Persistence of Methyl Parathion Residues on Sunflower Seeds¹

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Growers of minor crops, or crops that yield marginal profits, face a particular problem in their efforts to control insect pests. Potential insecticide usage is often not great enough to stimulate widespread interest in securing data necessary to obtain a label for its use on the crop. Sunflower is a crop that falls within this category.

The sunflower moth, <u>Homoeosoma electellum</u> (Hulst.) is a major pest of sunflower. Damage is caused by the larva feeding in the maturing seedhead and thereby yields are drastically reduced. Performance tests have indicated that methyl parathion could be used to control this pest provided residues on the seeds at time of harvest were not excessive.

Experimental

Methyl parathion, diluted to deliver 5 gallons of spray per acre, was applied by use of a high clearance spray machine to sunflower plants at rates of 0.5 and 1.0 pounds per acre. Treatments were made 28, 21, 14, 7, 3, and 1 day before the predetermined harvest date. Samples were collected immediately after the final treatment (0-day)

applications) by random cutting of 15 sunflower heads from each treated and check plot. Seeds were threshed from the heads, placed in plastic bags and held in a deep freeze until analyzed.

Precipitation on the third, eighteenth and twenty-first day following the initial application was 0.89, 1.40 and 1.45 inches, respectively.

Methyl parathion was extracted from the seeds by blending 100-gram samples in 300 ml. of acetonitrile, filtering the homogenate and concentrating the filtrate to about 10 ml. The residue was taken up in 50 ml. of benzene and dried over anhydrous sodium sulfate before adding the extract to a chromatographic column. The column, 2 x 30 cm., was prepared by adding successive layers of the following components: (1) 12 grams of acid alumina, Brockmann Activity 1, 80-200 mesh; (2) five grams of a 1:1 mixture of Hyflo Super-Cel and Darco G-60 carbon; (3) 12 grams of acid alumina. Methyl parathion was eluted from the column with 180 ml. of benzene and the eluate concentrated to a suitable volume, minimum of one ml., for injection of aliquots into a gas chromatograph.

Gas chromatographic analyses were made with a Barber Colman Series 5000 instrument equipped with a sodium thermionic detector (1). A six foot, 1/4 inch i.d. glass column packed with 10 per cent DC 200 silicone fluid on 80-90 mesh Anakrom ABS was used. Operating temperatures were as follows: column 215°C, injector 240°C, and detector 220°C. Nitrogen was used as the carrier gas at 60 ml./min. and air was supplied to the burner at 300 ml./min. Hydrogen flow was adjusted

to maintain a baseline current of 5×10^{-9} amp. The sensitivity scale was set at 10⁻⁸ AFS and the recorder chart speed at 15 inches/hour. The retention time for methyl parathion was 4.75 minutes.

Results and Discussion

Following extraction and clean-up, an equivalent of 100 mg. of untreated seeds injected into the gas chromatograph gave no peaks with retention times similar to methyl parathion. When methyl parathion was added to 100 grams of the seeds at the 0.10 ppm level, recovery of the insecticide ranged from 94 to 102 per cent. on a peak height of one cm. for two nanograms of material, the sensitivity of the method was 0.02 ppm.

Analysis of treated sunflower seeds revealed that relatively low deposits were present immediately following treatment (Table 1); only 2 ppm methyl

TABLE 1 Levels of Methyl Parathion in Sunflower Seeds Following Treatments at 0.5 and 1.0 Pounds Per Acre.

	P.P.M. After Treatment at					
Days After	0.5 lbs/a			1.0 1bs/a		
Treat- ment	Max.	Min.	Ave. ²	Max.	Min.	Ave. ²
01	0.800	0.541	0.696	2.120	1.737	2.068
1	0.318	0.278	0.304	0.933	0.846	0.970
3	0.114	0.076	0.100	0.490	0.352	0.401
7	0.093	0.040	0.060	0.197	0.135	0.184
14	0.023	0.019	0.023	0.095	0.086	0.089
21	ND^3	ND	ND	0.068	0.053	0.059
28	ND	ND	ND	ND	ND	ND

Samples collected immediately after treatment.

Average of four analyses. None detectable.

parathion residues were found on the seeds from plants treated at the higher application rate. Sunflower seedheads grow in such a manner that the seeds face the ground and are protected in umbrella fashion from above. The low insecticide deposits on the seeds probably resulted because the seeds were shielded from the downward spray. Such distribution of the toxicant on the plant would be highly desirable since the sunflower moth larva enters the heads at the stem end and would be subjected to much higher residues than present on the seeds.

Dissipation rates of the methyl parathion residues were similar following treatment at the two different levels. In both cases, over 50 per cent of the initial deposits were lost from the seeds within 24 hours. Based on the 1.0 ppm methyl parathion residue that is presently allowed on many agricultural crops, it was evident that dissipation was sufficiently rapid to allow harvest of seeds three days after treatment at one pound per acre. Residues of this magnitude were not deposited when plants were treated at 0.5 pounds per acre. If allowable residues were set as low as 0.10 ppm, the maximum interval between methyl parathion treatments of 0.5 and 1.0 pounds per acre and harvest would be seven and 14 days, respectively, as indicated by the results of this study.

References

(1) L. Giuffrida, JAOAC <u>47</u>, 293(1964).