

US EPA ARCHIVE DOCUMENT

Sample # 223975 was taken from the field study in Brawley, CA. in which the field had been treated at the rate of 0.5 lb.a.i./A with ten(10) applications of experimental XRM-4656 at 3 to 5 day intervals and a 40 day PHI. No detectable residue was reported. A fortified sample containing 0.01ppm chlorpyrifos showed a 90% recovery. The detection limit for chlorpyrifos was 0.01ppm.

Chlorpyrifos in cottonseed was analyzed according to Method ACR 76.3. Briefly, the active ingredient was extracted from the cottonseed sample by maceration in acetone. After filtration, dilution with hexane and the addition of sodium sulfate to remove water - the chlorpyrifos was extracted from the hexane solution with acetonitrile. After the solution was reduced to dryness, the dry residue was dissolved in hexane and the chlorpyrifos was eluted through a silica gel column with hexane. After again evaporating the eluted hexane solution to dryness, the dry sample was again taken up in hexane and analyzed by GLC using a FP detector.

[Note: This procedure is similar to the enforcement Method I, PAM II, (10/77)]

Typical Chromatograms of Standard, control, Recovery, and Treated sample from the Determination of 3,5,6-Trichloro-2-Pyridinol(TCP) in cottonseed.

Sample # 228301 was taken from the field study in Dysart, AZ in which the field had also been treated at the rate of 0.5 lb a.i./A with ten(10) applications of the experimental XRM-4656 at 4 day intervals with a 30 day PHI. No detectable residues of TCP were reported. A spiked sample containing 0.05ppm TCP showed a 96% recovery. The detection limit for TCP was 0.05 ppm.

TCP in cottonseed was analyzed according to Method ACR 71.19. Any chlorpyrifos present was hydrolyzed to TCP. The amount of TCP in the sample was the difference between the total amount determined here and the amount of TCP from the chlorpyrifos determined by Method ACR 76.3.

Briefly the samples were hydrolyzed with alcoholic-NaOH. After filtration, the filtrate was brought to a known volume, NaCl was added and mixture extracted with benzene. After discarding the benzene layer, the aqueous layer was acidified with HCl and again extracted with benzene. The latter was added to an acidic alumina column and total pyridinol (chlorpyrifos plus TCP) was eluted with buffered diethyl ether. The TCP was then extracted with NaHCO₃ which was partitioned with benzene. An aliquot of this benzene solution was treated with [N,O-bis(trimethylsilyl)acetamide] to form the trimethylsilyl derivative. Determination was by GLC using an EC detector. [Note: Method is a modification of enforcement Methods II & VII, PAM II].

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RCB Response

Raw data and chromatograms validating the previously submitted data are considered adequate. The deficiency is resolved.

Conclusion and Recommendation

We have no objection to the registration of this new product.

cc: SF(Chlorpyrifos);RF; Reviewer;PM#12;Circulation;PMSD/ISB
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TS-769;RCB;W.Anthony;wa;CM-2;Rm812;557-4351;6/18/87