Residues in Three Peanut Varieties Grown in Dieldrin Treated Soil

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Three commercial peanut varieties (Starr Spanish, Florunner, and Florigiant) were grown in field plots treated with 0, 1, and 4 kg dieldrin per hectare. Soil samples were taken at planting; soil and aerial portions were sampled 10 weeks into the growing season. Peanuts were grown to maturity (16 weeks), harvested, and dried before shelling. Soils, hay, shells, and meats were analyzed for dieldrin content. All soil levels of insecticide caused detectable residues in all plant parts; furthermore, the dieldrin content of each plant part was proportional to the soil level in which it was grown. Varietal difference was detected only in the residue content of the shells where the Florunner variety contained significantly greater dieldrin levels than Starr Spanish and Florigiant. It appears possible to predict dieldrin levels in peanuts when soil residue levels are known.

Evidence is accumulating that dieldrin is taken up from the soil into a number of crop plants (Beestman *et al.*, 1969; Lichtenstein *et al.*, 1965; Wheeler *et al.*, 1967). The persistence of this and other organochlorine insecticides in agricultural soils is well known (Wilkinson *et al.*, 1964) and crop residues from this source will continue to appear.

Morgan *et al.* (1967) reported that 0.02 to 0.08 ppm of dieldrin remained in soil 15 mo following soil treatment with aldrin at 2 kg per hectare. Whole peanuts grown in the same soil contained 0.03 to 0.09 ppm of dieldrin residue. Peanuts planted in soil immediately after treatment with 2 kg per hectare aldrin contained dieldrin residue of 0.3 to 0.9 ppm in shells and 0.5 to 0.6 ppm in meats. These same authors reported similar results for heptachlor-hepatachlor epoxide soil treatment. In these latter tests there were also detectable levels of aldrin and heptachlor in shells and meats. Earlier, Beck *et al.* (1962), in a similar study, had shown dieldrin and heptachlor epoxide residues in hay and shells and meats of peanuts resulting from contaminated soil. Sheets *et al.* (1969) reported a linear relationship between dieldrin and DDT concentrations in peanuts and tobacco, and rates of soil applications.

Residue resulting from contaminated soil is particularly a problem in a crop such as peanut where the major commodity develops within the soil. The purpose of this investigation was to determine whether there are varietal differences in soil-incurred dieldrin residues, and to determine whether the residue level in a given plant part is predictable on the basis of soil residue analysis.

MATERIALS AND METHODS

Field Experiment. Dieldrin, 18.7% emulsifiable concentrate (Shell Chemical Co.) was sprayed over field experimental plots at 1 and 4 kg per hectare active ingredient, and incorporated into the soil with a roto-tiller on May 17, 1968, immediately prior to planting. Seeds of Florunner, Florigiant, and Starr Spanish varieties were planted by hand approximately 3 in. apart in two rows in Arredondo fine sand. Insecticide treatments of plots, 6.3 ft x 14 ft with 6-ft alleys between plots, followed a completely randomized block design with three replications. Soil had been fertilized on April 24, 1968, with 530 kg per hectare 5-10-15 (N-P-K) into which was incorporated 3 kg per hectare of active chlordane and 21 kg per hectare of No. 503 fritted trace elements. During the growing season, maneb and carbaryl were used when necessary for leaf spot and insect control. Soils were sampled at planting and 70 days later. Two plants were randomly selected from each plot on the same day as the final soil sample. The entire plants were frozen and stored for analysis of dieldrin residues in shoots. The remaining plants were harvested 112 days after planting, using a digger-shaker with an attached inverter, cured on stack poles, and picked with a carding type picker on Oct. 25, 1968.

SAMPLE PREPARATION AND ANALYSIS

Soil. Soils were brought to 12% moisture and extracted by tumbling 100 g of soil for 1 hr with 200 ml of hexane-acetone (1 to 1) in a Mason jar at 64 rpm. The extract was filtered through glass wool into a 500-ml separatory funnel and washed three times with 100-ml portions of distilled water. Washings were discarded and the hexane layer was filtered through a small Buchner funnel containing Whatman No. 42 filter paper and a mixture of 30 ml of sodium sulfate, 15 ml of Super Cel, and 15 ml of Celite 545. The filtrate was injected directly into a gas chromatograph.

Hay. Hay was chopped and 50-g portions were extracted with 200 ml of hexane-isopropanol (2 to 1). Extracts were filtered through a Buchner funnel and washed with water in a separatory funnel. The water was discarded and the hexane layer was filtered through anhydrous sodium sulfate. An aliquot representing 1.0 g of crop was concentrated to 1 ml and cleaned up using a Sweep Co-distillation (Kontes) apparatus (Storherr and Watts, 1965). The cleanup procedure was modified as follows: the heated (250° C) Storherr tube pre-rinsed with 1 ml of hexane; 1 ml of hexane containing crop was injected (in 250-μl pulses) into the Storherr tube; immediately 1 ml of hexane was injected (in 250-μl pulses); the tube was rinsed with four 1-ml portions of hexane (250-μl pulses); at 3 min intervals; the Teflon tubing was rinsed with 1 ml of hexane.

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Shells and Meats. Shells were separated mechanically from meats, extracted, and cleaned up by the method for nonfatty samples of low moisture content (Bertuzzi *et al.*, 1967). Meats were extracted and cleaned up by the method of Mills (1961), extracting comminuted meats by the procedure recommended for cheese. The Florisil cleanup of peanut oil was modified by omitting the petroleum ether–acetonitrile partitioning and placing the petroleum ether extract directly on the Florisil column as follows. Chromatographic column was packed (approx. 1.0 cm, i.d.) with 2.0 g of Florisil, and topped with 2.5 cm of anhydrous sodium sulfate. The packed column was placed in a 160° C oven for at least 5 hr. The columns were allowed to cool to room temperature and pre-wetted with 10 ml of redistilled hexane. Oil, (100 mg ± 10 mg) weight known to nearest 0.1 mg, was rinsed onto the column with small quantities of hexane; any eluate was discarded. Dieldrin was eluted from the column with 10 ml of Nanograde acetonitrile. Eluate was concentrated, being certain to evaporate the hexane, to 1.0 ml (or greater) for analysis.

Instrumental Parameters. Dieldrin analysis was performed using an F&M Model 700 gas chromatograph equipped with a glass 6 ft × 1/4 in. o.d., 3% QF-1 on 60/80 mesh Gas Chrom Q column at 190° C and a pulsed EC detector at 210° C. The injection port was at 210° C and the flow rate of 5% argon in methane was 60 ml per min. Recoveries of dieldrin from fortified samples averaged 80 to 90% throughout.

Statistical data were obtained by analysis of covariance.

RESULTS AND DISCUSSION

Dieldrin residue averages of three replicates are shown in Table I. Soil residues of 0.7 ppm in 0 kg per hectare treatments resulted from an aldrin treatment of 3.5 kg per hectare on March 1, 1966, a period of about 2 yr prior to this investigation. It can be assumed that other treated soil plots contained this base level of dieldrin contamination prior to beginning the experiment. There was no apparent decrease in dieldrin content in the soil during the experimental period in the 0 kg and 1 kg per hectare treatments; however, the higher 4 kg per hectare rate showed a measurable decrease. This slow degradation or elimination rate of dieldrin from soil substantiates the well known persistence of this class of insecticides.

In all varieties at all treatment levels, the amount of dieldrin showed the progression: shells > meats > hay. An important factor is that peanut meats contained substantial insecticide, even where soil levels were the lowest. A study of Table I indicates there is a linear relationship between the quantity of insecticide in the soil and the concentration of dieldrin detected in shells, meats, and hay. Owing to the presence of residual dieldrin in the soil, a covariance analysis was performed. An adjustment of the treatment totals, for the respective amounts of residue, enabled the comparison of the treatment levels 0, 1, and 4 kg per hectare of the insecticide. The mean effect of application rate is significant at the 0.01 level in the amount of dieldrin in shells, meats, and hay of all varieties. The only statistically significant difference at the 0.01 level among varieties appeared in residue content of shells. The Florunner variety contained significantly greater residue than Starr Spanish or Florigiant.

This difference cannot easily be explained on the basis of morphological characteristics of the shells. The pods and seed of Florunner are intermediate in size between Florigiant, which has much larger pods, and Starr Spanish, which has smaller pods. The pods of Florigiant are more pubescent and have more prominent indentations than the pods of Florunner. The shells of Florunner, however, are thinner than those of Florigiant and the seeds are more compressed within the shell. The difference in dieldrin levels in shells may be related to shell composition, although there are no supporting data for this.

Table I. Mean Dieldrin Levels (ppm) in the Soil and in Three Plant Varieties Following Pre-Planting Soil Fortifications

Dieldrin Application rate (kg/ha)	Mean Dieldrin Level (ppm) Soil		Plant		
	Planting	Preharvest[a]	Hay[a,b]	Meat[c]	Shell[c]
		Starr Spanish			
0	0.7	0.9	0.06	0.45	1.15
1	2.1	1.4	0.14	1.33	4.16
4	5.6	3.5	0.24	2.50	9.48
		Florunner			
0	0.7	0.8	0.06	0.65	1.56
1	1.5	1.7	0.15	1.35	4.55
4	3.9	3.4	0.27	3.00	11.21
		Florigiant			
0	0.7	0.9	0.07	0.80	1.56
1	2.0	2.1	0.10	1.13	3.10
4	4.5	3.1	0.28	3.53	9.64

[a] 70 day samples. [b] p.p.m. based on fresh weight. [c] 112 day samples—dried.

The legal tolerance of dieldrin in peanut meats and peanut hay for cattle is 0. Extrapolation of data in Table I reveals that approximately 0.05 ppm in the soil will produce 0.05 ppm in meats. The correlation coefficient between hay and meats is r = 0.912, and between hay and shells is r = 0.881. These coefficients indicate clearly that if dieldrin is present in peanut hay, it is also present in the meats and the shells in predictable quantities.

It appears on the basis of this work that the quantity of dieldrin in peanut plant parts can be predicted by the level of soil contamination, at least in the soil type used in these studies. Soil type can be expected to affect the quantity of dieldrin residues (Beestman *et al.*, 1969).

A check of soil levels in peanut growing areas prior to planting could assist the grower by providing a warning of possible above tolerance crop residues.

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LITERATURE CITED

Beck, E. W., Dawsey, L. H., Woodham, D. W., Leuck, D. B., Morgan, L. W., *J. Econ. Entomol.* **55,** 953 (1962).
Beestman, G. B., Keeney, D. R., Chesters, G., *Agron. J.* **61,** 390 (1969).
Bertuzzi, P. A., Kamps, L., Miles, C. I., Burke, J. A., *J. Ass. Offic. Anal. Chem.* **50,** 623 (1967).
Lichtenstein, E. P., Schulz, K. R., Skrentny, R. F., Stitt, P. A., *J. Econ. Entomol.* **58,** 742 (1965).
Mills, P. A., *J. Ass. Offic. Anal. Chem.* **44,** 171 (1961).
Morgan, L. W., Leuck, D. B., Beck, E. W., Woodham, D. W., *J. Econ. Entomol.* **60,** 1289 (1967).
Sheets, T. J., Jackson, M. D., Mistric, W. J., Campbell, W. V., *Pestic. Monit. J.* **3,** 80 (1969).
Storherr, R. W., Watts, R. R., *J. Ass. Offic. Anal. Chem.* **48,** 1154 (1965).
Wheeler, W. B., Frear, D. E. H., Mumma, R. O., Hamilton, R. H., Cotner, R. C., J. Agr. Food Chem. **15,** 231 (1967).
Wilkinson, A. T. S., Finlayson, D. G., Morley, H. V., *Science* **143,** 681 (1964).

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