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

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FILE

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Command® Technical (EPA Reg. No. 279-3052). Residue Analytical Method and Storage stability in RACs and Soils. MRID No.408809-01 & -02. Branch No. 4655.

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FMC Corporation has resubmitted (in MRID 408809-02) the additional storage stability data determining the stability of Command® [2-(2-chlorophenyl)methyl-4,4-dimethyl-3-isoazolidinone; proposed common name = clomazone] residues in cottonseed, soybeans, tobacco, and soils and a description (in MDID 408809-01) of the methodology for determining clomazone residues in/on various crops and soils. We deferred to EFED for review of the soil data, which see K. Dockter 12/16/88 review of MRID 406333-01; Branch Nos. 4510 & 4511; and now similarly defer the current soil methodology.

The previously referenced residue GC/NP method as described now in MRID 408809-01 involves the following procedures for the determination of clomazone residues in/on green & cured tobacco, soybeans and cottonseed.

Briefly, crop samples from storage at -18°C were thawed at room temperature. Some samples were spiked at this time for recovery determination. Then 5 g (or 3 g of cured tobacco) were refluxed for 1 hour in dilute (0.25N) hydrochloric acid, filtered hot through glasswool and allowed to cool to room temperature. An aliquot was then extracted 3x with hexane, after which the combined extracts were washed once with saturated sodium bicarbonate solution, concentrated in a Kuderna-Danish evaporator, and cleaned up through a Florisil column using

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5%(v:v) ethyl acetate in hexane, which was discarded, followed by elution with 10%(v:v) ethyl acetate in hexane, which is then concentrated on a nitrogen evaporator, agitated, and transferred to sample injection vials for gas chromatographic analysis. Both packed- (3% ov-17 on 100/120 mesh Chromosorb W-HP) and capillary-column GCs equipped with nitrogen/phosphorus (NP) detectors were used. The inlet, oven, and detector temperatures were 250, 160 (capillary 170), and 300°C, respectively. Parent retention times were 6.3-6.4 min.; on packed column, and 3.6-3.8 (capillary). The system was calibrated after every 2 sample injections using the external standard method. The Company claims no visible interference was noted in any check samples, and no interference due to other pesticides is expected. It also claims that one analyst can complete a set of 8 samples in 7-8 hours. Additionally, sample calculations were provided. The integrator responses as area units were calculated as ng of parent based on a 0.5 ng/µl injection of run standard.

Samples of soybeans and cottonseed fortified at 0.2 ppm, and of tobacco at 0.5 ppm showed recoveries of 48-90 and 72-92% using packed and capillary columns, respectively. The method sensitivity of 0.2 ppm was reported for soybeans and cottonseed; and 0.5 ppm for tobacco. The Company claims previous studies indicated 0.05 ppm sensitivities can be achieved. Detectabilities of 0.05 ppm for soybeans and cottonseed, and 0.1 ppm for tobacco were also reported. Copies of various chromatograms were provided. Analyses of single standards (parent compound) at 0.5 and 1.0 ng are given; a standard curve is not. All samples showed no significant decline in levels over the 40-month interval; 15 months for cottonseed, a late addition to the study. The results are summarized below.

RAC	mo.	Residue, ppm
Cottonseed	0	0.20 ± 0.02
	15	0.18 " 0.01
Soybeans	0	0.20 " 0.01
	3	0.21 " "
	5	0.18 " "
	12	0.23 " "
	24	0.18 " 0.02
	40	0.17 " 0.03
Tobacco (cured & green)	0	0.51 " 0.04
	3	0.44 " 0.03
	5	0.43 " 0.04
	12	0.47 " "
	24	0.54 " "
	39	0.49 " 0.06

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Conclusion

These data are adequate to show that the residue analytical method is valid; degradation of clomazone residues in experimental samples is not a problem for up to 40 months.

cc: K. Dockter (DEB), Command S.F., PP# 4G3128, EFED,
E. Eldredge (ISB/PMSD), Circulation (7), RF.
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