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OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Mepiquat Chloride. Interim Storage Stability Study for Cottonseed Processed Commodities. Reregistration Case No. 2375. Chemical No. 007969. MRID #42892201. DP Barcode D195902. CBRS #12,702.

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BASF Corporation has submitted an interim freezer storage stability study for mepiquat chloride in cottonseed and its processed commodities (MRID #42892201). Results for samples stored up to 14.5 months are reported, and the study is scheduled to continue for a storage period of 24 months.

Tolerances for mepiquat chloride 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).

Conclusions

1. In the final report, the registrant should include copies of information concerning characterization of test material used in sample fortification and for reference standards.



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2. The analytical method was adequately described. Adequate representative raw data and chromatograms were provided for standards, controls, fortified controls, and treated samples. For all matrices and fortification levels, procedural recovery levels averaged $81 \pm 7\%$ ($n = 40$).

3. The interim storage stability results indicate that mepiquat chloride is stable in cottonseed and its processed commodities for at least 10.5 to 14.5 months of frozen storage (< -5 C). The study will continue until samples have been stored for 24 months.

Detailed Considerations

Samples

Treated samples of delinted seed, meal, and hulls obtained from a previously conducted cotton processing study (MRID #42426803) were used in this study. The meal and hulls were from a trial in CA (RCN 90136) and the delinted seed from a trial in LA (RCN 90137). In both trials, the cotton was treated at an exaggerated rate (0.2 lb ai/A, 3X), at the green boll growth stage, in order to ensure finite residue levels in the seed. The cotton from these trials was collected at normal harvest time (PHI for CA 74 days and in LA 63 days) and immediately frozen. Samples were shipped to the processing facility where they were stored frozen until being processed. After processing, all fractions were returned to frozen storage and shipped to BASF's Agricultural Research Center, Research Triangle Park, NC.

The control crude and refined oils and soapstock used for the fortification part of the study were representative samples obtained from Texas A&M University Food Protein R&D Center. Aliquots of each matrix were removed from the bulk samples and fortified at the 1 ppm level with an analytical standard of mepiquat chloride (lot #CH 39/153-1). At each time period, duplicate fortified stored samples were removed from the freezer and analyzed, along with control material and fortified control material to provide procedural recovery results.

Samples remained in frozen storage (< -5 C) until analysis. For the initial analysis, subsamples of each matrix were removed and analyzed. The remainder of the sample was left in frozen storage until use in this storage stability study. At the initiation of this study, aliquots were removed, relabeled, and returned to frozen storage. At appropriate time intervals, two subsamples for each matrix were removed and analyzed along with control material and fortified control material to provide procedural recovery results.

Test Material

The registrant stated that the test substance used for the fortified storage samples and reference standard was characterized according to Good Laboratory Practices. This information is located at Landwirtschaftliche Versuchsstation der BASF, Limburgerhof, Germany and is available to BASF.

For the final report, the registrant should include copies of information concerning characterization of test material used to fortify samples and for reference standard.

Analytical Method

All samples were analyzed by BASF Method Number A9106. Previously submitted data (see J.Stokes, 10/12/93, CBTS #10671, 106890, and 10690) indicate that method A9106 is adequate for collecting data on residues of mepiquat chloride per se in or on cottonseed.

Briefly, residues were extracted from all matrices except refined oil by blending in a 25% 0.5 N HCl (aq) in methanol solution. Matrix impurities were removed by precipitation and filtering in a basic medium followed by washing the filtrate with hexane and dichloromethane. For refined oil, after dissolving in hexane, residues were extracted into 0.5 N HCl and the aqueous layer was washed with dichloromethane. Mepiquat chloride was isolated in the form of a dipycrylamine-complex which was partitioned into dichloromethane. Mepiquat chloride was then decomplexed and extracted from the dichloromethane with an aqueous acidic solution and the residues passed through an aluminum oxide column. Final determination was performed using ion pair chromatography with conductivity detection and a suppressor system.

Adequate representative raw data and chromatograms were provided for standards, controls, fortified controls, and treated samples.

Results

Method Recovery Data - For all matrices and fortification, procedural recovery levels averaged $81 \pm 7\%$. Individual recoveries for each matrix/fortification level are presented in Table 1.

CBRS concludes that the analytical method is adequate up to this point in the storage stability study.

Table 1. Percent Recoveries of Mepiquat Chloride for each matrix/fortification level.

Matrix	Fortification Level (ppm)	% Recovery
Delinted Seed	0.1	92
	3.0	85, 84
	6.0	77
Hulls	0.1	85
	1.0	78, 76, 70, 68
	2.0	91

Matrix	Fortification Level (ppm)	% Recovery
Meal	0.1	91
	1.0	82, 82
	3.0	78, 77
	8.0	88
Crude Oil	1.0	91, 88, 87, 83, 79, 78, 76, 76
Refined Oil	1.0	90, 87, 87, 86 83, 83, 82, 82
Soapstock	1.0	81, 78, 76, 73 73, 71, 71, 88

Storage Stability

A summary of interim storage stability results is presented in Table 2. These results indicate that mepiquat chloride is stable in cottonseed and its processed commodities for at least 10.5 to 14.5 months of frozen storage (<-5 C). The study will continue for 24 months.

Results are the average of duplicate analyses except: for the 0-month crude oil, refined oil, and soapstock, which involved quadruplicate analyses; and for the 11-month analysis of delinted seed for which only one value was obtained.

Table 2. Summary of Mepiquat Chloride Storage Stability Data.

Storage Period (Months)	Residue Found (ppm)	Uncorrected Percent Recovery	Procedural Percent Recovery	Residue Found Corrected for Procedural Recovery (ppm)	Corrected Percent Recovery
Delinted Seed					
0	2.87	--	84	3.42	--
11	2.62	91.2	84	3.12	91.2
Hulls					
0	0.61	--	88	0.69	--
5	0.55	90.2	69	0.80	115
11.5	0.60	98.4	77	0.78	113
Meal					
0	5.01	--	90	5.57	--
5	4.90	97.8	82	5.98	107
11	4.70	93.8	77	6.10	110

5

Storage Period (Months)	Residue Found (ppm)	Uncorrected Percent Recovery	Procedural Percent Recovery	Residue Found Corrected for Procedural Recovery (ppm)	Corrected Percent Recovery
Crude Oil					
0	0.87	--	87	1.00	--
3	0.80	92.0	79	1.01	101
12	0.71	81.6	76	0.93	93
Refined Oil					
0	0.85	--	85	1.00	--
2.5	0.82	96.5	88	0.93	93
12	0.82	96.5	83	0.99	99
Soapstock					
0	0.76	--	76	1.00	--
2.5	0.72	94.7	75	1.00	100
14.5	0.70	92.1	73	0.96	96

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