Residues of Fumigant Mixture in Cerals Fumigated and Aired at Two Different Temperatures

Eugenia Alumot and Rachel Bielorai

Residues of a fumigant mixture (carbon disulfide, carbon tetrachloride, chloroform, and trichlorethylene) were determined in cereals aired at two different temperatures. Lowering the temperature enhanced the airing process. At 17° C., the grains were almost free of residual fumigants after about 3 weeks, while at 30° C., residues were still detectable after 2 months.

The problem of fumigant residues in foods and feeds is receiving renewed interest since the development of new analytical methods for determination of residues at nanogram levels (Bielorai and Alumot, 1966; Heuser and Scudamore, 1967).

As previously shown (Bielorai and Alumot, 1966), cereals fumigated with a mixture of carbon disulfide, carbon tetrachloride, chloroform, and trichlorethylene are not free of residues even after prolonged airing at room temperature (about 22° C.). Airing curves were established at controlled conditions of relative humidity and temperature to find the airing conditions at which the smallest residue levels could be reached in the shortest time. The present study reports the temperature effect.

METHODS

Whole grain wheat, barley, corn, and sorghum were fumigated with a mixture containing carbon tetrachoride, carbon disulfide, choloroform, and trichlorethylene at 5, 26, 32, and 37 volume %, respectively, at a concentration of 300 grams per cu. meter, for 48 hours. The fumigation and airing were performed as described previously (Bielorai and Alumot, 1966). Two independent experiments were carried out, each at two temperatures, 17° and 30° C. The fumigations were conducted in duplicate, four fumigation vessels being used in each experiment.

The first experiment was carried out in two constant-temperature rooms at 17° and 30° C., with air circulation. The relative humidities were 65 and 45%, respectively. The second experiment was carried out in two steps in the same constant-temperature room. The room was first held at 17° C. and then at 30° C., with the relative humidity maintained at 65%.

Determination of fumigant residues during airing was carried out by the gas chromatographic method (Bielorai and Alumot, 1966). Sample size varied from 5 grams at the beginning to 50 grams at the end of airing, when only small residues remained.

RESULTS AND DISCUSSION

The two experiments gave similar results; differences between samples from duplicate vessels did not exceed 10%. The data represent the average of these experiments.

The Volcani Institute of Agricultural Research, Rehovot, Israel

Table I. Amounts (P.P.M.) of Fumigants Initially Sorbed by Cereals at Two Temperatures (°C.)

	Wheat		Barley		Corn		Sorghum	
Fumigant	**17**	**30**	**17**	**30**	**17**	**30**	**17**	**30**
Chloroform	117	135	123	132	189	224	176	178
Trichlorethylene	119	136	105	97	194	187	129	120
Carbon disulfide	120	140	50	54	500	400	360	200
Carbon tetrachloride	11	12	11	8	9	14	9	8

The results presented in Table I show that all the cereals tested sorbed appreciable amounts of the fumigants, roughly proportional to the concentration of the components in the fumigant mixture. In each cereal species, chloroform and trichlorethylene levels were similar. Carbon tetrachloride was sorbed in small amounts, according to its low percentage in the mixture. The data for carbon disulfide are not so reliable as the others because of its high volatility, which causes losses during determination (Bielorai and Alumot, 1966).

There were no substantial differences between the amounts initially sorbed at the two temperatures, although, according to sorption laws, sorption should decrease with a temperature rise. The difference in temperature was apparently too small to influence initial sorption significantly. Other factors, such as grain structure, fat content, etc., should probably be considered in this regard (Alumot and Calderon, 1965). Barley sorbed the lowest amounts of the fumigants, probably owing to its hull structure; corn and sorghum sorbed the highest amounts, possibly with relation to fat levels; wheat sorbed intermediate amounts.

In contrast to species differences in initial sorption, the process of airing was similar for all cereals, with characteristic variations for each fumigant.

The most general phenomenon observed was the enhancement of the airing process at the lower temperature. This trend is illustrated in Figure 1, showing the disappearance of chloroform and trichlorethylene residues at both temperatures from fumigated wheat.

The data for the other cereals are presented in Table II for chloroform and trichlorethylene residues.

It is clear from these data that a sharp decline of the residue levels occurred during the first two days of airing. Thereafter, at 30° C., a characteristic slowdown of the airing process took place, whereas at 17° C. the residue levels continued to drop steadily. At 30° C.,

Table II. Residues (P.P.M.) of Chloroform and Trichlorethylene in Barley, Corn, and Sorghum during Airing at Two Temperatures (°C.)

Days of Airing	Barley		Corn		Sorghum	
	17	30	17	30	17	30
			Chloroform			
0	123	132	189	224	176	178
1	37	59	79	122	88	103
2–3	25	51	72	116	52	94
6–7	18	47	52	110	19	73
13–14	3	42	33	75	13	61
20	2	35	—	—	5	50
25	—	—	10	71	—	—
31	—	33	2	—	—	43
60–66	—	16	—	16	—	22
			Trichlorethylene			
0	105	97	194	187	129	120
1	23	48	72	87	38	84
2–3	18	44	34	84	36	77
6–7	10	30	14	74	31	61
13–14	3	31	10	55	15	44
20	2	23	3	—	6	34
25	—	—	—	45	—	—
31	—	19	—	—	—	26
60–66	—	7	—	14	—	11

Table III. Residues (P.P.M.) of Carbon Tetrachloride and Carbon Disulfide in Cereals during Airing at Two Temperatures (°C.)

Days or Airing	Wheat		Barley		Corn		Sorghum	
	17	30	17	30	17	30	17	30
			Carbon Tetrachloride					
0	11.0	12.0	11.0	8.0	9.0	14.0	9.0	8.0
1	4.0	9.0	3.0	6.0	2.8	5.0	6.8	6.0
2–3	2.0	8.0	2.0	5.0	2.5	5.0	5.5	6.0
6–7	—	8.0	1.5	3.6	1.6	7.0	1.7	4.0
13–14	0.7	6.0	0.6	4.5	0.9	3.6	1.1	4.0
20	—	—	—	2.8	—	—	—	3.0
25	0.8	6.0	0.4	—	—	3.4	—	—
31	0.5	—	0.3	3.0	—	—	—	2.8
60–66	—	1.9	—	0.5	—	0.8	—	1.6
			Carbon Disulfide					
0	150	140	80	54	500	360	360	200
1	58	42	30	25	228	110	104	56
2–3	7	32	4	7	104	62	48	50
6–7	4	13	2	—	10	37	12	36
13–14	1	—	Traces	4	7	31	6	34
25	—	6	—	—	Traces	20	Traces	24
31	—	—	—	1	—	—	—	10
66	—	Traces	—	—	—	3	—	9

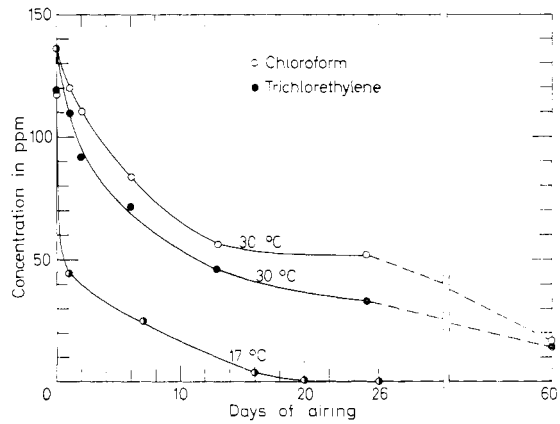

Figure 1. Residues of chloroform and trichlorethylene during airing of wheat at two temperatures

appreciable residues of the two fumigants were still found after two months of airing, while at 17° C., after three weeks practically no residues were present.

The airing process of carbon tetrachloride and carbon disulfide can be followed from Table III.

In spite of the differences in the initial levels and desorption rate of carbon tetrachloride and carbon disulfide (Table III), both fumigants disappeared at a much higher rate at 17° than at 30° C.

The more rapid desorption at the lower temperature found for all fumigants and in all cereal species tested was rather unexpected. It was assumed that the volatile substances would disappear more quickly from the grain with an increase of temperature.

This phenomenon is probably a general one for whole grain and does not depend upon airing conditions. Airing in commercial mills may be more intensive than in these experiments, and therefore low residue levels may be reached in a shorter time, but it seems desirable to perform the airing at as low a temperature as possible.

ACKNOWLEDGMENT

The skillful technical assistance of Helen Geler, Chava Genige, and Eliahu Mandel is gratefully acknowledged.

LITERATURE CITED

Alumot, E., Calderon, M., *J. Sci. Food Agr.* **16,** 464 (1965).
Bielorai, R., Alumot, E., J. AGR. FOOD CHEM. **14,** 622 (1966).
Heuser, S. G., Scudamore, K. A., *Chem. Ind.* **1967,** p. 1557.

Received for review November 7, 1968. Accepted January 10, 1969. This work was financed by a grant from the U. S. Department of Agriculture under P.L. 480. Contribution from the Volcani Institute of Agricultural Research (N.U.I.A.), Bet Dagan, Israel, 1968 Series, No. 1441-E.