Effect of Soil Systemic Insecticides on Flavor and Residue in Coffee

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Flavor tests and residue analyses were made on coffee beans harvested from trees treated with the systemic insecticides phorate, disulfoton, Bidrin granular, Bidrin resin pellets, as well as an untreated check. Residue analyses were performed on samples harvested 30 days after treatment; harvests for flavor tests ranged from 53 to 86 days. Expert tasters ranked the samples numerically, attempted to separate some treatments by the triangular method, rated the samples by differences and preference scales, and subjectively evaluated the flavor. Green coffee beans from all treatments were analyzed for total phosphorus, and those treated with disulfoton were further analyzed by paper chromatography for the pesticide and its metabolites. No foreign odor or flavor was present in any of the samples; in some series, treated samples appeared slightly superior to those untreated. Absorbances of phosphorus from all samples were essentially the same, and no detectable residues of disulfoton or its metabolites were found.

The history of the coffee leaf miner, *Leucoptera coffeella* (Guerin-Meneville and Prrott.), in Guatemala denotes that this microlepidoptera has been the most important insect pest of coffee during the past 50 years, and has been especially persistent since 1949. This insect is a major pest throughout the coffee-growing regions of Africa and Central and South America.

The senior author first had contact with the coffee leaf miner in 1961. In 1964, an intensive study was initiated in Guatemala to find a more practical solution to this problem, and some recommendations resulted from studies in which oil was used in combination with insecticides in spray applications (Rodriguez *et al.*, 1966), and from experiments in which various insecticides were used as sprays (Campbell *et al.*, 1967).

Another method of control that merited investigation was the use of systemic insecticides used as soil applications to the coffee tree. One of the advantages in using such a method of attack on the coffee leaf miner is that control of a preventive type is long-lasting (Perez Escolar, 1965). Many biological tests were conducted in Guatemala by technical representatives of commercial companies, and some corroborating tests were conducted by the Asociación Nacional del Cafe (1964). Soil applications of phorate or disulfoton were effective in preventing or controlling infestations of the coffee leaf miner. Grower acceptance would come about unless good reasons were found not to recommend this practice.

The objective of this work is to investigate possible residues that might occur from soil application of the long-lasting systemic insecticides and, concomitantly, to test for any detectable off-flavor that might result in the coffee brew.

EXPERIMENTAL

Granular formulations of phorate, *O,O*-diethyl *S*-[(ethylthio) methyl]phosphorodithioate, disulfoton, *O,O*-diethyl *S*-[2-(ethylthio)ethyl)]phosphorodithioate, Bidrin, 3-hydroxy-*N,N*-dimethyl-*cis*-crotonamide dimethyl phosphate, and a resin pellet formulation of Bidrin were used. This was designated as Bidrin, XP657 resin pellets (20%). The materials were sprinkled at the base of trees (12- to 18-inch radius from the trunk) 5 to 6 feet tall in a block that had been lightly attacked by the leaf miner the previous year; in general, the whole plantation had been heavily attacked since about 1950. This plantation, Finca Parraxe, in Samayac, Suchitepequez, is situated at elevations of 2300 to 2700 feet.

Phorate and disulfoton (10% granular) were applied at 12.5, 25, 37.5, and 50 grams per tree. Bidrin 5% granular was applied at 25, 50, 75, and 100 grams per tree, while the Bidrin 20% XP657 resin pellet formulation was applied at 8, 16, 24, and 32 grams per tree. There were 10 trees per plot and these were replicated 3 times at random. The materials were applied August 22, 1964, during the rainy season. The coffee berries were picked as they ripened on September 21, October 14 and 28, and November 16, 1964. These pickings will be referred to henceforth as the first, second, third, or fourth pickings or harvests. The elapsed time between treatment and harvest for each interval was 30, 53, 67, and 86 days.

Processing. In preparation for the taste tests, the harvested coffee berries were treated as follows: On the same day that the berries were picked, they were cleaned of extraneous material and abnormal berries, and were depulped. The beans were then put to ferment in plastic bags, partially washed at the end of 24 hours, leaving some of the fermenting liquor in the bags, then washed further 24 hours later. They were set to dry in the sun in open-mesh fabric bags until they reached a state of approximately 10% moisture.

After drying, the parchment skins were hulled off the beans, then the beans were screened to remove abnormally large or subnormally small sizes, and those having off

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colors and odd shapes were sorted out, resulting in a homogeneous sample of green coffee beans, which were then roasted in the usual fashion for market coffee. A 250-gram sample was drawn out, ground, and mixed.

The second, third, and fourth harvests of the experiment were prepared in this manner for taste tests. In the first harvest, the quantity of material was sufficient only for residue analysis.

Flavor Tests. The coffee was tasted by two groups of expert tasters, first at the Asociación Nacional del Cafe (ANC) in Guatemala and again at General Foods Corp., Tarrytown, N.Y.

In the ANC tests, all the coffee was brewed by a uniform method, and each cup contained 7.2 grams of ground coffee. Each taster was presented a series of 5 cups of coffee, 1 cup taken from the check plot and 1 from each of the 4 insecticide treatments. The samples were given numerical ranks from 1 to 5 (best to worst). The tasters also evaluated each cup subjectively as to whether a flavor different from or abnormal to the coffee of the region was present. At least four separate tastings were made by each taster of the samples from each harvest. Two of the treatments appeared in a few tests to possess a flavor superior to that of the others, and these two were further scrutinized in triangular tests against the check plot.

The rank sums of a given taster's rankings of a particular harvest were considered as a single test, and subjected to analysis for significance of differences at the level $P = 0.05$ according to the method developed by Kramer (1960). The rank sum method is particularly good for flavor tests, since all tasters rank the samples by numbers 1 through 5; thus the method eliminates the tendency any individual may have to score all the samples either high or low in comparison with the other tasters. The results of the triangular taste tests were submitted to analysis for significance ($P = 0.05$) of the number of correct separations according to the method of Roessler *et al.* (1948).

At the General Foods laboratories, coffee samples taken at the third harvest date from the same four treatments were compared with the check. The green coffee beans were subjected to uniform roasting, grinding, and brewing processes, and since they were ground to specifications for fine grind coffee, 13.5 grams of ground coffee was used with 8 ounces of spring water for each cup of brewed coffee. The taster compared the treatments to the check according to a difference scale rating, a preference scale rating, and a flavor description rating. The difference scale ratings ranged from 10 (identical to control) to 6 (extremely different from control). The preference scale rating ranged from 9 (like extremely) through 5 (neither like nor dislike) to 1 (dislike extremely). The flavor description rating was an individual comment by the taster. A photometric technique to measure roast color was also employed on these samples.

Residue Analysis. The green coffee beans for pesticide residues were analyzed in the laboratories of the U.S Department of Agriculture.

Harvest samples were taken from the trees which had received the maximum insecticide treatment, 5 grams per tree of actual phorate or disulfoton in granular form or 6.4 grams of actual Bidrin as a resin pellet. Samples of the first harvest, 30 days after treatment, were analyzed for total phosphorus, and the beans from the disulfoton-treated trees were also analyzed by paper chromatography.

Preparation of the beans for analysis was as follows: A 50-gram sample of ground coffee beans was extracted with 200 ml. of acetonitrile, the mixture was filtered, and the solvent removed with a Rinco evaporator. The residue was dissolved in acetone, and chromatographed on a 19-mm. column containing a mixture of 3 grams of acid

Table I. Results of Rankings of Coffee Samples

Harvest	Rank Sums		Highest and Lowest Significant Rank Sum	Conclusion
	1st taster	2nd taster		
Second			6–18[a]	No significant difference
Phorate	18	15		
Bidrin	12	12		
Bidrin-XP657	10	8		
Disulfoton	15	16		
Check	15	9		
Third			8–22[b]	No significant difference
Phorate	9	11		
Bidrin	18	14		
Bidrin-XP657	13	15		
Disulfoton	16	13		
Check	19	22		
Fourth			10–26[c]	No significant difference
Phorate	16	18		
Bidrin	25	24		
Bidrin-XP657	17	10		
Disulfoton	17	18		
Check	15	20		

[a] 5 treatments, 4 replicates. [b] 5 treatments, 5 replicates. [c] 5 treatments, 6 replicates.

washed Darco (activated carbon produced by Atlas Chemical Industries, Wilmington, Del.) and 10 grams of Celite 545 (diatomaceous earth product produced by Johns-Manville Corp., New York, N.Y.). The column was washed with 200 ml. of 5 to 1 acetone-benzene solution before the sample was added to the column; 150 ml. of the same solution was used to elute the sample from the column. The sample was then analyzed for total phosphorus using the methods of Saliman (1964). The disulfoton samples were analyzed by paper chromatography after chromatographic clean up on the charcoal-Celite column. The method used was that described by Getz (1962).

RESULTS AND DISCUSSION

According to the evaluation of the expert tasters at the ANC, no foreign flavor or odor was present in any of the samples. Table I shows the rank sums of each taster's rankings of a particular harvest and the highest and lowest significant rank sums as set up by Kramer (1960) for an experiment of the indicated number of treatments and replicates. Since none of the rank sums in any of the tests exceeded the highest significant rank sum or were lower than the lowest significant rank sum, apparently no significant differences were found in the flavor of the coffee samples tasted. The results of the triangular evaluations, in which Bidrin as resin pellets and phorate were compared with the check, are shown in Table II. The tests were rather limited as to the number of separations, but although one taster was able to separate phorate correctly from the check, the number of correct separations was not significant in relation to the total number of separations, and neither treatment was readily detectable from the check plot. The general conclusions from the taste tests conducted by the ANC were that the treatments did not impart any objectionable flavor and, in a few series, might even have seemingly imparted a somewhat better flavor to the beans from treated plots as compared with beans from the check.

The findings of the tasters at the General Food laboratories in general agreed with those of the ANC. The results of the roast color tests and difference and preference ratings are shown in Table III. A difference of from 4 to 8 units on the roast color scale is required before a difference can be detected by the human eye. The check measured 50, the treatments ranged from 52 to 57; therefore, the differences measured in the samples were either not detectable or barely detectable to the observer. On the difference scale, the treatments were rated either barely different from or moderately different from the check. The difference was attributed to a slight increase in the acidity. In the preference ratings, all samples including the check were given ratings from like slightly to like moderately. The tasters concluded that the minor differences were probably attributable to slight variations in roast color and not to the insecticide treatment of the coffee plant.

Results of the residue analyses performed for total phosphorus in the coffee samples from the first harvest are presented in Table IV. The absorbances of the check and the insecticide-treated samples are essentially the same. The background absorbance of 0.190 was equivalent to 0.02 p.p.m. of phosphorus, and would be considered the minimum sensitivity of the method. Additional analysis of the disulfoton sample by paper chromatography showed no detectable residues of the insecticide or its metabolites.

Perez Escolar (1965), in Puerto Rico, treated trees with disulfoton at dosages ranging from 1 to 12 grams of the 10% granular formulation per foot of tree height, and at various time intervals starting 7 months prior to harvest. The coffee samples were submitted to the formulator, Chemagro Corp., Kansas City, Mo., for chemical analysis. Analysis showed that none of the samples contained disulfoton residue irrespective of the time interval between the treatment and harvest. These general results were

Table II. Results of Triangular Evaluations of Flavor of Coffee Treated with Insecticides

Treatments Tested	Number of Separations	Number of Correct Separations	Number of Correct Separations Preferring Insecticide Treatment
Bidrin, XP657 (resin pellets, 20%) and check	8	1[a]	0
Phorate (10% granular) and check	4	2[a]	2

[a] Not significant.

Table IV. Absorbance of Total Phosphorus in Residue Analyses of Coffee Samples

Insecticide	Absorbance
Phorate	0.170
Bidrin	0.182
Bidrin-XP657	0.180
Disulfoton	0.183
Check	0.190[a]

[a] Background absorbance of 0.190 equivalent to 0.02 p.p.m of phosphorus.

Table III. Roast Color, Flavor Difference, and Preference Ratings

	Roast Color	Ratings: Difference, Taster 1	Difference, Taster 2	Difference, Taster 3	Difference, Mean	Preference, Taster 1	Preference, Taster 2	Preference, Mean
Phorate	54	8.0	9.0	9.0	8.7	6.0	7.0	6.5
Bidrin	52	8.0	9.0	8.0	8.3	6.0	7.0	6.5
Bidrin-XP657	57	8.0	9.0	8.0	8.3	6.0	7.0	6.5
Disulfoton	54	8.0	9.0	10.0	9.0	6.0	7.0	6.5
Check	50	10.0	10.0	10.0	10.0	7.0	7.0	7.0

corroborated; the dosage ranges of disulfoton were comparable, since apparently the Puerto Rican trees were about 4 feet tall.

Treatment with phorate or disulfoton has been shown to be effective against coffee leaf miner within two weeks of application, and the metabolites of these compounds remain effective for several months after treatment. In this latter fact lies much of the value of the materials, since systemic insecticides must be applied to the trees prior to the end of the rainy season, which also means that any potential infestation in the planting is several months away. Moreover, the principle of integrated control can be applied with this type of treatment, since soil application will not harm or impede the effect of parasites against the larvae.

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