# Absorption of Insecticidal Residues from Contaminated Soils into Five Carrot Varieties

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To determine whether varieties of one vegetable would absorb different amounts of insecticidal residues from soils, five varieties of carrots (*Daucus carota*) differing in size and color (dark red to white) were field-grown during 1961 and 1962 in soils treated at various rates with aldrin or heptachlor. Considerable differences in the rate of absorption of pesticidal residues by the five varieties were noticed. The concentrations of insecticidal residues in the carrots varied from 22 to 80% of the concentration in the soil. The white carrots (White Belgium) contained 50% of the absorbed insecticides within the pulp, while the four remaining varieties contained 70 to 86% of the totally recovered residue within their peels. The vertical distribution of the insecticidal residues within the carrot tissue corresponded to the vertical distribution within the various soil layers. Less insecticidal residue was found in soils along the carrot rows than in areas where no carrots had grown. Although significant differences were noticed in the amounts of ether-extractable substances within the five varieties, no correlation could be established between the amounts of these substances in carrots and the ability of the carrot tissue to absorb, or store, insecticidal residues. The effect of boiling carrots in water on the stability of translocated aldrin or heptachlor residues was also investigated.

Under certain conditions some insecticidal residues in soils may be translocated into various crops (*4*, *6*). The amount of insecticides absorbed and translocated into various plant tissues is dependent on the insecticide employed, the particular crop, and the soil type in which the crop is grown (*3*). Carrots are outstanding in their ability to absorb pesticidal residues from soils (*2*, *4*, *5*, *11*). Residue tolerances for insecticides on or in crops usually refer to a particular crop without considering possible differences between varieties, a factor that could influence the residue level within the plant tissue.

To test whether varieties of one vegetable would absorb different amounts of insecticidal residues from soils, five cultivated varieties of carrots (*Daucus carota*, Umbelliferae) were field-grown in insecticide-contaminated soils. An attempt was also made to determine the distribution of the toxicants within the edible parts of carrots, as related to the location of the insecticidal residue in the soil and to individual carrot characteristics.

## Experimental

**Translocation of Insecticidal Residues into Five Carrot Varieties from Soils Treated with Abnormal Rates of Aldrin and Heptachlor.** To study the persistence and the fate of insecticidal residues in soils, two Carrington silt loam plots (each 30

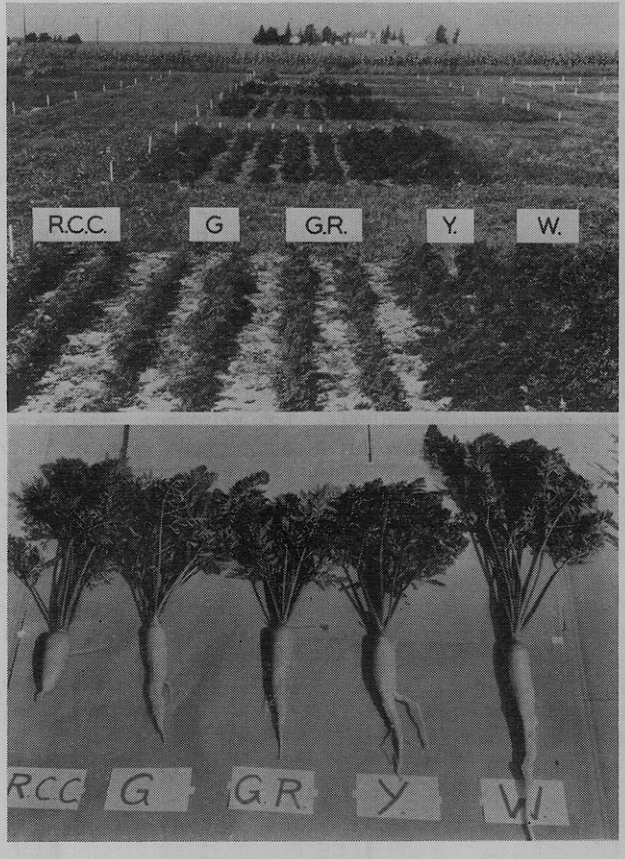


Figure 1. Five carrot varieties grown in 1961 in Carrington silt loam plots

R.C.C. Red Cored Chantenay
G. Gold Pak
G.R. German Red
Y. Yellow Belgium
W. White Belgium

**Table I. Recoveries of Aldrin (A), Dieldrin (D), Heptachlor (H), and Heptachlor Epoxide (HO) Residues from a Carrington Silt Loam and Five Carrot Varieties, Grown on Aldrin- and Heptachlor-Treated Plots (5 Lb./5-Inch Acre) from 1958 through 1962**

| | Recovered from Soils, P.P.M.[a] | | | |
|---|---|---|---|---|
| | A + D | % D[b] | H + HO | % HO[c] |
| August 1961[d] | 2.81 | 54 | 3.08 | 22 |
| August 1962[d] | 3.31 | 56 | 4.26 | 23 |
| May 1962[e] | 2.71 ± 0.19 | 63 | 2.53 ± 0.29 | 31 |
| August 1962[e] | 2.63 ± 0.13 | 63 | 2.41 ± 0.35 | 29 |

| | Recovered from Carrots, P.P.M. | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Whole | | Pulp | | Peel | | | | Whole | | Pulp | | Peel | | | |
| | A + D | % D | A + D | % D | A + D | % D | P % T[f] | C % S[g] | H + HO | % HO | H + HO | % HO | H + HO | % HO | P % T[f] | C % S[g] |
| Red C. Chantenay | | | | | | | | | | | | | | | | |
| 1961 Color. anal. | 0.60 | 52 | | | 3.81 | 54 | 71.8 | 23.8 | 1.03[h] | | | | | | | |
| Bioassay | 0.66 | 52 | | | 3.72 | 59 | ± | ± | 1.09 | 28 | 0.39 | 26 | | | 77.0 | 37.5 |
| 1962 GLC[i] | 0.83 | 64 | 0.22 | 63 | 4.64 | 61 | 1.3 | 1.3 | 1.73 | 25 | 0.48 | 22 | 13.98 | 24 | ± 9.0 | ± 2.5 |
| Gold Pak | | | | | | | | | | | | | | | | |
| 1961 Color anal. | 1.24 | 59 | | | 5.02 | 58 | 69.3 | 40.5 | 1.27[h] | | | | 7.87[h] | | | |
| Bioassay | 1.24 | 54 | | | 5.12 | 61 | ± | ± | 1.60 | 26 | | | 9.93 | 23 | 86.0 | 58.5 |
| 1962 GLC | 1.23 | 63 | 0.15 | 64 | 4.96 | 64 | 6.8 | 3.5 | 2.77 | 25 | 0.79 | 21 | 15.72 | 24 | ± 4.0 | ± 6.5 |
| German Red | | | | | | | | | | | | | | | | |
| 1961 Color. anal. | 0.62 | 52 | | | 3.88 | 50 | 78.5 | 22.3 | 0.96[h] | | | | 4.74[h] | | | |
| Bioassay | 0.59 | 54 | | | 4.12 | 47 | ± | ± | 1.20 | 25 | | | 6.38 | 24 | 73.5 | 42.0 |
| 1962 GLC | 0.76 | 67 | 0.17 | 65 | 2.91 | 62 | 13.5 | 0.8 | 1.92 | 23 | 0.27 | 17 | 9.04 | 25 | ± 0.5 | ± 3.0 |
| Yellow Belgium | | | | | | | | | | | | | | | | |
| 1961 Color. anal. | 0.72 | 48 | | | 4.19 | 49 | 80.8 | 32.3 | 0.97[h] | | | | | | | |
| Bioassay | 0.74 | 53 | | | 3.80 | 39 | ± | ± | 1.16 | 22 | 0.23 | 26 | | | 78.8 | 39.0 |
| 1962 GLC | 1.26 | 65 | 0.23 | 65 | 5.64 | 66 | 8.8 | 5.8 | 1.70 | 23 | 0.42 | 18 | 8.59 | 24 | ± 5.3 | ± 1.0 |
| White Belgium | | | | | | | | | | | | | | | | |
| 1961 Color anal. | 1.51 | 52 | | | 4.14 | 53 | 55.3 | 46.8 | 1.45[h] | | | | 5.29[h] | | | |
| Bioassay | 1.42 | 58 | | | 4.91 | 61 | ± | ± | 1.81 | 23 | | | 5.40 | 20 | 55.5 | 60.5 |
| 1962 GLC | 1.36 | 65 | 0.63 | 62 | 4.13 | 62 | 4.8 | 5.7 | 2.70 | 25 | 1.82 | 20 | 11.20 | 21 | ± 6.5 | ± 2.5 |

[a] Determined by colorimetric analyses.
[b] Dieldrin in per cent of totally recovered residues (A + D).
[c] Heptachlor epoxide in per cent of totally recovered residues (H + HO).
[d] One soil sample (30 cores) collected from 30- × 24-foot area on which all carrots grew.
[e] Mean and standard deviation of analyses of 5 soil samples, each collected along row where particular carrot variety grew.
[f] Residues found in peelings in per cent of residues recovered from whole carrots. Average of results obtained in 1961 and 1962.
[g] Residues found in whole carrots in per cent of total residue recovered from soil. Average of results obtained in 1961 and 1962.
[h] Heptachlor only; no data for heptachlor epoxide available colorimetrically.
[i] Gas liquid chromatography.

× 24 feet) were treated in May of each year, 1958 through 1962, with aldrin or heptachlor at 5 pounds per 5-inch acre. Application of the insecticides, performed as previously described (*4*), resulted in a total dosage of 25 pounds per acre over the 5-year period. Carrots were grown on these plots during the summers of 1961 and 1962.

PROCEDURES IN 1961. Immediately following the insecticidal application in May 1961, five carrot varieties (Red Cored Chantenay, Gold Pak, German Red, Yellow Belgium, and White Belgium) were seeded in an east-west direction in the contaminated and in two control plots. Each carrot variety was grown in two 30-foot rows (Figure 1). The carrots, known to differ in size and also in their carotene content, ranged in color from dark red (German Red) to white (White Belgium).

In August, a soil sample consisting of 30 cores (3/4 inch in diameter and 6 inches long) was collected from each plot. At the same time all the edible parts of each carrot variety were harvested and brought into the laboratory for processing. Each carrot was brushed in warm water to remove adhering soil particles. After that, the crop was rinsed with acetone from a wash bottle, followed by an additional rinsing with water. A subsample of each carrot variety was selected at random and then peeled with a potato peeler. The peels and pulp were then macerated separately with a food grinder (Hobart, Model T-215 food cutter), the ground material was thoroughly mixed, and aliquots were placed in plastic bags within 1-quart ice cream cartons, frozen, and held until extraction. The remaining whole carrots of each variety were macerated without peeling, mixed, subsampled, and frozen for future extraction and analyses.

ANALYTICAL PROCEDURES. The handling of the soil samples, including extraction, cleanup, and colorimetric analyses, was done as described (*7–9*). Carrots were extracted, and the extracts were purified and analyzed colorimetrically (*4, 6*).

In addition, fractions of extracts containing aldrin, dieldrin, heptachlor, or heptachlor epoxide were used for ascending paper chromatography (*10*). In all cases where any of the insecticides were detected colorimetrically, positive results were also obtained by paper chromatography: $R_f$ values secured with purified crop extracts were identical with $R_f$ values obtained with reference grade insecticides. The same extracts were also tested with vinegar flies (*Drosophila melanogaster*, Meig) (*1*).

PROCEDURES IN 1962. The results obtained in 1961 (Table I) revealed considerable differences in the rate of absorption of pesticidal residues by the five carrot varieties.

To determine if these differences were the result of an unequal distribution of insecticidal soil residues, soil samples were collected in May 1962 along the rows where carrots had grown during the previous summer. Fifteen

cores (3/4 inch in diameter, 6 inches long) were taken from an 8-inch-wide path along each row. Since each carrot variety was grown in two 30-foot rows, one soil sample consisted of 30 cores. The resulting five samples were then frozen and held for future analyses.

After the plots had been retreated with aldrin or heptachlor as described (*4*), the five carrot varieties used in 1961 were again seeded on these plots, but the carrot rows were established in a south-north direction and were only 24 feet long. In August the carrots were harvested, processed as in the previous year, and frozen for future analyses. One soil sample, consisting of 30 cores (3/4 inch in diameter, 6 inches long), was collected at the same time from the total growing area of 24 × 30 feet. Five individual soil samples were also collected along the rows where each of the five carrot varieties had grown.

Soil analyses were performed in the same way as in 1961. Carrots, however, were analyzed by gas liquid chromatography. For extraction, 75 grams of macerated mixed carrot material were placed in a 1-pint (473-ml.) Mason jar, containing 75 ml. each of redistilled acetone and hexane. To minimize formation of emulsions, 10 to 15 grams of anhydrous sodium sulfate were added. This mixture was macerated and mixed at medium speed for 5 minutes by means of a Volu-Mix Lourdes homogenizer, then filtered through glass wool into a 500-ml. separatory funnel. After the liquids had passed through the glass wool, 150 ml. of distilled water were added to the carrot material retained by the glass wool and allowed to percolate into the carrot extract within the separatory funnel.

The hexane-acetone-water mixture was then shaken. After separation of the phases the water-acetone layer was discarded. The hexane layer was dried over anhydrous sodium sulfate and used for analysis by gas liquid chromatography.

A Jarrell-Ash gas chromatograph, Model 700, equipped with a 100-mc. tritium electron affinity ionization detector was used. A 4-foot (1.22-meter) column (3/16 inch = 4.76-mm. inside diameter) containing 80- to 90-mesh Anakrom ABS (acid, alcoholic base, washed and siliconized) and 1% SE 30–neopentyl glycol adipate (terminated) was conditioned before use for 48 hours at 200° C. For aldrin and dieldrin determinations a column pressure of 25 p.s.i. of nitrogen, giving a flow rate of 100 ml. per minute, was employed. The injector temperature was held at 250° C., the column temperature at 150° C., and the detector cell at 208° C. The detector potential was 15 volts. Amounts of aldrin and dieldrin added to crop material were recovered to an extent of 90 to 98%. When analyses for heptachlor residues were performed, the column temperature was held at 129° C. A column pressure of 25 p.s.i. of nitrogen resulted in a flow rate of 110 ml. per minute. Temperatures of the injector and the detector cell were as described. The detector potential, however, was 11 volts. Amounts of heptachlor or heptachlor epoxide added to the crop material were recovered to an extent of 95 to 99%.

Because of the reapplication of aldrin and heptachlor in the spring of 1962, the concentration of the insecticidal residues in the soil plots had increased as compared to 1961. The concentrations of the insecticidal residues in the soil along each carrot row were very similar, as shown by the analyses of the soil samples collected in May and in August 1962 (Table I, soil residues, footnote [e]). Therefore, any difference in the translocation of insecticidal residues into the carrot tissue was a function of the particular carrot variety. Moreover, the amounts of aldrin and heptachlor residues in soils along the carrot rows were significantly smaller than in areas where no carrots had grown. This could have resulted from the absorption of insecticides from the soil into the carrots.

The amounts of chemicals found within the edible part of one carrot variety during the two growing seasons were very similar, especially when expressed in per cent of the residue concentration in the soil at harvest time. However, considerable differences existed in the rate of absorption among the carrot varieties themselves (Table I). The insecticidal concentrations ranged from 0.6 to 1.5 p.p.m. in carrots grown on aldrin-treated soil and from 1.1 to 2.8 p.p.m. in those grown on heptachlor-treated soil. The amounts of dieldrin or heptachlor epoxide were similar in all crops and soils when expressed in per cent of the totally recovered residue.

Most insecticidal residues were absorbed by carrots of the varieties White Belgium and Gold Pak. Concentrations of the residues in these carrots amounted to 41 and 47%, respectively, of the insecticidal concentrations in aldrin-treated soils and to 59 and 61%, respectively, of the residue levels in heptachlor-contaminated soils. Conversely, carrots of the Red Cored Chantenay and German Red varieties contained the smallest amounts of residues, which in comparison with other crops were still relatively large (*4*). The White Belgium variety was also unique in that close to 50% of the absorbed insecticidal residues were located within the inner part (pulp) of these carrots. The remaining carrot varieties, however, contained 70 to 86% of the total insecticidal residue content within their peels. This makes it possible to remove the major part of the toxicants by peeling.

**Translocation of Insecticidal Residues into Two Carrot Varieties from Soils Treated at Practical Levels of Aldrin and Heptachlor.** In May 1960 six experimental plots (each 30 × 24 feet) were established on a Carrington silt loam soil and treated with aldrin or heptachlor at 1, 2, or 3 pounds per acre. Treatment was performed as described (*4*), and was followed by rototilling of the soils to a depth of 4 to 5 inches. These soil treatments were repeated in May 1961 and in May 1962, resulting in total applications of either 3 (3 × 1), 6 (3 × 2), or 9 (3 × 3) pounds per acre of aldrin or heptachlor over the 3-year period. Carrots of the Red Cored Chantenay and White Belgium varieties were grown in 1962 on these plots to test the absorption of insecticidal residues into carrots from loam soils treated at "normal" dosages. The carrots were harvested in August and processed as described. At the same time, soil samples were collected from the total areas of each experimental plot. Both carrots and soils were extracted and analyzed by gas liquid chromatography, as previously mentioned (*5*).

The amounts of aldrin and heptachlor in the soil were very similar (Table II) and ranged from 0.7 pound per acre or 0.44 p.p.m. (3 × 1 pound per acre treatment) to 2.1 pounds per acre or 1.33 p.p.m (3 × 3 pounds per acre treatment). Carrots, however, absorbed more residues from the heptachlor-treated soils than from those containing aldrin and dieldrin. Also, carrots of the White Belgium variety absorbed nearly twice the amount of insecticidal residues as those of the Red Cored Chantenay variety. The increasingly higher residue levels within the different soils, due to the different insecticidal application rates, did not cause a proportional increase in the amount of toxicants translocated into the carrot tissue.

A residue level below 0.1 p.p.m. in carrots was observed only with the Red Cored Chantenay variety, grown in a soil that had been treated with aldrin at 1 pound per acre each year for three successive years.

**Distribution of Insecticidal Residues within Carrot Tissue.** LATERAL. Carrots of the German Red variety, grown in 1962 on aldrin- or heptachlor-treated soils (5 pounds per acre per year from 1958 through 1962), were harvested and cleaned as described (*4*). Afterward, seven concentric layers were secured from the upper 3-inch pieces of each of 20 carrots in the following way.

The outer layer (No. 1) was removed by means of a potato peeler. After that, the inner core (No. 7) was removed with a cork borer (o.d. 3/16 inch). The next layers (No. 6, 5, 4, and 3) were removed with cork borers which had outside diameters of 5/16, 7/16, 9/16, or 3/4 inch. Layer 2 was the carrot material that remained after removal of the peel and the five inside cores. Comparable layers from the 20 carrots were then weighed, extracted, and analyzed by gas liquid chromatography as described.

It was found (Table III) that the peel, representing 14% of the total carrot

weight, contained most of the insecticidal residues. The second layer, though representing half of the total carrot material, contained significantly less residues than the peelings. Practically all of the recovered insecticidal residues (96 or 99%) were located in these two outside layers of the carrot root.

VERTICAL. Two Carrington silt loam plots (24 × 30 feet) were established in 1963. One was treated with aldrin at 1 pound per acre, the other with dieldrin at 3 pounds per acre. After the insecticidal application (*4*) the soils were rototilled to a depth of 4 to 5 inches. Carrots were grown in these soils to determine the vertical distribution of insecticidal residues in the plant tissue as related to the insecticidal concentrations in comparable soil layers. Carrots of the White Belgium variety were seeded in a 30-foot row on both treated plots and the Red Cored Chantenay variety only on the dieldrin-contaminated soil. After harvesting and cleaning as described (*4*), the carrots were cut into either three (Red Cored Chantenay) or four (White Belgium) vertical sections. The total length of the Red Cored Chantenay variety was $4^1/_2$ to $5^1/_2$ inches, and of the White Belgium variety, 8 to 12 inches. The uppermost section ($^1/_2$ to 1 inch long) usually protruded out of the ground and contained some chlorophyll. The following sections were 2-inch-long vertical pieces of the whole carrot, which resulted in two additional sections (0 to 2 and 2 to 4 inches) from the Red Cored Chantenay variety, and in three (0 to 2, 2 to 4, 4 to 6 inches) from the White Belgium variety. All of the carrot material from one particular vertical level of each variety was then macerated, mixed, and frozen for future analyses. As a control, nonsectioned, whole carrots were also macerated, mixed, and frozen.

Soil samples, consisting of 15 cores each, were collected along each row where carrots had grown. Each core ($^3/_4$ inch in diameter and 6 inches long) was divided into three 2-inch vertical layers (0 to 2, 2 to 4, and 4 to 6 inches). The comparable soil layers of each row were then combined and frozen for future analyses. As a control, 15 additional soil cores (6 inches deep, but not subdivided) were collected along each row and combined into one sample. The analytical result of this sample should be similar to the sum of the data obtained from analyses of the upper three 2-inch soil layers. Soils and carrots were extracted and analyzed by gas liquid chromatography as previously described (*5*).

Results (Table IV) obtained with soils showed that more than 70% of the recovered insecticidal residues were located in the upper 2-inch soil layer, and 20 to 25% in the following 2-inch soil layer (2 to 4 inches). With carrots, the upper $^1/_2$- to 1-inch section, usually not consumed by humans and discarded, contained relatively high concentrations of translocated residues. The distri-

**Table II. Recoveries of Aldrin (A), Dieldrin (D), Heptachlor (H), and Heptachlor Epoxide (HO) Residues from Carrington Silt Loam and Carrots Grown in 1962 on Aldrin- and Heptachlor-Treated Plots**

| | | *Insecticides Applied to Soil in 1960, 1961, and 1962, Lb./5-Inch Acre* | | | | | |
|---|---|---|---|---|---|---|---|
| | | *Aldrin* | | | *Heptachlor* | | |
| | | *1* | *2* | *3* | *1* | *2* | *3* |
| | | *Recovered at Harvest, P.P.M.*[a] | | | | | |
| Soil | A + D | 0.47 | 0.77 | 1.34 | H + HO | 0.41 | 0.90 | 1.33 |
| | % D[b] | 64 | 62 | 62 | % HO[b] | 27 | 23 | 20 |
| Carrots | | | | | | | | |
| R. C. C.[c] | A + D | 0.08 | 0.30 | 0.27 | H + HO | 0.15 | 0.44 | 0.69 |
| | % D | 77 | 62 | 63 | % HO | 36 | 20 | 19 |
| | C % S[d] | 17 | 39 | 20 | C % S | 37 | 49 | 52 |
| W. B.[e] | A + D | 0.17 | 0.47 | 0.53 | H + HO | 0.43 | 0.71 | 0.98 |
| | % D | 68 | 59 | 64 | % HO | 23 | 30 | 17 |
| | C % S | 36 | 61 | 40 | C % S | 105 | 79 | 74 |

[a] Determined by gas liquid chromatography.
[b] Amount of dieldrin (% D) or heptachlor epoxide (% HO) in per cent of totally recovered residue in crop.
[c] Red Cored Chantenay.
[d] Total residue in carrots in per cent of total residue in soil.
[e] White Belgium.

**Table III. Lateral Distribution of Insecticidal Residues in Upper 3 Inches of Carrots (German Red), Grown in 1962 on Aldrin- or Heptachlor-Treated Loam Soils**

| | | *Recovered, P.P.M.*[a] | | | | | |
|---|---|---|---|---|---|---|---|
| | | *A + D*[c] | *% D*[d] | *% T*[e] | *H + HO*[f] | *% HO*[g] | *% T* |
| *Soil, 6 Inches Deep* | | 3.31 | 56 | | 4.26 | 23 | |
| *Carrot, Concentric Layers* | *Wt., % T*[b] | | | | | | |
| 1[h] | 14 | 4.76 | 63 | 79 | 8.80 | 24 | 56 |
| 2 | 51 | 0.30 | 61 | 17 | 1.61 | 27 | 43 |
| 3 | 16 | 0.13 | 63 | 3 | 0.08 | 58 | 0.5 |
| 4 | 8 | 0.06 | 64 | 0.6 | 0.08 | 57 | 0.2 |
| 5 | 5 | Trace | | | Trace | | |
| 6 | 4 | Trace | | | Trace | | |
| 7[i] | 2 | Trace | | | Trace | | |

[a] Determined by gas liquid chromatography.
[b] Weight of particular layer in per cent of total weight of carrot.
[c] Total of aldrin and dieldrin.
[d] Dieldrin in per cent of totally recovered residues (A + D).
[e] Residues recovered from one particular layer in per cent of residues recovered from whole carrot 3 inches long.
[f] Total of heptachlor and heptachlor epoxide.
[g] Heptachlor epoxide in per cent of totally recovered residues (H + HO).
[h] Outside layer of carrot.
[i] Inner core of carrot.

**Table IV. Vertical Distribution of Insecticidal Residues in Soils and Carrots Grown in 1963 on Aldrin (A)- or Dieldrin (D)-Treated Silt Loam Soils**

| | *Insecticides Applied to Soil* | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | *Aldrin, 1 Lb./5-Inch Acre* | | | | *Dieldrin, 3 Lb./5-Inch Acre* | | | |
| | *Recovered, P.P.M.*[a] | | | | | | | |
| *Vertical Sections, Inches* | *Soil*[b] | | *W. B.*[c] | | *D* | | | |
| | *A + D* | *% D*[e] | *A + D* | *% D* | *Soil* | *W. B.* | *Soil* | *R. C. C.*[d] |
| Upper $^1/_2$–1[f] | ... | .. | 0.05 | 62 | ... | 0.43 | ... | 0.34 |
| 0–2[g] | 0.30 | 43 | 0.05 | 60 | 1.86 | 0.51 | 2.21 | 0.18 |
| 2–4 | 0.07 | 46 | 0.02 | 67 | 0.55 | 0.35 | 0.83 | 0.03 |
| 4–6 | 0.02 | 79 | 0.01 | 83 | 0.09 | 0.10 | 0.08 | [h] |
| 0–6 | 0.13 | 55 | 0.02[i] | 50 | 1.28 | 0.13[i] | 1.39 | 0.15[i] |

[a] Determined by gas liquid chromatography.
[b] Soil sampled after harvest along row where carrots had grown.
[c] Carrot variety White Belgium.
[d] Carrot variety Red Cored Chantenay.
[e] Dieldrin in per cent of total residue (aldrin and dieldrin) recovered from one section.
[f] Upper $^1/_2$–1 inch of carrot, usually above ground.
[g] 0- to 2-inch soil layer or upper 2-inch carrot section, grown in 0- to 2-inch soil layer.
[h] Only upper $4^1/_2$ inch of carrots analyzed.
[i] Nonsectioned edible part of whole carrots.

bution of the insecticidal residues in the following edible carrot sections corresponded to some extent to the distribution of the residues in the soil. Fifty-five to 62% of the recovered residues from the edible parts of carrots of the White Belgium variety were located in the 0- to 2-inch section and 25 to 36% in the 2- to 4-inch section. Only 10% of the totally recovered residues were in the 4- to 6-inch carrot section. Carrots of the Red Cored Chantenay variety contained 92% of the totally recovered residues from the edible parts within the 0- to 2-inch section.

It appears from these data that the distribution of the insecticidal residues depends not only on the particular carrot variety, but also on the concentration and location of the residues within the soil.

**Ether-Extractable Substances in Carrots.** Since aldrin and heptachlor residues are fat-soluble, it was felt that a relationship might exist between ether-extractable substances in carrots and the ability of carrots to absorb insecticidal residues from soils.

Carrots grown in 1961 and 1962 on insecticide-free soil were used to measure the amounts of ether-extractable substances in the five carrot varieties. Three hundred to 500 grams of macerated carrot tissue (whole carrots, peel, or pulp) were dried at 50° C. for 48 hours in a drying oven, while warm air was passing over the plant material. Ten grams of the dried carrot tissues were then extracted for 10 hours with diethyl ether within a Soxhlet apparatus. After that the extracts were adjusted to a volume of 100 ml. A 1% concentration of the plant material in ether was then prepared for determination of absorbance characteristics in a spectrophotometer. Each extract had an absorption peak at 448 mμ, which is a characteristic of carotene (*12*). Absorbance figures were then determined for each extract, read at this wave length against a blank of diethyl ether (Table V).

The remaining ether extract (99 ml.) was then evaporated to dryness within a beaker, on a hot sand bath. The weight of the ether-extractable substances in per cent of the dry carrot material extracted was then determined (Table V).

Significant differences in the amounts of ether-extractable substances within the five carrot varieties were noticed. Although carrots of the White Belgium variety absorbed more insecticidal residues than the other four, their carotene and the amount of ether-soluble substances found were among the lowest. Carrots of the German Red and Red Cored Chantenay varieties absorbed considerably less insecticidal residues, yet they contained more ether-soluble substances. No correlation could be established between the amount of these substances in carrots and the ability of the carrot tissue to absorb, or store, insecticidal residues. It appears that other reasons, such as the differently developed root systems of the carrot varieties and other genetic factors, are responsible for these individual properties.

**Table V. Diethyl Ether–Extractable Substances in Carrots**

| Carrot Varieties | Whole | | Pulp | | Peel | |
|---|---|---|---|---|---|---|
| | Wt. %[a] | Abs.[b] | Wt. % | Abs. | Wt. % | Abs. |
| Red C. Chantenay | 1.00 ± 0.11 | 0.306 | 0.99 ± 0.18 | 0.262 | 1.18 ± 0.03 | 0.520 |
| Gold Pak | 1.30 ± 0.03 | 0.475 | 1.40 ± 0.02 | 0.740 | 1.51 ± 0.03 | 0.730 |
| German Red | 1.70 ± 0.03 | 0.560 | 1.47 ± 0.66 | 0.240 | 1.69 ± 0.15 | 0.365 |
| Yellow Belgium | 0.66 ± 0.15 | 0.057 | 0.55 ± 0.18 | 0.007 | 0.78 ± 0.10 | 0.038 |
| White Belgium | 0.65 ± 0.07 | 0.003 | 0.65 ± 0.20 | 0.003 | 0.85 ± 0.17 | 0.010 |

[a] Weight of ether-extractable substances in per cent of total dry carrot material extracted. Average of determinations obtained with carrots grown in 1961 and 1962 on insecticide-free soil.

[b] Absorbance at 448 mμ of diethyl ether extract (concentration 1%) of carrot material, read against diethyl ether in spectrophotometer.

**Table VI. Effect of Boiling Carrots (Red Cored Chantenay) in Water on Persistence of Translocated Aldrin (A), Dieldrin (D), Heptachlor (H), and Heptachlor Epoxide (HO) Residues**

| Growing Season | Carrot Condition | Recovered from Soils, P.P.M.[a] | | | |
|---|---|---|---|---|---|
| | | A + D | % D[b] | H + HO | % HO[c] |
| 1962 | | 3.73 | 55 | 3.81 | 25 |
| 1963 | | 2.25 | 85 | 1.96 | 28 |
| | | Recovered from Carrots, P.P.M.[a] | | | |
| 1962 | Raw | 0.71 | 62 | 1.41 | 28 |
| | Boiled | 0.72 | 62 | 0.64 | 39 |
| 1963 | Raw | 0.41 | 80 | 1.56 | 41 |
| | Boiled | 0.40 | 77 | 1.20 | 53 |

| Water Condition | % Mortality of Aedes aegypti Larvae after Exposure for 24 Hours to Water in Which Carrots Were Boiled | |
|---|---|---|
| | AD[d] | HHO[e] |
| Unfiltered | 10 | 90 |
| Filtered | 15 | 90 |
| Centrifuged | 30 | 55 |

[a] Determined by gas-liquid chromatography.
[b] Dieldrin in per cent of total residue.
[c] Heptachlor epoxide in per cent of total residue recovered.
[d] Carrots grown in 1963 in aldrin-treated soil.
[e] Carrots grown in 1963 in heptachlor-treated soil.

**Effect of Boiling Carrots in Water on Stability of Translocated Aldrin or Heptachlor Residues.** Carrots (Red Cored Chantenay) were grown in 1962 and 1963 on replicated Carrington silt loam plots (30 × 24 feet) which had been treated as described (*4*) with aldrin or heptachlor at 5 pounds per acre each year from 1958 through 1962. After harvest the edible parts of the carrots were washed with water, rinsed with acetone, and rewashed with water. Each carrot was then cut lengthwise into two equal parts. One half of each carrot was combined into one sample, macerated in a raw stage, and frozen for future analyses. The other half was boiled in water for 30 minutes within a covered aluminum pot. The cooked carrots were then macerated and frozen pending subsequent analyses.

Soils were sampled at harvest time in 1962 and 1963 from the total plot areas. Each sample consisted of 30 cores. Carrots and soils were extracted and analyzed by gas liquid chromatography as described (*5*).

The possible toxicity of the water, in which carrots from either aldrin- or heptachlor-treated plots had been boiled for 30 minutes, was also tested. Ten third instar mosquito larvae (*Aedes aegypti*) were placed in 18 ml. of this water within duplicated test tubes. Mortality counts were made after an exposure of 24 hours. Mosquito larvae were also exposed to water from which suspended carrot particles had been removed either by filtering the water through glass wool or by centrifuging.

The boiling of carrots from heptachlor-treated soils caused an appreciable loss of insecticidal residues from the carrot tissue (Table VI). This was also indicated by the high toxicity of the water in which these carrots had been boiled, after mosquito larvae had been introduced. Centrifuging this water resulted in a lower mortality of the exposed mosquito larvae. No mortalities were registered when mosquito larvae were placed in water in which insecticide-free carrots had been boiled.

The concentration of aldrin residues in carrots, however, was not affected by boiling. Only slight mortalities of mosquito larvae were observed 24 hours

after being placed in water in which these carrots had been boiled.


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**FOOD FLAVOR CHANGES**

# Relationship between Monocarbonyl Compounds and Flavor of Potato Chips


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**Eighteen monocarbonyl compounds have been identified in fresh potato chips and 19 in stale potato chips. The quantitative change of each individual carbonyl compound during storage has been investigated. Among saturated aldehydes the largest increase was in hexanal and next in pentanal; among 2-alkanones the important increase was in 2-pentanone and next in 2-propanone; and among 2-enals the largest increase was in 2-heptenal and 2-octenal. Only one 2,4-dienal—viz., 2,4-decadienal—was found in both fresh and stale potato chips. Its amount was greatly decreased during storage. The mechanism of the formation of these carbonyl compounds and their relationship to the flavor of potato chips are discussed.**


CARBONYL compounds are an important group of compounds which contribute to both the savory and off-flavors of foods, particularly those rich in fat content. Potato chips contain 30 to 35% of their total weight in fat and possess a distinct, characteristic flavor, which, on storage, is gradually replaced by an unpalatable off-flavor. The chemical nature of the monocarbonyl compounds which might contribute to the fresh potato chip flavor and cause the development of this stale flavor have not been characterized.

Literature concerning the flavor components of potato chips is very scanty. Buttery (*3*, *4*) studied the autoxidation of potato granules and identified acetaldehyde, propanal, 2-methylpropanal, butanal, 2- and 3-methylbutanal, pentanal, hexanal, octanal, 2-pentenal, 2-octenal, methane, ethane, propane, butane, and pentane as autoxidation products. Self, Rolley, and Joyce (*28*) investigated the volatiles of cooked potatoes and identified several carbonyl and sulfur compounds. In 1963 Dornseifer and Powers (*8*) reported the changes in volatile carbonyls of potato chips during storage. They positively identified 2,3-butanedione and tentatively identified ethanal, propenal, 2-propanone, *n*-butanal, 2-pentenal, 2-hexenal, *n*-heptanal, and 2-heptenal. Their isolation technique involved the steam distillation of potato chips under vacuum.

Work by Gaddis, Ellis, and Currie (*12*) has indicated that a large number of the determinable carbonyls in rancid fat do not originally exist in the oxidized fat as free carbonyls. They are apparently produced through the breakdown of precursors under reaction conditions used in the isolation process. This fact was supported by Buttery (*3*). Furthermore, Crossley and Thomas (*7*), on the basis of their work on oil, stated that the carbonyl determinations used by many workers have estimated not only carbonyls but hydroperoxides as well.

Recently, Schwartz developed a procedure for the investigation of monocarbonyl compounds in an oil (*26*, *27*). This method has several advantages over the conventional methods. Carbonyl compounds can be converted quantitatively into their 2,4-dinitrophenylhydrazones without the necessity of separating them from the oil; a carbonyl present in an oil in a concentration as low as 0.1 p.p.m. can be determined; and hydroperoxides of fat do not react with reactants under the chromatographic conditions. The present paper reports the use of this technique to study the relation between monocarbonyl compounds and flavor of potato chips.

### Experimental

**Materials.** Potato chips used were made from Kennebec potatoes by frying in a mixture of 50% cottonseed and 50% corn oils at 365° F. The chips had a color of 4 to 5 according to the proposed color reference standard of the Potato Chip Institute. The freshly fried potato chips were packed in standard 2-pound coffee tin cans under an atmosphere of nitrogen containing only 1.70% oxygen and stored at −10° C. before using.

The fresh potato chips had an excellent flavor as evaluated by an expert taste panel. They were devoid of any harsh and burnt odor and had a clean, pleasant aftertaste. They had a moisture content of 1.70% as determined by TAPPI (Technical Association of the Paper and