PESTICIDES AND FLAVOR

Benzene Hexachloride Content and Flavor of Peanuts Grown in Rotation with Cotton Dusted with This Insecticide

IRWIN HORNSTEIN, Entomology Research Branch HOWARD REYNOLDS and GLADYS L. GILPIN, Human Nutrition Research Branch Agricultural Research Service, U. S. Department of Agriculture, Beltsville, Md.

Benzene hexachloride analyses and palatability studies were made on peanut butter prepared from peanuts that followed cotton which was dusted with 42 to 168 pounds per acre of 3% gamma benzene hexachloride dust formulated with approximately 8.3% of 36% gamma benzene hexachloride. Soil samples, taken before the peanuts were planted, and at harvest time, were also analyzed. None of the soil or peanut butter samples had significant amounts of benzene hexachloride and none of the peanut butter samples had off-flavors that could be attributed to carry-over in the soil of benzene hexachloride applied to the preceding cotton plants.

CHEMICAL ANALYSES FOR BENZENE HEXACHLORIDE and palatability evaluations of peanut butter made from peanuts, grown in soils previously used to grow cotton that had been dusted with this insecticide, showed measurable amounts of benzene hexachloride and definite off-flavor characteristics of this insecticide when unusually heavy applications were made (4). The dust contained 3% of gamma benzene hexachloride (24% of technical benzene hexachloride containing 12 to 14% of gamma), 5% of DDT, and 40% of sulfur.

The recent trend is to use grades of benzene hexachloride of higher gamma content, because products containing 36%and higher of this isomer are now readily available.

Musty odors associated with benzene hexachloride have been attributed to overchlorinated materials such as heptachlorocyclohexanes and octachlorocyclohexanes (\mathcal{S}) and to polychlorocyclohexenes derived by the removal of chlorine from the polychlorocyclohexanes (\mathcal{I}), rather than to the gamma isomer.

Studies with the pure isomers of benzene hexachloride have shown, however, that the gamma isomer may cause some off-flavor (2). Flavor defects appear to be related to the total amount of these isomers to which foods are exposed during growth. Dusts prepared from benzene hexachloride containing 36% of gamma should therefore be less likely to cause off-flavor than preparations from benzene hexachloride of lower gamma content because of the smaller quantity of benzene hexachloride needed to provide insecticidal quantities of the gamma isomer.

This paper reports a continuation of the palatability tests and chemical analyses of peanut butter, which was made from peanuts that followed cotton on which dust made from a benzene hexachloride containing 36% of the gamma isomer was used. Beginning in May 1951 cotton, growing on 13 plots at the Pee Dee Experiment Station at Florence, S. C., and at two nearby farms, was treated with 4 to 16 applications of this dust. Five plots were left as untreated controls. None of the plots had been previously exposed to benzene hexachloride.

The prepared dust contained 3.08% of gamma benzene hexachloride, 5.25% of the other isomers, and 91.67% of the diluent. Neither DDT nor sulfur was used in preparing this dust.

Soil samples were obtained from 12 of these plots in the spring of 1952 prior to the planting of peanuts, and from all plots at the time of harvesting. These soil samples were screened, mixed, and subsampled at the Moorestown, N. J., laboratory of the Bureau of Entomology and Plant Quarantine and sent to the Beltsville, Md., laboratory for analysis. After harvesting, the peanuts were processed into peanut butter in a laboratory of the Bureau of Plant Industry, Soils, and Agricultural Engineering.

Chemical Data

The soil and peanut butter samples were analyzed by modifications (3) of the Schechter-Hornstein colorimetric method (6) for benzene hexachloride. Briefly, peanut butter samples were extracted with methylene chloride, the methylene chloride was evaporated on a steam bath, and glacial acetic acid was added to the residual peanut oil. Soil samples were extracted directly with acetic acid. The benzene hexachloride in acetic acid was dechlorinated by the use of zinc and the benzene formed nitrated to m-dinitrobenzene. This compound in the presence of methyl ethyl ketone and strong alkali gives a violet red color that can be measured photometrically. This analytical procedure is reliable to as little as 0.1 p.p.m. of benzene hexachloride when applied to soils, and to about 0.2 p.p.m. when applied to peanuts (3). All but one of the concentrations in the soil samples, as shown in Table I, were at or below this level of reliability. In this one case (Turner Farm, 8 applications, fall sampling) the amount found, 0.12 p.p.m., was of borderline significance, particularly because a sample from this plot analyzed

in the spring had only 0.05 p.p.m. of benzene hexachloride. All the benzene hexachloride concentrations for the peanut butter samples were under 0.2 p.p.m. Within the limits of the method no significant amounts of benzene hexachloride were therefore found in the peanut butter samples.

Palatability Data

The peanut butter was stored in screw-capped jars for approximately 2 weeks at 38° F. before palatability tests were begun. Samples were brought to room temperature before being served to judges and one-half-teaspoon portions were served on coded china plates.

The experimental designs used for these studies permitted analysis of the data by the incomplete-block method of Cochran and Cox(1) as well as by the ranked-pair method of Terry, Bradley, and Davis (7). Samples from each field block were evaluated separately, so that the results could be combined in one analysis at each location subject to tests of homogeneity. The design used with the samples from the Experiment Station provided four replications of scores per sample per judge; that used with samples from the Hill Farm, three replications per sample per judge; and from the Turner Farm, four replications of each sample in each of three scoring sessions. The judging panel consisted of five persons experienced in rating peanut butter, who were selected on the basis of tests previously described (4).

Each judge scored two pairs of samples per session. Each pair of samples was scored independently, and in case of a tie score for any pair the judge was instructed to circle the score of the sample to be considered the better within the 1point range. A 10-point scale was used, 10 indicating no off-flavor, 8 barely perceptible, 6 perceptible, 4 slightly strong, and 2 strong off-flavor.

Mean flavor scores for all the peanut butter samples evaluated are given in Table II and those that are significantly low are indicated. The ranked-pair analyses are not presented, but significant findings were the same for both this method and the analysis of variance. The ranked-pair analyses also indicated

significantly lower than the samples from the untreated field and from a field receiving 132 pounds. Because the comparable sample from plot A was not significantly different from the other samples with which it was compared, it seems probable that the off-flavor. found in the sample from plot B, was due to other factors than the benzene hexachloride treatment. The roast of this sample was probably one factor, because the most frequent comment of the judges

Table II. Flavor of Peanut Butter

Benzene
Hexachloride

Dosage, Pounds per Acre	Mean Flavor Score ^a								
	Experiment Station		Hill Farm		Turner	General			
	Plot A	Plot B	Plot A	Plot B	Form	mean			
0	9.2	9.6	9.9	9.1	8.5	9.3			
42.4					8.8	8.8			
48.8			7.70	9.4		8.5			
67.8	8.6	6.2%			<i>.</i>	7.4			
84.0					8.9	8.9			
88.0			9.4	7.3 ^b		8.4			
132.0	9.4	9.6			•	9.5			
142.8			9.5	7.2%		8.3			
168.0					8.9	8.9			
168.0°					8.5	8,5			

^a 10, no off-flavor; 8, barely perceptible off-flavor; 6, perceptible off-flavor; 4, slightly strong off-flavor; 2, strong off-flavor. ^b Significantly lower, at 5% level, than other means in same column.

^e Activated carbon applied at rate of 200 pounds per acre before planting peanuts.

that the judges were scoring consistently with each other.

The samples from the Experiment Station were not significantly different from each other, with one exception. The sample in plot B from a field receiving 67.8 pounds of the insecticide was scored

in describing its flavor was that it was green or raw. This sample was a lighter roast than either of the other samples with which it was compared, a factor that makes flavor analysis of peanut butter very difficult and probably accounts, in part, for the low score of 6.2.

Table I. Benzene Hexachloride Content of Soils and of Peanuts

				Benzene Hexachloride Found, P.P.M. ^a						
Dosage, Pounds per Acre Total Total Gamma		No. of	In Soil							
		Gamma	Appli- cations	Spring		Foll		In Peanut Butter		
dust isomers	isomer	Plot A		Plot B	Plot A	Plot B	Plot A	Plot		
				Experim	nent Station					
67.8	5.4	2.0	6			0.04	0.02	0.01	0.08	
132.0	11.0	4.0	12		• • • •	0.02	0.03	0.09	0.07	
				Hil	l Farm					
48.8	4.5	1.5	4	0.00	0.03	0.00	0.05	0.07	0.00	
88.0	7.3	2.6	8	0.10	0.00	0.00	0.02	0.00	0.00	
142.8	11.9	4.3	12	0.08	0.01	0.09	0.10	0.15	0.15	
				Turr	ner Farm					
42.0	3.5	1.3	4	0.00		0.00		0.00		
84.0	7.0	2.5	8	0.05		0.12		0.10		
168.0	14.0	5.1	16	0.10		0.10		0.00		
168.0	14.0	5.1	16 ^b					0.00		

" Corrected for apparent values in untreated controls.

^b Activated carbon applied at rate of 200 pounds per acre prior to peanut planting.

Among the Hill Farm samples the flavor scores, even when significantly low, indicate only slight off-flavor and show no regular correlation with the dosage of insecticide to which soil was exposed or consistency among samples from replicate plots. In the samples from the Turner Farm only barely perceptible off-flavors were noted; there were no significant differences among these flavor scores. The sample from the plot treated with activated carbon did not differ significantly from the others. The general means of all flavor scores for each treatment also show no regular correlation with the quantities of insecticide to which soils were exposed.

Conclusions

In general, the results provide no evidence that the benzene hexachloride used on cotton accumulated appreciably in the soils or caused any flavor defects or accumulation in peanuts grown on the plots the following year. Because of its high gamma content, 36%, the heaviest dosage of 5.1 pounds of the gamma isomer per acre deposited only 14.0 pounds of total isomers per acre.

In tests on samples grown at Holland, Va., in 1951 (4), benzene hexachloride averaging 0.60 p.p.m. was found and offflavors were observed when 5.1 pounds of the gamma isomer per acre was used; however, the total dosage of benzene hexachloride isomers was 41.1 pounds per acre.

Acknowledgment

The authors are indebted to Lorin C. Fife, Bureau of Entomology and Plant Quarantine, for supplying the peanut and soil samples, and to Robert D. Chisholm and Louis Koblitsky of the same bureau for preparing the soil samples; to the Bureau of Plant Industry, Soils, and Agricultural Engineering for preparing the peanut butter; and to the following members of the Bureau of Human Nutrition and Home Economics: Elsie H. Dawson for advice on planning the palatability studies, Eleanor L. Geissenhainer and Ruth A. Redstrom for assistance in carrying out the palatability studies, and Albert B. Parks and Elsie F. Dochterman for statistical analyses of the data.

Literature Cited

- Cochran, W. G., and Cox, G. M., "Experimental Designs," Chap. 11, New York, John Wiley & Sons, 1950.
- (2) Dawson, E. H., Gilpin, G. L., Kirkpatrick, M. E., and Weigel, C. A., J. Agr. Food Chem., 1, 399-403 (1953).
- (3) Hornstein, Irwin, Anal. Chem., 24, 1036-7 (1952).
- (4) Reynolds, H., Gilpin, G. L., and Hornstein, I., J. Agr. Food Chem., 1, 772-6 (1953).
- (5) Riemschneider, R., Anz. Schädlingskunde, 26, 37-8 (1953).
- (6) Schechter, M. S., and Hornstein,
 I., Anal. Chem., 24, 544-8 (1952).
- (7) Terry, M. E., Bradley, R. A., and Davis, L. L., Food Technol., 6 (7): 250-4 (1952).
- (8) Wilson, J. K., and Choudri, R. S., J. Agr. Research, 77, 25-32 (1948).

Received for review February 12, 1954. Accepted June 8, 1954. Work done in cooperation with the South Carolina Pee Dee Experiment Station.

PESTICIDES AND FLAVOR

Flavor of Peanut Butter as Affected by Aldrin, Chlordan, Dieldrin, Heptachlor, and Toxaphene Used as Insecticides in Growing Peanuts

GLADYS L. GILPIN, RUTH A. REDSTROM, and HOWARD REYNOLDS, Human Nutrition Research Branch FRED W. POOS, Entomology Research Branch

Agricultural Research Service, U. S. Department of Agriculture, Beltsville, Md.

FLAVOR DAMAGE TO SOME FOODS from use of benzene hexachloride as an insecticide in growing the crops has emphasized the importance of determining the effect of new insecticides on flavor of food before they are recommended for specific application.

While soil applications of comparatively low dosages of either technical benzene hexachloride or lindane give excellent control of southern corn rootworm on peanuts, previous studies made in this laboratory (1) have shown that peanuts grown in soils treated with either of these insecticides develop characteristic off-flavors and that these off-flavors may be present in peanut butter or carried over into potatoes french-fried in the peanut oil (made in the pilot plant of the Planters Nut and Chocolate Co., Suffolk, Va.). Other investigators have reported similar results with peanuts and peanut butter (2).

In limited studies on peanut butter (1) from peanuts grown with aldrin, dieldrin, and toxaphene, insecticides which also give very effective control of the southern corn rootworm on peanuts, it appeared that, in the formulations and dosages used, these insecticides did not affect the flavor of the product. Because the previous tests were limited, further studies were carried out in 1951 and 1952 to evaluate the flavor of peanuts grown in soils treated with these insecticides and with heptachlor and chlordan. Tests with aldrin, toxaphene, and heptachlor included variations in dosage rates, insecticide formulations, and methods of application with different kinds of farm equipment.

Peanuts used in these studies were

grown at the Tidewater Field Station (Holland, Va.) of the Virginia Agricultural Experiment Station under carefully controlled conditions or at outlying farms in Nansemond County and other counties in the Virginia peanut-growing area. In several instances, the same insecticide treatment was used at two of the locations. The peanuts were grown in heavy or moderately heavy soils, with the exception of samples No. 7 and 20, Table I, which were grown in light, sandy soil.

The peanuts for the 1951 study (Table I) were of Virginia Jumbo variety, except for samples 6, 11, and 19. which were Virginia Runner variety. Eighteen samples were from treated fields and three were check samples from untreated fields. The treated samples included nine grown with aldrin, six