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EFFECTS OF DRYING PROCEDURES, PLANT GROWTH, AND
WEATHERING ON INSECTICIDE RESIDUES ON SPRAYED ALFALFA

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In studies of insecticide residues on forage and other crops it is common practice to dry the green forage samples before stripping off the residue for quantitative determination. Since some insecticides are more volatile than others, in 1950 experiments were conducted to determine the effects of several methods of drying on the residues remaining on alfalfa from applications of emulsion sprays of chlordane, lindane, methoxychlor, and toxaphene. The loss of residues due to normal weathering in the field over a period of 13 days was also studied.

Methods

Spraying. --Plots 8 feet wide and 228 feet long were set up in an alfalfa field on the farm of the Bureau of Dairy Industry at Beltsville, Md. The plots apparently had a uniform stand of alfalfa 12 to 15 inches high and were located so that there was little possibility of spray drift from one to another.

On August 16 one plot was sprayed with a chlordane emulsion at the rate of 1.35 pounds of toxicant per acre, and another plot was similarly sprayed with methoxychlor at 1.33 pounds per acre. In like manner on August 17 one plot was sprayed with lindane at 0.36 pound and another with toxaphene at 1.7 pounds per acre.

The applications were made with a specially constructed sprayer having an 8-foot boom. Uniformity in application was greatly facilitated by installation of a special speedometer to regulate the speed of the sprayer and a pressure regulator to insure a constant delivery rate at

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a pressure of 30 pounds per square inch. The sprayer was operated at 2.6 miles per hour and delivered approximately 12 gallons of spray per acre.

Sampling and drying. --At five locations uniformly distributed in each plot samples were collected for residue determinations. Each sample consisted of all the vegetation within 1 square yard. Cutting approximated that with the customary mowing machine. The residue on one sample taken at each location was stripped off for analysis as soon as collected. Four other samples from each location were dried by one of the following methods before the residue was stripped off for analysis: (1) In a small cabinet in which the temperature was thermostatically controlled at 50°-60° C. and through which a slow current of air was pulled by an exhaust fan, 2 to 3 days required for drying; (2) in a large drier at 60°-70° C. with a strong current of air, less than 24 hours required for drying when operated continuously; (3) spread out on benches in a well shaded and ventilated greenhouse, about 3 days required for drying as the maximum temperature reached was not over 40° C.; and (4) spread out on papers out-of-doors in the sunshine during 8 hours of the day and in the greenhouse in the interim, 2 full days of sunny weather required for drying. After drying, the samples were stored for 2 to 3 months in 25-pound paper bags in tightly closed cardboard cartons in a room having little circulation of air at a temperature probably not exceeding 26° C.

One set of samples was collected within 4 hours after spraying, and a second set was collected from contiguous areas 13 days later.

Chemical analysis of samples. --All the samples were analyzed for organic chlorine by the method described by Poos et al. (1). The samples of undried vegetation were analyzed as soon as they were brought into the laboratory, and the dried samples 2 to 3 months later. All residues were calculated to a dry-weight basis. No correction was made for untreated checks, because it was not practical to conduct the volume of work required to do this adequately.

Discussion of Results

The results given in table 1 indicate first of all that in most tests less residue was found on dried samples than on comparable undried samples. The loss of residue in drying may be expected to be greater following applications of lindane and chlordane than of methoxychlor and toxaphene.

Table 1. --Insecticide residues on green and dried samples of sprayed alfalfa.
Figures in parts per million.

Condition of samples	Chlordane at 1.35 lb. per acre		Methoxychlor at 1.33 lb. per acre		Lindane at 0.36 lb. per acre		Toxaphene at 1.7 lb. per acre	
	Range	Average	Range	Average	Range	Average	Range	Average
Samples collected within 4 hours after treatment								
Undried	54-73	65.9	193-357	276.5	42-120	66.7	467-1024	774.6
Dried--								
At 50°-60° C.	32-47	39.3	148-212	170.5	14-19	17.0	382-977	724.7
At 60°-70° C.	17-75	46.1	199-370	258.4	13-28	19.1	272-903	552.6
In shaded greenhouse	6-66	28.3	159-246	201.6	18-22	19.5	201-610	453.2
In outdoor sunshine	13-29	19.9	139-246	201.8	16-20	17.7	154-807	475.6
LSD for dried samples at 5% level	---	<u>1/</u>	---	<u>1/</u>	---	<u>1/</u>	---	147.8
Samples collected 13 days after treatment								
Undried	5-12	7.8	13-56	35.8	3-10	6.3	72-237	132.2
Dried--								
At 50°-60° C.	13-18	15.4	36-49	40.9	2-4	3.2	61-151	99.4
At 60°-70° C.	0-2	1.1	1-15	5.9	1-20	8.9	43-175	89.4
In shaded greenhouse	1-8	4.7	20-43	31.3	1-19	8.4	35-116	68.8
In outdoor sunshine	1-7	2.7	4-122	37.3	1-16	8.1	43-109	74.0
LSD for dried samples at 5% level	---	4.5	---	10.3	---	<u>1/</u>	---	<u>1/</u>

1/ Difference not significant according to the F test.

In general the losses of residues in drying of the sprayed alfalfa were not nearly so large as the losses due to weathering over the 13-day period. During this period 5.68 inches of precipitation was recorded at the U. S. Weather Bureau Station located within a few hundred yards of the sprayed areas. On August 19, 4.85 inches of rainfall was recorded. This was no doubt a major cause for the losses of residues during the 13-day period. On August 23 and again on August 29, 0.34 inch of precipitation was recorded.

No clear-cut distinction can be drawn between the four drying methods. The loss of residue from cut alfalfa cured in the field would be expected to follow a pattern similar to that of the samples dried in the greenhouse or outdoor sunshine. Additional rainfall and the loss of leaves during curing and storing would further reduce the amount of residue, especially if the alfalfa were stored unbaled. On the basis of present knowledge, the best procedures that can be recommended for drying laboratory samples are those using a minimum of heat and circulation of air consistent with the prevention of spoilage.

Separate analyses of variance were conducted for each of the eight tests of the four drying methods, as no combination seemed advisable.^{2/} In these analyses of variance the data for undried samples were not included, because the residues found were usually very much higher than on the dried samples. The design of the experiment did not permit separation of analytical error from sampling error or calculation of experimental error.

Table 2 compares the weight of the alfalfa samples and the insecticide residues on them at the beginning and at the end of the 13-day period. The natural growth and increase in weight of the alfalfa plants in association with weathering are reflected in a decrease in parts per million of residue. It is evident that the increase in weight of the plants was not sufficient to account for the decrease in the amounts of residue found. When calculated on an area basis, the results indicate that approximately 80 to 88 percent of the insecticides was lost from the undried samples of alfalfa during the 13-day period, irrespective of the growth of the plants. The heavy rainfall during the period undoubtedly contributed to the rather rapid disappearance of the insecticides from the alfalfa.

Summary

A study was made of the effect of drying procedures, plant growth, and weathering on residues of chlordane, lindane, methoxychlor, and toxaphene remaining on alfalfa sprayed with emulsions of these insecticides.

^{2/} The authors express their appreciation to F. M. Wadley for advice on the statistical treatment of these data.

Table 2. --Effects of 13 days' growth and weathering on insecticide residues on undried alfalfa

Insecticide	Weight of alfalfa (average of 10 samples)			Residue (average on 5 samples)					
				Quantity basis			Area basis (1 sq. yd.)		
	Within 4 hours	After 13 days	Increase	Within 4 hours	After 13 days	Loss	Within 4 hours	After 13 days	Loss
	<u>Grams</u>	<u>Grams</u>	<u>Percent</u>	<u>P.p.m.</u>	<u>P.p.m.</u>	<u>Percent</u>	<u>Milligrams</u>	<u>Milligrams</u>	<u>Percent</u>
Chlordane	750	796	6.1	66	8	87.8	11.1	1.4	87.4
Methoxychlor	853	882	3.4	277	36	87.0	54.1	7.9	85.4
Lindane	793	1,020	28.6	67	6	91.0	13.8	1.6	88.4
Toxaphene	861	1,006	16.8	775	132	82.9	184.7	36.9	80.0



In general less residue was present on dried samples than on comparable undried samples. Losses of chlordane and lindane residues were greater than those of methoxychlor and toxaphene. Losses of residues in laboratory drying of sprayed alfalfa were less than those from green alfalfa in the field subjected to a 13-day period of weathering. From 80 to 88 percent of the insecticides was lost from the green alfalfa during this period, irrespective of the growth of the plants. No clear-cut differences were found between drying methods that utilized temperatures of 50° to 70° C. and forced air currents and those that utilized ordinary air temperatures and natural air currents.

Laboratory samples should be dried with the minimum of heat and circulation of air, consistent with the prevention of spoilage. Samples should be stored in tightly closed containers as soon as drying is completed.

Literature Cited

Poos, F. W., Dobbins, T. N., and Carter, R. H.

1950. Sampling forage crops treated with organic insecticides for determination of residues. U. S. Bur. Ent. and Plant Quar. E-793, 12 pp.