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EVALUATION OF FUMIGANTS FOR CONTROL OF INSECTS ATTACKING WHEAT
WALKDEN, H H SCHWITZGEBEL, R B 1 OF 1

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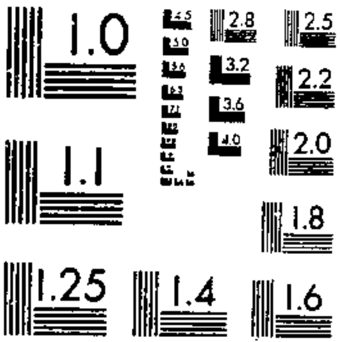


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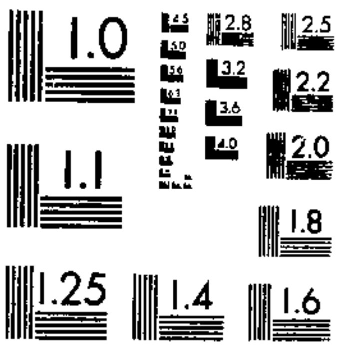


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# Evaluation of Fumigants for Control of Insects Attacking Wheat and Corn in Steel Bins[1]

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## INTRODUCTION

The protection of stored grains from insect attack has long been a serious problem. The ever-normal granary program, started some years ago, provided for the storage of a reserve stock of grain on the farm to help stabilize prices in years of surplus and to be a safeguard in years of scarcity. Soon after the Federal Government began to acquire large quantities of grain under the Agricultural Adjustment Act of 1938, an investigation was begun to determine the best methods of storing and caring for grain in farm-type bins.[3]

[1] Submitted for publication May 18, 1951.

[2] Resigned June 26, 1945.

[3] This investigation was conducted, under the auspices of the Commodity Credit Corporation, by the Agricultural Adjustment Administration, the Bureau of Plant Industry, Soils, and Agricultural Engineering, and the Bureau of Entomology and Plant Quarantine, in cooperation with the State agricultural experiment stations of Kansas, Iowa, and North Dakota.

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Because fumigation was considered the best means of combating insect infestation in stored grain, large-scale tests were conducted with various fumigants known or thought to be of value for this purpose. Owing to the scarcity of the common grain fumigants during World War II, many other materials were tested to discover effective substitutes. Approximately 600,000 bushels of wheat and 300,000 bushels of shelled corn were fumigated in steel bins to determine the minimum lethal dosage, the rate of penetration, the retention of vapors by the grain, and the effect on viability of seed and on the milling and baking qualities of the wheat. The toxicity of the fumigants to insects was established by laboratory tests. The performance of promising materials under practical storage conditions is reported in this bulletin.

## MATERIAL AND APPARATUS

The wheat was stored in calked steel bins (fig. 1) having capacities of 1,000, 2,740, and 5,000 bushels, at Hutchinson, Kans., and Jamestown, N. Dak. The corn was stored in uncalked steel bins holding 2,000 and 2,740 bushels, in Boone, Hamilton, Henry, Linn, and Story Counties, Iowa.

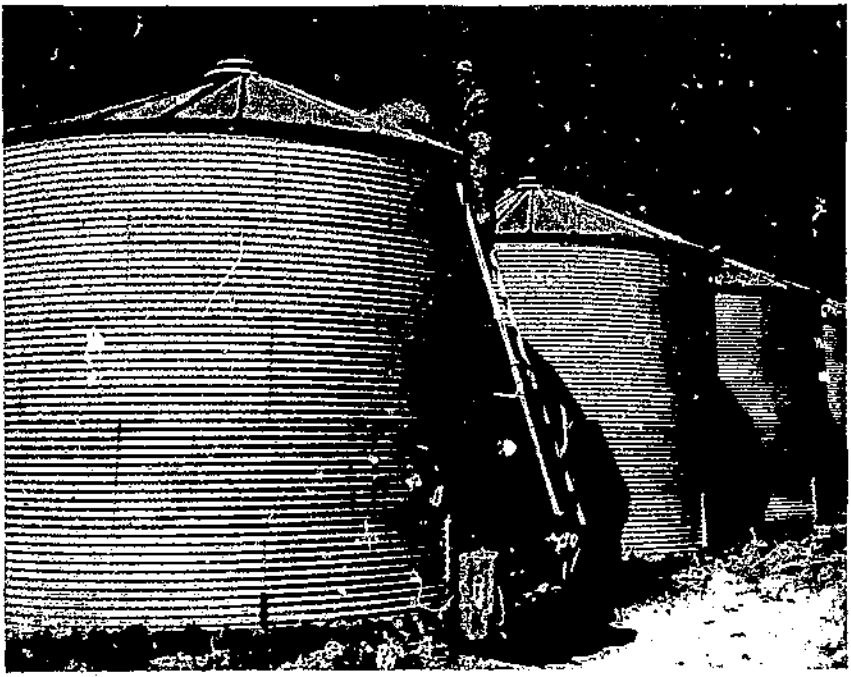

FIGURE 1.—Steel bins used for the fumigation tests.

### FUMIGANTS

Several factors must be considered in the selection of a fumigant for controlling insects in stored grain. Its vapors must be toxic to grain-infesting insects of all species and capable of penetrating the grain rapidly enough to produce a lethal concentration in all parts of the bin. It should be nonexplosive and nonflammable, and should have no deleterious effect on the viability of the grain or on its milling and baking qualities. It should leave no objectionable odor on the grain.

**CAUTION: Chemicals used in grain fumigation are poisonous to human beings and animals. In handling them, the operator should take all necessary precautions and follow all directions given on manufactured products. Care should be taken not to breathe the vapors or spill the fumigants on the skin or clothing. A suitable gas mask should always be worn by anyone who is exposed to the concentrated vapors of a fumigant.**

Nineteen liquid fumigants and various mixtures of them were tested (table 1).

TABLE 1.—*Physical characteristics of liquids used in experimental fumigation of stored grain*

| Fumigant | Boiling point | Specific gravity | Weight per gallon | Range of flammability in air |
|---|---|---|---|---|
| | *°C.* | | *Pounds* | *Percent by volume* |
| Acrylonitrile | 78.0 | 0.797 | 6.6 | 3 to 17. |
| Allyl bromide | 71.3 | 1.398 | 11.7 | No information. |
| Carbon disulfide | 46.3 | 1.261 | 10.5 | 1.06 to 50+. |
| Carbon tetrachloride | 76.8 | 1.595 | 13.3 | Nonflammable. |
| Chloropicrin | 112.4 | 1.692 | 13.8 | Do. |
| Dichloroethyl ether | 178.5 | 1.221 | 10.2 | Flammable above 131° F. |
| 1,1-Dichloro-1-nitroethane | 124.0 | 1.415 | 11.8 | 10. |
| Ethylene dibromide | 131.6 | 2.170 | 18.1 | Nonflammable. |
| Ethylene dichloride | 83.7 | 1.257 | 10.5 | 6.2 to 15.9. |
| Isopropyl formate | 71.3 | .883 | 7.4 | 2.4 to 8.6. |
| β-methylallyl chloride | 72.0 | .925 | 7.71 | [1]. |
| Methyl bromide | 4.6 | 1.732 | 14.4 | 13.5 to 14.5. |
| Methylene chloride | 40.1 | 1.336 | 11.1 | Nonflammable at laboratory temperatures. |
| Propylene dichloride | 96.8 | 1.166 | 9.7 | 3.4 to 14.5. |
| Tetrachloroethane | 130.5 | 1.588 | 13.2 | Nonflammable. |
| Tetrachloroethylene | 121.2 | 1.631 | 13.6 | Do. |
| Trichloroacetonitrile | 85.0 | 1.439 | 12.0 | Do. |
| Trichloroethane | 74.1 | 1.325 | 11.0 | Do. |
| Trichloroethylene | 88.0 | 1.477 | 12.2 | Nonflammable at laboratory temperatures. |

[1] The explosive range of air mixtures lies between 93 and 375 grams per cubic meter.

## TEST INSECTS

The test insects were adults of the red flour beetle (*Tribolium castaneum* (Hbst.)), the flat grain beetle (*Laemophloeus pusillus* (Schönh.)), the rice weevil (*Sitophilus oryza* (L.)), the lesser grain borer (*Rhyzopertha dominica* (F.)), the saw-toothed grain beetle (*Oryzaephilus surinamensis* (L.)), and the long-headed flour beetle (*Latheticus oryzae* (Waterh.)). Eggs of the confused flour beetle (*Tribolium confusum* Duv.) were included in some tests.

Test insects were reared in grain stored in 20-gallon garbage cans. The grain in the culture was screened, and only insects able to walk out of the screenings were used.

### Cages for Test Insects

Cages of two types were used in exposing the test insects to the fumigant (fig. 2): (1) One-ounce gelatin capsules with silk bolting-cloth ends (*A*), described by Farrar,[4] which proved unsatisfactory under Kansas conditions, because they became brittle and were broken easily in handling; (2) cylinders of 45-mesh brass-wire cloth (*B*)

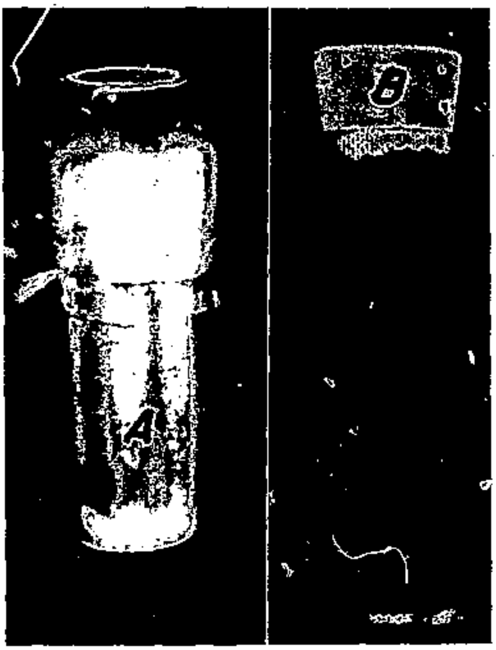


Figure 2.—Cages for exposing test insects to fumigants: *A*, Gelatin capsule with silk bolting-cloth ends; *B*, wire-cloth cylinder of 45-mesh brass wire.

closed at one end with wire cloth and at the other with a cork stopper. A cage of the second type was constructed by forming a rectangular piece of wire cloth, 2½ by 2⅜ inches, into a cylinder by soldering the longer sides together. A glass test tube was ideal for shaping the cage and for holding it while it was being soldered, as the solder did not adhere to the glass. A small square of wire cloth was then soldered to one end of the cylinder, and the protruding edges were trimmed.

From 30 to 70 insects were placed in each cage, with a little grain for food.

### Check Probes and Surface Cages

The caged insects were exposed to the fumigants by means of check probes (fig. 3) composed of dowels 1 inch in diameter fastened to-

[4] Farrar, M. D. small insect cage. Jour. Econ. Ent. 35: 76. 1942.

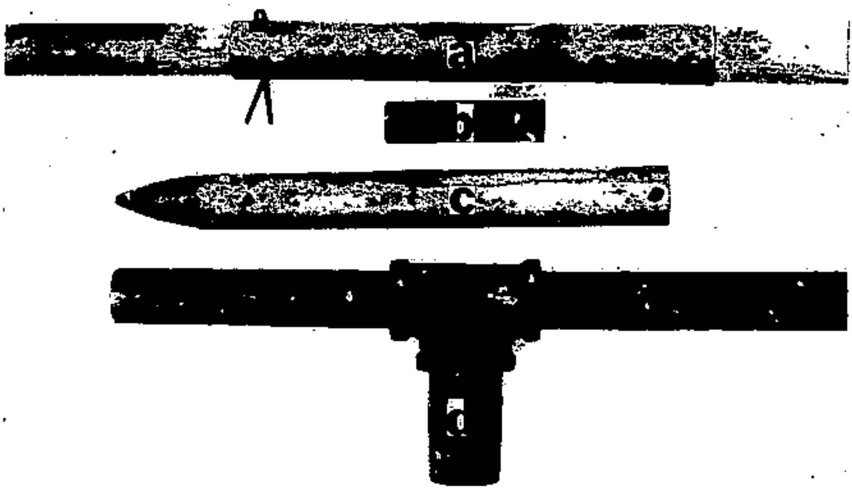

FIGURE 3.—Construction details of check probes: *a*, Perforated metal sleeve showing method of attachment to dowels; *b*, cage for exposing test insects; *c*, bottom sleeve fitted with wooden point; *d*, handle used for thrusting probes into the grain.

gether with sheet-metal sleeves (*a* and *c*) of the same diameter and 9 inches long, so that at each union a cell was formed in which a cage (*b*) could be placed. The sleeves were perforated to admit the fumigant. The bottom sleeve was fitted with a wooden point to facilitate insertion of the probe into the grain. The dowels were cut so as to make the distance between the cells about 3 feet when the sections were joined. In this manner the probes could be made up in any desired number of 3-foot lengths, and when thrust vertically to the bottom of the bins the test insects in the cells were exposed at 3-foot intervals (fig. 4).

A handle for pushing the check probes into the grain was constructed with iron-pipe nipples and a tee connection (fig. 3, *d*). This handle was fitted over the end of each probe as it was being inserted in the grain and removed when the probe was properly located.

The distribution of the fumigant within the grain mass and, to some extent, its concentration could be determined by noting the mortality of the test insects at the different levels and locations.

Three probes thrust vertically into the grain to the bottom of the bin gave a good indication of the efficiency of the fumigant. One probe was inserted in the center and the others 2 to 3 feet from the walls on opposite sides of the bin.

Because some fumigants caused comparatively low mortality of insects near the surface of the grain, five cages were inserted from 3 to 6 inches beneath the surface—one cage in the center and one in each of the four cardinal directions—about 3 feet from the walls of the bin. An 18-inch length of twine was attached to each cage, with the end remaining above the grain surface to insure easy recovery after fumigation.

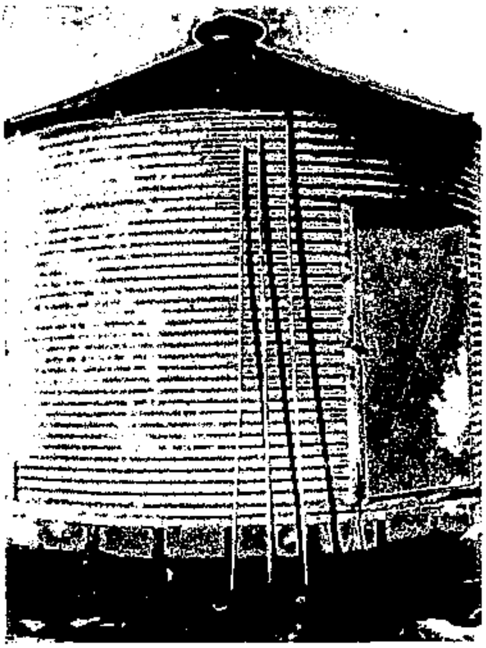

FIGURE 4.—Check probes ready to be placed in the grain.

As a check on each day's work, a probe containing several cages of insects was placed in an untreated bin of grain to determine the mortality in the absence of fumigation.

## APPARATUS FOR APPLYING LIQUID FUMIGANTS TO GRAIN

Fumigators of two types were designed for use in the fumigation tests. One was a combined air compressor and fumigant tank (fig. 5), consisting of an air-cooled ½-horsepower gasoline engine connected by a V-belt to a single-stage 1-cylinder air compressor, and an 18-gallon upright galvanized-steel tank with a glass gage on one side calibrated from 0 to 16 gallons. A gate-valve inlet was fitted to one side of the tank near the top and connected to the air compressor with copper tubing. A side connection for attaching air hose and chuck and a pressure gage were also provided in this line. The filling opening 1½ inches in diameter at the top center of the tank was fitted with a threaded bushing for a plug bearing a metal handle. A gate valve low on the side of the tank was provided with a pipe-to-hose connection for attaching the discharge hose. The air-compressor unit and the fumigant tank were mounted on a steel base with metal carrying handles.

The other fumigator was similar, except that an air-pressure tank was substituted for the air-compressor unit. This apparatus provides the required energy when mechanical or electrical energy is not available. The air-pressure tank can be filled at a gasoline station. The construction details of this fumigator have been described by Walkden and Barre.[5]

[5] WALKDEN, H. H., and BARRE, H. J. AN AIR-PRESSURE TANK FOR THE PNEUMATIC APPLICATION OF LIQUID FUMIGANTS. U. S. Bur. Ent. and Plant Quar. ET-210, 4 pp., illus. 1943. [Processed.]

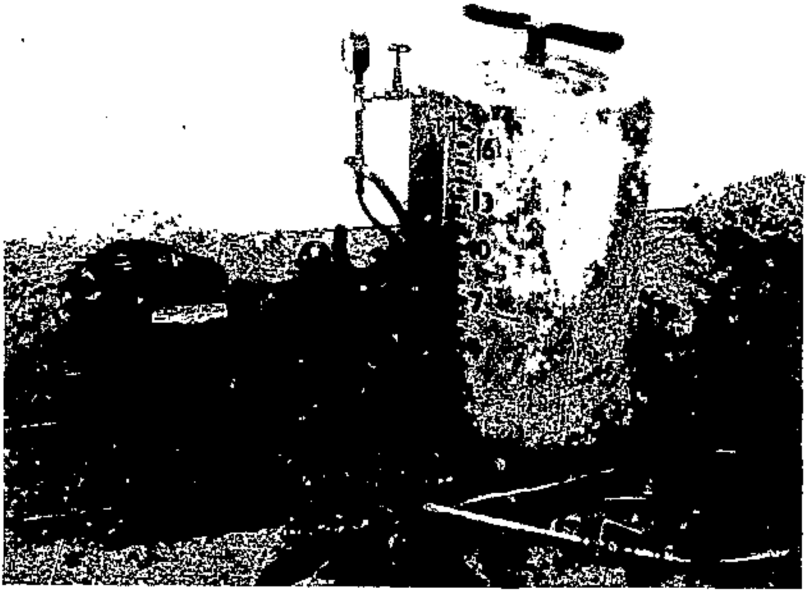

FIGURE 5.—Air compressor and fumigant tank used in applying liquid fumigants to the grain.

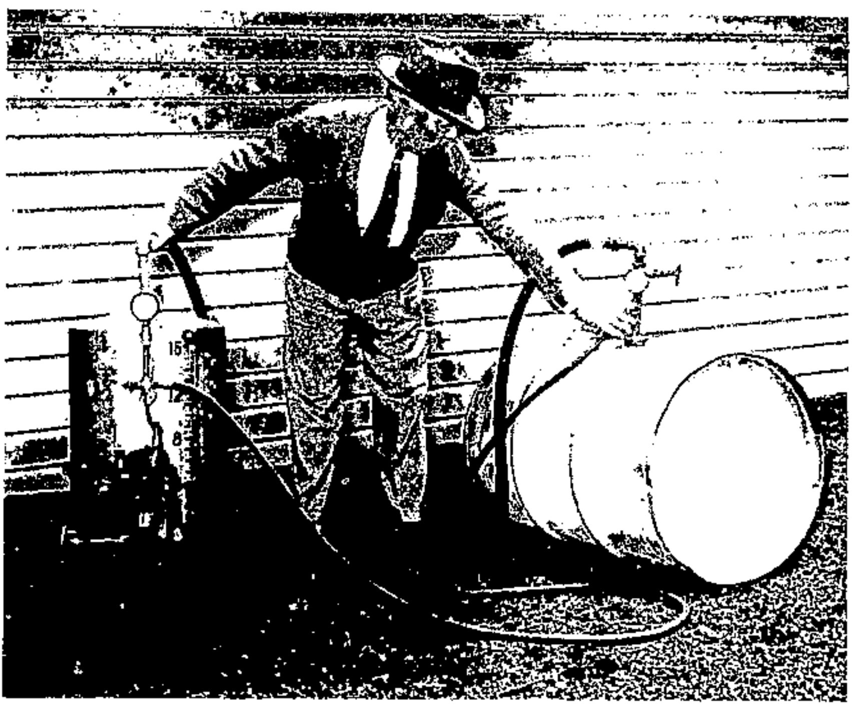

FIGURE 6. Device used in transferring liquid fumigant from 55-gallon drums to a fumigant tank by compressed air.

A device was constructed for transferring liquid fumigants from a 55-gallon drum into the fumigant tank by means of compressed air available on both types of fumigators (fig. 6). Its use protected the operators from fumes.

Because many of the common grain fumigants are solvents for rubber, natural rubber hose cannot be used to deliver the fumigant from the tank to the grain. Neoprene hose strong enough to withstand pressures up to 100 pounds per square inch was selected for the work.

The nozzles used to distribute the fumigant over the surface of the grain (fig. 7) were fashioned out of iron pipe and couplings by flatten-

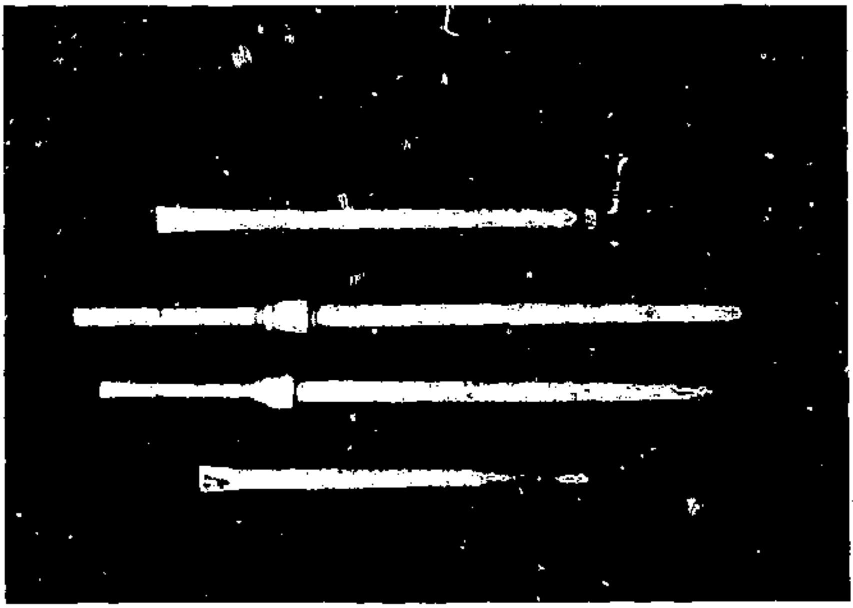

FIGURE 7.—Nozzles used in applying fumigants to the grain.

ing the ends of pipe of different diameters to form slots of different lengths and widths. The sizes of the nozzles were varied according to the capacity of the grain bins and the dosage of the fumigant.

## ASPIRATING APPARATUS

Large numbers of insects were handled in making up the test cages. Their caging was expedited by the use of a siphon-type aspirator, operated with compressed air. The aspirator consisted of a vial, a rubber stopper, a right-angle glass tube of 1/8-inch inside diameter, a T-shaped glass connection tube of the same diameter, and a glass jet (fig. 8). The arm of the tee holding the jet was connected with rubber tubing to the air supply. When compressed air was released through the jet, the air pressure in the vial was lowered, causing air to be drawn into it through the intake tube. The insects to be collected were sifted from infested grain into a shallow pan and drawn into the vial through this intake tube. A 55-gallon drum was employed as a reservoir for the compressed air, pressure being renewed when necessary. A drum of this type is not safe for a pressure of more than 10 pounds per square inch.

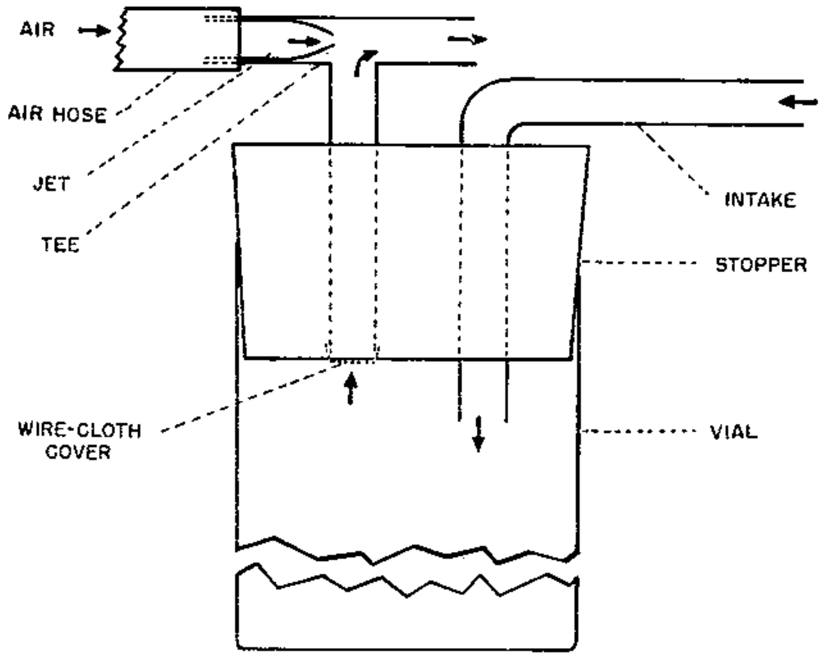


FIGURE 8.—Construction of siphon-type aspirator for caging test insects.

## METHODS

A fumigation procedure was followed which permitted observations on the rate of penetration and the retention of the different fumigants, as determined from the mortality of test insects. To determine the natural insect population, samples were taken with a grain probe from different levels in the center and in each quadrant of the bin. Although the effect of the fumigant on the natural population was found to be an excellent index of its efficiency, bins with sufficient natural insect populations were seldom available, and check probes containing caged insects had to be introduced in all of the experimental work.

To determine the effect of the fumigants on the viability of the grain, probe samples of grain were taken vertically from the top to the bottom of the bin about halfway between the wall and the center, before and at different intervals after each fumigation. Subsamples were cut from them with a Boerner grain divider for the germination tests.

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Care was taken to select grain of uniform quality when comparisons were being made between different dosages of fumigant. Grain containing more than 12 percent of moisture or over 3 percent of dockage, or that was caked or moldy or had recently been turned and cleaned, was found to be unsuitable.

In general, a 72-hour exposure was adequate for comparing the different fumigants. However, mixtures containing high percentages of ethylene dichloride required a longer time to effect the maximum kill, especially when applied at low dosages, and in such cases the exposure period was increased to 6 days.

At the end of the exposure period the probes were removed from the grain and identified by attaching a manila tag bearing the bin number and the location of the probe.

## FACTORS AFFECTING EFFICIENCY OF FUMIGANTS

Some factors affecting the efficiency of a fumigant are (1) the manner of application, (2) turning of the grain before fumigation, (3) the amount of dockage in wheat or of cracked kernels and foreign material in corn, (4) the moisture content of the grain, (5) the temperature of the grain, and (6) the tightness of the bins.

### Manner of Application

The manner of applying the fumigant to the grain affected the diffusion of the vapors in the grain mass. A mixture of methyl bromide, carbon tetrachloride, and ethylene dichloride was applied in four ways: (1) On the center surface; (2) on the center surface under reinforced kraft paper cover; (3) one-fourth of the dosage in the center of each quadrant; and (4) uniformly on the grain surface. A uniform distribution of the fumigant over the entire grain surface gave the best results (table 2).

About 1 gallon of fumigant per 100 square feet was the minimum that would give an even distribution of the material over the grain

TABLE 2.—*Effect of manner of application on efficiency of a 1:2.25:6.75 mixture of methyl bromide, carbon tetrachloride, and ethylene dichloride in killing insects in grain*

| Manner of application [1] | Dosage per 1,000 bushels | Mortality of insects |
|---|---|---|
| In corn (2 tests): | *Gallons* | *Percent* |
| On center surface under paper cover | 1 | 48 |
| Uniformly on grain surface | 1 | 99 |
| On center surface | 2 | 67 |
| Uniformly on grain surface | 2 | 100 |
| In wheat (1 test): | | |
| On center surface | 2 | 77 |
| One-fourth of dosage in center of each quadrant | 2 | 80 |
| Uniformly on grain surface | 2 | 100 |

[1] No cover except where indicated.

surface. It was delivered from the nozzle in a coarse, fan-shaped stream at a tank pressure of 40 to 60 pounds per square inch.

### Turning Grain Before Fumigation

The efficiency of carbon tetrachloride and the 3:1 mixture of ethylene dichloride and carbon tetrachloride was greatly increased in wheat turned immediately before being fumigated. The mortality of the insects ranged from 11 to 26 percent higher in the turned wheat than in the undisturbed wheat (table 3). Both lots contained less than 1 percent of dockage. The cost of turning, however, was much greater than the saving of fumigant.

Table 3.—*Effect of turning wheat immediately before treatment on efficiency of two fumigants*

| Fumigant | Dosage per 1,000 bushels | Bin capacity | Wheat turned | | Wheat undisturbed | |
|---|---|---|---|---|---|---|
| | | | Tests | Mortality of insects | Tests | Mortality of insects |
| | *Gallons* | *Bushels* | *Number* | *Percent* | *Number* | *Percent* |
| Carbon tetrachloride | 1 | 2,740 | 1 | 100 | 1 | 87 |
| Ethylene dichloride-carbon tetrachloride (3:1) | 2 | 1,000 | 1 | 95 | 3 | 84 |
| | 3 | 1,000 | 1 | 100 | 5 | 74 |
| | 2.5 | 2,740 | 2 | 98 | | ... |
| | 3 | 2,740 | | | 5 | 77 |

### Dockage in Wheat

Hard winter wheat seldom contains more than 1 percent of dockage, which does not affect the dosage of fumigant required. However, the efficiency of an ethylene dichloride-carbon tetrachloride mixture was considerably reduced in wheat containing 3 or more percent of dockage (table 4).

Table 4.—*Effect of dockage in wheat on efficiency of fumigation with 3:1 mixture of ethylene dichloride and carbon tetrachloride (bin capacity 2,740 bushels)*

| Dockage (percent) | Dosage per 1,000 bushels | Tests | Mortality of insects |
|---|---|---|---|
| | *Gallons* | *Number* | *Percent* |
| 0.2 to 0.9 | 4 | 16 | 96 |
| 4.0 to 5.3 | 4 | 1 | 36 |
| 3.2 to 5.5 | 6 | 1 | 78 |
| 3.4 to 5.7 | 8 | 1 | 83 |

## Cracked Kernels and Foreign Material in Corn

The amount of cracked kernels and foreign material—husks, cob chaff, rodent feces, and dirt—in shelled corn stored in steel bins affects the amount of fumigant required. When the bins are filled, this material is distributed unevenly throughout the grain mass, and the vapors of fumigants do not penetrate uniformly. The concentration is lowest in the portion of the grain containing the most of such materials, and here insect populations are usually heaviest. Therefore, it is important that shelled corn be put in storage as nearly free as possible from such material.

## Moisture Content of Grain

As the moisture content of the grain increases above 12 percent a proportionally higher dosage is required. No differences were noted in grain of low-moisture content. Bins containing surface grain having a moisture content of 15 to 20 percent could not be fumigated satisfactorily because the fumigant failed to penetrate the moist layer.

## Temperature of Grain

The mean temperature of the grain was calculated from thermocouple readings taken in 20 places in the grain mass before the fumigant was applied. The data for three fumigants (table 5) showed no marked differences in the insect mortality in grain temperatures ranging from 36° to 85° F.

Table 5.—*Effect of temperature of stored wheat on mortality of insects resulting from treatment with three fumigants*

| Mean temperature of wheat (°F.) | Mortality of insects from treatment with— | | |
|---|---|---|---|
| | Ethylene dichloride-carbon tetrachloride (3:1), 4 gallons | Carbon tetrachloride, 3 gallons | Carbon disulfide, 3 gallons |
| | *Percent* | *Percent* | *Percent* |
| 36-40 | | 93 | 93 |
| 41-45 | | | 92 |
| 46-50 | 93 | | 98 |
| 51-55 | | 100 | |
| 56-60 | 99 | 100 | 100 |
| 61-65 | 97 | 100 | |
| 66-70 | 94 | | |
| 71-75 | 96 | 91 | |
| 76-80 | 95 | 100 | |
| 81-85 | 98 | | 100 |

### Tightness of Bins

The tightness of the bins materially affects the length of time the vapors of the fumigants are retained in lethal concentrations in the grain. In uncalked steel bins the vapors could be detected seeping through the lower joints of the bin walls a few minutes after the fumigant was applied. In calked bins this did not occur.

## RATE OF PENETRATION OF FUMIGANTS

The penetration of the vapors in the grain mass was observed by putting 11 probes in the center of the bin and removing 1 probe every hour for the first 8 hours and then 24, 48, and 72 hours after fumigation. The numbers of living and dead test insects were recorded 24 hours after removal of each probe. The mortalities showed that the vapors reached the bottom of the 1,000-bushel calked bin within 1 to 2 hours after the fumigant had been applied (table 6). However, a maximum mortality was not reached until some time later.

Table 6.—*Penetration of two fumigants in wheat stored in 1,000-bushel calked steel bins (grain 8 feet deep)*

CARBON TETRACHLORIDE, 2 GALLONS

| Exposure period (hours) | Mortality of insects 24 hours after removal | | | |
|---|---|---|---|---|
| | 6 feet above floor | 3 feet above floor | At floor | Mean |
| | *Percent* | *Percent* | *Percent* | *Percent* |
| 1 | 52 | 20 | 24 | 31 |
| 2 | 100 | 46 | 40 | 64 |
| 3 | 100 | 100 | 74 | 92 |
| 4 | 100 | 96 | 93 | 96 |
| 5 | 100 | 100 | 97 | 99 |
| 6 | 100 | 100 | 100 | 100 |
| 7 | 100 | 100 | 100 | 100 |

ETHYLENE DICHLORIDE AND CARBON TETRACHLORIDE (3 : 1), 4 GALLONS

| Exposure period (hours) | 6 feet above floor | 3 feet above floor | At floor | Mean |
|---|---|---|---|---|
| 1 | 100 | 16 | 15 | 56 |
| 2 | 100 | 34 | 15 | 62 |
| 3 | 100 | 70 | 10 | 58 |
| 4 | 100 | 53 | 55 | 69 |
| 5 | 100 | 56 | 42 | 63 |
| 6 | 100 | 100 | 62 | 83 |
| 7 | 100 | 97 | 60 | 86 |
| 24 | 100 | 99 | 93 | 98 |
| 48 | 100 | 100 | 100 | 100 |

## RETENTION OF FUMIGANTS

As pointed out by Cotton and coworkers,[6] the fumigation of grain in steel bins is influenced by sorption. Because of the possible effect of the fumigants on the viability of the grain, the period that they were retained in the grain after fumigation was determined. At weekly intervals after fumigation from one to three check probes containing caged insects were placed in the grain and allowed to remain for 3 to 6 days. This procedure was continued until the mortality approximated that of insects in an unfumigated bin. The retention period of lethal concentrations in wheat stored in calked steel bins ranged from 2 to 32 weeks, depending upon the fumigant, the dosage, and the capacity of the bin (table 7). In shelled corn stored in uncalked steel bins the retention period was less than 72 hours.

TABLE 7.—*Retention of fumigants in lethal concentrations in stored wheat*

| Fumigant | Dosage per 1,000 bushels | Bin capacity | Retention period |
|---|---|---|---|
| | *Gallons* | *Bushels* | *Weeks* |
| Carbon tetrachloride | 2 | 1,000 | 2–3 |
| | 3 | 2,740 | 4–5 |
| β-methylallyl chloride-carbon tetrachloride (1:4) | 2 | 1,000 | 2–3 |
| Carbon disulfide-carbon tetrachloride (1:4) | 2 | 1,000 | 3–4 |
| | 2 | 2,740 | 3–4 |
| | 3 | 2,740 | 4–5 |
| Chloropicrin-carbon tetrachloride: | | | |
| 1:8.1 | 2 | 1,000 | 4–5 |
| 1:5.7 | 2 | 1,000 | 10–12 |
| 1,1-Dichloro-1-nitroethane-carbon tetrachloride: | | | |
| 1:8.1 | 2 | 1,000 | 4–5 |
| 1:5.7 | 1.5 | 1,000 | 5–6 |
| Ethylene dichloride-carbon tetrachloride (3:1) | 3 | 1,000 | 4–5 |
| | 4 | 1,000 | 6–8 |
| | 4 | 2,740 | 12–16 |
| | 6 | 2,740 | 24–28 |
| | 3 | 5,000 | 24–32 |

## RESULTS OF FUMIGATION TESTS

### IN WHEAT

The results of the fumigation tests in stored wheat are given in table 8.

Carbon tetrachloride alone and the 4:1 mixture of it with carbon disulfide were satisfactory fumigants at 2 gallons per 1,000 bushels.

[6] COTTON, R. T., WALKDEN, H. H., and SCHWITZGEBEL, R. B. THE ROLE OF SORPTION IN THE FUMIGATION OF STORED GRAIN AND MILLED CEREAL PRODUCTS. Kans. Ent. Soc. Jour. 17: 98–103. 1944.

With carbon tetrachloride there is no fire or explosion hazard.

The 3:1 mixture of ethylene dichloride and carbon tetrachloride gave consistently good results at 4 gallons per 1,000 bushels. The vapors were retained in the grain longer than were those of the other fumigants tested, giving adequate protection from reinfestation for several months.

The 1:4 mixture of β-methylallyl chloride and carbon tetrachloride in a limited number of tests was adequate at 1.5 gallons per 1,000 bushels.

The 1:19 mixture of ethylene dibromide and carbon tetrachloride was more toxic than carbon tetrachloride alone, particularly in the surface layer of grain.

The 1:1:18 mixture of acrylonitrile, trichloroacetonitrile, and carbon tetrachloride was more toxic than carbon tetrachloride alone.

Trichloroethylene was slightly less toxic than carbon tetrachloride at 3 gallons per 1,000 bushels.

Although carbon disulfide is extremely toxic to stored-grain insects, the hazards from explosion and fire are too great to warrant its use alone.

Dichloroethyl ether, trichloroethane, tetrachloroethane, and allyl bromide imparted a persistent objectionable odor to the grain. The odor of dichloroethyl ether was carried over into the bread made from wheat fumigated with it.[7]

In tests with tetrachloroethylene and methylene chloride control was not satisfactory at the dosages used.

Ethylene dichloride was more toxic than propylene dichloride. Neither of these materials can be used alone, however, because of their flammability. The fire hazard can be sufficiently reduced by using 1 part of carbon tetrachloride to 3 parts of either of these fumigants.

## In Shelled Corn

The results of fumigation tests made in shelled corn are shown in table 9.

In the tests with carbon tetrachloride and the 3:1 mixture of ethylene dichloride and carbon tetrachloride, 4 gallons per 1,000 bushels were required to give satisfactory results.

The following mixtures gave good results when used at 2 gallons per 1,000 bushels of stored shelled corn:

- Carbon tetrachloride-methyl bromide—
  - Alone (9:1)
  - Plus carbon disulfide (9:1:2.5)
    - Ethylene dichloride (2.25:1:6.75)
    - Propylene dichloride (2.25:1:6.75)
- Carbon tetrachloride-β-methylallyl chloride (5.25:1)
- Carbon tetrachloride-acrylonitrile (7:1 and 4.3:1)
- Carbon tetrachloride-trichloroacetonitrile—
  - Alone (19:1, 15.6:1, and 9:1)
  - Plus acrylonitrile (27:1:5.3 and 18:1:1)
- Methylene chloride-methyl bromide (9:1)

[7] Milling and baking tests were made through the cooperation of the Department of Milling Industry, Kansas State College.

TABLE 8.—*Mortality of insects in stored wheat treated with various fumigants*

| Fumigant | Dosage per 1,000 bushels | Tests | Mortality of insects |
|---|---|---|---|
| | *Gallons* | *Number* | *Percent* |
| Carbon disulfide | 1 | 2 | 91 |
| | 2 | 2 | 98 |
| | 3 | 2 | 100 |
| Carbon tetrachloride | 1 | 1 | 37 |
| | 1.5 | 3 | 98 |
| | 2 | 16 | 99 |
| | 3 | 11 | 99 |
| | 4 | 1 | 100 |
| | 5 | 1 | 100 |
| Dichloroethyl ether | 3 | 1 | 90 |
| Ethylene dichloride | 4 | 4 | 94 |
| Methylene chloride | 2 | 4 | 69 |
| | 3 | 5 | 87 |
| Propylene dichloride | 4 | 4 | 75 |
| Tetrachloroethane | 3 | 5 | 66 |
| Tetrachloroethylene | 3 | 5 | 74 |
| Trichloroethane | 3 | 3 | 77 |
| Trichloroethylene | 2 | 4 | 91 |
| | 3 | 5 | 97 |
| Acrylonitrile-carbon tetrachloride: | | | |
| 1:15.6 | 2 | 1 | 100 |
| 1:7 | 2 | 6 | 94 |
| 1:4.3 | 2 | 1 | 100 |
| Acrylonitrile-trichloroacetonitrile-carbon tetrachloride: | | | |
| 5.3:1:27 | 2 | 1 | 100 |
| | 1 | 5 | 94 |
| 1:1:18 | 1.5 | 3 | 100 |
| | 2 | 6 | 100 |
| Allyl bromide-carbon tetrachloride: | | | |
| 1:27.6 | 2 | 1 | 98 |
| 1:17.9 | 2 | 1 | 92 |
| 1:13 | 2 | 1 | 98 |
| 1:11 | 2 | 1 | 100 |
| β-methylallyl chloride-carbon tetrachloride: | | | |
| 1:5 | 1.5 | 3 | 88 |
| | 2 | 4 | 100 |
| 1:4 | 1.5 | 4 | 100 |
| 1:3 | 1.5 | 5 | 100 |
| Carbon disulfide-carbon tetrachloride: | | | |
| 1:1 | 2 | 1 | 100 |
| | 4 | 1 | 100 |
| 1:2 | 3 | 1 | 99 |
| 1:3 | 4 | 1 | 100 |
| 1:4 | 1.5 | 2 | 100 |
| | 2 | 11 | 99 |
| | 3 | 7 | 99 |
| | 3.5 | 1 | 100 |
| | 4 | 3 | 100 |
| | 5 | 1 | 99 |
| | 6 | 1 | 100 |
| Methyl bromide-carbon tetrachloride (1:9) | 1.5 | 2 | 100 |
| | 2 | 4 | 91 |

TABLE 8.—*Mortality of insects in stored wheat treated with various fumigants*—Continued

| Fumigant | Dosage per 1,000 bushels | Tests | Mortality of insects |
|---|---|---|---|
| | *Gallons* | *Number* | *Percent* |
| Chloropicrin-carbon tetrachloride: | | | |
| 1:12.3 | 2 | 1 | 87 |
| | 3 | 1 | 100 |
| 1:8.1 | 2 | 3 | 97 |
| 1:5.7 | 1 | 3 | 74 |
| | 1.25 | 1 | 89 |
| | 1.5 | 3 | 100 |
| | 2 | 3 | 88 |
| 1:3.3 | 1 | 2 | 100 |
| Chloropicrin-ethylene dichloride: | | | |
| 1:5.7 | 1 | 2 | 70 |
| 1:4 | 1 | 2 | 88 |
| 1,1-Dichloro-1-nitroethane-carbon tetrachloride: | | | |
| 1:12.3 | 2 | 4 | 92 |
| 1:8.1 | 2 | 4 | 95 |
| 1:5.7 | 1 | 1 | 100 |
| | 1.5 | 5 | 99 |
| 1:4.3 | 1 | 2 | 98 |
| 1:3.3 | 1 | 2 | 98 |
| Ethylene dibromide-carbon tetrachloride: | | | |
| 1:19 | 2 | 2 | 100 |
| 1:9 | 2 | 4 | 100 |
| Ethylene dibromide-methyl bromide-carbon tetrachloride (1:1:18) | 1 | 2 | 96 |
| Ethylene dichloride-carbon tetrachloride (3:1) | 1 | 1 | 35 |
| | 2 | 3 | 84 |
| | 3 | 11 | 76 |
| | 4 | 32 | 96 |
| | 5 | 13 | 96 |
| | 6 | 6 | 98 |
| Ethylene dichloride-carbon tetrachloride-ethylene dibromide (14.2:4.8:1) | 2 | 2 | 95 |
| Ethylene dichloride-carbon tetrachloride-methyl bromide (6.8:2.3:1) | 1.5 | 7 | 90 |
| | 2 | 15 | 95 |
| | 2.5 | 1 | 100 |
| Ethylene dichloride-methyl bromide (9:1) | 2 | 2 | 89 |
| | 2.5 | 1 | 57 |
| | 3 | 1 | 78 |
| Methylene chloride-methyl bromide (9:1) | 1.5 | 4 | 96 |
| | 2 | 4 | 89 |
| Propylene dichloride-carbon tetrachloride (3:1) | 4 | 4 | 87 |
| | 5 | 4 | 99 |
| | 6 | 1 | 100 |
| Propylene dichloride-carbon tetrachloride-methyl bromide (6.8:2.3:1) | 1 | 1 | 70 |
| | 1.5 | 5 | 70 |
| | 2 | 6 | 94 |
| Propylene dichloride-methyl bromide (9:1) | 1.5 | 1 | 77 |
| | 2 | 5 | 87 |
| | 2.5 | 2 | 79 |
| Trichloracetonitrile-carbon tetrachloride: | | | |
| 1:19 | 2 | 1 | 75 |
| 1:15.6 | 2 | 1 | 94 |
| 1:9 | 2 | 1 | 100 |
| 1:7 | 2 | 1 | 100 |

TABLE 9.—*Mortality of insects in stored shelled corn treated with various fumigants*

| Fumigant | Dosage per 1,000 bushels | Tests | Mortality of insects |
|---|---|---|---|
| | *Gallons* | *Number* | *Percent* |
| Carbon tetrachloride | 1 | 4 | 56 |
| | 2 | 4 | 75 |
| | 4 | 2 | 94 |
| Acrylonitrile-carbon tetrachloride: | | | |
| 1:7 | 2 | 1 | 94 |
| 1:4.3 | 2 | 2 | 94 |
| Acrylonitrile-trichloroacetonitrile-carbon tetrachloride: | | | |
| 5.3:1:27 | 2 | 1 | 100 |
| 1:1:18 | 2 | 1 | 100 |
| β-methylallyl chloride-carbon tetrachloride: | | | |
| 1:9 | 2 | 4 | 88 |
| 1:7 | 2 | 4 | 93 |
| 1:5.25 | 2 | 3 | 94 |
| Carbon tetrachloride-methyl bromide (9:1) | 1 | 3 | 96 |
| | 2 | 3 | 96 |
| Carbon tetrachloride-carbon disulfide-methyl bromide: | | | |
| 12.3:3.3:1 | 2 | 4 | 92 |
| 9:2.5:1 | 2 | 4 | 98 |
| Choropicrin-carbon tetrachloride: | | | |
| 1:25.3 | 2 | 3 | 100 |
| 1:12.3 | 1 | 3 | 100 |
| 1,1-Dichloro-1-nitroethane-carbon tetrachloride: | | | |
| 1:25.3 | 2 | 3 | 99 |
| 1:12.3 | 1 | 3 | 96 |
| Ethylene dichloride-carbon tetrachloride (3:1) | 4 | 10 | 94 |
| Ethylene dichloride-carbon tetrachloride-methyl bromide (6.75:2.25:1) | 1 | 5 | 99 |
| | 2 | 5 | 100 |
| Ethylene dichloride-methyl bromide (9:1) | 0.64 | 1 | 53 |
| | 1 | 2 | 63 |
| | 2 | 3 | 89 |
| Ethylene dichloride-carbon tetrachloride-dichloroethyl ether-methyl bromide (7:2.5:2:1) | 2.4 | 1 | 90 |
| Methylene chloride-carbon disulfide (9:1) | 2 | 4 | 44 |
| Methylene chloride-isopropyl formate (9:1) | 2 | 2 | 56 |
| Methylene chloride-methyl bromide (9:1) | 1 | 3 | 89 |
| | 2 | 3 | 99 |
| Propylene dichloride-carbon tetrachloride (3:1) | 4 | 2 | 87 |
| Propylene dichloride-carbon tetrachloride-methyl bromide (6.75:2.25:1) | 1 | 3 | 78 |
| | 2 | 3 | 92 |
| Propylene dichloride-methyl bromide (9:1) | 1 | 3 | 94 |
| | 2 | 3 | 99 |
| Trichloroacetonitrile-carbon tetrachloride: | | | |
| 1:19 | 2 | 1 | 94 |
| 1:15.6 | 2 | 1 | 95 |
| 1:9 | 2 | 1 | 100 |

More information on β-methylallyl chloride is given by Richardson and Walkden.[5]

## RESULTS OF GERMINATION TESTS

The 3:1 ethylene dichloride-carbon tetrachloride mixture containing 10 percent of methyl bromide at dosages sufficient to kill the test insects (2 gallons per 1,000 bushels) reduced the viability of wheat stored at Jamestown, N. Dak. (table 10). Similar effects on viability were noted in wheat stored at Hutchinson, Kans. Germination injury was also observed with mixtures of chloropicrin or 1,1-dichloro-1-nitroethane with carbon tetrachloride. Moreover, their lachrymatory effect made them disagreeable for use under conditions imposed by steel-bin storage. When the effect on viability is not a factor in grain fumigation, such mixtures may be used. None of the other compounds tested in this investigation adversely affected the viability of wheat. In some instances fumigation with the 3:1 mixture of ethylene dichloride and carbon tetrachloride stimulated germination.

TABLE 10.—*Effect of fumigation with a 3:1 mixture of ethylene dichloride and carbon tetrachloride containing 10 percent of methyl bromide on the viability of stored wheat, Jamestown, N. Dak. (grain fumigated November 1941 and August 1942; dosage 2 gallons per 1,000 bushels)*

| Date of test | Viability of stored wheat | | | |
|---|---|---|---|---|
| | In 1,000-bushel bins (2 tests) | | In 2,740-bushel bins (7 tests) | |
| | Fumigated | Not fumigated | Fumigated | Not fumigated |
| | *Percent* | *Percent* | *Percent* | *Percent* |
| 1941: | | | | |
| August | 92 | 90 | 91 | 93 |
| 1942: | | | | |
| February | 93 | 87 | 82 | 90 |
| August | 87 | | 63 | 85 |
| November | 54 | 82 | 42 | 88 |
| 1943: | | | | |
| February | 57 | 88 | 40 | 89 |
| May | 45 | 88 | 29 | 87 |
| August | 39 | 88 | 22 | 84 |
| Total change | −53 | −2 | −69 | −9 |

A series of 22 bins of shelled corn were fumigated twice 1 month apart with the 3:1 mixture of ethylene dichloride and carbon tetrachloride containing 10 percent of methyl bromide. Germination tests made before the second fumigation and 1 and 6 months thereafter indicated no adverse effect on the viability of the corn.

[5] RICHARDSON, C. H., and WALKDEN, H. H. β-METHYLALLYL CHLORIDE AS A FUMIGANT FOR INSECTS INFESTING STORED CORN. Jour. Econ. Ent. 38: 471–477. 1945.

## SUMMARY

Large-scale tests with 19 fumigants and various mixtures of them were made to determine their efficiency for the control of insects attacking wheat and corn stored in farm-type steel bins. The test insects were adults of the red flour beetle (*Tribolium castaneum* (Hbst.)), the flat grain beetle (*Laemophloeus pusillus* (Schönh.)), the rice weevil (*Sitophilus oryza* (L.)), the lesser grain borer (*Rhyzopertha dominica* (F.)), the saw-toothed grain beetle (*Oryzaephilus surinamensis* (L.)), and the long-headed flour beetle (*Latheticus oryzae* (Waterh.)). Eggs of the confused flour beetle (*Tribolium confusum* Duv.) were included in some tests.

Best results were obtained when the fumigant was applied as a coarse spray uniformly over the surface of the grain, rather than at one to four restricted locations on the surface. Wheat treated immediately after being turned required less fumigant than undisturbed grain, but the cost of turning was much greater than the saving in fumigant. Wheat containing from 3 to 6 percent of dockage required more than twice as much fumigant as wheat with less than 1 percent. No differences in dosage requirements were noted in grain of less than 12-percent moisture content, but above this percentage proportionately higher dosages were required. Bins containing surface grain having a moisture content of 15 to 20 percent could not be fumigated satisfactorily because the fumigant failed to penetrate the moist layer.

The temperature of the grain had no observable effect on the efficiency of fumigation within the range of temperatures observed.

In penetration tests the lethal concentrations of the vapors of carbon tetrachloride and the 3:1 mixture of ethylene dichloride and carbon tetrachloride reached the bottom of bins with 8 feet of grain within 1 to 2 hours after application to the grain surface.

The vapors of the fumigants were retained in the wheat in lethal concentrations for 2 to 32 weeks, depending on the fumigant, the dosage, and the capacity of the bin. The vapors of the 3:1 mixture of ethylene dichloride and carbon tetrachloride were retained longest in 5,000-bushel bins at a dosage of 3 gallons per 1,000 bushels.

Although most of the fumigants tested were effective, some had properties that made them undesirable for fumigation of wheat stored in steel bins. When used at dosages sufficient to control the infestation, mixtures containing methyl bromide, chloropicrin, or 1,1-dichloro-1-nitroethane reduced the viability of the wheat. Dichloroethyl ether, trichloroethane, tetrachloroethane, and allyl bromide imparted an objectionable odor to the wheat and the odor of dichloroethyl ether was carried over into the flour and bread. Where the odor or the effect on viability is not a factor in grain fumigation, such materials can be used.

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