interchanged since the control sample had quantifiable residues and the treated samples had no quantifiable residues.



| Mepiquat Chloride Residues on Cottonseed After 3 Foliar Applications |     |           |              |                         |                  |
|--|-----|-----------|--------------|-------------------------|------------------|
| Sample No.   | Loc | lb a.i./A | PHI, in days | Mepiquat chloride (ppm) | Storage, in days |
| 90086-2  | CA  | 0.132     | 30           | 0.86                    | 282              |
| 90086-2dup   | CA  | -         | -            | 0.72                    | 289              |
| 90087-2  | CA  | 0.132     | 30           | 1.19                    | 286              |
| 90087-2dup   | CA  | -         | -            | 1.4                     | 296              |
| 90088-2  | AZ  | 0.132     | 30           | 0.36                    | 269              |
| 90089-2  | TX  | 0.132     | 33           | 0.48                    | 263              |
| 90090-2  | TX  | 0.132     | 32           | 0.27                    | 263              |
| 90091-2  | TX  | 0.132     | 30           | 1.49                    | 365              |
| 90091-2dup   | TX  | -         | -            | 1.33                    | 375              |
| 90092-2  | MS  | 0.132     | 30           | 0.79                    | 293              |
| 90093-2  | LA  | 0.132     | 30           | 1.66                    | 306              |
| 90093-2dup   | LA  | -         | -            | 1.62                    | 316              |
| 90094-2  | AR  | 0.132     | 30           | 0.44                    | 301              |
| 90095-2  | TN  | -         | -            | 0.84                    | 299              |
| 90096-2  | AL  | 0.132     | 30           | 0.44                    | 314              |
| 90097-2  | GA  | 0.132     | 30           | 0.43                    | 303              |
| 90098-1  | NC  | 0.132     | 30           | 0.73                    | 337              |
| 90098-2dup   | NC  | -         | -            | 1.25                    | 313              |
| 90098-2dup   | NC  | -         | -            | 1.02                    | 337              |
| 90098-2dup   | NC  | -         | -            | 0.65                    | 337              |

Residues of mepiquat chloride are analyzed by the method described above. Representative chromatograms and sample calculations of residues are provided.

| Recoveries of Fortified Mepiquat Chloride from Cottonseed Samples. |                |                               |
|--|----------------|-------------------------------|
| Fortified Level (ppm) <sup>a</sup>                                 | No. of Samples | Percent Recovery <sup>b</sup> |
| 0.10   | 6              | 68-103                        |
| 1.0  | 4              | 79-90                         |
| 2.0  | 2              | 78, 80                        |

<sup>a</sup> Fortifications were added prior to extraction and were run concurrently with control samples.

<sup>b</sup> The petitioner stated that recoveries were corrected for any residues found in the untreated samples.

Previously, CBTS has reviewed field residue data submitted in PR-160 (Acc#096637) (See memo of 3/21/78, M. Nelson) and in PR-191 (Acc#098032) (See memo of 7/19/83, A. Smith).

Data in PR-160 were reported for 50 field trials conducted in the states of AR (4), AZ (9), CA (15), GA (5), NC (4), and MS (13). The seasonal application totals of mepiquat chloride ranged from 0.019 to 0.132 lb a.i./A, with PHI's from 43 to 175 days. Residues ranged from <0.10 to 1.05 ppm. One sample was provided for a 0.99 lb a.i./A application, 71 day PHI, 4.26 ppm. The total mepiquat chloride application of at least 0.088 lb a.i./A (label rate of 1.5X the current maximum 0.066 lb a.i./A rate) was only in 10 trials, while the 0.132 lb a.i./A (2X the current maximum label rate) was only used in 5 of these 10 trials. However, none of this data can be used to support a 30 day PHI.

Data in PR-191 were reported for 41 field trials [25 with either commercial or CO<sub>2</sub> backpack ground applications (15 - 20 gpa), and 16 by aerial applications (3 - 6 gpa) conducted in the states of AL (2), AR (3), LA (3), MO (1), MS (2), OK (4), SC (4), and TX (22)]. The seasonal application totals of mepiquat chloride ranged from 0.022 to 0.176 lb a.i./A, with PHI's from 8 to 131 days. Residues ranged from <0.10 to 1.63 ppm. The total mepiquat chloride application of at least 0.088 lb a.i./A (label rate of 1.5X the current maximum 0.066 lb a.i./A rate) was in 18 trials (PHI's from 8 to 79 days), while the 0.132 lb a.i./A (2X the current maximum label rate) was used in 5 (PHI's from 28 to 39 days) of these 18 trials. There appeared to be no difference in mepiquat chloride residues in cottonseed between ground vs. aerial application, or ground application with commercial equipment vs. CO<sub>2</sub> backpack or other type of hand sprayer.

Residue data from 7 aerial field trials conducted in the states of AR (1), MS (2), SC (1), and TX (3) have been submitted [Acc#147113,

Report no. PR-259, "Determination of Mepiquat Chloride and its Metabolite Residues in Cottonseed (Comparison of Aerial Treatment in Water and Oil)". The application rate involved the first treatment at 0.044 lb a.i./A, followed by a second application at 0.022 lb a.i./A at 15 to 37 days. Samples were taken for analysis with PHI's ranging from 35 to 88 days. Mepiquat chloride residues are given in the following table.

| Mepiquat Chloride and its Metabolite Residues in Cottonseed. Aerial Treatments in Water and Oil. |                       |       |       |             |      |                           |                   |      |
|--|-----------------------|-------|-------|-------------|------|---------------------------|-------------------|------|
| Loc  | Appl. rate, lb a.i./A |       |       | Aerial, gpa |      | PHI, in days <sup>a</sup> | Residue (ppm)     |      |
|  | #1                    | #2    | total | water       | oil  |                           | water             | oil  |
| AR   | 0.044                 | 0.022 | 0.066 | 5           | 0.25 | 48(75)                    | 0.85 <sup>b</sup> | 1.0  |
| MS   | 0.044                 | 0.022 | 0.066 | 5           | 0.25 | 57(76)                    | 0.76              | 0.30 |
| MS   | 0.044                 | 0.022 | 0.066 | 5           | 0.25 | 60(82)                    | 0.13              | 0.18 |
| SC   | 0.044                 | 0.022 | 0.066 | 5           | 0.25 | 88(105)                   | 0.57              | 0.71 |
| TX   | 0.044                 | 0.022 | 0.066 | 5           | 0.25 | 35(56)                    | 0.43              | 1.2  |
| TX   | 0.044                 | 0.022 | 0.066 | 5           | 0.25 | 41(56)                    | 1.7               | 0.80 |
| TX   | 0.044                 | 0.022 | 0.066 | 5           | 0.25 | 49(72)                    | 0.38              | 0.34 |

<sup>a</sup> Numbers in ()'s are interval days from first treatment.

<sup>b</sup> Average from 3 determinations.

Of the 7 trials, 4 showed no differences with water vs. oil as the diluent. In 2 trials the measured residues were twice as high in the water application vs. the oil, while only in 1 trial were residue values higher in the ULV oil application vs. the water application. Therefore, it appears that mepiquat chloride residues in cottonseed will be similar whether the aerial application is performed with water or oil. No additional data are needed for ULV oil applications of mepiquat chloride on cotton, because data from water applications will adequately cover any residues that will occur with the aerial ULV oil applications.

The data from the above trials (MRID#424268-02, PR-191, and PR-259) can be used to support either of the proposed label increases, i.e., 1.3X (0.088 lb a.i./A) or 2X (0.132 lb a.i./A).

#### Storage Stability Data for Field Trials

The petitioner cited a previously submitted storage stability study "Freezer Storage Stability of BAS 083 in Cottonseed, Cotton Forage,

and Soil" [MRID # 00140864 (submitted 1979), but reformatted and submitted under MRID#92091025]) depicting the stability of mepiquat chloride in an unspecified number of cottonseed samples fortified at 1.0 ppm and stored at <-5 C for 25 months. Recovery was 86% from the stored sample with concurrent method recovery of 84%.

All field samples from the current study were stored frozen at unspecified temperatures within 24 hours of harvest. Samples were shipped frozen to the analytical laboratory (BASF Corporation Research Triangle Park, NC), and upon receipt were stored at -5 C until analyzed. Field samples were stored frozen for 9-13 months between harvest and analysis. These data show that mepiquat chloride residues are stable in the cottonseed matrix for up to 25 months.

Processing Study:

BASF submitted a processing study of cottonseed (MRID#42426803) depicting mepiquat chloride residues in processed fractions of cottonseed from two tests, one each in CA and LA. Cottonseed was harvested 74 days (CA) and 63 days (LA) following a single foliar application of the 0.35 lb/gal FLC formulation (PIX<sup>®</sup> Plant Regulator; EPA Reg. No. 7969-52) at 0.2 lb ai/A. This rate is equivalent to 1.5x the maximum proposed seasonal application rate (0.132 lb ai/A) for the 0.35 lb/gal FLC formulation. Applications were made using ground equipment (CO<sub>2</sub> backpack sprayer) in 20 gal/A of finished spray.

The harvested cotton was processed prior to shipment to BASF for residues analysis. Briefly, the cotton was ginned and delinted. The resulting delinted seed was mechanically cracked and screened to remove hulls. The resulting kernels were heated, flaked, and were extracted with hexane to remove crude oil. Spent flakes were desolventized by with warm forced air to generate meal. The hexane:crude oil (60:40, v:v) mixture was micella refined and then mixed with sodium hydroxide at 50 C. After refining, the refined oil and hexane were separated from the precipitated soapstock. Soapstock was slightly heated to remove solvent. Refined oil and hexane were separated by a laboratory evaporator.

| Mepiquat Chloride Residues in Cottonseed After 0.2 lb a.i./A |     |               |              |                         |                  |
|--|-----|---------------|--------------|-------------------------|------------------|
| Sample No.   | Loc | RAC           | PHI, in days | Mepiquat chloride (ppm) | Storage, in days |
| 6  | CA  | Cottonseed    | 74           | 2.79                    | 300              |
| 6dup   | CA  | Cottonseed    | 74           | 3.11                    | 300              |
| 8  | CA  | Delinted seed | 74           | 3.05                    | 302              |

| Mepiquat Chloride Residues in Cottonseed After 0.2 lb a.i./A |     |               |              |                         |                  |
|--|-----|---------------|--------------|-------------------------|------------------|
| Sample No.   | Loc | RAC           | PHI, in days | Mepiquat chloride (ppm) | Storage, in days |
| 8dup   | CA  | Delinted seed | 74           | 3.35                    | 302              |
| 10   | CA  | Hulls         | 74           | 0.6                     | 306              |
| 10dup  | CA  | Hulls         | 74           | 0.62                    | 306              |
| 14   | CA  | Meal          | 74           | 5.27                    | 310              |
| 14dup  | CA  | Meal          | 74           | 4.75                    | 310              |
| 16   | CA  | Crude oil     | 74           | <0.1                    | 562              |
| 16dup  | CA  | Crude oil     | 74           | <0.1                    | 562              |
| 18   | CA  | Refined oil   | 74           | <0.1                    | 329              |
| 18dup  | CA  | Refined oil   | 74           | <0.1                    | 329              |
| 22   | CA  | Soapstock     | 74           | 0.26                    | 367              |
| 22dup  | CA  | Soapstock     | 74           | 0.26                    | 367              |
| 22dup  | CA  | Soapstock     | 74           | 0.28                    | 373              |
| 6  | LA  | Cottonseed    | 63           | 1.92                    | 308              |
| 6dup   | LA  | Cottonseed    | 63           | 2.24                    | 308              |
| 8  | LA  | Delinted seed | 63           | 2.88                    | 310              |
| 8dup   | LA  | Delinted seed | 63           | 2.86                    | 310              |
| 10   | LA  | Hulls         | 63           | 0.81                    | 314              |
| 10dup  | LA  | Hulls         | 63           | 0.6                     | 314              |
| 14   | LA  | Meal          | 63           | 4.02                    | 318              |
| 14dup  | LA  | Meal          | 63           | 4.55                    | 318              |
| 16   | LA  | Crude oil     | 63           | 0.1                     | 570              |
| 16dup  | LA  | Crude oil     | 63           | 0.11                    | 570              |
| 18   | LA  | Refined oil   | 63           | <0.1                    | 337              |
| 18dup  | LA  | Refined oil   | 63           | <0.1                    | 337              |
| 22   | LA  | Soapstock     | 63           | 1.05                    | 375              |
| 22dup  | LA  | Soapstock     | 63           | 1.06                    | 375              |
| 22dup  | LA  | Soapstock     | 63           | 0.78                    | 375              |

These data indicate that mepiquat chloride residues concentrated approximately 2x in cottonseed meal, and about 1.5X in delinted seed. Based upon this data, the established feed additive tolerance of 3.0 ppm for cottonseed meal must be increased to 4.0 ppm. A feed additive petition must be submitted.

In addition, any secondary mepiquat chloride residues that might occur from these proposed labels will be adequately covered by the established tolerances for livestock, meat, fat, and meat byproducts (0.1 ppm), milk (0.05 ppm), and eggs (0.05 ppm).

Residues of mepiquat chloride were analyzed by the method described above. Representative chromatograms and sample calculations of residues were provided by the petitioner. Procedural recoveries were also provided along with storage stability data and are presented as follows:

| Recoveries from Fortified Cottonseed Processed Fractions |     |               |                         |                     |
|--|-----|---------------|-------------------------|---------------------|
| Sample No.   | Loc | RAC           | Mepiquat chloride (ppm) | Fortification (ppm) |
| 90136-7  | CA  | Cottonseed    | 0.1, 5.00               | 83, 82              |
| 90136-9  | CA  | Delinted seed | 0.1, 6.00               | 92, 77              |
| 90136-11   | CA  | Hulls         | 0.1, 2.00               | 85, 91              |
| 90136-15   | CA  | Meal          | 0.1, 8.00               | 92, 88              |
| 90136-17   | CA  | Crude oil     | 0.1, 2.00               | 75, 71              |
| 90136-19   | CA  | Refined oil   | 0.1, 2.00               | 93, 85              |
| 90136-23   | CA  | Soapstock     | 0.1-2.00                | 64-81               |

Storage Stability Data for the Processing Study:

The petitioner submitted storage stability data to accompany the cottonseed processing study. Meal and hull stability samples bearing weathered residues (already stored for 10 months) were taken from the processing study, analyzed concurrently with the residue samples reported above, and were placed in storage at -5 C and analyzed again 5 months later. Uncorrected residues in meal were reported as 5.01 ppm (method recovery 90%) when placed in storage and 4.90 ppm (method recovery 82%) 5 months later. Uncorrected residues in hulls were reported as 0.61 ppm (method recovery 88%) when placed in storage and 0.55 ppm (method recovery 69%) 5 months later. If these data are corrected for the respective concurrent recoveries, they indicate that weathered residues did not decline during the 5-month storage interval. Control samples of crude oil, refined oil, and soapstock were

fortified with mepiquat chloride at 1 ppm and placed in storage at -5 C. Uncorrected residues recovered were 0.82 ppm from crude oil stored for 3 months (method recovery 79%), 0.75 ppm from soapstock stored for 2.5 months (method recovery 72%), and 0.83 ppm from refined oil stored for 2.5 months (method recovery 89%). If these data are corrected for the respective concurrent recoveries, they indicate that mepiquat chloride did not decline in the fortified samples during the 2.5- to 3-month storage intervals. The petitioner indicated that the storage stability study on cottonseed processed commodities would be ongoing.

Processed samples from both states were not frozen prior to shipment to the processing laboratory; CA samples were shipped frozen 24 hours after harvest and LA samples were shipped frozen the day of harvest after an unspecified period. All samples were stored frozen at unspecified temperatures upon receipt at the processing laboratory, and after processing, the processed fractions were returned to freezer storage and shipped frozen to the analytical laboratory (BASF Corporation, Research Triangle Park, NC) and stored at <-5 C until analyzed. All processing fractions were stored ca. 10-13 months, except for crude oil which was stored for ca. 17 months.

Therefore, adequate storage stability data for the processing study have been submitted to support the proposed label increases of mepiquat chloride on cotton.

cc: J. Stokes (CBTS); mepiquat chloride List B File; mepiquat chloride S.F.; R.F.; Circu  
RDI: PErrico:10/7/93:RLoranger:10/7/93  
7509C:CBTS:JStokes:js:Rm 803:CM#2:305-7561:10/9/93