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Exothermic Inert-Atmosphere Generators for Control of Insects in Stored Wheat^{1,2}

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ABSTRACT

Use of exothermic inert-atmosphere generators for displacing the normal atmosphere in stored wheat was demonstrated. The time required to displace normal atmosphere with an oxygen-deficient atmosphere was dependent upon the rate of flow of gases released into the tank and the degree of pretreatment purging achieved prior to loading the tank with grain. Atmospheric concentrations containing

less than 1% O₂ for 24 hours killed adult confused flour beetles, *Tribolium confusum* Jacquelin duVal, but were not effective against immature stages of the rice weevil, *Sitophilus oryzae* (L.), for time periods of 72 to 96 hours. No significant differences between treated and untreated wheat were found in tests to determine the effect of the inert atmosphere on grain quality.

Research to develop use of inert atmosphere as a possible substitute for conventional chemical fumigation has gained emphasis because of the growing concern about pesticide residues. Various combinations of atmospheric gases have been tested, all involving restriction of O₂ available to the insect. Several methods have been used to manipulate the O₂ concentrations. Proxate B[®], a mixture of methyl formate and CO₂, was applied to 6000-bu bins of wheat in 1935 by researchers in the Cereal and Forage Insect Investigations of the Bureau of Entomology and Plant Quarantine, USDA (unpublished data). The gas was released from pressurized cylinders and passed through a copper coil immersed in hot water. The vaporized gas was released into the grain mass through a pipe which was pushed below the surface. Bailey (1955, 1956, 1957, 1965) conducted tests with a gas-mixing apparatus designed to produce various mixtures of N₂, O₂, and CO₂ which were passed over test insects in a continuous flow. Harein and Press (1968) used premixed binary and ternary mixtures of N₂, O₂, and CO₂ released from pressurized cylinders and passed through glass jars containing cages of test insects. Similar tests were conducted by Lindgren and Vincent (1970) with static conditions (no air movement). Nitrogen containing 0.5 and 1% O₂ was used to purge 1-lb containers of sorghum in tests by Person and Sorenson (1970). A unique system was developed by Jay et al. (1970) for applying a large volume of CO₂ in peanut silos. In this system a mobile liquid CO₂ storage tank with a capacity of about 9000 lb was equipped with electrically operated vaporizers for rapid conversion of the liquid CO₂ to a gas. The CO₂ was released into the overhead space above the peanuts and recirculated through the silo. Results obtained in these studies were inconclusive; however, some general observations can be made. Most stored-grain insects are affected by atmospheres containing less than 3% O₂ or more than 40% CO₂. The lethal effect of atmospheres producing mortality is increased by increased treatment temperatures or extended exposure periods. Atmospheres of N₂ require much lower O₂ concentrations to be effective than are required in atmospheres of CO₂. Immature stages of stored-product insects are more tolerant of O₂-deficient atmospheres than are the adult stages. The composition of inert atmospheres used by the feed industry for the preservation of vitamin content, color,

weight, and palatability of alfalfa pellets (Kruger 1960) appears to be capable of restricting the amount of O₂ available to stored-grain insects. This atmosphere is nearly devoid of O₂ and is produced by gas-fired exothermic inert-atmosphere generators. The capacities of these generators usually range from 50 ft³/hr to 100,000 ft³/hr of inert gas. This paper describes tests conducted with atmospheres produced by this type of generator.

PROCEDURE.—Each test was conducted in an 18-ft-diam, 130-ft-high concrete elevator tank containing about 20,000 bu of wheat. The inert atmosphere was produced by 2 exothermic inert-atmosphere generators⁴ (Fig. 1), each capable of generating 15,000 ft³ of inert atmosphere per hour. Composition of the inert atmosphere was <0.1% O₂, 8.5–11.5% CO₂, and the balance was principally N₂. The generators were situated in a small building adjacent to the main elevator complex and interconnected with a large supply duct placed beneath the storage tanks. For Test 1, feeder pipes, 1½ in. diam, were attached to the supply duct and extended about 2 ft through the floor to each tank. The ends of the pipe were screened to prevent grain from entering the system. A ¾-in.-diam bypass pipe was attached to each feeder line to permit application of the gas at a reduced rate by restricting the volume released into the tank. Air samples for gas chromatographic analysis were obtained from within the grain mass from 2 tiers of ¼-in. plastic sampling lines. One tier was situated next to the inner wall of the tank and the other near the center. Each tier of lines included sample points and small cloth bags containing test insect cages spaced at 20-ft intervals at depths of 10, 30, 50, 70, 90, 110, and 130 ft (bottom) in the grain. An additional sampling point was situated in the 3 ft of overhead space above the grain surface and just beneath a hinged outlet for release of the air from the top of the tank (Fig. 2).

Air samples in Test 1 were collected at 4-hr intervals during the 24-hr period following release of the inert atmosphere through the 1½-in.-diam pipe. Additional samples were taken after 48 hr and just prior to completion of the 72-hr exposure period. The air samples were trapped in 125-ml glass collection tubes and returned to the Manhattan laboratory for analysis.⁵ Test 2 utilized the inert atmosphere released through the ¾-in. diam bypass pipe during an overnight period in an attempt to purge a tank of normal atmosphere prior to filling it

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² Mention of a proprietary product does not imply endorsement by the USDA.

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⁴ Model XH 1500, manufactured by Gas Atmospheres Inc., a division of Alco Standard Corp., Cleveland, Ohio.

⁵ Loren I. Davidson, of the Manhattan laboratory, conducted the gas chromatographic analyses.

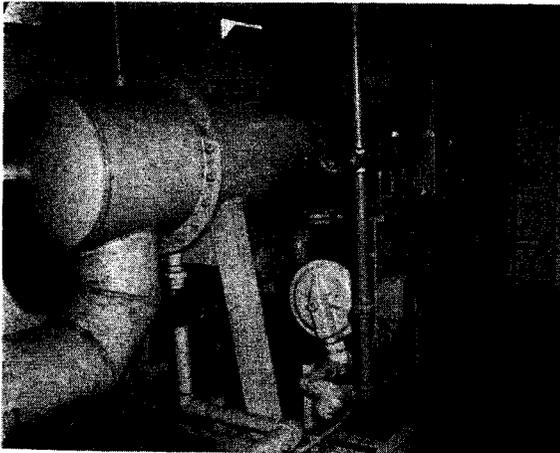


FIG. 1.—Exothermic inert atmosphere generator used in the treatment of bulk-stored wheat for insect control.



FIG. 2.—View of hinged outlet for release of inert atmosphere from the top of a storage tank and plastic air sampling lines.

with wheat. Air samples were collected just before the tank was filled with grain and again immediately after it was filled. Thereafter, samples were taken at 4, 8, 24, 48, and 72 hr. The tank in the 3rd test was also purged prior to loading; however, the inert atmosphere was released through the larger 1½-in.-diam pipe. Air samples were taken before and after loading and following 4, 8, 24, and 96 hr of treatment.

Gross analyses of the O₂ content were made periodically throughout each test with a Fyrite O₂ indicator.

Insects used in each test were adult confused flour beetles, *Tribolium confusum* Jacquelin duVal, and immature stages of the rice weevil, *Sitophilus oryzae* (L.). Two brass-screened test insect cages were used at each sampling point. One cage contained 33 adult flour beetles plus a small amount of suitable food material and the other cage contained 9 g of wheat infested with the immature stages of the rice weevil. The infested wheat was a blended sample of equal amounts of grain known to contain insects in the egg, larval, and pupal stages of development. The cages were placed in small muslin cloth bags and taped to the sample lines at each air-sampling point. The sample lines were placed in the elevator tank prior to filling the tank with wheat. After completion of each test the lines were released at the

top of the tank and recovered at the base of the tank as the grain was turned. To delineate the effect of the inert atmosphere on the egg, larval, and pupal stages, 4 separate weekly counts were made of the emerging rice weevil adults beginning with the 1st emergence observed. This occurred between the 2nd and 3rd week following treatment. Mortality estimates were based on the relative numbers of adults emerging from treated and untreated samples. Progeny from the 1st count were assumed to be in the pupal or prepupal stage during exposure, progeny from the 2nd and 3rd counts from larval stages, and the 4th count primarily from eggs exposed during treatment.

As the wheat was turned a composite grain sample was obtained before and after each test for germination studies and grade information. The germination tests were conducted by the State Seed Laboratory, Kansas State Board of Agriculture, Topeka, and the grade information was determined by the Grain Division, Consumer and Marketing Service, USDA, Kansas City, Mo. Additional samples were taken during Test 3 (96-hr exposure) for milling and baking studies. Physical measurements of flour milled from wheat samples obtained before and after the treatment included farino-

Table 1.—Average O₂ concentrations and test insect mortality obtained at center and wall locations at indicated levels in a 20,000-bu tank of wheat treated for 72 hr with an inert atmosphere^a produced by an exothermic inert-atmosphere generator and introduced through the floor by using a 1½-in. pipe without purging prior to treatment (Test 1).

Location (Depth)	% oxygen concentrations after indicated hours—					% mortality	
	4	8	24	48	72	Adult confused flour beetle	Immature rice weevil
Overhead ^b	18.3	17.5	2.6	0.4	0.6	100	85.4
10 ft	18.6	17.1	0.4	1.0	.2	100	85.3
30 ft	18.2	7.2	.3	0.7	.2	100	60.2
50 ft	17.2	3.2	.3	.4	.2	100	39.4
70 ft	15.9	1.0	.3	.4	.3	100	97.8
90 ft	3.9	1.0	.2	.3	.2	100	100
110 ft	0.9	0.5	.2	.5	.2	100	100
130 ft	.4	.4	.2	.3	.2	100	100

^a Composition of the inert atmosphere, <0.1%, 8.5–11.5% CO₂, and the balance principally N₂.

^b Single sample location.

Table 2.—Average O₂ concentrations and test insect mortality obtained at center and wall locations at indicated levels in a 20,000-bu tank of wheat treated for 72 hr with an inert atmosphere^a produced by an exothermic inert-atmosphere generator and introduced through the floor by using a 3/4-in. pipe. Tank purged overnight prior to treatment (Test 2).

Location (Depth)	% oxygen concentrations					% mortality			
	Before filling	After filling	After indicated hours				Adult confused flour beetle	Immature rice weevil	
			4	8	24	48			72
Overhead ^b	10.9	19.0	17.6	20.2	12.7	2.8	1.2	15	45.4
10 ft	8.2	19.0	17.3	16.1	14.2	2.5	1.2	9	17.3
30 ft	8.2	16.8	15.7	14.8	11.6	1.0	1.0	50	22.7
50 ft	8.6	15.0	14.4	13.2	9.3	0.7	0.7	100	10.9
70 ft	8.2	13.3	12.7	11.5	2.9	1.0	1.7	100	23.6
90 ft	8.1	11.7	10.9	9.7	0.7	.5	.4	100	17.3
110 ft	7.9	9.9	8.8	1.7	.5	.4	.3	100	11.9
130 ft	7.8	7.9	0.8	0.5	.2	.2	.3	100	30.0

^a Composition of the inert atmosphere, <0.1% O₂, 8.5–11.5% CO₂, and the balance principally N₂.

^b Single sample location.

graph tests, mixing times, moisture content, and protein analysis. Baking tests included flour-absorption studies and loaf-volume measurements following potassium bromate treatments. These studies were conducted by the USDA Hard Winter Wheat Quality Laboratory and the Grain Science Department of Kansas State University, Manhattan.

Temperature of the wheat in each test was determined from thermocables permanently installed in each tank. Thermocouples were spaced at 7-ft intervals along a single cable situated near the center of the tank. Average grain temperatures in the 3 tests ranged from 75°F in Test 1 to 71°F in Test 3. Moisture content of the wheat was 12.5% in Test 1, 11.5% in Test 2, and 11.7% in Test 3. No significant change was detected in either the grain temperature or moisture following the 72- to 96-hr treatment periods.

RESULTS.—An O₂-deficient atmosphere was obtained throughout the grain mass in each test and was successfully maintained during the balance of 72- and 96-hr exposure periods (Tables 1, 2, 3). The time required to achieve displacement of the normal atmosphere was dependent upon the rate of flow of the gas released into

the tank and the degree of pretreatment purging achieved prior to filling the tank with grain.

In Test 1 (Table 1) with delivery through the 1½-in.-diam pipe, but no prior purging, the O₂-deficient zone moved upward through the grain mass at about 8 ft/hr. Static pressures measured from the sampling lines with a 1-in. scale magnehelic gauge indicated an estimated time of about 17 hr for one complete air change within the tank. The intermix of inert atmosphere and the existing atmosphere as the gas passed through the grain mass was surprisingly low. Carbon dioxide concentrations in the range of 9–10% accompanied the O₂-deficient zone. After 72 hr, the CO₂ concentrations throughout the entire grain mass were within the 8.5–11.5% range produced by the inert atmosphere generators.

In Test 2 (Table 2) with delivery through the ¾-in.-diam pipe, pretreatment purging resulted in a reduction of the O₂ in the empty tank to about 8% just prior to filling. Loading the tank restored near-normal atmospheric conditions to the upper 1/3 of the tank, but the reduced flow through the ¾-in. pipe delayed the subsequent displacement of the normal atmosphere.

Table 3.—Average O₂ concentrations and test insect mortality obtained at center and wall locations at indicated levels in a 20,000-bu tank of wheat treated for 96 hr with an inert atmosphere^a produced by an exothermic inert-atmosphere generator and introduced through the floor by using a 1½-in. pipe. Tank purged overnight prior to treatment (Test 3).

Location (Depth)	% oxygen concentrations					% mortality		
	Before filling	After filling	After indicated hours				Adult confused flour beetle	Immature rice weevil
			4	8	24	96		
Overhead ^b	4.3	18.3	11.7	2.5	0.9	7.5 ^c	88	59.2
10 ft	4.0	16.3	7.9	0.5	.3	0.7	100	54.7
30 ft	3.9	12.5	4.1	.4	.2	.5	100	49.0
50 ft	3.9	10.0	1.2	.4	.2	.4	100	43.5
70 ft	3.9	8.1	0.5	.3	.3	.3	100	46.3
90 ft	3.9	5.8	.3	.3	.2	.2	100	47.0
110 ft	3.9	0.9	.2	.2	.2	.2	100	67.6
130 ft	2.4	.1	.1	.1	.1	.1	100	70.4

^a Composition of the inert atmosphere, <0.1% O₂, 8.5–11.5% CO₂, and the balance principally N₂.

^b Single sample location.

^c Strong shifting winds prior to taking of this sample resulted in some diffusion of outside air into the top of the tank.

In Test 3, with the 1½-in. pipe, pretreatment purging resulted in O₂ concentrations ranging from 2.4% in the bottom of the tank to 4.3% in the top of the tank. Filling the tank left a stratification of O₂ concentrations ranging from <1% below the 110-ft level to near normal atmosphere in the top of the tank. At the 4-hr reading, O₂ concentrations were <1% in the bottom half of the tank, and 8 hr after filling, the O₂ concentrations were reduced to <1% throughout the entire grain mass.

Control of the test insects exposed to the O₂-deficient atmosphere was only partially successful (Tables 1, 2, 3). Oxygen concentrations of less than 1% for 24 hr killed the adult confused flour beetles, but were not effective against the immature stages of the rice weevil for time periods of 72 to 96 hr. Although some overlapping in the development cycle of each immature stage would be expected, the indicated order of susceptibility in each test was pupae > larvae > eggs. It is not known whether this apparent difference was due to physiological factors or simply to a physical barrier relationship involving the size of the cavity in the individual kernel of wheat and gas interchange between cavity and interspace. Contrasting results were obtained by Lindgren and Vincent (1970), who found the number of days required to obtain 95% mortality in an atmosphere of N₂ at 80°F was 9.6 for rice weevil eggs, 9.0 for larvae, 18.8 for pupae, and 4.0 for adults.

Results of tests to determine the effect of the inert atmosphere on grain quality showed no significant differences between treated and untreated wheat. Germination of the wheat before treatment was 84% in Test 1, 85% in Test 2, and 80% in Test 3. After treatment, the germination was 81% in Test 1, 88% in Test 2, and 84% in Test 3. Treated and untreated wheat samples were graded the same, and factors comprising the total defects remained virtually the same within each test. Results obtained in the baking and milling studies did not show a significant difference between untreated controls and the corresponding treated wheat in any of the factors measured.

DISCUSSION.—Use of exothermic inert-atmosphere generators for displacing the normal atmosphere in stored wheat was demonstrated. Further study is needed to establish the minimum time periods of exposure to the O₂-deficient atmosphere required to produce a high mortality of the immature stages of stored-product insects.

Pretreatment purging was effective in reducing the treatment time required to achieve displacement of the existing atmosphere, but a considerable amount of inert atmosphere was required during the purging period. The advisability of purging prior to treatment would depend on the generating capacity of the inert atmosphere equipment. The total inert atmosphere produced

per hour by small generators could perhaps be more effectively used in direct application to grain, whereas the total volume of inert atmosphere produced by larger units might well exceed the output required for immediate treatment or maintenance of inert atmosphere in previously treated tanks. This surplus which might normally be vented to the outside atmosphere could be effectively used to prepurge tanks for future loading and treatment. The cost of consumables (natural gas, electricity, water) for operation of each 15,000 CFH generator was estimated at \$1.70/hr. Establishing comparative costs of inert-atmosphere treatments and conventional chemical fumigation requires information beyond the scope of the study reported here. It is evident, however, that treatment costs with the inert-atmosphere generators will depend on how effectively the total amount of inert atmosphere generated each hour can be used.

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