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Comparison of Two Dislodgable Residue Extraction Methods[1]

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Dislodgable or easily removed surface insecticide residues on cotton are an important aspect in establishing safe reentry intervals for personnel working in treated fields. This study was done as an attempt to resolve the ongoing analytical question of which method of extraction is best for establishing dislodgable residue data: The method of Ware (1-min shaking in the most efficient solvent) or that of IWATA et al. (1977) (detergent/water extraction). The compounds tested represented each of the currently used insecticide classifications: Organophosphates, sulprofos (Bolstar); carbamates, methomyl (SD 14999); and pyrethroids, permethrin (Pounce).

METHODS AND MATERIALS

Test plots were located in a block of vigorously growing 'Stoneville 825' short staple cotton at the Agricultural Experiment Station, Marana, Arizona. Cotton plant heights averaged 45 cm on the day of insecticide applications, July 7, 1980. Plots consisted of 4 treated rows, with 102 cm spacing, 30.5 m long. Sprays were applied at 122 L/ha, 4.2 km/h, and 276 kPa pressure (13 gal/acre, 2.6 mph, and 40 psi). The manually drawn spray rig treated two rows, using 3 DC 2-13 Spraying Systems nozzles per row. The pressure was maintained from a 6.8 kg CO_2 tank with a two-stage regulator.

The formulation and rate of active ingredient (AI) per ha were: Bolstar 6 (6 EC) @ 1.1 kg/ha, SD 14999 (1.8 # methomyl/gal) @ 0.495 kg/ha, and Pounce (3.2 EC) @ 0.165 kg/ha.

Minimum and maximum air temperatures during the test were: July 7, 16.7°–40.0°; July 8, 21.1°–41.7°; July 9, 23.3°–38.9°; and July 10, 21.7°–39.4°C. There was no precipitation during this study.

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Triplicate samples for each extraction procedure were collected from each treated plot at 0, 24, 48, and 72 h after treatment. Controls were collected at 0 and 48 h post treatment for Ware's method and at all sampling times for Iwata's method. Each sample consisted of 100 (Ware's method) or 80 (Iwata's method) leaf disks, 2.54-cm diameter, taken singly and consecutively from the top, middle, and bottom portions of plants in all 4 rows.

The 100-disk samples were extracted in the field with 100 mL of the appropriate solvent. A sample was shaken for 1 min, the extract transferred to a labeled storage bottle, the solvent level marked, and the bottle placed in an ice chest until transferred to the laboratory refrigerator. The extracting solvents were redistilled benzene for sulprofos, redistilled hexane for permethrin, and tap water for methomyl. Controls were extracted with each of the solvents as described above.

The 80-disk samples were placed in labeled jars and covered with ice until their return to the laboratory for processing. They were processed according to the protocol for dislodgable residues (IWATA et al. 1977) as described by BUCK et al. (1980). The samples were ultimately taken up in redistilled hexane for permethrin, redistilled benezene for sulprofos, and acidified distilled water for methomyl.

Permethrin extracts were cleaned on activated Florisil and analyzed by glc as described by WARE et al. (1980). Methomyl extracts were hydrolyzed and analyzed by glc as described by WARE et al. (1980) with the following exceptions: No initial hexane partioning was done on the tap water extracts and the last aqueous phase of all samples was extracted 5 times, instead of 4, with ethyl acetate. Sulprofos and its metabolites were analyzed without cleanup by gc using a flame photometric detector in the phosphorus mode. An 89 cm x 4 mm I.D. Pyrex column containing 2% OV-101 on 100/120 mesh Chromosorb W (H.P.) was used. Carrier gas flow was 120 mL/min and temperatures were 190°, 225°, and 230°C for column, detector, and inlet respectively. Quantitation was by peak height.

RESULTS AND DISCUSSION

The results are presented in Table 1, expressed as $\mu g/cm^2$ of cotton leaf, one surface only. The Ware method gives significantly higher recoveries for sulprofos and permethrin, but not for methomyl. This is explained by the duplication of solvents with methomyl, e.g., water vs water + surfactant.

Table 2 was prepared to demonstrate the consistency of variability of the two methods. It will be noted that the Ware method has a considerably lower coefficient of variability percentage, indicating a greater degree of extraction reliability.

TABLE 1

Comparison of extraction methods for dislodgable residues of three insecticides on cotton plants.[1/][2/] Marana, Arizona, July 7, 1980.

	Sulprofos 1.1 kg/ha					
	Sulprofos		Sulfoxide		Sulfone	
Hours	Ware	Iwata	Ware	Iwata	Ware	Iwata
0	6.0a[3/]	4.6b	0.39a	0.66a	<0.028a	<0.038a
24	2.6a	1.8b	1.3a	1.6a	0.27a	0.27a
48	1.1a	0.31b	1.6a	1.8a	0.92a	0.82a
72	0.42a	0.16a	1.4a	1.5a	0.99a	0.83b
Controls	<0.001	<0.001	<0.008	<0.004	<0.003	<0.001

	Permethrin 0.16 kg/ha						Methomyl 0.50 kg/ha	
	cis isomer		trans isomer		Total			
Hours	Ware	Iwata	Ware	Iwata	Ware	Iwata	Ware	Iwata
0	0.36a	0.35a	0.51a	0.49a	0.87a	0.84a	2.1a	1.7a
24	0.32a	0.25b	0.43a	0.35b	0.75a	0.60b	0.45a	0.56a
48	0.30a	0.19b	0.40a	0.25b	0.70a	0.44b	0.23a	0.28a
72	0.24a	0.17b	0.32a	0.23b	0.56a	0.40b	0.11a	0.13a
Controls	<0.006	<0.007	<0.006	<0.007	-	-	<0.003	<0.017

1/ No corrections made for recovery.

2/ Residues expressed as $\mu g/cm^2$ for one surface only of cotton leaf.

3/ Common letters on the same line for the same compound indicate no difference in residue at the 0.05 level (t test).

TABLE 2

Variation, expressed as coefficient of variability, in two extraction methods for insecticides.

	Coefficient of Variability (%)	
Material	Ware	Iwata
Sulprofos	8.5	19.1
Sulprofos sulfoxide	8.4	15.9
Sulprofos sulfone	8.1	12.4
cis Permethrin	8.7	10.0
trans Permethrin	8.0	8.7
cis + trans Permethrin	8.1	8.7
Methomyl	18	11

Additionally, the Ware method of extraction is much faster. For example, to extract most insecticides requires no more than 15 minutes for 12 samples using the Ware method, and approximately 3.5 hours using the Iwata method. In summary, it would appear expedient to adopt the 1-min solvent shaking method to establish a faster, more consistent dislodgable residue extraction, that could ultimately permit and encourage other labs to become involved.

REFERENCES

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