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FUMIGATION AGAINST GRAIN WEEVILS WITH VARIOUS VOLATILE ORGANIC COMPOUNDS

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PURPOSE OF INVESTIGATION

Weevils destroy many million dollars' worth of wheat and other grains annually. Carbon disulphide is extensively used as a fumigant against these insects, but, although efficacious, it has serious disadvantages. It has an extremely disagreeable odor and in moderate concentrations its vapor is poisonous to man. Although carbon disulphide is volatile, millers occasionally complain that wheat which has been treated with it retains its odor, and it has been shown that the baking quality of flour from carbon disulphide fumigated wheat is sometimes injured (8).³

The really serious objection to the use of carbon disulphide as a fumigant, however, arises from the fact that it is readily inflammable and that its vapor when mixed with air is highly explosive. For this reason fire insurance companies refuse to carry the fire risk on elevators during the time carbon disulphide is being used to treat the grain contained in them. Even more important is the action taken by the General Managers' Association of Chicago, representing the leading railway systems of the United States, in adopting the following resolution:

That because of the highly inflammable character of solution of carbon disulphide and tetrachloride, its use for fumigating cars to be loaded with grain be prohibited, except that the Illinois Central may continue its use at New Orleans

¹ Deceased June 19, 1923.

² Credit is due Maj. A. Gibson, formerly of the Chemical Warfare Service, for obtaining poisonous materials from the laboratories of Edgewood Arsenal; G. W. Kirby, of the Insecticide and Fungicide Laboratory, Bureau of Chemistry, for assistance in the chemical work; J. H. Cox, Harold Anderson, and the Milling and Baking Laboratory of the Grain Division, Bureau of Agricultural Economics, for assistance in carrying out the car-fumigation tests.

³ Italic numbers in parentheses refer to Literature Cited, p. 39.

and the Baltimore & Ohio at Locust Point, Md., when cars are isolated and protected, until some other satisfactory suitable substance can be provided, and further, that each carrier, member of this association, shall issue, without delay, necessary instructions prohibiting its use.

The following resolution was also adopted at the same time by this association:

That because of the presence of bran bug and weevil in grain and the great danger being done thereby, the chairman of this association communicate with the Bureau of Agriculture, Washington, D. C., advising of the action taken by this association and recommending that such investigations as may be necessary be made by the Government to produce a substance for this purpose which can be used with safety.

The investigation reported in this bulletin was undertaken because of the action taken by these railroad officials.

Carbon tetrachloride, hydrocyanic acid gas, sulphur dioxide, chloropicrin, naphthalene, phosgene, arsine, cyanogen chloride, and many other substances have been tested as fumigants for grain weevils by various investigators, who have reached the following conclusions: Carbon tetrachloride is ineffective under practical conditions; hydrocyanic acid gas fails to kill weevils very far below the surface of the grain; sulphur dioxide has low toxicity, injures ironwork, destroys the germinating power of wheat, makes a sticky dough (4), and retards fermentation, the bread obtained being heavy and unfit for consumption; carbon dioxide is effective only in tightly sealed containers and at relatively high concentrations; chloropicrin shows promise of being a practical fumigant, but is not yet commercially available; naphthalene is not very effective and has an objectionable odor; phosgene is poisonous to man, comparatively nonpoisonous to insects, and, because of its high vapor pressure, difficult to control; the toxicity of arsine to insects is comparatively low; the effect of cyanogen chloride as an insecticide is practically the same as that of hydrocyanic acid.

It is evident, therefore, that there is great need for a fumigant which will be effective against injurious insects in wheat and other cereals and also noninflammable and nonexplosive and neither dangerous nor highly disagreeable to those who handle it. The object of the investigation here reported was to discover such a fumigant, which could be used in place of carbon disulphide. In connection with this investigation, much information on the relation of the chemical constitution of compounds to their toxicological action on insects was acquired.

EXPERIMENTAL PROCEDURE

The rice weevil (*Sitophilus oryza* L.), the flour weevil (*Tribolium confusum* Fab.), and the granary weevil (*Sitophilus granarius* L.) were used in most of the tests, and the Indian meal moth (*Plodia interpunctella* Hbn.) was used in a great many. Adult insects of the weevils and the larvæ of the Indian meal moth were used.

The first series of experiments (Tables 1 and 2) were conducted in the apparatus described by Neifert (18). Four-liter glass jars, containing 10 to 20 live weevils of each species tested, were filled with a mixture of air of 40 per cent relative humidity and the vapor of the compound to be tested. After standing for 24 hours at room temperature (21° to 32° C.), the percentage of dead weevils was determined. (All specimens were examined after 24 hours, and also after 48 hours, to avoid reporting as dead those which might

have been only stupefied.) To obtain the desired humidity, air was bubbled through bottles of sulphuric acid of the proper density, the vapor pressure of water in sulphuric acid-water mixtures being known for mixtures of various densities. In the less volatile compounds, the quantity of the compound present in the jar in the form of vapor was determined by passing a known volume of air over a weighed quantity of the compound. The loss in weight (in grams) of the compound divided by the quantity of air (in liters) gives the quantity (in grams) of substance present in the form of vapor per liter of air.

The following factors were used in calculating the results reported:

Grams per liter $\times 62.43$ = pounds per 1,000 cubic feet.

Grams per liter $\times \frac{22.4 \times 100}{\text{gram-molecular weight}}$ = percentage concentration.

Percentage concentration $\times \text{gram-molecular weight} \times 0.02787$ = pounds per 1,000 cubic feet.

1 cubic foot = 28.32 liters.

1 kilogram = 2.20462 pounds.

Assuming the vapor of a compound to be a perfect gas, the molecular weight (in grams) of this vaporized compound will occupy 22.4 liters under a pressure of 760 millimeters of mercury and at a temperature of 0° C. For example, the molecular weight of chloroform is approximately 119.4. Therefore, if 22.4 liters of space contains 119.4 grams of chloroform vapor (equivalent to 5.33 grams per liter), the percentage concentration is 100.

Since the tests were made at about the same temperature on compounds differing greatly in vapor pressure, it follows that the more volatile compounds were tested in high molar concentrations and the slightly volatile compounds were tested in low molar concentrations, which, however, were the maximum concentrations of the vapors possible at the temperature of the test.

EFFECT OF VOLATILE ORGANIC COMPOUNDS ON WEEVILS

The results of the fumigation of weevils in glass flasks at room temperature (21° to 32° C.) are shown in Table 1. Table 2 gives the formula, molecular weight, boiling point, and minimum lethal concentration for each compound tested.

TABLE 1.—Results of fumigation tests on weevils in glass flasks at room temperature (21° to 32° C.)

Test No.	Tem- pera- ture	Fumigant	Concentration of fumigant		Weevils killed after exposure for 24 hours			
			Molar per- cent- age	Pounds per 1,000 cu. ft.	<i>S.</i> <i>oryza</i>	<i>S.</i> <i>gran- arius</i>	<i>Tribo- lium</i>	<i>Plodia</i>
HYDROCARBONS:	° C.				<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
311.....	25.0	Amylene.....	40.31	78.76	100	100	100	-----
343.....	21.5	do.....	14.73	28.78	0	0	0	-----
347.....	22.5	do.....	21.26	41.54	0	0	0	-----
94.....	28.5	Kerosene.....	1.40	1.43	0	0	0	10
83.....	23.0	Cyclohexane.....	13.00	30.48	100	100	100	85
139.....	30.5	do.....	14.00	32.82	100	100	100	70
35.....	28.5	Benzene.....	13.00	28.29	100	100	100	100
130.....	28.5	do.....	.90	1.96	0	0	0	0
133.....	29.0	do.....	1.60	3.48	10	30	80	0
136.....	30.5	do.....	3.80	8.27	100	100	100	70
37.....	28.5	Toluene.....	3.00	7.70	75	50	100	80
23.....	30.0	o-Xylene.....	1.40	4.14	60	0	0	-----
14.....	24.0	Naphthalene (25 per cent) and carbon tetrachlo- ride (75 per cent).	2.50	1.79	75	25	-----	25
39.....	28.5	Anthracene.....	.01	.05	0	0	0	0

¹ Molecular weight of kerosene assumed to be 128.

² Concentration of naphthalene only.

TABLE 1.—Results of fumigation tests on weevils in glass flasks at room temperature (21° to 32° C.)—Continued

Test No.	Temperature	Fumigant	Concentration of fumigant		Weevils killed after exposure for 24 hours			
			Molar per-cent-age	Pounds per 1,000 cu. ft.	<i>S. oryza</i>	<i>S. gran-arius</i>	<i>Tribolium</i>	<i>Plodia</i>
BROMIDES:		° C.			<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
50	31.0	Bromoform	0.94	6.62	100	80	90	100
365	23.0	Ethyl bromide	53.50	162.48	100	100	100	100
373	25.5	do	16.80	51.02	100	100	100	100
376	25.0	do	14.20	43.13	100	100	100	100
378	25.5	do	8.80	26.73	0	0	0	100
380	24.0	do	12.70	38.57	100	100	100	100
382	24.0	do	8.00	24.30	100	100	100	100
404	24.0	do	7.60	23.08	100	100	100	100
405	25.0	do	4.00	12.15	100	100	100	100
410	27.0	do	4.30	13.06	100	100	100	100
412	28.0	do	3.30	10.02	100	100	100	100
414	28.0	do	2.90	8.81	100	100	100	100
419	24.0	do	1.70	5.16	0	0	0	100
420	24.0	do	1.20	3.64	100	0	0	100
421	24.0	do	.70	2.13	0	0	0	100
366	23.0	Ethylene bromide	1.30	6.81	100	100	100	100
374	25.5	do	.50	2.62	100	100	100	100
377	25.0	do	.20	1.05	100	30	80	100
379	25.5	do	.30	1.57	0	0	80	100
381	24.0	do	.30	1.57	75	50	100	100
383	24.0	do	.20	1.05	100	0	80	100
278	26.0	<i>n</i> -Propyl bromide	12.79	43.84	100	100	100	100
280	26.0	do	13.84	47.44	100	100	100	100
308	25.0	do	5.50	18.85	100	100	100	100
313	25.0	do	7.26	24.89	100	100	100	100
326	24.5	do	5.54	18.99	100	100	100	100
331	24.0	do	4.00	13.71	100	50	100	100
353	24.0	do	4.01	13.75	0	0	0	100
367	24.0	do	4.90	16.80	100	100	100	100
369	24.0	do	4.10	14.05	100	100	100	100
385	24.0	do	2.20	7.54	0	0	0	100
483	26.0	Allyl bromide	15.30	51.59	100	100	100	100
486	26.0	do	8.00	26.97	100	100	100	100
489	25.0	do	3.90	13.15	100	100	100	100
275	26.0	<i>n</i> -Butyl bromide	5.27	20.12	100	100	100	100
277	26.0	do	5.30	20.24	100	25	100	100
307	25.0	do	2.64	10.08	100	100	100	100
312	25.0	do	2.56	9.78	100	100	100	100
325	24.5	do	1.63	6.22	80	25	100	100
330	24.0	do	1.09	4.16	0	25	15	100
54	32.0	Bromobenzene	.77	3.37	20	0	0	0
295	23.0	do	.56	2.45	0	0	0	0
297	23.5	do	.55	2.41	0	0	0	0
58	30.0	Benzyl bromide	.12	.57	0	0	0	20
CHLORIDES:								
518	25.0	Methylene chloride	39.50	93.51	100	100	100	100
520	25.0	do	8.10	19.18	0	0	100	100
524	25.0	do	16.80	39.77	100	100	100	100
525	25.0	do	14.20	33.62	100	100	100	100
541	26.0	do	4.40	10.42	100	100	100	100
580	28.0	do	3.40	8.05	100	0	0	100
581	28.0	do	2.20	5.21	100	0	0	100
582	28.0	do	1.20	2.84	100	0	0	100
583	28.0	do	1.10	2.60	100	0	0	100
19	31.0	Chloroform	20.00	66.55	100	100	100	100
69	28.5	do	2.30	7.65	0	0	0	100
79	30.0	do	15.00	49.91	100	100	100	100
104	28.5	do	3.20	10.65	95	45	100	50
106	29.0	do	7.00	23.29	100	100	100	80
109	29.0	do	13.90	46.25	100	100	100	100
11	30.0	Carbon tetrachloride	2.00	8.58	0	0	0	0
239	23.0	do	10.05	43.09	100	100	100	100
240	24.0	do	12.21	52.35	100	100	100	100
249	24.0	do	10.31	44.21	100	100	100	100
250	23.5	do	6.86	29.41	100	100	100	100
89	28.5	Ethylidene chloride	20.00	55.16	100	100	100	100
153	25.5	do	10.20	28.13	100	100	100	100
156	26.0	do	10.90	30.06	100	100	100	100
159	26.5	do	2.00	5.52	0	0	20	100
162	27.0	do	1.30	3.59	0	0	75	100
88	28.5	Trichloroethane	4.00	14.87	100	100	100	100
114	28.0	do	3.80	14.13	100	100	100	100
117	29.0	do	1.90	7.06	100	90	100	65
120	30.0	do	.90	3.35	95	0	0	65
123	30.0	do	.48	1.78	0	0	100	10

TABLE 1.—Results of fumigation tests on weevils in glass flasks at room temperature (21° to 32° C.)—Continued

Test No.	Temperature	Fumigant	Concentration offumigant		Weevils killed after exposure for 24 hours			
			Molar per-cent-age	Pounds per 1,000 cu. ft.	<i>S. oryza</i>	<i>S. gran-arius</i>	Tribolium	Plodia
CHLORIDES—Con.	° C.				<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
126	30.0	Trichloroethane	0.22	0.82	0	0	70	0
497	24.0	do	2.80	10.41	100	100	100	100
499	26.0	do	1.60	5.95	100	100	100	100
501	26.0	do	.80	2.97	100	100	100	100
503	24.0	do	.40	1.49	0	0	0	0
505	24.0	do	.20	.74	100	100	100	100
512 513	23.0	do	.40	1.49	0	0	0	0
516	24.5	do	.20	.74	0	0	0	0
526	25.0	do	.80	2.97	50	50	50	50
527	25.0	do	.80	2.97	100	100	75	75
528, 529	26.0	do	.50	1.86	50	50	50	50
530	25.0	do	1.10	4.09	100	100	100	100
531	25.5	do	1.10	4.09	100	100	100	100
66	28.5	s-Tetrachloroethane	1.00	4.68	60	0	0	50
78	29.5	do	1.10	5.15	100	100	100	100
96	30.0	do	1.10	5.15	100	60	100	90
98	30.0	do	.46	2.15	60	50	100	90
100	30.0	do	.23	1.08	70	50	100	60
87	28.5	Ethylene chloride	10.00	27.58	100	100	100	100
113	28.0	do	12.00	33.10	100	100	100	100
116	29.0	do	6.00	16.55	100	100	100	100
119	29.0	do	3.00	8.27	95	50	100	85
122	30.0	do	1.40	3.86	0	0	100	25
125	30.0	do	.73	2.01	90	20	100	20
84	31.0	Trichloroethylene	10.00	36.62	100	100	100	100
95	30.0	do	10.00	36.62	100	100	100	100
97	30.0	do	5.00	18.31	10	0	0	10
99	30.0	do	2.10	7.69	100	100	100	100
102	30.0	do	1.05	3.85	0	0	0	70
146	30.0	do	5.00	18.31	65	60	100	20
150	30.0	do	2.50	9.16	50	15	80	0
574	26.0	Tetrachloroethylene	2.60	12.02	100	100	100	100
575	26.0	do	1.30	6.01	50	0	100	100
576	26.0	do	.60	2.77	0	0	0	0
577	26.0	do	.30	1.39	0	0	0	0
55	32.0	Acetylene dichloride	9.02	24.37	100	100	100	80
141	29.0	do	28.00	75.65	100	100	100	100
145	30.0	do	16.80	45.39	100	100	100	100
149	30.0	do	6.90	18.64	60	0	90	0
536	26.0	Propylene dichloride	6.10	19.21	100	100	100	100
537	26.0	do	2.80	8.82	100	100	100	100
538	26.0	do	1.20	3.78	100	100	100	100
556	27.0	do	1.10	3.46	100	100	70	70
557	27.0	do	1.00	3.15	100	100	80	80
558	27.0	do	.70	2.20	100	100	60	60
559	27.0	do	.70	2.20	100	100	0	0
92	28.5	Isopropyl chloride	18.00	39.40	0	0	65	50
1	26.0	Monochlorobenzene	1.00	3.14	100	100	100	100
2	26.0	do	.25	.78	100	100	100	100
3	26.0	do	.10	.31	100	100	100	100
4	26.0	do	.50	1.57	100	100	100	100
8	30.0	do	2.00	6.27	100	100	100	100
9	30.0	do	1.00	3.14	100	50	100	100
15	29.5	do	2.00	6.27	75	32	20	20
16	30.5	do	2.00	6.27	90	50	20	20
65	28.5	do	7.70	24.15	100	100	100	95
138	30.5	o-Dichlorobenzene	.60	2.46	0	0	0	0
5	26.0	p-Dichlorobenzene	1.00	4.10	100	50	50	50
10	30.0	do	1.00	4.10	85	75	100	100
7	30.0	o and p-Dichlorobenzene	2.00	8.19	100	100	100	100
13	30.0	do	1.00	4.10	100	50	100	100
FLUORIDES:								
465	24.0	Fluorobenzene	14.30	38.29	100	100	100	100
467	24.0	do	7.20	19.28	100	100	100	100
469	24.0	do	3.40	9.10	100	100	100	100
471	24.5	do	1.90	5.09	100	100	100	100
474	23.0	do	2.20	5.89	0	0	100	100
476	23.0	do	1.90	5.09	100	100	25	25
60	30.5	Difluorodiphenyl	.002	.01	0	0	0	10
IODIDES:								
302	25.0	n-Butyl iodide	1.61	8.26	100	100	100	100
303	25.0	do	1.54	7.90	100	100	100	100
316	24.5	do	.79	4.05	100	100	100	100
337	21.5	do	.43	2.21	0	0	0	0
340	24.0	do	.36	1.85	0	0	0	0

TABLE 1.—Results of fumigation tests on weevils in glass flasks at room temperature (21° to 32° C.)—Continued

Test No.	Temperature	Fumigant	Concentration of fumigant		Weevils killed after exposure for 24 hours			
			Molar per-cent-age	Pounds per 1,000 cu. ft.	<i>S. oryza</i>	<i>S. gran-arius</i>	Tribolium	Plodia
ALCOHOLS AND PHENOLS:								
34	28.5	Methyl alcohol	16.00	14.28	100	100	100	100
129	28.5	do	1.00	.89	0	0	0	0
132	29.0	do	2.00	1.79	0	0	0	0
135	30.0	do	4.50	4.02	30	15	5	10
64	30.5	Ethyl alcohol	8.00	10.27	0	0	0	15
21	30.5	n-Propyl alcohol	3.50	5.86	100	100	100	100
71	32.0	do	1.70	2.85	96	50	100	100
111	29.0	do	1.70	2.85	100	100	100	100
137	30.5	do	1.50	2.51	20	0	15	85
81	30.0	n-Butyl alcohol	1.70	3.51	100	60	100	100
20	31.0	Isoamyl alcohol	.80	1.96	100	100	100	80
142	29.0	do	.77	1.89	80	60	100	100
144	30.0	do	.33	.81	0	0	0	0
148	30.0	do	.20	.49	0	0	0	0
68	28.5	Geraniol	.10	.43	10	0	0	0
59	30.5	Menthol	.08	.35	0	0	0	80
46	30.0	Thymol	.09	.38	0	0	0	0
ALDEHYDES:								
67	28.5	Chloral hydrate	.20	.92	20	0	0	0
289	25.0	n-Butyraldehyde	11.15	22.40	100	100	100	100
291	25.0	do	9.46	19.00	100	100	100	100
321	26.0	do	5.53	11.11	100	100	100	100
335	21.5	do	3.27	6.57	0	0	100	100
338	24.0	do	2.59	5.20	0	0	90	100
464	24.0	Crotonaldehyde	4.00	7.81	100	100	100	100
466	24.0	do	2.00	3.91	100	100	100	100
468	24.0	do	1.30	2.54	100	100	100	100
470	24.5	do	.50	.98	100	100	100	100
473	23.0	do	.60	1.17	100	100	100	100
475	23.0	do	.50	.98	100	100	75	100
18	30.5	Furfural	.80	2.14	100	100	100	20
52	31.0	Benzaldehyde	.27	.80	100	100	55	75
KETONES:								
33	28.5	Acetone	15.00	24.27	100	100	100	100
128	28.5	do	2.00	3.24	5	10	10	0
131	28.5	do	4.10	6.63	95	100	15	25
134	30.0	do	8.20	13.27	100	100	100	100
219	24.0	Chloroacetone	1.53	3.94	100	100	100	100
220	23.0	do	.70	1.80	100	100	100	100
232	24.0	do	.42	1.08	100	100	100	100
243	26.0	do	.22	.57	0	0	0	0
268	25.0	do	.42	1.08	100	100	100	100
270	25.0	do	.26	.67	100	100	100	100
80	23.0	Ethyl methyl ketone	15.00	30.13	100	100	100	100
140	29.0	do	13.30	26.72	100	100	100	100
143	29.0	do	7.20	14.46	100	100	100	100
147	30.0	do	3.40	6.83	100	70	100	80
151	30.0	do	1.80	3.62	100	80	100	0
304	25.0	Mesityl oxide	1.53	4.18	100	100	100	100
309	25.0	do	1.42	3.88	100	100	100	100
341	21.0	do	.39	1.07	0	0	0	0
345	22.5	do	.55	1.50	100	50	0	0
352	23.0	do	.60	1.64	100	100	0	0
53	31.0	Acetophenone	.16	.54	100	45	0	50
6	30.0	Chloroacetophenone (5 per cent) in carbon tetrachloride (95 per cent).	.10	.43	100	0	0	100
12	30.0		.01	.04	0	0	0	0
63	30.5		.17	.73	0	0	0	5
ESTERS:								
217	26.0	Methyl formate	56.00	93.71	100	100	100	100
222	23.0	do	20.37	34.09	100	100	100	100
234	24.5	do	11.62	19.44	100	100	100	100
241	25.5	do	6.35	10.63	100	100	100	100
259	26.0	do	4.54	7.60	100	100	100	100
260	26.0	do	3.64	6.09	100	100	100	100
271	24.5	do	3.64	6.09	0	0	0	0
272	24.5	do	4.10	6.86	100	100	100	100
273	24.5	do	2.65	4.43	100	100	100	100
474A, 475A	24.5	do	3.50	5.86	100	100	100	100
476A	24.5	do	2.00	3.35	100	100	100	100
506	24.0	do	1.30	2.18	100	100	100	100
507	24.0	do	.90	1.51	100	100	100	100
284	24.5	Ethyl formate	20.37	42.05	100	100	100	100
286	24.5	do	22.41	46.26	100	100	100	100
319	26.0	do	15.04	31.04	100	100	100	100
328	24.0	do	10.00	20.64	100	100	100	100

* Concentration of chloroacetophenone only.

TABLE 1.—Results of fumigation tests on weevils in glass flasks at room temperature (21° to 32° C.)—Continued

Test No.	Temperature	Fumigant	Concentration of fumigant		Weevils killed after exposure for 24 hours			
			Molar per-cent-age	Pounds per 1,000 cu. ft.	<i>S. oryza</i>	<i>S. gran-arius</i>	Tribolium	Plodia
ESTERS—Con.					<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
333.	24.0	Ethyl formate	6.41	13.23	100	100	100	
357.	24.0	do	4.89	10.09	100	100	100	
359.	23.0	do	3.71	7.66	100	100	100	
389.	25.0	do	1.80	3.72	100	100	100	
391.	25.0	do	.60	1.24	100	100	100	
406.	25.0	do	1.30	2.68	100		100	
407.	25.0	do	1.10	2.27	100		80	
408.	25.0	do	.90	1.86	100		100	
411A.	23.0	do	1.10	2.27	100		100	
412A, 422.	23.0	do	.80	1.65	100		100	
423.	23.0	do	.40	.83	0		0	
424.	23.0	do	.60	1.24	100		0	
426, 428.	25.0	do	.50	1.03	0		0	
484.	26.0	Isopropyl formate	15.50	38.05	100		100	
487.	26.0	do	8.20	20.13	100		100	
490.	25.0	do	3.90	9.57	100		100	
492.	25.5	do	2.00	4.91	100		100	
494.	25.5	do	1.00	2.45	50		0	
514.	24.0	do	1.20	2.95	100		100	
515.	25.0	do	.90	2.21	100		100	
532.	26.5	do	1.10	2.70	100		100	
533.	26.5	do	.90	2.21	100		100	
542.	26.0	do	.80	1.96	100		80	
543.	26.0	do	.50	1.23	100		0	
166.	27.0	Methyl cyano formate	.20	.47	16	0	100	100
167.	27.5	do	.32	.76	88	90	100	100
169.	28.0	do	.08	.19	24	55	15	95
175.	27.0	do	.90	2.13	100	100	100	100
176.	27.5	do	.68	1.61	100	100	100	100
177.	27.5	do	.40	.95	95	90	100	100
178.	27.5	do	.30	.71	50	50	100	100
38.	27.5	<i>n</i> -Propyl acetate	3.50	9.96	100	100	100	100
73.	32.0	do	2.50	7.11	100	100	100	100
155.	25.5	do	3.70	10.53	95	90	100	100
158.	26.0	do	1.90	5.41	90	50	100	100
161.	27.0	do	1.00	2.85	0	0	0	0
44.	30.0	<i>n</i> -Butyl acetate	.76	2.46	70	20	95	100
282.	26.0	Isobutyl acetate	1.68	5.44	0	0	0	0
283.	26.0	do	1.56	5.05	0	0	0	0
45.	30.0	Isoamyl acetate	1.50	5.44	100	100	100	100
75.	31.0	do	.90	3.26	100	100	100	100
101.	30.0	do	.54	1.96	20	50	75	85
103.	30.0	do	.24	.87	0	0	0	0
294.	23.0	Ethyl- <i>n</i> -valerate	.77	2.79	0	0	0	0
296.	23.5	do	.66	2.39	0	0	0	0
ETHERS:								
292.	25.0	Acetal	4.86	16.00	100	100	100	
293.	25.0	do	4.34	14.29	100	100	100	
322.	26.5	do	2.27	7.47	50	20	100	
344.	21.5	do	1.72	5.66	0	0	80	
348.	22.5	do	1.70	5.60	100	100	100	
351.	23.0	do	1.64	5.40	50	20	100	
521.	27.0	do	4.40	14.49	100		100	
523.	27.0	do	1.10	3.62	100		0	
274.	26.0	Chloromethyl ether	2.34	5.25	100	100	100	
275.	26.0	do	2.22	4.98	100	100	100	
218.	26.0	<i>s</i> -Dichloromethyl ether	4.87	15.60	100	100	100	
221.	23.0	do	3.89	12.46	100	100	100	
233.	24.0	do	1.13	3.62	100	100	100	
244.	26.0	do	.62	1.99	50	50	90	
267.	25.0	do	1.21	3.88	100	100	100	
269.	25.0	do	.70	2.24	0	0	0	
CHLOROHYDRINS:								
517.	25.0	Ethylene chlorohydrin	1.40	3.14	0		0	
519.	25.0	do	.30	.67	0		0	
288.	24.5	Epichlorohydrin	1.95	5.03	100	100	100	
290.	25.0	do	1.93	4.98	100	100	100	
320.	26.0	do	1.19	3.07	100	100	100	
329.	24.0	do	.73	1.88	100	100	100	
334.	24.0	do	.59	1.52	100	100	100	
358.	23.0	do	.42	1.08	100	100	100	
360.	23.0	do	.41	1.06	100	100	100	
363.	23.0	do	.25	.64	100	100	100	
388.	25.0	do	.20	.52	100	100	100	
390.	25.0	do	.10	.26	100	100	100	
397.	25.0	do	.09	.23	100		50	
398.	25.0	do	.09	.23	100		0	

TABLE 1.—Results of fumigation tests on weevils in glass flasks at room temperature (21° to 32° C.)—Continued

Test No.	Temperature	Fumigant	Concentration of fumigant		Weevils killed after exposure for 24 hours			
			Molar per-cent-age	Pounds per 1,000 cu. ft.	<i>S. oryza</i>	<i>S. gran-arius</i>	<i>Tribo-lium</i>	<i>Plodia</i>
SULPHUR COM-POUNDS:								
115.	28.0	Carbon disulphide	46.00	97.59	<i>P. ct.</i> 100	<i>P. ct.</i> 100	<i>P. ct.</i> 100	<i>P. ct.</i> 100
118.	29.0	do.	24.70	52.40	100	100	100	100
121.	30.0	do.	12.30	26.10	100	100	100	100
124.	30.0	do.	6.40	13.58	100	100	100	100
127.	30.0	do.	3.00	6.36	100	100	100	100
164.	27.0	do.	3.30	7.00	100	100	100	100
165.	27.0	do.	2.50	5.30	100	100	100	100
168.	27.5	do.	1.10	2.33	100	100	100	100
170.	26.0	do.	1.00	2.12	100	35	70	100
171.	26.0	do.	.50	1.06	0	0	0	0
172.	27.0	do.	.47	1.00	0	0	0	0
173.	27.0	do.	.50	1.06	0	0	0	0
174.	27.0	do.	.11	.23	0	0	0	0
93.	28.5	Ethyl mercaptan	19.00	32.89	100	100	100	100
29.	31.5	<i>n</i> -Butyl mercaptan	3.00	7.54	100	100	100	100
72.	32.0	do.	2.00	5.03	100	100	100	100
152.	25.5	do.	5.40	13.57	100	100	100	100
154.	25.5	do.	2.30	5.78	100	100	100	100
157.	26.0	do.	.90	2.26	100	20	80	0
160.	26.5	do.	.30	.75	0	0	0	10
163.	27.0	do.	.10	.25	0	0	0	0
216.	26.0	Methyl sulphide	53.80	93.14	100	100	100	---
223.	23.0	do.	20.98	36.32	100	100	100	---
235.	25.0	do.	9.45	16.36	50	0	100	---
242.	25.0	do.	10.44	18.07	100	100	100	---
256.	23.5	do.	9.75	16.88	90	0	100	---
257.	24.0	do.	9.49	16.43	100	100	100	---
258.	23.5	do.	5.00	8.66	0	0	50	---
265.	25.0	do.	12.87	22.28	60	0	100	---
266.	25.0	do.	11.54	19.98	100	100	100	---
215.	26.0	Ethyl sulphide	4.91	12.34	100	100	100	---
224.	24.0	do.	2.92	7.34	100	100	100	---
236.	25.0	do.	1.50	3.77	20	10	90	---
245.	25.5	do.	1.72	4.32	95	80	80	---
261.	24.5	do.	3.65	9.17	100	100	100	---
262.	25.0	do.	3.72	9.35	100	100	100	---
264.	25.0	do.	2.34	5.88	50	0	100	---
213.	26.0	Methyl thiocyanate.	1.84	3.75	100	100	100	---
226.	24.0	do.	.78	1.59	100	100	100	---
237.	25.0	do.	.36	.73	100	100	100	---
246.	26.0	do.	.20	.41	100	100	100	---
253.	26.5	do.	.20	.41	100	100	100	---
254.	26.5	do.	.14	.29	100	100	100	---
255.	26.0	do.	.09	.18	0	0	0	---
263.	26.0	do.	.10	.20	100	20	30	---
214.	26.0	Ethyl thiocyanate.	1.04	2.53	100	100	100	---
225.	24.0	do.	.46	1.12	100	100	50	---
229.	23.0	do.	.46	1.12	5	0	100	---
231.	24.0	do.	.19	.46	100	100	50	---
238.	23.0	do.	.71	1.72	0	0	0	---
402.	24.0	do.	.80	1.94	100	---	100	---
51.	31.0	Allyl isothiocyanate.	1.14	3.15	100	100	100	100
77.	29.5	do.	.70	1.93	100	100	100	100
107.	29.0	do.	.34	.94	100	100	100	100
110.	29.0	do.	.62	1.71	100	100	100	100
30.	28.5	Cyanogen sulphide	.10	.23	0	0	0	0
31.	28.5	Mercaptol	.16	1.16	0	0	0	0
28.	30.5	Perchloromethyl mercaptol	1.00	5.18	60	100	25	0
90.	28.5	<i>p</i> -Toluenesulphochloride	.15	.80	0	0	0	100
NITRILES AND ISO-NITRILES:								
305.	25.0	Acetonitrile	11.60	13.27	100	100	100	---
310.	25.0	do.	12.13	13.87	100	100	100	---
342.	21.0	do.	3.47	3.97	0	0	0	---
346.	22.5	do.	4.97	5.68	100	100	100	---
371.	26.5	do.	3.90	4.46	50	0	0	---
372.	26.5	do.	5.70	6.52	100	100	100	---
430.	25.0	Propionitrile	1.30	1.99	0	---	0	---
414A.	24.0	<i>n</i> -Valeronitrile	1.10	2.55	100	---	100	---
416.	24.0	do.	.60	1.39	50	---	30	---
418.	24.0	do.	.30	.69	0	---	0	---
26.	30.0	Phenylisonitrile	.12	.34	0	0	0	0
425.	25.0	Phenylacetoneitrile	.10	.33	100	---	90	---
427.	25.0	do.	.03	.10	0	---	0	---
429.	25.0	do.	.02	.07	0	---	0	---
437.	25.0	do.	.07	.23	0	---	0	---
438.	25.0	do.	.03	.10	0	---	0	---

TABLE 1.—Results of fumigation tests on weevils in glass flasks at room temperature (21° to 32° C.)—Continued

Test No.	Temperature	Fumigant	Concentration of fumigant		Weevils killed after exposure for 24 hours			
			Molar per-cent-age	Pounds per 1,000 cu. ft.	<i>S. oryza</i>	<i>S. gran-arius</i>	Tribolium	Plodia
° C.					<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
NITRITES:								
413A	24.0	<i>n</i> -Butyl nitrite	9.10	26.15	100		100	
415A	24.0	do	4.30	12.36	90		90	
417	24.0	do	2.20	6.32	100		100	
434	25.0	do	1.60	4.60	100		100	
435	25.0	do	1.10	3.16	100		100	
436	25.0	do	1.00	2.87	100		100	
442	24.0	do	.60	1.72	100		100	
443	24.0	do	.60	1.72	100		66	
444	24.0	do	.30	.86	50		8	
279	26.0	Isoamyl nitrite	4.70	15.34	100	100	100	
281	26.0	do	4.84	15.80	100	100	100	
318	24.5	do	2.36	7.70	100	100	100	
327	24.0	do	1.46	4.77	100	100	100	
332	24.0	do	1.02	3.33	100	50	100	
354	24.0	do	1.13	3.69	0	0	0	
368	24.0	do	1.40	4.57	100	100	100	
370	26.0	do	1.10	3.59	100	75	100	
384	24.0	do	.80	2.61	100	100	100	
399	25.0	do	1.20	3.92	100		100	
400	25.0	do	.80	2.61	100		100	
411	23.0	do	.70	2.28	100		50	
413	24.0	do	.60	1.96	100		100	
415	24.0	do	.80	2.61	100		100	
NITRO COMPOUNDS:								
298	23.5	Nitromethane	4.35	7.40	100	100	100	
300	25.0	do	4.59	7.81	100	100	100	
323	26.5	do	3.57	6.07	100	100	100	
336	21.5	do	1.27	2.16	0	0	0	
339	24.0	do	1.03	1.75	0	0	0	
32	28.5	Nitrobenzene	.10	.34	0	0	0	0
49	31.0	do	.12	.41	0	0	0	0
82	30.0	<i>p</i> -Chloronitrobenzene	.12	.53	0	0	0	0
85	31.0	<i>m</i> -Dinitrobenzene	.28	1.31	0	0	0	0
86	28.5	α -Nitronaphthalene	.10	.48	0	0	0	0
AMINES:								
496	24.0	Diethylamine	20.60	41.98	100		100	
498	24.0	do	11.80	24.04	100		100	
500	26.0	do	5.80	11.82	100		100	
502	25.0	do	2.60	5.30	100		100	
504	23.5	do	1.10	2.24	100		100	
509	24.0	do	.90	1.83	100		100	
511	23.0	do	.50	1.02	100		100	
534	26.0	do	.80	1.63	50		50	
535	26.0	do	.50	1.02	50		100	
482	26.0	<i>n</i> -Butylamine	10.20	20.78	100		100	
485	26.0	do	5.30	10.80	100		100	
488	27.0	do	2.80	5.71	100		100	
491	25.5	do	1.30	2.65	100		100	
493	25.5	do	.60	1.22	100		100	
508	24.0	do	.30	.61	50		0	
510	24.0	do	.20	.41	0		0	
43	29.5	Aniline	1.89	4.90	0	0	0	0
47	29.0	Methylaniline	.13	.39	0	0	0	0
56	30.0	Dimethylaniline	.15	.51	0	0	0	0
57	30.0	Ethylbenzylaniline	.03	.18	0	0	0	0
62	30.5	Acetphenylenediamine	.07	.29	0	0	0	0
91	28.5	α -Naphthylamine	.016	.06	0	0	0	0
MISCELLANEOUS NITROGENOUS COMPOUNDS:								
48	30.0	Pyridine	1.69	3.72	100	100	100	100
76	31.0	do	.84	1.85	100	100	100	100
105	29.0	do	.17	.37	0	0	0	0
108	29.0	do	.39	.86	10	5	10	20
112	29.0	do	.83	1.83	100	100	75	65
61	30.5	Hexamethylenetetramine	.002	.008	0	0	0	0
INORGANIC COMPOUNDS:								
477A	23.5	Carbon dioxide	100.00	122.64	100		100	
478A	24.5	do	100.00	122.64	65		40	
479	24.5	do	50.00	61.32	100		25	
480	24.5	do	33.00	40.47	0		0	
481	24.5	do	50.00	61.32	100		100	
495	25.5	do	50.00	61.32	100		0	
522	27.0	do	50.00	61.32	100		0	
41	29.0	Thionyl chloride	5.00	16.58		100	100	100
74	32.0	do	4.50	14.92	100	100	100	100

TABLE 2.—*Formulæ, molecular weights, boiling points, and lethal concentrations (from results in Table 1) of fumigants*

Fumigant	Formula	Molecular weight ¹	Boiling point ²	Lethal concentration ³ (molar percentage)			
				S. oryza	S. gran-arius	Tri-bol-ium	Plo-dia
HYDROCARBONS:							
Amylene	C ₅ H ₁₀	70.105	22-37	40.3	40.3	40.3	
Kerosene	Mixture		150-280				
Cyclohexane	C ₆ H ₁₂	84.126	80.8	43.0	43.0	43.0	
Benzene	C ₆ H ₆	78.078	79-81	3.8	3.8	3.8	13.0
Toluene	C ₆ H ₅ CH ₃	92.099	109-110			3.0	
o-Xylene	C ₆ H ₄ (CH ₃) ₂	106.120	143.5-144.5				
Naphthalene	C ₁₀ H ₈	128.114	218				
Anthracene	C ₁₄ H ₁₀	178.150	351				
BROMIDES:							
Bromoform	CHBr ₃	252.773	142-151	.9			
Ethyl bromide	C ₂ H ₅ Br	108.970	38-40	2.9	48.0	2.9	
Ethylene bromide	C ₂ H ₄ Br ₂	187.882	127-132	.5	.5	.5	
n-Propyl bromide	C ₃ H ₇ Br	122.991	70-71	4.1	4.1	4.1	
Allyl bromide	C ₃ H ₅ Br	120.975	69-71	43.9		43.9	
n-Butyl bromide	C ₄ H ₉ Br	137.012	100-101	2.6	2.6	1.6	
Bromobenzene	C ₆ H ₅ Br	156.990	154-155				
Benzyl bromide	C ₆ H ₅ CH ₂ Br	171.011	198-199				
CHLORIDES:							
Methylene chloride	CH ₂ Cl ₂	84.941	40.5-42	4.4		4.4	
Chloroform	CHCl ₃	119.393	58-61.5	7.0	7.0	3.2	13.9
Carbon tetrachloride	CCl ₄	153.845	76-77	6.9	6.9	6.9	
Acetylene dichloride	C ₂ H ₂ Cl ₂	96.946	55	9.0	9.0	9.0	16.8
Ethylene chloride	C ₂ H ₄ Cl ₂	98.962	83-84	6.0	6.0	4.7	6.0
Ethylidene chloride	C ₂ H ₄ Cl ₂	98.962	59.5-61.5	10.2	10.2	10.2	10.2
Trichloroethane	C ₂ H ₃ Cl ₃	133.414	74.5	.8	3.8	1.1	3.8
Trichloroethylene	C ₂ HCl ₃	131.398	85.5-87	10.0	10.0	10.0	10.0
Tetrachloroethane	C ₂ Cl ₄	167.866	144-146	1.1	1.1	1.1	1.1
Tetrachloroethylene	C ₂ Cl ₄	165.850	119-121	2.6	2.6	1.3	
Propylene dichloride	C ₃ H ₄ Cl ₂	112.983	95-96	4.7		1.2	
Isopropyl chloride	(CH ₃) ₂ CHCl	78.531	34-36				
Monochlorobenzene	C ₆ H ₅ Cl	112.530	128-132	2.0	2.0		2.0
o-Dichlorobenzene	C ₆ H ₄ Cl ₂	146.982	178-181				
p-Dichlorobenzene	C ₆ H ₄ Cl ₂	146.982	172-174				
o and p-Dichlorobenzene	C ₆ H ₄ Cl ₂	146.982		1.0	2.0		1.0
FLUORIDES:							
Fluorobenzene	C ₆ H ₅ F	96.070	84.9	41.9		2.2	
Diffuorodiphenyl	C ₆ H ₄ F-C ₆ H ₄ F	190.124	254-255				
IODIDES:							
n-Butyliodide	C ₄ H ₉ I	184.012	129-131	.8	.8	.8	
ALCOHOLS AND PHENOLS:							
Methyl alcohol	CH ₃ OH	32.037	65	16.0	16.0	16.0	16.0
Ethyl alcohol	C ₂ H ₅ OH	46.058	78				
n-Propyl alcohol	C ₃ H ₇ OH	60.079	96-98	1.7	3.5	1.7	1.7
n-Butyl alcohol	C ₄ H ₉ OH	74.100	114-118	41.7		41.7	41.7
Isoamyl alcohol	C ₅ H ₁₁ OH	88.121	130-132	.8	.8	.8	.8
Geraniol	C ₁₅ H ₁₆ CH ₂ OH	154.194	230				
Thymol	(CH ₃) ₂ CHC(CH ₃) ₂ OH	150.162	228-232				
Menthol	C ₁₀ H ₁₈ OH	156.210	210				
ALDEHYDES:							
Chloral hydrate	CCl ₃ CH(OH) ₂	165.414	97.5				
n-Butyraldehyde	C ₄ H ₇ CHO	72.084	75-77.5	5.5	5.5	3.3	
Crotonaldehyde	CH ₃ CH=CHCHO	70.068	104-105	4.5		.6	
Furfural	C ₄ H ₃ OCHO	96.057	161	4.8	4.8		
Benzaldehyde	C ₆ H ₅ CHO	106.083	179	4.3	4.3		
KETONES:							
Acetone	CH ₃ COCH ₃	58.063	55-56	8.2	4.1	8.2	8.2
Chloroacetone	CH ₂ ClCOCH ₃	92.515	117-119	.26	.26	.26	
Ethyl methyl ketone	C ₂ H ₅ COCH ₃	72.084	79-80	41.8	7.2	41.8	7.2
Mesityl oxide	(CH ₃) ₂ C=CHCOCH ₃	98.110	130-131	.55	.6	1.4	
Acetophenone	C ₆ H ₅ COCH ₃	120.104	202	4.16			
Chloroacetophenone	C ₆ H ₅ COCH ₂ Cl	154.556	247	.10			.10
ESTERS:							
Methyl formate	HCOOCH ₃	60.042	31.5-32.5	4.9	42.65	4.9	
Ethyl formate	HCOOC ₂ H ₅	74.063	53.5-55	.6	4.6	.8	
Isopropyl formate	HCOOCH(CH ₃) ₂	88.084	63-64	4.5		.9	
Methyl cyano formate	CNCOOCH ₃	85.047	100-101	.7	.7	.2	.2
n-Propyl acetate	CH ₃ COOC ₃ H ₇	102.105	99-102	2.5	2.5	1.9	1.9
n-Butyl acetate	CH ₃ COOC ₄ H ₉	116.126	124-126				4.76
Isobutyl acetate	CH ₃ COOC ₄ H ₉	116.126	115-117				
Isoamyl acetate	CH ₃ COOC ₅ H ₁₁	130.147	138-140	.9	.9	.9	.9
Ethyl-n-valerate	C ₄ H ₉ COOC ₂ H ₅	130.147	144-145.5				

¹ Based on international atomic weight values for 1922.² The boiling points given are those of the substances as tested, not those of these compounds of the highest degree of purity.³ The lethal concentration here reported represents the minimum percentage concentration which consistently caused 100 per cent mortality after exposure for 24 hours. Since in some cases the concentrations tested varied decidedly and because of some variation in the experimental results, the results here given do not necessarily represent the exact minimum lethal concentration.⁴ Minimum concentration tested.

TABLE 2.—*Formulae, molecular weights, boiling points, and lethal concentrations (from results in Table 1) of fumigants—Continued*

Fumigant	Formula	Molecular weight	Boiling point	Lethal concentration (molar percentage)			
				<i>S. oryza</i>	<i>S. granarius</i>	<i>Tribolium</i>	<i>Plodia</i>
				° C.			
ETHERS:							
Acetal	$\text{CH}_3\text{CH}(\text{OC}_2\text{H}_5)_2$	118.142	102-104	4.3	4.3	1.7	
Chloromethyl ether	$\text{CH}_3\text{ClOCH}_3$	80.510	55-60	⁴ 2.2	⁴ 2.2	⁴ 2.2	
s-Dichloromethyl ether	$\text{CH}_2\text{ClOCH}_2\text{Cl}$	114.962	100-108	1.1	1.1	1.1	
CHLOROHYDRINS:							
Ethylenechlorohydrin	$\text{CH}_2\text{ClCH}_2\text{OH}$	80.510	126-127				
Epichlorohydrin	$\text{C}_3\text{H}_5\text{ClO}$	92.515	115-117	⁴ .09	⁴ .10	.10	
SULPHUR COMPOUNDS:							
Carbon disulphide	CS_2	76.125	46	1.0	1.1	1.1	1.0
Ethyl mercaptan	$\text{C}_2\text{H}_5\text{SH}$	62.118	34.5-35.5	⁴ 19.0	⁴ 19.0	⁴ 19.0	⁴ 19.0
n-Butyl mercaptan	$\text{C}_4\text{H}_9\text{SH}$	90.160	96-98	.9	2.0	2.0	2.0
Methyl sulphide	$(\text{CH}_3)_2\text{S}$	62.118	38-39	10.4	10.4	9.5	
Ethyl sulphide	$(\text{C}_2\text{H}_5)_2\text{S}$	90.160	92-93	2.9	2.9	2.3	
Methyl thiocyanate	CH_3SCN	73.102	130-131	.1	.14	.14	
Ethyl thiocyanate	$\text{C}_2\text{H}_5\text{SCN}$	87.123	146-147	.8	1.0	.8	
Allyl isothiocyanate	$\text{C}_3\text{H}_5\text{NCS}$	99.128	143-151	⁴ .3	⁴ .3	⁴ .3	⁴ .3
Cyanogen sulphide	$(\text{CN})_2\text{S}$	84.086	(Sublimes 30-40°)				
Mercaptol	$(\text{CH}_3)_2\text{C}(\text{C}_6\text{H}_5\text{S})_2$	260.323					
Perchloromethyl mercaptol	CCl_3SCl	185.905	146.5-148		⁴ 1.0		
p-Toluenesulphochloride	$\text{C}_7\text{H}_7\text{SO}_2\text{Cl}$	190.611	145-146 (15 mm.)				⁴ .15
NITRILES AND ISONITRILES:							
Acetonitrile	CH_3CN	41.042	76-82	5.0	5.0	5.0	
Propionitrile	$\text{C}_3\text{H}_7\text{CN}$	55.063	96-97				
n-Valeronitrile	$\text{C}_5\text{H}_9\text{CN}$	83.105	139-141	1.1		1.1	
Phenylisonitrile	$\text{C}_6\text{H}_5\text{NC}$	103.083	165-166				
Phenylacetonitrile	$\text{C}_6\text{H}_5\text{CH}_2\text{CN}$	117.104	231.7	.1			
NITRITES:							
n-Butyl nitrite	$\text{C}_4\text{H}_9\text{NO}_2$	103.100	77-79	.6		1.0	
Isoamyl nitrite	$\text{C}_5\text{H}_{11}\text{NO}_2$	117.121	96-99	⁴ .6	1.4	.8	
NITRO COMPOUNDS:							
Nitromethane	CH_3NO_2	61.037	98-101	3.6	3.6	3.6	
Nitrobenzene	$\text{C}_6\text{H}_5\text{NO}_2$	123.078	209.4				
p-Chloronitrobenzene	$\text{ClC}_6\text{H}_4\text{NO}_2$	157.530	242				
m-Dinitrobenzene	$\text{C}_6\text{H}_4(\text{NO}_2)_2$	168.078	297				
α-Nitronaphthalene	$\text{C}_{10}\text{H}_7\text{NO}_2$	173.114	304				
AMINES:							
Diethylamine	$(\text{C}_2\text{H}_5)_2\text{NH}$	73.116	55-56	.9		⁴ .5	
n-Butylamine	$\text{C}_4\text{H}_9\text{NH}_2$	73.116	76-78	.6		.6	
Aniline	$\text{C}_6\text{H}_5\text{NH}_2$	93.094	182				
Methylaniline	$\text{C}_6\text{H}_5\text{NHCH}_3$	107.115	193.5				
Dimethylaniline	$\text{C}_6\text{H}_5\text{N}(\text{CH}_3)_2$	121.136	192				
Ethylbenzylaniline	$\text{C}_6\text{H}_5\text{N}(\text{C}_2\text{H}_5)\text{CH}_2\text{C}_6\text{H}_5$	211.219	285-286				
Acetphenylenediamine	$\text{NH}_2\text{C}_6\text{H}_4\text{NHCOCH}_3$	150.136					
α-Naphthylamine	$\text{C}_{10}\text{H}_7\text{NH}_2$	143.130	300				
MISCELLANEOUS NITROGENOUS COMPOUNDS:							
Pyridine	$\text{C}_5\text{H}_5\text{N}$	79.073	116-118	.83	.83	.83	.83
Hexamethylenetetramine	$\text{C}_6\text{H}_{12}\text{N}_4$	140.158					
INORGANIC COMPOUNDS:							
Carbon dioxide	CO_2	44.005	-78.5	50.0		100.0	
Thionyl chloride	SOCl_2	118.980	78	⁴ 4.5	⁴ 4.5	⁴ 4.5	⁴ 4.5

⁴ Minimum concentration tested.

HYDROCARBONS

Against grain weevils, a 100 per cent kill was obtained with amylenes at a concentration of 40.31 per cent, with cyclohexane at a concentration of 13 per cent, and with benzene at a concentration of 3.8 per cent. Against all of the insects except *Tribolium*, kerosene, toluene, o-xylene, naphthalene, and anthracene were not more than 75 per cent effective.

Trillat and Legendre (26) found that benzene and toluene vapors at a concentration of 10 grams per cubic meter were insufficient to kill mosquitoes after exposure for an hour at 20° to 28° C.

Holt (9) tried the effect of benzene, toluene, xylene, naphthalene, anthracene, "benzoline," heptane, petrol, petroleum ether, and paraffin oil (boiling point, 150° to 289° C.), in concentrations of 1 drachm

to 1,250 cubic centimeters, on roaches (*Periplaneta orientalis* L.) confined in a glass-stoppered 1,250-cubic centimeter bottle. Benzene and toluene showed the same toxicity. In general, the higher the boiling point the longer the time necessary to kill. "Benzoline" (boiling point, 60° C.), which killed in 17 minutes, was the most toxic.

McClintock, Hamilton, and Lowe (13) compared the toxicity of the vapors of naphthalene, kerosene, Australene, Oregon fir turpentine, oil turpentine, Michigan wood turpentine, and oil of *Pinus palustris* on bedbugs, cockroaches, house flies, clothes moths, and mosquitoes in an 800-liter hood. Naphthalene was the most toxic.

Jewson and Tattersfield (10) found that naphthalene vapor had no apparent effect on mites (*Aleurobius farinæ* De G.), even after exposure for 16 hours.

Lloyd (12) found pure naphthalene, free from tarry acids, to be a poor fumigant against adult greenhouse white flies (*Asterochiton vaporariorum* Westw.). At a temperature of 69° to 72° F., 0.5 gram of pure naphthalene killed only 3 per cent of the adult white flies confined in a half-gallon glass-stoppered jar during exposure for an hour.

Lefroy (11) found that xylene, turpentine, cymene, and pseudocumene killed 100 per cent, and that eucalyptus oil killed some mealworms dipped in the liquid.

Russell (22) found toluene more effective than carbon disulphide in the partial sterilization of sick soil in which tomatoes were growing.

Titschack (25) found the vapor of benzene to be very much less effective than carbon disulphide against the eggs, larvæ, and moths of the clothes moth (*Tineola bisellrella* Hum.). Xylene also showed a low toxicity, but naphthalene was effective.

Richardson and Smith (21) found the toxicity toward the black aphid (*Aphis rumicis* L.) to increase from benzene through toluene to xylene, but in all cases the tolerance of the host plant, nasturtium, was much less than the minimum toxic concentration. Cyclohexane was twice as toxic as benzene.

Tattersfield and Roberts (24) found that anthracene and phenanthrene were nontoxic to wireworms; that mesitylene, *p*-cymene, and naphthalene had marginal toxicity; and that *n*-pentane, *n*-hexane, *n*-heptane, benzene, toluene, and *m*- and *p*-xylene had low toxicity. Of the hydrocarbons examined only pseudocumene had moderate toxicity. Moore (15) found that the toxicity of petroleum ether, benzene, toluene, xylene, gasoline, camphene, naphthalene, and kerosene toward flies increased with diminishing volatility.

The low toxicity and ready inflammability of all the hydrocarbons tested make this class of organic compounds unpromising in the search for a practical fumigant.

BROMIDES

The most effective bromide tested during the investigation reported in this bulletin was ethylene bromide, which killed 100 per cent of the weevils at a concentration of only 0.5 per cent. The order of toxicity of the other bromides follows: Bromoform, *n*-butyl, ethyl, allyl, *n*-propyl, and benzyl bromide, and bromobenzene.

Bromoform and monobromobenzene were found by Tattersfield and Roberts (24) to be moderately toxic to wireworms. Moore (14) tested the action on flies of bromobenzene, *p*-dibromobenzene, *o*- and *p*-bromotoluene, and bromoxylene. The disubstitution products were more toxic than the monosubstitution products, and the bromine

compounds were more toxic than the corresponding chlorine compounds. From tests on potato-beetle eggs with bromoform, trimethylene bromide, *o*-bromotoluene, and bromoxylene, Moore and Graham (17) concluded that the toxicity of organic compounds to insect eggs increased with decreasing volatility. Moore (15) also tested the action of bromoform, brometone (tribromo tertiary butyl alcohol), and ethylene bromide on flies.

Bertrand and Rosenblatt (2) found benzyl bromide to be more toxic than carbon disulphide, but less toxic than monochloroacetone to the larvæ of (*Bombyx*) *Malacosoma neustria* L.

Holt (9) found that it took nine minutes for the vapor of bromoform (0.8 gram per liter) to kill cockroaches (*Periplaneta orientalis* L.), as compared with eight minutes for carbon disulphide at the same concentration.

Organic bromides have the disadvantage of being costly. While the bromides are in general more effective than the corresponding chlorides, they can not be expected to give an economical fumigant, because liquid bromine is usually quoted at a price four to five times that of chlorine; furthermore, 35.5 units by weight of chlorine are the chemical equivalent of about 80 units by weight of bromine.

Ethylene bromide and ethyl bromide (Tables 3, 4, 5, 6, 7, 8) are the only bromides which seem worth a further trial.

CHLORIDES

Trichloroethane, *s*-tetrachloroethane, propylene dichloride, and a mixture of *o*- and *p*-dichlorobenzene gave 100 per cent mortality in concentrations of 2 per cent or less. Other chlorides showed the following order of toxicity: Monochlorobenzene, *p*-dichlorobenzene, tetrachloroethylene, methylene chloride, ethylene chloride, carbon tetrachloride, chloroform, acetylene dichloride, trichloroethylene, ethylidene chloride, and isopropyl chloride.

Tattersfield and Roberts (24) experimented with wireworms. Dichloroethylene, trichloroethylene, carbon tetrachloride, chloroform, and tetrachloroethane, in the aliphatic series, and monochlorobenzene and *o*-chlorotoluene, in the aromatic series, had a low toxicity; 1, 2, 4-trichlorobenzene, monochloroxylene, *p*-dichlorobenzene, and benzotrichloride had a marginal toxicity; *o*-dichlorobenzene and benzal chloride had a moderate toxicity; and benzyl chloride had a high toxicity.

Parker and Long (19), using several chlorides against the larvæ of *Trogoderma khapra* Arrow, at a concentration of 10 ounces per 1,000 cubic feet and an exposure of 1,000 minutes, obtained a mortality of 11 per cent with carbon tetrachloride, 27.7 per cent with trichloroethylene, 73.3 per cent with tetrachloroethane, and 77.7 per cent with pentachloroethane.

Holt (9), Trillat and Legendre (26), Bertrand and Rosenblatt (2), Lefroy (11), McClintock, Hamilton, and Lowe (13), Titschack (25), and many other investigators report that chloroform has a low toxicity against various insects.

Altson (1), using the vacuum method commonly employed with hydrocyanic acid gas, found tetrachloroethane useless for the fumigation of beetles in wood. He recommends both *o*- and *p*-dichlorobenzene as a deterrent to timber beetles.

Lloyd (12) reports that tetrachloroethane gives good results against the white fly in greenhouse fumigation, but is without

effect on the eggs. About half a pint per 1,000 cubic feet should be used, and the fumigation should be continued for not less than 18 hours. Lloyd states that:

It has been used for a wide variety of plants, including tomatoes, and no damage has resulted except in one case when the foliage of three young sycamores (*Acer pseudo-planatus*)⁴ growing in pots turned brown the day after the fumigation and was subsequently shed.

Speyer (23) also found tetrachloroethane effective against adult white flies, but its effect on the red spider was only temporary. The tetrachloroethane, when used at the rate of 10 ounces per 1,000 cubic feet, severely injured several varieties of chrysanthemum.

Richardson and Smith (21) found that 34 and 31 grams per 100 cubic centimeters of chloroform and carbon tetrachloride, respectively, were necessary to kill aphids, and that the tolerance of the nasturtium plant for these two compounds varied from 5 to 8. Chlorobenzene and commercial trichlorobenzene were less toxic to aphids than carbon disulphide, and their toxic concentrations exceeded the tolerance of the plant.

Moore (14, 15) used chlorobenzene, *p*-dichlorobenzene, chloroform, carbon tetrachloride, and chloreton (trichloro tertiary butyl alcohol) in various tests on flies.

FLUORIDES

Fluorobenzene at a concentration of 1.9 per cent showed 100 per cent mortality against *Sitophilus oryza*, but difluorodiphenyl exhibited almost no toxicity, owing probably to its slight volatility at the temperature of the test.

Organic fluorine compounds have not been tested by many investigators, probably because they are rare and expensive.

IODIDES

The only iodide tested, normal butyl iodide, killed all the weevils at a concentration of 0.8 per cent.

Moore (14) found iodobenzene to be more toxic to house flies than the corresponding bromine and chlorine compounds.

Tattersfield and Roberts (24) found iodoform to be nontoxic to wireworms, while iodobenzene was moderately toxic.

According to Holt (9), cockroaches dusted with iodoform did not succumb until a period of 9 hours had elapsed.

ALCOHOLS AND PHENOLS

Methyl alcohol was more toxic than ethyl alcohol, but with this exception the toxicity increased with increasing molecular weight through isoamyl alcohol. Thymol, menthol, and geraniol are so slightly volatile at ordinary temperature as to be practically nontoxic.

Richardson and Smith (21) found methyl, ethyl, *n*-propyl, *n*-butyl, capryl, isoamyl, benzyl, and furfuryl alcohols to be ineffective against aphids. Even pure methyl and ethyl alcohols killed less than 95 per cent of the insects.

Moore (15) found the toxicity of methyl, ethyl, and amyl alcohols, menthol, and thymol on house flies to increase with decreasing volatility.

Trillat and Legendre (26) showed that methyl, ethyl, propyl, and amyl alcohols had only a feeble toxicity to mosquitoes.

⁴Sycamore maple.

Titschack (25) showed that ethyl alcohol was ineffective against the eggs and larvæ of clothes moths.

McClintock, Hamilton, and Lowe (13) found the effectiveness of methyl and ethyl alcohols to be low against bedbugs, cockroaches, house flies, clothes moths, and mosquitoes. Menthol was about half as toxic to bedbugs as carbon disulphide.

Holt (9) found methyl alcohol to be more toxic to cockroaches than ethyl or amyl alcohol. All required at least 45 minutes to produce death, however.

Lefroy (11) reported that 70 per cent ethyl alcohol failed to kill any mealworms dipped in it.

Burmeister in 1836 (3, p. 39) recorded instances of beetles which after being immersed in spirits of wine for 12 hours recovered all their functions when removed from it.

Apparently alcohols of the fatty acid series have low toxicity to insects and are not suited for fumigants.

ALDEHYDES

Crotonaldehyde was much more toxic than *n*-butyraldehyde. Furfural and benzaldehyde showed marked toxicities at concentrations of less than 1 per cent. The chlorine-substituted aldehyde, chloral hydrate, had low toxicity.

Richardson and Smith (21) tested paraldehyde, aldehyde ammonia, chloral hydrate, furfural, and benzaldehyde against aphids. Even benzaldehyde, the most effective, was not a practical aphicide, as a 5 per cent solution was required to kill.

None of the many experimenters with formaldehyde, an effective fungicide, has discovered any practical value for it as a fumigant against insects. Phelps and Stevenson (20) found a 0.5 per cent solution to be effective as a stomach poison to flies, possessing a coefficient of 2.32 as compared with a coefficient of 1 for 0.001 normal arsenite solution. Four and 8 per cent formaldehyde solutions were less effective than the 0.5 per cent solution; a 1 per cent solution had a coefficient of 2.36. The addition of molasses and brown sugar diminished the effectiveness of the formaldehyde solutions. Dry powdered paraformaldehyde showed a coefficient of only 0.14, while a saturated solution had a coefficient of 1.90.

Moore (15) found that the toxicity of acetaldehyde, chloral hydrate, and furfural to house flies in general increased as their volatilities decreased.

According to Davis (5), acetaldehyde is ineffective against white grubs in soil. Trillat and Legendre (26) report that it has only a feeble toxicity for mosquitoes.

Lefroy (11) found a solution of chloral hydrate in water to have no effect on mealworms. Holt (9) reports that roaches dusted with powdered chloral hydrate survived for 4 hours.

McClintock, Hamilton, and Lowe (13) found benzaldehyde to be more effective than carbon disulphide against bedbugs, cockroaches, and house flies, equally effective against clothes moths, and less than half as toxic to mosquitoes.

KETONES

Ethyl methyl ketone was more effective than acetone (dimethyl ketone). The introduction of chlorine into a ketone, as in chloroacetone, enormously increased its toxicity.

Richardson and Smith (21) found that even 100 per cent acetone and ethyl methyl ketone killed less than 95 per cent of aphids, and acetal was effective only in concentrations over 50 per cent.

Moore (15) found the toxicity of acetone, bromomethyl phenyl ketone, and menthone to house flies to increase with diminishing volatility.

Holt (9) found that it took acetone vapor 32 minutes to kill cockroaches, as against 8 minutes for carbon disulphide.

Lefroy (11) found that acetone killed no mealworms dipped in it.

According to McClintock, Hamilton, and Lowe (13), acetone has very little effect against bedbugs, cockroaches, house flies, clothes moths, and mosquitoes.

Titschack (25) found acetone less effective than carbon disulphide against the eggs and larvæ of clothes moths.

Guérin and Lormand (7) found that bromoacetone had no apparent effect on plants at a concentration of 1 to 2,000 exposed for an hour.

Bertrand and Rosenblatt (2) found monochloroacetone to be very much more active against the larvæ of (*Bombyx*) *Malacosoma neustria* L. than ether, chloroform, carbon tetrachloride, or carbon disulphide, but less active than hydrocyanic acid or chloropicrin.

ESTERS

The esters showed a higher toxicity to weevils than any other class of organic compounds tested. Methyl, ethyl, isopropyl, and methyl cyano formates and isoamyl acetate are more toxic, molecule for molecule, than carbon disulphide. Propyl acetate is less toxic than carbon disulphide; *n*-butyl acetate, isobutyl acetate, and ethyl-*n*-valerate are so slightly volatile at the temperatures used that they do not give 100 per cent mortality in the concentrations obtained.

Richardson and Smith (21) tested the action on aphids of amyl acetate, methyl salicylate, and benzyl acetate. Benzyl acetate, at a concentration of about 1 per cent, was the most effective.

Moore (15) tried the effect on house flies of methyl salicylate, ethyl malonate, ethyl acetoacetate, amyl acetate, amyl valerate, and propyl acetate.

Lefroy (11) found that amyl acetate killed all mealworms dipped in it, while methyl salicylate and ethyl acetate killed some.

Moore (16) tried the effect of a wide variety of substances on the clothes louse. He concluded that liquids less volatile or more volatile than creosote are not as successful, and that, while not quite as good, crude phenol and methyl salicylate are the best substitutes.

ETHERS

s-Dichloromethyl ether had an efficiency comparable to that of carbon disulphide; chloromethyl ether was about half as effective. Acetal was effective at a concentration of slightly over 4 per cent.

Ethyl ether was the most volatile and least toxic of all materials used by Moore (15) against house flies.

Ethyl ether has been tested by a number of other experimenters, but in all cases it showed only a low toxicity against insects.

CHLOROHYDRINS

Epichlorohydrin, the most toxic substance tested in this investigation, killed 100 per cent of *S. oryza* at a concentration of only 0.09 per cent. Ethylene chlorohydrin at the maximum concentration tested, 1.4 per cent, was ineffective.

Apparently chlorohydrins have not been tested by other experimenters with insecticides.

SULPHUR COMPOUNDS

Methyl and ethyl thiocyanates and allyl isothiocyanate were more effective than carbon disulphide; methyl and ethyl sulphides and ethyl mercaptan were less effective than carbon disulphide. Butyl mercaptan was as effective as carbon disulphide against *S. oryza*, but only half as effective against the other species of weevils.

Mercaptol is so slightly volatile as to be ineffective. Cyanogen sulphide killed no weevils at a concentration of 0.1 per cent; perchloromethylmercaptol and *p*-toluenesulphochloride gave variable kills.

Richardson and Smith (21) found that a concentration exceeding 5 per cent of carbon disulphide was necessary to kill aphids, whereas a solution containing less than 2 per cent injured the nasturtium plant.

According to Moore (15), molecule for molecule, allyl isothiocyanate is more toxic against house flies than chloropicrin, and carbon disulphide and ethyl mercaptan are more toxic than their relative volatilities would indicate.

Tattersfield and Roberts (24) found allyl isothiocyanate to be the most toxic to wireworms of all compounds tested by them. The toxicity of carbon disulphide was equal to that of benzene.

Speyer (23) found ethyl mercaptan to have no effect on red spiders. It also failed to kill adult white flies.

NITRILES AND ISONITRILES

n-Valeronitrile had a toxicity as great as that of carbon disulphide; acetonitrile was about one-fifth as toxic as carbon disulphide. Propionitrile and phenylisonitrile were ineffective. Phenylacetonitrile was the most toxic of this class of compounds, killing all *S. oryza* at a concentration of 0.10 per cent.

Moore (14) found that the toxicity of benzonitrile to house flies compared with that of iodobenzene and xylene.

NITRITES

n-Butyl nitrite and isoamyl nitrite were about equally toxic.

Moore (15) found that amyl nitrite had about the same toxicity to flies as gasoline.

Tattersfield and Roberts (24) found that the toxicity of amyl nitrate to wireworms was low and that the toxicity of amyl nitrite was moderate.

Speyer (23) found that the grubs of a chironomid fly (*Orthocladius*) came to the surface of the soil of cucumber pot plants and were killed in a short time when amyl nitrite and amyl nitrate were used. He also found that methyl nitrite had a more permanent effect on red spiders than amyl nitrate and tetrachlorethane, but that it was necessary to use concentrations which hurt the plants. The adults of the white fly were killed by all these vapors.

NITRO COMPOUNDS

Nitromethane was the only nitro compound which killed all of the insects; a concentration of 3.6 per cent was necessary. Nitrobenzene, *p*-chloronitrobenzene, *m*-dinitrobenzene, and α -nitronaphthalene at saturation concentrations killed none of the weevils.

Moore (15) found nitrobenzene even more toxic than nicotine to house flies.

Several nitro compounds have been tested against wireworms by Tattersfield and Roberts (24). *m*-Nitroaniline, *o*-nitroaniline, *p*-nitrophenol, nitronaphthalene, dinitrobenzene, and nitrobenzaldehyde were nontoxic; *o*- and *p*-nitrochlorobenzene, *o*- and *p*-nitrotoluene, *p*-nitroaniline, and nitroxylylene (mixed derivatives) had marginal toxicity; nitromethane had low toxicity; nitrobenzene had moderate toxicity; and *o*-nitrophenol and nitrochloroform (chloropicrin) had high toxicity.

AMINES

Diethylamine and *n*-butylamine were more toxic to weevils than carbon disulphide. None of the other amines tested (aniline, methylaniline, dimethylaniline, ethylbenzylaniline, acetphenylenediamine, and α -naphthylamine), however, showed any toxicity.

Richardson and Smith (21) tested the action of the following compounds on aphids: Trimethylamine hydrochloride, tetramethylammonium chloride, *p*-phenylenediamine, phthalimidine, methylamine hydrochloride, diethylamine, triethylamine, triethylamine hydrochloride, tetraethylammonium chloride, tetrapropylammonium hydroxide, isobutylamine, diamylamine, triacetoneamine, hexamethylenetetramine, formamide, dicyanodiamide, choline hydrochloride, betaine hydrochloride, nitroguanidine, succinimide, aniline, benzylamine, benzidine hydrochloride, *m*-phenylenediamine hydrochloride, camphylamine, and tetrahydrobetanaphthylamine hydrochloride. None of these approached nicotine in toxicity. Tetramethylammonium chloride, the most effective, required a concentration of 0.35 per cent, compared with an effective concentration for nicotine of only 0.007 per cent.

Tattersfield and Roberts (24), in tests on wireworms, found that *o*- and *m*-nitroaniline, *m*-phenylenediamine, phenylhydrazine, naphthylamine, and diphenylamine were nontoxic; that *p*-nitroaniline and *p*-chloroaniline had marginal toxicity; that trimethylamine, ethylamine, dimethylamine, monomethylamine, aniline, and *o*-chloroaniline had moderate toxicity; and that *o*- and *p*-toluidine, xylydine, dimethylaniline, and monomethylaniline had high toxicity.

Foreman and Graham-Smith (6, p. 113) found aniline (saturated aqueous solution) and aniline hydrochloride to be toxic to flies when taken by the mouth. The hydrochloride of *o*-toluidine had a similar effect, but *p*-toluidine hydrochloride was nontoxic. Flies taking monomethylaniline hydrochloride appeared to be dead in 10 minutes, but recovered in 2 hours. Hydroxylamine hydrochloride in 2 per cent solution had little effect, or none, and a 1 per cent solution of *m*-phenylenediamine had no effect.

Jewson and Tattersfield (10) tested the action on mites (*Aleurobius farinæ*) of aniline, monomethylaniline, and dimethylaniline. Most of the large mites remained alive, but moved sluggishly.

MISCELLANEOUS NITROGENOUS COMPOUNDS

Pyridine at a concentration of 0.8 per cent killed all the weevils, but hexamethylenetetramine showed no killing power.

Richardson and Smith (21) sprayed aphids with the following pyridine and quinoline derivatives: Nicotinic acid nitrate, 4-dimethylaminoantipyrine, *n*-ethyl piperidine sulphate, *n*-ethyl piperidine, crude chloropiperidine, piperidine sulphate, pyridinium ethyl hydroxide, pyridine, pyridinium ethyl iodide, piperidine, methylene dipiperidine, $\gamma\gamma$ dipyridyl, α -picoline, pyrrole, quinoline, tetrahydro-

quinoline, quinaldine, and piperazine. The minimum toxic concentration of pyridine was 25 grams per 100 cubic centimeters of solution. None of these compounds was more than one-hundredth as effective as nicotine.

McClintock, Hamilton, and Lowe (13) found little difference in the toxicity of pyridine, pyridine bases, and quinoline for bedbugs, cockroaches, house flies, clothes moths, and mosquitoes.

Jewson and Tattersfield (10) found pyridine effective against mites.

Lefroy (11) found that quinoline and pyridine killed some of the mealworms dipped in the liquids, but that a 1 per cent aqueous solution of nicotine killed none.

Tattersfield and Roberts (24) found that pyridine was moderately toxic against wireworms.

Moore (15) found pyridine somewhat more toxic than its position in the table of organic compounds arranged according to volatility would indicate. Its toxicity was about the same as that of furfural.

INORGANIC COMPOUNDS

None of the inorganic compounds tested proved suitable as a fumigant. Thionyl chloride killed all weevils at a concentration of 4.5 per cent; concentrations of carbon dioxide ranging from 50 to 100 per cent were required to effect a complete kill.

RELATION BETWEEN VOLATILITY AND TOXICITY OF FUMIGANTS

The compounds causing 100 per cent mortality to *S. oryza* (selected on account of its high resistance to fumigants) exposed to them for 24 hours, together with their boiling points, are listed in Table 3.

TABLE 3.—*Toxicity of fumigants to Sitophilus oryza* (arranged in order of decreasing effect)

Fumigant	Minimum concentration causing 100 per cent mortality in 24 hours	Boiling point ¹	Minimum quantity causing 100 per cent mortality in 24 hours	Cost per pound ²	Cost per 1,000 cubic feet
	Per cent	° C.	Lbs. per 1,000 cu. ft.	Dollars	Dollars
Epichlorohydrin.....	³ 0.09	116.6	0.23	27.22	6.26
Phenylacetoneitrile.....	.10	231.7	.33	18.14	5.99
Chloroacetophenone.....	.10	247.0	.43	9.07	3.90
Methyl thiocyanate.....	.10	133.0	.20	27.22	5.44
Acetophenone.....	³ .16	202.0	.54	5.90	3.18
Chloroacetone.....	.26	119.0	.67	27.22	18.24
Benzaldehyde.....	³ .27	178.3	.80	1.13	.90
Allyl isothiocyanate.....	³ .34	150.7	.94	4.08	3.84
Crotonaldehyde.....	³ .50	104.0	.98	-----	-----
Ethylene bromide.....	.50	129.0	2.62	.91	2.38
Isopropyl formate.....	³ .50	70.0	1.23	2.27	2.79
Mesityl oxide.....	.55	130.0	1.50	13.61	20.42
Isoamyl nitrite.....	³ .60	99.0	1.96	9.07	17.78
Ethyl formate.....	.60	54.4	1.24	.40	.50
n-Butyl nitrite.....	.60	75.0	1.72	3.63	6.24
n-Butylamine.....	.60	76.0	1.22	36.29	44.27
Methyl cyano formate.....	.68	100.0	1.61	-----	-----
Propylene dichloride.....	³ .70	96.8	2.20	1.27	2.79
Furfural.....	³ .80	161.0	2.14	.25	.54
Trichloroethane.....	.80	74.1	2.97	2.27	6.74
Isoamyl alcohol.....	.80	131.0	1.96	1.81	3.55
n-Butyl iodide.....	.80	129.9	4.10	9.07	37.19
Ethyl thiocyanate.....	.80	146.0	1.94	22.68	44.00
Pyridine.....	.83	116.0	1.83	.36	.66

¹ Boiling points taken from Beilstein.

² Most of the prices are taken from List No. 10 of the Eastman Kodak Co., Rochester, N. Y., issued September, 1923; some are from wholesale quotations by the Oil, Paint, and Drug Reporter, March 3, 1924; and some are quotations from the Miner Laboratories, Chicago, Ill.

³ Minimum concentrations tested.

TABLE 3.—*Toxicity of fumigants to Sitophilus oryza (arranged in order of decreasing effect)—Continued*

Fumigant	Minimum concentration causing 100 per cent mortality in 24 hours	Boiling point	Minimum quantity causing 100 per cent mortality in 24 hours	Cost per pound	Cost per 1,000 cubic feet
	Per cent	° C.	Lbs. per 1,000 cu. ft.	Dollars	Dollars
Methyl formate.....	³ .90	32.3	1.51	1.59	2.40
Isoamyl acetate.....	.90	139.0	3.26	.91	2.97
Diethylamine.....	.90	55.0	1.83	16.33	29.88
<i>n</i> -Butyl mercaptan.....	.90	97.0	2.26	36.29	82.02
Bromoform.....	³ .94	150.5	6.62	2.04	13.50
<i>o</i> - and <i>p</i> -Dichlorobenzene.....	1.00	175.0	4.10	.15	.615
Carbon disulphide.....	1.00	46.0	2.12	.06	.127
<i>s</i> -Tetrachloroethane.....	1.10	147.0	5.15	1.13	5.82
<i>n</i> -Valeronitrile.....	1.10	141.0	2.55	27.22	69.41
<i>s</i> -Dichloromethyl ether.....	1.13	105.0	3.62	27.22	98.54
<i>n</i> -Propyl alcohol.....	1.70	97.4	2.85	1.36	3.88
<i>n</i> -Butyl alcohol.....	³ 1.70	117.0	3.51	.76	2.67
Ethyl methyl ketone.....	³ 1.80	79.6	3.62	.30	1.09
Fluorobenzene.....	³ 1.90	84.9	5.09		
Chlorobenzene.....	2.00	132.0	6.27	.09	.56
Chloromethyl ether.....	³ 2.20	59.5	4.94	13.61	67.23
<i>n</i> -Propyl acetate.....	2.50	101.6	7.11	2.27	16.14
<i>n</i> -Butyl bromide.....	2.60	101.0	9.93	4.54	45.08
Tetrachloroethylene.....	2.60	121.0	12.02	1.13	13.58
Ethyl sulphide.....	2.90	92.0	7.29	2.27	16.54
Ethyl bromide.....	2.90	38.4	8.81	.40	3.52
Nitromethane.....	3.60	101.0	6.12	9.07	55.51
Benzene.....	3.80	80.2	8.27	4.04	.33
Allyl bromide.....	³ 3.90	70.0	13.15	15.88	208.82
<i>n</i> -Propyl bromide.....	4.10	70.8	14.05	13.61	191.22
Acetal.....	4.30	102.2	14.15	18.14	256.68
Methylene chloride.....	4.40	47.6	10.42	6.80	70.86
Thionyl chloride.....	³ 4.50	78.0	14.92	9.07	135.32
Acetonitrile.....	5.00	81.6	5.71	18.14	103.58
<i>n</i> -Butyraldehyde.....	5.50	77.0	11.05	13.61	150.39
Ethylene chloride.....	6.00	83.5	16.55	.68	11.25
Carbon tetrachloride.....	6.90	76.7	29.58	.082	2.44
Chloroform.....	7.00	61.2	23.29	.32	7.45
Acetone.....	8.20	56.5	13.27	.20	2.65
Acetylene dichloride.....	9.00	55.0	24.31		
Trichloroethylene.....	10.00	88.0	36.62	.36	13.18
Ethylidene chloride.....	10.20	59.2	28.13	22.68	637.99
Methyl sulphide.....	10.40	38.0	18.00	45.36	816.48
Cyclohexane.....	³ 13.00	80.8	30.48	22.68	691.29
Methyl alcohol.....	16.00	64.7	14.28	⁵ .15	2.14
Ethyl mercaptan.....	³ 19.00	37.0	32.89	9.07	298.31
Amylene.....	40.30	22-37	78.76	9.07	714.35
Carbon dioxide.....	50.00	-78.5	61.32	.07	4.29

³ Minimum concentrations tested.⁴ Chemically pure, in drums, sold for 30 cents a gallon.⁵ Purified, in drums, sold for \$1 a gallon.

While the volatility of organic compounds at ordinary temperatures is not proportional to their boiling points, very few data on the vapor pressure of these compounds at ordinary temperatures are available. For that reason the boiling points were used in this investigation. The compounds are arranged in the order of decreasing toxicity.

Moore (15) states:

In general, the toxicity of a volatile organic compound is correlated closely with its volatility. A decreasing volatility is accompanied by an increased toxicity. The boiling point of the chemical is a general index of its volatility. Compounds with boiling points of 225° to 250° C. are usually so slightly volatile that they do not produce death except after very long exposures.

If this theory is true, the boiling points of the compounds should show a decrease, indicating increasing volatility, but no well-defined relation between the toxicity and the boiling point is shown in Table 3. This is not in agreement with Moore's theory.

TABLE 4.—Results of fumigation tests on weevils in grain in 19-liter glass bottles

[illegible]

[illegible]

¹ Same results obtained for tests Nos. 604, 631 and 632.

EFFECT OF FUMIGATION ON WEEVILS IN THE PRESENCE OF GRAIN IN BOTTLES, BOXES, AND BARRELS

In order to ascertain the action of the compounds as they would be used in practical fumigation work, weevils were placed in pill boxes, with perforations to permit the entrance of the vapor, and the boxes were placed at different levels in grain, usually wheat. The weevils, usually about 10 to the box, were exposed to the action of the compounds for 24 hours, at temperatures from 21° to 32° C.

One series of tests was carried out in large glass bottles of 19 liters capacity (Table 4), another in a wooden box of 100 cubic feet capacity (Table 5), and another in a barrel of 6 cubic feet capacity (Table 6). The fumigant or mixture of fumigants was applied in all these tests by pouring the calculated quantity over the top of the grain and then closing the receptacle. The bottle had a stopper, the barrel had a wooden head, and the box was provided with felt striping and a tight-fitting cover, which could be clamped down.

(In Tables 4 and 5 the more toxic substance, with its concentration, is given first; the less toxic substance, used principally as a diluent, with its concentration, is given next.)

TABLE 6.—Results of fumigation tests on weevils in grain in barrels (6 cubic feet capacity)

Test No.	Fumigant	Concentration of fumigant	Average mortality	Weevils killed after exposure for 24 hours								
				Bottom of barrel			Middle of barrel			Top of barrel		
				<i>S. oryza</i>	<i>S. granarius</i>	<i>Tribolium</i>	<i>S. oryza</i>	<i>S. granarius</i>	<i>Tribolium</i>	<i>S. oryza</i>	<i>S. granarius</i>	<i>Tribolium</i>
		<i>Lbs. per 1,000 cu. ft.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>
386	Carbon tetrachloride (75 per cent) and turpentine (25 per cent).	15.0	17	0	0	95	0	0	25	0	0	30
387	do.	30.0	14	30	0	100	0	0	0	0	0	0
394	Carbon disulphide	7.0	52	100	-----	0	50	-----	0	100	-----	60
409	do.	10.0	87	100	-----	100	100	-----	100	100	-----	0
410A	do.	10.0	71	100	-----	100	25	-----	0	100	-----	100
¹ 441	do.	10.0	100	100	-----	100	100	-----	100	100	-----	100
403	{ Ethylene bromide	3.1	50	100	-----	100	100	-----	100	100	-----	100
	{ Carbon tetrachloride	10.0										
431	{ Ethylene bromide	3.1										
	{ Carbon tetrachloride	10.0										
450	{ Ethyl bromide	9.1	96	100	-----	100	100	-----	100	50	-----	25
456	{ do.	18.2	100	100	-----	100	100	-----	100	100	-----	100
447	{ Epichlorohydrin	1.0	100	100	-----	100	100	-----	100	100	-----	100
	{ Carbon tetrachloride	10.0										
458	{ <i>n</i> -Butyl nitrite	2.9										
	{ Carbon tetrachloride	10.0	92	50	-----	100	100	-----	100	100	-----	100

¹ Barrel made as tight as possible in this test.

The insecticidal action of a gas is greatly lessened by the presence of grain, probably because the grain absorbs many vapors in large quantities and because the grain mechanically interferes with the diffusion of the gas throughout the receptacle. For example, in a glass vessel containing nothing but weevils and the vapor of the compound (mixed with air), epichlorohydrin killed 100 per cent of the insects at a concentration equivalent to 0.23 pound per 1,000 cubic feet; when the weevils were planted in wheat, a concentration

of epichlorohydrin equivalent to 2.06 pounds per 1,000 cubic feet, combined with carbon tetrachloride at the rate of 10 pounds per 1,000 cubic feet, did not kill all of the insects in every test.

The compounds consistently causing 100 per cent mortality in the small-scale tests in wheat (Tables 4, 5, and 6) are shown in Table 7.

TABLE 7.—*Most effective fumigants for weevils (based on results in Tables 4, 5, and 6)*

Test No.	Fumigant	Minimum concentration consistently causing 100 per cent mortality of all species after exposure for 24 hours	Test No.	Fumigant	Minimum concentration consistently causing 100 per cent mortality of all species after exposure for 24 hours
		<i>Lbs. per 1,000 cu. ft.</i>			<i>Lbs. per 1,000 cu. ft.</i>
456	Ethyl bromide.....	18.2	439	Ethylene bromide.....	6.2
462	Ethyl bromide.....	4.5	603	Carbon tetrachloride.....	10.0
630	Carbon tetrachloride.....	10.0	604	Ethylene bromide.....	8.0
615	Ethyl bromide.....	4.5	631	Carbon tetrachloride.....	8.0
616	Carbon disulphide.....	2.2	632		
	Carbon tetrachloride.....	10.0	589	Ethylene bromide.....	5.3
	Ethyl bromide.....	4.5	590	Carbon tetrachloride.....	12.0
611	Carbon disulphide.....	2.2	594	Ethylene bromide.....	5.3
612	Carbon tetrachloride.....	10.0	597	Chlorobenzene.....	10.0
	Ethylene bromide.....	5.3	598	Carbon tetrachloride.....	10.0
599	Ethyl bromide.....	13.2		Crotonaldehyde.....	2.9
600	Ethylene bromide.....	5.3	472	Carbon tetrachloride.....	10.0
540	Ethyl bromide.....	1.7		Ethyl formate.....	2.5
545	Methyl formate.....	6.1	445	Carbon tetrachloride.....	10.0
585	Ethyl bromide.....	11.2	546		
586	Methyl formate.....	3.5	547	Carbon disulphide.....	6.4

With the exception of carbon disulphide, the most economical fumigant at 1924 prices, is the combination of ethyl formate with carbon tetrachloride. (The ethyl acetate-carbon tetrachloride mixture,⁵ which was the most suitable and economical fumigant of all those tested, was not tried on a small scale.)

IN BOX CARS

Many tests were also carried out in box cars of grain. Weevils in perforated pill boxes (10 in each box) were planted at 10 different levels in the grain in a box car, by putting the boxes in the compartments of a grain sampler, which was then plunged into the grain at an angle of about 45°.

The fumigant was applied to the grain by sprinkling the liquid over the surface from a small sprinkler-top watering can as quickly as possible. The door of the car was then closed and sealed. Twenty-four hours later the pill boxes were withdrawn and the number of dead weevils was determined (Table 8). (The more toxic substance, with its concentration, is given first; the less toxic substance, used principally as a diluent, with its concentration, is given next in Table 8.)

⁵ The use of ethyl acetate and ethyl acetate-carbon tetrachloride mixture was suggested by R. C. Roark.

TABLE 8.—Results of fumigation tests on weevils in wheat in box cars

[illegible]

[illegible]

1 (g), *Sitophilus granarius*: (o), *S. oryza*: (T), *Tribolium*.

Level 1 is the bottom and level 10 the top of the grain.

³ All but those on top were killed.

Some were killed.

Grain was covered with canvas.

⁶ Some were killed: strong odor present.

7 Ounces per 1,000 cubic feet.

⁸ None were killed.

Many of the fumigants and combinations of fumigants which operated successfully in glass jars and in the presence of wheat in bottles, boxes, and barrels gave disappointing results when used on a large scale. A mixture, in equal proportions by weight, of ethylene bromide and carbon tetrachloride, used at the rate of 16 pounds per 1,000 cubic feet, was successful when applied to weevils in wheat in a box, but this mixture, even with the addition of 50 per cent more carbon tetrachloride, was ineffective when used in box cars. This is probably explained by the facts that in a box car the weevils may be in the grain at a much greater depth than in a small box or barrel and that the grain is much more tightly packed. Also, a box car is not a tight container and the vapors of the fumigant may not be long retained.

The most successful fumigants used in the box-car tests were ethyl formate in combination with carbon tetrachloride (12.1 pounds each per 1,000 cubic feet) and ethyl acetate (12.5⁶ pounds per 1,000 cubic feet) in combination with carbon tetrachloride (25 pounds per 1,000 cubic feet).

FIRE HAZARD FROM FUMIGANTS

The vapor pressures of ethyl acetate and carbon tetrachloride are very close for all temperatures up to their boiling points (Table 9, Fig. 1).

TABLE 9.—*Vapor pressures of ethyl acetate and carbon tetrachloride*

Temperature	Observed vapor pressure ¹		Temperature	Observed vapor pressure ¹	
	Ethyl acetate ²	Carbon tetrachloride ³		Ethyl acetate ²	Carbon tetrachloride ³
°C.	mm.	mm.	°C.	mm.	mm.
-20	6.55	9.92	40	186.20	210.90
-10	12.95	18.81	50	282.20	309.00
0	24.30	33.08	60	415.40	439.00
10	42.70	55.65	70	596.30	613.80
20	72.80	89.55	80	832.70	⁴ 836.35
30	118.70	139.60			

¹ Sydney Young. The vapor-pressures, specific volumes, heats of vaporization, and critical constants of 30 pure substances. *In Sci. Proc. Roy. Dublin Soc., n. s. (1909-10), 12:374-443.*

² Boiling point at 760 millimeters, 77.15° C.

³ Boiling point at 760 millimeters, 76.75° C.

⁴ Calculated.

A mixture of 3 volumes of carbon tetrachloride and 2 volumes of ethyl acetate (equivalent to 72.5 per cent carbon tetrachloride and 27.5 per cent ethyl acetate by weight) is noninflammable at ordinary temperatures. Moreover, in this mixture the vapors of the two components tend to separate but very slightly, thus making the mixture safe from fire hazard.

On the other hand, carbon disulphide has a much higher vapor pressure than carbon tetrachloride at ordinary temperatures, thus making mixtures of these compounds unsafe. The experiments of the Underwriters Laboratories at Chicago (Grain Dealers Journal, December 10, 1921, vol. 47, p. 798) show that a mixture of 75 per cent carbon tetrachloride and 25 per cent carbon disulphide by vol-

⁶ A mixture of ethyl acetate and carbon tetrachloride containing 33½ per cent by weight of the acetate will flash slightly at ordinary temperatures but will not continue to burn.

ume (equivalent to 79.1 per cent and 20.9 per cent by weight) gives off a highly inflammable vapor. The use of such a mixture to kill weevils or other insects is condemned as dangerous.

A mixture of carbon tetrachloride and carbon disulphide containing as little as 5 per cent by volume of carbon disulphide will flash at 20° C., although it will not continue to burn.

FIG. 1.—Vapor pressures of ethyl acetate and carbon tetrachloride (Table 9).

TABLE 10.—Results of milling tests with wheat exposed to fumigants

Sample No.	Fumigant	Concentration of fumigant	Odor of fumigant on				Flour	Remarks
			Wheat	Bran	Shorts			
		<i>Lbs. per 1,000 cu. ft.</i>						
8687	Tetrachloroethane	4.60	Slight	Strong	Slight		Very slight	Milled soft; flour creamy.
8697	Untreated		Natural					Milled soft; flour white.
8698	Tetrachloroethane	4.60	do	None	None		None	Do.
8699	do	7.30	Very strong	Strong	Slight		Slight	Milled soft; flour slightly creamy.
8700	do	2.90	Strong	Very slight	Very slight		None	Milled granular; flour creamy white.
8704	Monochlorobenzene	2.90	Very slight	do	do		do	Do.
8688	do	5.85	Strong	Strong	Strong		Very slight	Milled soft; flour white.
8702	do	5.85	do	do	Slight		Slight	Do.
8703	do	2.90	Very slight	Slight	do		Very slight	Milled granular; flour creamy white.
8701	Carbon tetrachloride	10.00	Slight	None	None		None	Milled granular; flour slightly creamy.
10285	Untreated		Natural					Milled soft (very dry); flour white; grain badly damaged by Angoumois grain moth.
10286	Ethylene bromide	13.40	Very slight					Milled soft (very dry).
10287	Untreated		Natural					Milled soft; flour white; grain damaged by moth.
10288	(Ethylene bromide	10.5	Very slight					Milled soft; flour white (very dry); grain damaged by moth.
10376	Carbon tetrachloride	5.0	Slight garlicky					Milled soft and dry; flour gray.
10377	(Ethyl acetate (85 per cent)	13.10	Very slight					Do.
10378	Carbon tetrachloride	15.10	Very slight					Milled soft; flour white.
10379	Untreated		Natural					Do.
10380	(Ethyl formate	12.10	Strong					Milled soft; flour white.
10381	Carbon tetrachloride	12.10	Slight garlicky					Do.
10433	Untreated		Strong	Noticeable	Noticeable		None	Milled soft; flour specky white.
10434	(Ethyl acetate (85 per cent)	15.00	Slight garlicky	Noticeable	Noticeable		Noticeable	Do.
10435	Untreated		Strong	Noticeable	Noticeable		Noticeable	Milled soft; flour white.
10436	(Ethyl acetate	11.80	Strong	Noticeable	Noticeable		Noticeable	Do.
10455	Untreated		Natural					Do.
10456	(Ethyl acetate (85 per cent)	12.50	Strong					Do.
10457	Carbon tetrachloride	25.00	Strong					Do.
10458	Untreated		Natural					Do.
10459	(Ethyl acetate (85 per cent)	10.00	Strong	Slight	Slight		Slight	Milled granular; flour slate gray; cookies present.
10460	Carbon tetrachloride	20.00	Musty and garlicky.					Milled granular; flour slate gray.
10461	Untreated		Strong					Milled soft; flour white.
10462	(Ethyl acetate (85 per cent)	5.00	Strong					
10463	Carbon tetrachloride	10.00	Garlicky					

		12.50 25.00	Strong	Very slight	Very slight	Very slight	
10537	{ Ethyl acetate (99 per cent)		{ Strong				Milled soft; flour creamy white.
	{ Carbon tetrachloride	15.0	{ Natural				Milled soft; flour white.
10866	{ Ethyl acetate (new process)	30.0	{ Slight				Do.
10867	{ Carbon tetrachloride	15.0	{ Slight				Do.
10868	{ Ethyl acetate (old process)	30.0	{ Slight				Do.
10869	{ Carbon tetrachloride	12.5	{ Nearly natural				Do.
10870	{ Ethyl acetate (new process)	25.0	{ Natural				Do.
10871	{ Carbon tetrachloride	12.5	{ Natural				Do.
10872	{ Ethyl acetate (old process)	30.0	{ do				Do.
10873	{ Carbon tetrachloride	60.0	{ do				Do.
11023	{ Untreated		{ do				Do.
	{ do		{ do				Milled soft; flour gray white.
11024	{ Ethyl acetate (new process)	15.0	{ Very faint; no odor				Milled soft; flour creamy white.
	{ Carbon tetrachloride	30.0	{ faint; no odor				Milled soft; flour creamy white.
11025	{ Ethyl acetate (old process)	15.0	{ odor noticeable while milling.				Milled soft; flour white.
	{ Carbon tetrachloride	30.0	{ do				Do.
11026	{ Ethyl acetate (commercial, 85 per cent)	15.0	{ do				Do.
11027	{ Carbon tetrachloride	30.0	{ Very noticeable				Do.
	{ Ethyl acetate (99 per cent)	15.0	{ do				Do.
	{ Carbon tetrachloride	30.0	{ do				Do.

TABLE 11.—Results of baking tests with flour from fumigated wheat

Sample No.	Fumigant	Concentration of fumigant	Absorption	Volume of loaf	Weight of loaf	Color of loaf	Texture of loaf	Crumb of loaf	Remarks
		<i>Lbs. per 1,000 cu. ft.</i>	<i>Per cent</i>	<i>Cc.</i>	<i>Grams</i>				
8687	Tetrachloroethane	4.60	52.1	2,080	483	52.0	85.5	Very gray	Strong odor of fumigant in mixing; garlic odor only in hot and cold loaf.
8688	Monochlorobenzene	5.85	52.1	2,200	484	78.0	92.0	Gray	No odor of fumigant in mixing; very slight odor in hot loaf but none in cold; flavor normal.
8697	Untreated	51.8	51.8	2,130	478	91.0	92.0	Slight cream	
8698	Tetrachloroethane	4.60	52.6	2,080	482	89.5	91.0	Creamy gray	
8699	do.	7.30	52.4	2,080	481	88.5	88.5	Dark cream	Strong odor in mixing; slight odor in hot loaf but none in cold; flavor of fumigant.
8700	do.	7.30	53.5	2,130	484	90.0	92.0	Creamy gray	Very strong odor of fumigant in mixing and in hot loaf but none in cold.
8701	Carbon tetrachloride	10.00	52.6	2,120	485	91.0	92.0	Slight cream	No odor of fumigant in mixing nor in hot or cold loaf; very slight flavor of fumigant.
8702	Monochlorobenzene	5.85	52.4	2,070	482	87.0	87.5	Dark cream	Strong odor of fumigant in mixing; very slight odor in both hot and cold loaf; slight flavor of fumigant.
8703	do.	2.90	53.2	2,080	492	90.5	90.0	Slight cream	Strong odor of fumigant in mixing and in hot loaf; slight odor in cold loaf; strong flavor.
8704	do.	2.90	53.2	2,120	492	92.0	93.0	do.	Very slight odor in mixing and in hot and cold loaf; very slight flavor.
10285	Untreated	13.40	54.1	1,930	483	82.5	88.0	Dark creamy gray	Odor of garlic in hot loaf.
10286	Ethylene bromide	54.7	54.7	1,930	495	83.3	83.0	do.	
10287	Untreated	10.50	54.4	1,940	487	86.3	91.5	Creamy gray	
10288	Ethylene bromide	5.00	55.3	2,070	485	85.0	91.3	do.	Odor of fumigant in hot loaf but not in cold.
10376	Carbon tetrachloride	15.10	55.3	2,030	486	82.0	88.0	Very dark gray	
10377	{Ethyl acetate (85 per cent)}	15.10	55.0	2,060	485	81.5	89.3	do.	
10378	{Carbon tetrachloride}	15.10	55.0	1,990	486	82.8	87.8	Dark creamy gray	Odor of fumigant in dough and slight odor in hot loaf.
10379	{Ethyl formate}	12.10	54.7	2,020	473	81.5	88.3	{Very dark creamy gray}	
10433	{Carbon tetrachloride}	12.10	57.6	1,920	498	82.0	89.3	Dark gray	
10434	Untreated	15.00	57.6	1,980	503	85.0	90.3	do.	Slight odor of fumigant in dough.
10435	Ethyl acetate (85 per cent)	11.80	58.8	2,030	499	87.5	91.3	Gray	
10436	Untreated	11.80	55.6	1,960	497	91.0	91.0	do.	
10455	Untreated	12.50	56.2	1,860	483	81.5	87.0	Slight creamy gray	Strong odor of garlic in hot loaf and slight odor in cold loaf.
10456	{Ethyl acetate (85 per cent)}	25.00	57.1	2,110	490	88.0	91.5	Creamy gray	{Slight odor of fumigant in dough; odor of garlic in hot loaf.
10457	Untreated								

10458	{ Ethyl acetate (85 per cent) }	{ 10.00 }	59.1	2,100	501	88.8	90.8	do.	Very slight odor of fumigant in hot loaf.
	{ Carbon tetrachloride }	{ 20.00 }							Putrid odor in dough and hot loaf.
10459	{ Untreated }		57.6	1,970	496	4.5	87.0	Black purple	Do.
10460	{ Ethyl acetate (85 per cent) }	{ 5.00 }	57.1	1,930	492	3.0	87.5	do.	
10536	{ Carbon tetrachloride }	{ 10.00 }	56.2	2,090	490	89.3	91.8	Gray	
10537	{ Untreated }		57.4	2,160	488	88.0	92.3	do.	
	{ Ethyl acetate (99 per cent) }	{ 12.50 }							
	{ Carbon tetrachloride }	{ 23.00 }							
10866	{ Ethyl acetate (new process) }	{ 15.00 }	62.1	2,000	510	82.8	86.8	{ Very dark creamy }	No odor of fumigant in hot or cold loaf; flavor normal.
	{ Carbon tetrachloride }	{ 30.00 }						{ gray. }	
10867	{ Ethyl acetate (old process) }	{ 15.00 }	63.2	1,990	515	87.5	88.5	Creamy gray	Do.
	{ Carbon tetrachloride }	{ 30.00 }							
10868	{ Ethyl acetate (new process) }	{ 12.50 }	62.9	2,000	513	88.3	88.0	do.	
	{ Carbon tetrachloride }	{ 25.00 }							
10869	{ Ethyl acetate (old process) }	{ 12.50 }	62.6	2,070	512	88.0	87.8	do.	
	{ Carbon tetrachloride }	{ 25.00 }							
10870	{ Untreated }		63.2	1,990	510	87.5	87.0	do.	
10871	{ Ethyl acetate (old process) }	{ 30.00 }	62.9	2,030	509	89.5	87.5	do.	
10872	{ Carbon tetrachloride }	{ 60.00 }	62.9	2,090	510	89.0	89.0	Dark creamy gray	Do.
10873	{ Untreated }		62.4	2,010	509	88.5	90.0	Creamy gray	Odor and flavor normal
11023	{ do }		61.5	1,880	512	80.0	86.3	Very dark cream	Do.
	{ Ethyl acetate (new process) }	{ 15.00 }	60.9	2,060	510	80.0	81.0	Dark creamy gray	{ No odor of fumigant in hot or cold loaf; flavor normal. }
11024	{ Carbon tetrachloride }	{ 30.00 }						do.	
11025	{ Ethyl acetate (old process) }	{ 15.00 }	61.2	1,960	514	81.5	85.8	do.	Distinct odor of fumigant in hot loaf.
	{ Carbon tetrachloride }	{ 30.00 }							
11026	{ Ethyl acetate (commercial) (85 per cent) }	{ 15.00 }	60.9	1,990	514	82.0	85.5	do.	
	{ Carbon tetrachloride }	{ 30.00 }							
11027	{ Ethyl acetate (99 per cent) }	{ 15.00 }	60.6	2,040	516	81.8	85.8	Gray	Slight odor of fumigant in hot loaf.
	{ Carbon tetrachloride }	{ 30.00 }							

EFFECT OF FUMIGATION ON MILLED AND BAKED PRODUCTS

The results of the milling and baking tests on wheat and rye treated with various fumigants are given in Tables 10, 11, 12, and 13.

TABLE 12.—Results of milling tests with rye exposed to fumigants

Sample No.	Fumigant	Concentration of fumigant	Odor of fumigant on grain
		<i>Lbs. per 1,000 cu. ft.</i>	
10073	Untreated.....		Natural.
10074	{Methyl formate.....	4.0	Do.
	{Ethyl bromide.....	11.2	
10075	{Ethylene bromide.....	8.0	Do.
	{Carbon tetrachloride.....	12.0	
10076	{Ethyl formate.....	8.8	Do.
	{Ethyl bromide.....	18.5	
10077	Untreated.....		Do.
10078	do.....		Do.
10079	{Ethylene bromide.....	8.8	None.
	{Carbon tetrachloride.....	12.0	
10080	Untreated.....		Natural.
10081	{Ethyl formate.....	6.4	Do.
	{Ethyl bromide.....	13.6	
	{Ethyl bromide.....	4.5	Do.
10082	{Carbon disulphide.....	2.2	
	{Carbon tetrachloride.....	10.0	

TABLE 13.—Results of baking tests with flour from fumigated rye¹

Sample No.	Fumigant	Concentration of fumigant	Absorption	Volume of loaf	Weight of loaf	Color of loaf	Texture of loaf
		<i>Lbs. per 1,000 cu. ft.</i>	<i>Per cent</i>	<i>Cc.</i>	<i>Grams</i>		
10073	Untreated.....		51.5	1,630	462	73.0	90.8
10074	{Methyl formate.....	4.00	51.8	1,720	485	74.0	90.3
	{Ethyl bromide.....	11.20					
10075	{Ethylene bromide.....	8.00	51.5	1,800	488	75.5	91.8
	{Carbon tetrachloride.....	12.00					
10076	{Ethyl formate.....	8.80	51.8	1,740	486	73.0	90.3
	{Ethyl bromide.....	18.50					
10077	Untreated.....		51.8	1,710	483	70.8	89.3
10078	do.....		51.8	1,730	483	71.5	89.5
10079	{Ethylene bromide.....	8.00	51.8	1,730	485	75.0	91.0
	{Carbon tetrachloride.....	12.00					
10080	Untreated.....		52.1	1,760	484	72.5	90.5
10081	{Ethyl formate.....	6.40	52.1	1,710	479	73.0	89.5
	{Ethyl bromide.....	13.60					
	{Ethylene bromide.....	4.50	51.8	1,730	485	70.8	87.5
10082	{Carbon disulphide.....	2.20					
	{Carbon tetrachloride.....	10.00					

¹In all the tests the crumb of the loaf was gray. No odor of the fumigant was detected in samples 10074, 10076, 10081, or 10082. A slight odor of the fumigant in the dough, but none in the loaf, either hot or cold, was detected in samples 10075 and 10079.

Tetrachloroethane and monochlorobenzene gave a disagreeable odor to the flour and bran and other by-products. This disagreeable odor in the flour was carried through to the finished loaf. Ethyl bromide, ethylene bromide, carbon tetrachloride, and ethyl formate left very little or no odor in the flour. If present in the flour, it was completely volatilized during baking, giving a loaf free from foreign odor. The commercial grades of ethyl acetate, both the 85 per cent and the 99 per cent, left a noticeable odor in the grain, bran, shorts, and flour; this odor appeared even in the hot loaf. The

"old process" and "new process" grades of ethyl acetate completely volatilized from the grain, so that no odor was noticeable in the process of milling, and the loaf, hot or cold, had only the natural odor and flavor (samples 11024 and 11025 in Tables 10 and 11).

The suitability of a sample of ethyl acetate for fumigating grain should be determined by the following test: Wet a sheet of filter paper (11-centimeter diameter is a convenient size) with the ethyl acetate, and allow it to evaporate, noting the odor from time to time. No foreign odor should be present, and the liquid should volatilize completely without leaving any odor. The carbon tetrachloride to be mixed with the ethyl acetate should be similarly tested, and should likewise be free from odoriferous constituents of low volatility. The presence of sulphur compounds in carbon tetrachloride is particularly objectionable because they give a garlicky odor to the fumigated grain. Ethyl acetate and carbon tetrachloride of a grade which satisfies this test are now commercially available in large quantities at a price but slightly higher than that asked for the commercial grades.

ADDITIONAL FUMIGATION TESTS WITH ETHYL ACETATE AND CARBON TETRACHLORIDE

In August and September, 1924, car fumigation tests were made with a mixture of ethyl acetate and carbon tetrachloride combined in a ratio that would make a noninflammable product at ordinary temperatures. The results are shown in Table 14.

TABLE 14.—*Results of fumigation tests on weevils in wheat in box cars, using a mixture of 40 volumes of ethyl acetate with 60 volumes of carbon tetrachloride*

Date	Mean temperature	Relative humidity (noon)	Concentration	Average mortality ¹	Insects killed at different levels after exposure for 24 hours										Insects in grain ²
					Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	Level 10	
	° F.	Per cent	Lbs. per 1,000 cu. ft.	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
1924															
Aug. 26	78	68	30	80	100	100	50	50	50	100	50	100	100	100	100
26	78	68	40	96	100	100	100	100	100	80	80	100	100	100	(³) 100
27	78	41	40	99	100	100	100	100	100	100	90	100	100	100	100
27	78	41	40	99	100	100	100	100	100	90	100	100	100	100	100
27	78	41	40	100	100	100	100	100	100	100	100	100	100	100	99
28	80	61	50	100	100	100	100	100	100	100	100	100	100	100	100
Sept. 3	66	53	40	100	100	100	100	100	100	100	100	100	100	100	100
3	66	53	40	100	100	100	100	100	100	100	100	100	100	100	100
4	66	45	40	86	100	100	100	100	100	80	90	90	80	20	100
6	60	41	40	98	100	100	100	100	100	100	100	80	100	100	(⁴) 100
6	60	41	40	97	90	100	90	100	100	100	100	100	100	90	(⁵) 100
2	78	51	40	-----	No weevils; sampled for milling and baking tests.										
2	78	51	40	-----	Do.										
2	78	51	40	-----	Do.										
2	78	51	40	-----	Do.										

¹ For insects exposed in pill boxes.

² The grain in all but six of the cars fumigated was moderately infested with weevils. The percentages given in this column represent the kill of this infestation.

³ A few alive in ends of car.

⁴ Fumigation interrupted when halfway through for 10 or 15 minutes, during which time the car door remained open.

⁵ No weevils.

TABLE 15.—Results of milling tests with wheat before and after fumigation in box cars with a mixture of 40 volumes of ethyl acetate and 60 volumes of carbon tetrachloride

Sample No.	Before or after fumigation	Concentration of fumigant	Odor of fumigant on—				Remarks
			Wheat	Bran	Shorts	Flour	
11043.....	Before	Lbs. per 1,000 cu. ft.	Natural.....	Natural.....	Natural.....	Natural.....	Milled granular; flour creamy white.
11044.....	After		Slight.....	Very slight.....	Very slight.....	(1).....	Do.
11049.....	Before	40	Natural.....	Natural.....	Natural.....	Natural.....	Do.
11050.....	After		Slight.....	Very slight.....	Very slight.....	(1).....	Do.
11041, 11045, 11047.....	Before	40	Natural.....	Natural.....	Natural.....	Natural.....	Milled granular; flour creamy.
11042, 11046, 11048.....	After		Slight.....	Very slight.....	Very slight.....	(1).....	Milled granular; flour creamy white.
11051.....	After second fumigation.	(2)	do.....	do.....	do.....	(1).....	Do.
11052, 11063.....	Before	40	Garlicky ³	Garlicky.....	Garlicky.....	Garlicky.....	Milled soft; flour creamy white.
11053, 11064.....	After		do ³	do.....	do.....	do.....	Milled granular; flour creamy white.
11062, 11066, 11068.....	Before	40	Garlicky ³	do.....	do.....	do.....	Milled soft; flour creamy white.
11065, 11067, 11069.....	After		do ³	do.....	do.....	do.....	Do.
11056.....	Before	40	Natural.....	Natural.....	Natural.....	Natural.....	Milled soft; flour white.
11057.....	After		do.....	do.....	do.....	do.....	Do.
11058.....	Before	40	do.....	do.....	do.....	do.....	Milled very soft; flour white.
11059.....	After		Slight.....	Very slight.....	Very slight.....	(1).....	Milled soft; flour creamy white.
11060.....	Before	40	Smut.....	Smut.....	Smut.....	Smut.....	Milled granular; flour gray.
11061.....	After		Slight.....	Very slight.....	Very slight.....	(1).....	Milled granular; flour grayish white.
11054.....	Before	40	Natural.....	Natural.....	Natural.....	Natural.....	Milled granular; flour creamy white.
11055.....	After		do.....	do.....	do.....	do.....	Milled soft; flour white.

¹ Odor of fumigant in flour so slight as to be detected only by comparison with normal flour. The odor left by this fumigant is almost indistinguishable from that present in slightly fermented grain and is not objectionable from the standpoint of the baker because no odor remains in the bread baked from this flour.

² This car of wheat was fumigated with the mixture at the rate of 40 pounds per 1,000 cubic feet; two days later the same wheat was again fumigated at the rate of 50 pounds per 1,000 cubic feet.

³ This wheat contained some seed of garlic, the odor of which was so pronounced that it masked all other odors.

TABLE 16.—Results of baking tests with flour from wheat before and after fumigation in box cars with a mixture of 40 volumes of ethyl acetate and 60 volumes of carbon tetrachloride

Sample No.	Before or after fumigation	Concentration of fumigant Lbs. per 1,000 cu. ft.	Absorption Per cent	Volume of loaf Cc.	Weight of loaf Grams	Color of loaf	Texture of loaf	Crumb	Remarks
11643	Before	53.8	56.8	1,620	484	84	85	Dark cream	Flavor normal; odor normal.
11644	After	54.1	56.8	1,810	486	84	87.3	do	Do.
11649	Before	40	56.5	2,080	497	85.3	88	Dark creamy gray	Do.
11650	After	40	56.5	2,120	496	85.3	88.8	do	Do.
11641	Before	57.4	57.1	1,950	492	84.8	87.5	do	Do.
11642	After	40	57.1	2,160	491	85	87.3	do	Do.
11645	Before	56.2	56.2	2,060	492	84.3	87	do	Do.
11646	After	40	56.8	2,130	493	84.3	87.3	do	Do.
11647	Before	56.5	56.5	2,140	497	85.3	88	do	Do.
11648	After	40	56.5	2,180	492	86.5	89	do	Do.
11651	After second fumigation.	(1)	57.1	2,080	504	85.5	88.8	do	Do.
11652	Before	51.5	51.5	2,140	480	82.5	90.3	Gray	Garlic in dough and cold bread.
11653	After	40	51.5	2,040	479	82.8	90.3	do	Do.
11663	Before	40	51.5	1,940	481	85.3	90.8	Dark creamy gray	Flavor normal; odor normal.
11664	After	40	51.5	1,990	480	85	90.5	Dark cream	Do.
11662	Before	40	51.5	2,010	482	83	88.5	do	Do.
11665	After	40	51.5	2,010	481	85.5	90.5	do	Do.
11660	Before	40	51.5	1,530	485	78.5	20	Very dark creamy gray	Do.
11667	After	40	51.5	1,490	483	75.8	20	do	Do.
11668	Before	40	51.5	2,010	486	85	90.5	Dark cream	Do.
11669	After	40	51.5	2,000	477	80	89.3	Very dark creamy gray	Do.
11656	Before	40	52.4	1,700	476	82	81	Dark cream	Do.
11657	After	40	52.9	1,850	488	85.5	88.5	Dark creamy gray	Do.
11658	Before	40	52.9	2,050	486	86	90.3	Creamy gray	Do.
11659	After	40	53.5	2,080	488	86	90.8	do	Do.
11660	Before	40	52.4	2,080	485	39	90.3	Very gray	Do.
11661	After	40	52.9	2,230	485	59	92	Gray	Do.
11654	Before	40	52.9	1,900	490	84.5	88	Creamy gray	Do.
11655	After	40	52.9	1,970	492	89.3	91.3	Light cream	Do.

¹ This car of wheat was fumigated with the mixture at the rate of 40 pounds per 1,000 cubic feet; 2 days later the same wheat was again fumigated at the rate of 50 pounds per 1,000 cubic feet.

With the exception of four cars fumigated for milling and baking tests, the rice weevil (*S. oryza*) was the test insect used in the pill boxes. Furthermore, the grain in all but two of the cars was moderately infested with weevils. The results given in the last column in Table 14 show the percentage kill of this natural infestation.

The results of the car fumigation reported in Table 14 confirm those reported in Table 8.

With the exception of one case, where fumigation was interrupted, all fumigations with a mixture of 40 per cent by volume of ethyl acetate and 60 per cent by volume of carbon tetrachloride, in which not less than 40 pounds of the mixture per 1,000 cubic feet of inclosed space was used, gave satisfactory kills.

The results of the milling tests with the wheat fumigated in these box-car experiments are shown in Table 15; the results of the baking tests with flour made from this wheat are shown in Table 16. The data indicate that both the flavor and odor of bread baked from flour made from wheat fumigated in box cars with a mixture of 40 volumes of ethyl acetate and 60 volumes of carbon tetrachloride, at the rate of 40 pounds per 1,000 cubic feet, are normal.

EFFECT OF ETHYL ACETATE-CARBON TETRACHLORIDE FUMIGATION ON GERMINATION OF SEEDS

The seeds of wheat, barley, rye, winter oats, and corn in tightly closed bell jars were exposed to the vapor of ethyl acetate and carbon tetrachloride at a concentration of 40 pounds per 1,000 cubic feet for 24 hours. The percentage germination before and after fumigation is shown in Table 17.

TABLE 17.—*Effect of ethyl acetate-carbon tetrachloride fumigation on germination of seeds*

Seed	Germination		Seed	Germination	
	Before fumigation	After fumigation		Before fumigation	After fumigation
	<i>Per cent</i>	<i>Per cent</i>		<i>Per cent</i>	<i>Per cent</i>
Wheat.....	83	85	Winter oats.....	95	93
Barley.....	97	98	Corn.....	97	94
Rye.....	89	90			

The results in Table 17 show that exposure at 31° C. in a tight container for 24 hours to the fumes of a 40-60 mixture (by volume) of ethyl acetate and carbon tetrachloride at the rate of 40 pounds per 1,000 cubic feet does not injure the germinating power of wheat, barley, rye, winter oats, and corn. The grain would not be subjected to as severe a test in ordinary box cars.

SUMMARY

The action of more than 100 organic compounds on weevils was tested under conditions permitting a control of the factors of concentration, time, and humidity, and with observations of the temperature.

The following 30 compounds were more toxic to the rice weevil (*S. oryza* L.) than carbon disulphide: Two out of 8 bromides tested;

3 out of 15 chlorides tested; the only iodide tested; 1 alcohol out of 8 alcohols and phenols tested; 3 out of 4 aldehydes tested; 2 out of 4 ketones tested; both of the chlorine-substituted ketones tested; 1 of the 2 chlorohydrins tested; 5 out of 9 esters tested; 4 out of 12 sulphur compounds tested; 1 nitrile out of 5 nitriles and isonitriles tested; both of the nitrites tested; 2 out of 8 amines tested; and pyridine.

The relative toxicity of the different classes of compounds can not be given because the low volatility of several of those tested gave only very low vapor concentrations. As a class, the hydrocarbons showed the lowest insecticidal efficiency, not one of the eight tested equaling carbon disulphide in fumigating power. The most effective fumigant in the glass-jar tests was epichlorohydrin, which killed the rice weevil at a concentration of 0.09 per cent, equivalent to 0.23 pound per 1,000 cubic feet. It was, however, an unsatisfactory fumigant in the presence of grain.

There is no constant relationship between the boiling points and the lethal concentrations of the compounds killing 100 per cent of the rice weevils after exposure for 24 hours.

A much greater concentration of fumigant is required to kill weevils in wheat than to kill those exposed directly to the vapors in glass jars. A still higher concentration is necessary to kill weevils in wheat in box cars. Ethyl formate and ethyl acetate were the only promising fumigants for grain in box cars. The acetate, however, costs only about one-third as much as the formate.

Odoriferous constituents of low volatility from commercial grades of ethyl acetate (both the 85 per cent and the 99 per cent grades) are carried through from the fumigated wheat to the flour, and even to the bread baked from it. A pure grade of ethyl acetate, however, leaves practically no odor in the fumigated grain or in the bran or shorts made from the grain, and none in the flour or in the bread baked from the flour.

The insecticidal efficiency of ethyl acetate under practical fumigating conditions is increased by the addition of carbon tetrachloride.

The most effective fumigant, other than carbon disulphide, against insects in wheat, in grain cars, under practical fumigating conditions, is a mixture of about 40 volumes of ethyl acetate and about 60 volumes of carbon tetrachloride. It is noninflammable at ordinary temperatures. The proper dosage of this mixture for fumigating box cars is about 45 pounds per 1,000 cubic feet. Both the ethyl acetate and the carbon tetrachloride must be tested to make sure that they are free from odoriferous constituents of low volatility before they are used in grain fumigating.

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