

Behavior of DDT in Potatoes during Commercial and Home Preparation

F. C. Lamb,¹ R. P. Farrow,² E. R. Elkins,² R. W. Cook,² and J. R. Kimball¹

Potatoes grown in soil treated over a 5-year period with DDT were harvested and prepared for serving by commercial canning and home preparative procedures. Low concentrations of *o,p'*-DDT, *p,p'*-DDT, and *p,p'*-DDE were present at harvest. Commercial washing operations removed about 20% of the total DDT residue from potatoes and lye peeling plus washing removed about 94%. Com-

mercial processing further reduced the residue to insignificant levels. During home preparative procedures, peeling removed more than 91% of the residue. There was no significant decrease from the original residue when potatoes with skins were boiled or pressure cooked. Potatoes stored at 45° F. for a period of 6 weeks showed no significant loss of residue.

Some data have been collected on the effect of washing and processing on residues of DDT [1,1,1-trichloro-2,2-bis(*p*-chlorophenyl) ethane] in fruits and vegetables (Bohm *et al.*, 1950; Brittin and Fairing, 1950; Halter and Carter, 1950; Lamb *et al.*, 1950; Lamb *et al.*, 1948; Manalo *et al.*, 1946; Miller *et al.*, 1957; Tressler, 1947; Walker, 1949). Most of this work was carried out more than 16 years ago, utilizing colorimetric analytical methods that could not separate and detect all the isomers and breakdown products with the ease now available from chromatographic methods. Carter (1948) studied the effect of cooking on DDT in beef. Other work published recently reflects growing interest in the effect of food preparative steps on pesticide residues (Carlin *et al.*, 1966; Koivistoinen and Karinpää, 1965; Koivistoinen *et al.*, 1964a, 1964b, 1964c; Koivistoinen *et al.*, 1965a, 1965b, 1965c). Studies in this laboratory demonstrated the conversion of *p,p'*-DDT to *p,p'*-TDE [2,2-bis(*p*-chlorophenyl)-1,1-dichloroethane] during the processing of canned spinach (Farrow *et al.*, 1966). The authors have also reported on the removal of malathion, DDT, and carbaryl from tomatoes by commercial and home preparative methods (Farrow *et al.*, 1968). The work described here is a further portion of this project, which was designed to obtain information on the effects of commercial and home preparative operations on permissible pesticide residues in selected commercially important crops.

EXPERIMENTAL

Pesticide Application. Potatoes of the White Rose variety were planted during the week of August 30, 1965, on an experimental plot at the Riverside Experiment Station, Riverside, Calif. The field had been treated with DDT during the period from 1952 to 1956 according to the following schedule.

Date	DDT, Lb./Acre
9-25-52	19.1
11-3-53	19.6
11-4-54	20.0
10-27-55	19.2
10-6-56	23.2

Analytical data on soil samples taken from this plot and from an adjacent plot over a period of 11 years were supplied by the Department of Entomology of the Riverside Experiment Station, University of California. On the last sampling date in September 1963, the organic chlorine content of the soil from the DDT-treated plot was 5.1 p.p.m. and that of the soil from the control plot was 0.9 p.p.m. Since DDT contains 50% chlorine, twice the above figures would represent the equivalent level of DDT—i.e., 10.2 and 1.8 p.p.m., respectively.

Potatoes grown on this plot were used for both the commercial and home preparative experiments.

Commercial Preparative Methods. Potatoes were subjected to commercial canning procedures using equipment available at the Berkeley Laboratory. This equipment includes an experimental washer specially constructed to simulate commercial operations on a pilot plant scale. Potatoes smaller than about 1/2-inch diameter were discarded and approximately 18 kg. (40 pounds) ranging from 1/2- to 1 1/2-inch diameter were used for commercial processing. Three samples consisting of at least 10 potatoes and weighing approximately 0.45 kg. (1 pound) each were taken for analysis before washing. Each sample was homogenized, and two 100-gram subsamples were extracted.

The remaining portion of the 18-kilogram sample lot was washed in the experimental washer in the following sequence: reel washer, 30 seconds, 80 pounds water spray pressure; spray-immersion washer, 65 seconds, 65 pounds pressure; spray washer, 30 seconds, 65 pounds pressure; reel washer, 30 seconds, 80 pounds pressure.

At the end of the washing sequence, three additional

¹ National Canners Association, Berkeley, Calif. 94710
² National Canners Association, Washington, D. C. 20036

samples were taken for analysis and the remaining potatoes divided into two equal portions, which were peeled as follows: Potatoes were dipped in a 5% lye solution at 212° F. for 5 minutes. They were then run through the reel washer twice at full water pressure, about 98 pounds. Three samples of the peeled potatoes were taken for analysis.

Potatoes were dipped in a 15% lye solution at 145° to 155° F. for 2½ minutes. They were rinsed by running through the reel washer twice at full water pressure, and three samples were taken for analysis.

The peeled potatoes from both lots were filled into No. 303 cans using 312-gram (11 ounces) fill weight. The cans were filled with 2% salt brine and exhausted for 10 minutes at 210° F. They were then closed and processed in a still retort for 30 minutes at 240° F., water-cooled to about 100° F., and stored at room temperature until analyzed.

The 100-gram portions of washed and peeled analytical samples were chopped in a Hobart food cutter, mixed thoroughly, and held in frozen storage until they could be analyzed. In addition, portions of the ground sample were set aside for moisture determinations, thus enabling computation of test results on a moisture-free basis.

Home Preparative Methods. Home preparative steps and cooking procedures were carried out in the Washington Laboratory under the direction and general supervision of professional home economists. About 18 kg. (40 pounds) of raw potatoes were received by air express 1 week after harvest. After randomizing, three 0.45-kg. (1 pound) samples were withdrawn, weighed, and given a light rinse to remove any adhering soil. The samples were chopped separately in a Hobart mixer and two 100-gram subsamples from each sample extracted. Total solids determinations were made on each sample.

Three 0.68-kg. (1½ pounds) samples were boiled for 35 minutes in sufficient water to cover the potatoes. The water contained ½ teaspoon of salt. After cooking, each sample was homogenized in a blender, and two 100-gram subsamples were extracted.

Three samples were also cooked in a pressure cooker for 11 minutes and extracted. Total solids were determined on each sample.

Analytical Methods. The extraction and cleanup method used for DDT was essentially that of Mills (1959) and consisted of blending the sample in an Omni-Mixer with alcohol and petroleum ether and eluting the extract from Florisil using 6% ethyl ether in petroleum ether as the eluting solvent. Petroleum ether extracts were stored

at -10° F. until analyzed by electron-capture gas-liquid chromatography.

A Packard Model 800 gas chromatograph equipped with a 1.83-meter × 6-mm. (6 feet × ¼ inch) glass column and a Wilkens Hy Fi gas chromatograph with a 3.05-meter × 3-mm. (10 feet × ⅛ inch) glass column were used to obtain quantitative data. Both columns were packed with a mixture of equal amounts of 10% DC-200 silicone grease and 15% QF-1 coated on Gas Chrom-Q (Burke and Holswade, 1966). Columns were operated at a temperature of 200° C. and a nitrogen flow rate of 120 ml. per minute in the Packard instrument and 40 ml. per minute in the Wilkens instrument.

Sample Variation and Storage Effects. After completely randomizing the samples, the potatoes were stored in a constant temperature cabinet at 45° F. To monitor the DDT residue on the raw potatoes, samples were taken at about weekly intervals throughout the storage period. At each sampling three 0.68-kg. (1½ pounds) samples were taken and chopped in a Hobart mixer; two 100-gram subsamples were extracted.

A visual inspection of the total DDT values in Table I will suggest that there is no significant storage effect. However, an analysis of variance was carried out to isolate sample-to-sample variation (Table II). The analysis was carried out following a model used for a nested design suggested by Hicks (1956). Confidence limits at the 95% level have been calculated (Tables I, III, IV) to describe the variability of sample averages.

As expected, a significant storage effect is not demonstrated. The variation among samples is small, actually less than the error variance which in this design is due primarily to variations between duplicate subsamples. This is a relatively unusual situation and may be due to the fact that the potatoes were small and uniform in size. They were easily randomized, and the 0.68-kg. (1½ pounds) samples contained a large number of individual units. Larger potatoes could be expected to present some sampling difficulties.

DDT Removal by Commercial Preparative Methods. Data obtained on DDT residues in potatoes after various steps in the commercial processing experiments are shown in Table III. Owing to the adverse growing conditions encountered in this experiment, the amount of pesticide taken up from the soil may not be considered typical of what would be obtained under more normal circumstances; however, the results in Table III show that easily detectable amounts of two DDT isomers and of *p,p'*-DDE [1,1-dichloro-2-bis(*p*-chlorophenyl) ethylene] were

Table I. Behavior of DDT Residues in Potatoes during Storage at 45° F.^a

Days after Harvest	<i>p,p'</i> -DDE	<i>o,p'</i> -DDT	<i>p,p'</i> -DDT	Total DDT
1	0.18 (0.05) ^b	0.05 (0.02) ^b	0.11 (0.04) ^b	0.34 (0.11) ^b
6	0.18 (0.06)	0.07 (0.02)	0.14 (0.03)	0.39 (0.12)
13	0.13 (0.02)	0.05 (0.007)	0.11 (0.028)	0.29 (0.07)
21	0.17 (0.025)	0.07 (0.015)	0.13 (0.03)	0.37 (0.07)
33	0.12 (0.018)	Trace ^c	0.11 (0.028)	0.27 (0.07)
40	0.11 (0.04)	Trace	0.11 (0.03)	0.24 (0.12)

^a Residues in p.p.m., average of six determinations. ^b 95% Confidence limits (wet basis) = SD (Student's *t* at 0.05)/√*N*. ^c Trace, less than 0.05 p.p.m.

picked up by the potatoes. More *p,p'*-DDE (0.5 p.p.m.) than *p,p'*-DDT (about 0.2 p.p.m.) was found in the potatoes.

Washing by cold water removed about 20% of the total DDT residue. Five per cent lye peeling removed 94% of the residue, while 15% lye peeling removed 90% of the DDT residue. More than 96% of the total DDT residue was removed by washing, lye peeling, and commercial processing. Per cent removal has been calculated on a dry weight basis.

DDT Removal by Home Preparative Methods. Table IV lists data on the removal of DDT from potatoes by home preparative methods. In these samples, all detectable *o,p'*-DDT and *p,p'*-DDT was located in or adjacent to the skin. Mechanical peeling by means of a commonly used household potato peeler removed all detectable residues of these two DDT isomers. The only DDT-related com-

pound remaining after peeling was *p,p'*-DDE, and this residue was at a very low level. The gas chromatographic conditions used in the analysis are more sensitive to the presence of DDE than either of the two common DDT isomers.

Boiling the small potatoes with skins for 35 minutes resulted in no significant change in the total DDT. The slight apparent increase in DDE content is within experimental error.

Pressure cooking the potatoes with skins for 11 minutes also resulted in no significant change in the total DDT residue. The relative proportions of the residual DDT-related compounds did not change appreciably during cooking.

ACKNOWLEDGMENT

The authors are indebted to Elsie H. Dawson, Human Nutrition Research Division, Agricultural Research Service, U. S. Department of Agriculture, and to Katherine R. Smith and Gloria Hansen, Home Economics-Consumer Services, National Canners Association, for direction and general supervision of the home preparative procedures. The use of government-owned equipment (pilot washer), under contract AT (04-3)-536 with the Division of Isotopes Development, U. S. Atomic Energy Commission, for certain food preparation operations described in this paper is gratefully acknowledged. The potatoes were grown under the supervision of H. P. Hermanson, Research Entomologist of the Riverside Experiment Station.

Table II. Sample Variation and Storage Effects of DDT in Potatoes
Analysis of Variance

Source	Degrees of Freedom	Sum of Squares	Mean Square	F-Ratio	$F_{0.05}$	Std. Dev.
Total	35	0.3276
Storage	5	0.1057	0.0211	1.819	3.02	0.14
Sample	17	0.0714	0.0042	0.362	2.35	0.06
Error	13	0.1505	0.0166

Table III. Effect of Commercial Processing on DDT Residues in Potatoes^a

Treatment	Total Solids	<i>p,p'</i> -DDE	<i>o,p'</i> -DDT	<i>p,p'</i> -DDT	Total DDT	Confidence Limits, Six Samples	Decrease, %	
							Wet	Dry
Unwashed	14.61	0.54	0.08	0.16	0.77	(0.108)		
Washed	14.85	0.41	0.05	0.13	0.59	(0.14)	23	20
Peeled								
5% lye	16.01	0.05	Trace ^b	Trace	0.05	(0.01)	94	94
Peeled								
15% lye	13.03	0.07	Trace	Trace	0.07	(0.013)	91	90
Processed (canned)								
5% lye	14.86	Trace ^b	Trace	Trace	Trace		96+	96+
Processed (canned)								
15% lye	14.94	Trace	Trace	Trace	Trace		96+	96+

^a Residues in p.p.m., wet basis.

^b Trace, less than 0.05 p.p.m.; 0.03 used for averages.

Table IV. Removal of DDT from Potatoes by Home Preparative Methods^a

Treatment	Total Solids	<i>p,p'</i> -DDE	<i>o,p'</i> -DDT	<i>p,p'</i> -DDT	Total DDT	Confidence Limits	Decrease, %	
							Wet	Dry
Unwashed	11.52	0.18	0.05	0.11	0.34	(0.113)		
Peeled	11.07	Trace ^b	ND ^c	ND	Trace		91	90
Boiled with skins	8.76	0.21	Trace	0.10	0.32	(0.159)	0	28 ^d
Pressure cooked with skins	9.35	0.17	0.06	0.10	0.31	(0.106)	0	14 ^d

^a Residues in p.p.m., wet basis. Average of six determinations.

^b Trace, less than 0.05 p.p.m.; 0.03 used for averages.

^c Not detected.

^d Per cent increase.

LITERATURE CITED

- Bohm, R. O., Lamb, F. C., Lewis, L. D., White, D. G., "Insecticide Residue Studies on Raw and Canned Green Beans," Research Report No. 13600-C, National Cannery Association, February 14, 1950.
- Brittin, W. A., Fairing, J. D., *J. Assoc. Offic. Anal. Chemists* **33**, 599 (1950).
- Burke, J. A., Holswade, W., *J. Assoc. Offic. Anal. Chemists* **49**, 374 (1966).
- Carlin, A. F., Hibbs, E. T., Dahm, P. A., *Food Technol.* **20**, 80 (1966).
- Carter, R. H., *Science* **107**, 347 (1948).
- Farrow, R. P., Elkins, E. R., Cook, R. W., *J. AGR. FOOD CHEM.* **14**, 430 (1966).
- Farrow, R. P., Lamb, F. C., Cook, R. W., Kimball, J. R., Elkins, E. R., *J. AGR. FOOD CHEM.* **16**, 65 (1968).
- Haller, M. H., Carter, R. H., *Proc. Am. Soc. Hort. Sci.* **56**, 116-21 (1950).
- Hicks, Charles R., "Industrial Quality Control," p. 9, October 1956.
- Koivistoinen, P., Karinpää, A., *J. AGR. FOOD CHEM.* **13**, 459 (1965).
- Koivistoinen, P., Karinpää, A., Könönen, M., *J. AGR. FOOD CHEM.* **12**, 555 (1964a).
- Koivistoinen, P., Karinpää, A., Könönen, M., Roine, P., *J. AGR. FOOD CHEM.* **12**, 551 (1964b).
- Koivistoinen, P., Karinpää, A., Könönen, M., Roine, P., *J. AGR. FOOD CHEM.* **13**, 468 (1965a).
- Koivistoinen, P., Könönen, M., Karinpää, A., Roine, P., *J. AGR. FOOD CHEM.* **12**, 557 (1964c).
- Koivistoinen, P., Koskinen, A., Schulman, M., Karinpää, A., Roine, P., Salonen, A., *J. AGR. FOOD CHEM.* **13**, 463 (1965b).
- Koivistoinen, P., Vanhanen, L., Koskinen, E. H., *J. AGR. FOOD CHEM.* **13**, 344 (1965c).
- Lamb, F. C., Lewis, L. D., Bohm, R. O., "Insecticide Residue Studies on Raw and Canned Apricots," Research Report No. 13400-C, National Cannery Association, May 9, 1950.
- Lamb, F. C., Lewis, L. D., Lee, S. K., "Studies on Removal of Insecticide Residues from Apricots," Research Report No. 12441-A, National Cannery Association, October 7, 1948.
- Manalo, Gloria D., Hutson, R., Miller, E. J., Benne, E. J., *Food Packer* **27**, No. 13, 64-8 (1946).
- Miller, L. A., Miles, J. R. E., Sans, W. W., *Can. J. Plant Sci.* **37**, 280 (1957).
- Mills, P. A., *J. Assoc. Offic. Anal. Chemists* **42**, 734 (1959).
- Tressler, C. J., *J. Assoc. Offic. Anal. Chemists* **30**, 140 (1947).
- Walker, K. C., *J. Agr. Res.* **78**, 383-7 (1949).

Received for review November 29, 1967. Accepted January 12, 1968. Work conducted under contract No. 12-14-100-7780 (61) awarded to the National Cannery Association Research Foundation by the Human Nutrition Research Division, Agricultural Research Service, United States Department of Agriculture. Presented before the Division of Agricultural and Food Chemistry, 153rd Meeting, ACS, Miami Beach, Fla., April 1967.