

Effects of Fumigation on Wheat in Storage. I. Physical Measurements of Flour

RUTH H. MATTHEWS¹, C. C. FIFIELD², T. F. HARTSING³, C. L. STOREY³
and N. M. DENNIS³, Human Nutrition Research Division, ARS, USDA,
Beltsville, Maryland

ABSTRACT

A comprehensive study was conducted to determine effects of fumigation of wheat in storage on composition, quality, and baking performance of the wheats. Fumigations included methyl bromide; ethylene dichloride- CCl_4 (3:1); and phosphine. Two control samples, one held at ambient temperatures, the other at 32° F., were also studied. This paper gives results of physical quality tests and condition of the flours from the fumigated wheats milled at predetermined times.

Methyl bromide and ethylene dichloride- CCl_4 fumigations of the wheat kept the flours in the best condition, as shown by low insect-fragment counts. Inorganic bromide residue built up to 99 p.p.m. in the flour after 3 years' storage and repeated fumigations. Little accumulation of ethylene dichloride and CCl_4 was evident in the flour during the course of the research. Phosphine residue content increased somewhat near the end of the study.

Storage and treatment of the wheats caused changes in physical qualities of the flours. The amylograph showed highest viscosity in the flours from the phosphine treatment and lowest in the flours from methyl bromide and refrigerated control wheats. Farinograph readings showed the flours from the phosphine-fumigated grain to have the highest mixing tolerance at most test periods of the wheat. Near the end of the 3-year project, the methyl bromide sample showed some indication of lowered mixing tolerance. Together, storage and treatment of the wheat caused an over-all decrease in quality characteristics.

Only meager information has been published on the effects of fumigation of wheat during storage on the physical qualities of the milled flour. Measurements to predict flour performance due to fumigation of wheat have not been reported in the literature. Fumigants or other types of insect control are required to protect grain from insect damage and contamination during storage. Wheat for milling into white flour may be fumigated several times yearly and stored for 2 to 3 years or longer. Fumigants chemically reactive on wheat can change physical properties and baking performance of flours so that adjustments in formulations and preparation procedures may be required for optimum eating quality of baked products.

Past research on the effects of fumigation of wheat centered on amounts of residue accumulation, rates of insect kill, and viability of the grain. One or two applications of fumigant were used. Bread-baking quality evaluations gained some attention, but only a few reports were published (1,2,3,4,5).

This co-operative research of the Human Nutrition and Market Quality Research.

¹Present address: Consumer and Food Economics Research Division, ARS, USDA, Hyattsville, Maryland.

²Retired. Formerly with Market Quality Research Division, ARS, USDA, Beltsville, Maryland.

³Market Quality Research Division, ARS, USDA; Beltsville, Maryland, Manhattan, Kansas, and Savannah, Georgia, respectively.

Divisions of the U.S. Department of Agriculture was planned to evaluate the composition, quality, and baking performance of the wheats exposed to fumigations during storage. This paper reports procedures used and results of physical measurements of the flours. Results of baking tests and analyses for B-vitamin and tocopherol content of wheat, milling fractions, and baked products will be given in forthcoming papers. All samples of wheat, milling fractions, doughs, and baked products were analyzed for residue content by the Stored-Product Insects Research and Development Laboratory of the Market Quality Research Division in Savannah, Georgia. Detailed results of these analyses will be published by that laboratory.

MATERIALS AND METHODS

Wheat Procurement, Storage, and Fumigations

Early Triumph wheat with no previous fumigation or protectant treatment was purchased from a farm near Wichita, Kansas, and shipped directly to the Stored-Grain Insects Laboratory in Manhattan, Kansas, for storage. Four bins of 100-bu. capacity were especially constructed of 3/4-in. marine plywood for storage of the wheat. The separate bins, painted white with metal roofs, were exposed to the elements. Bins of wheat were fumigated in April and October for the first year, and in May, July, and September in the second and third years of storage. The additional fumigations in the second and third years were needed to keep the wheat in good condition and free from insect contamination.

Three fumigants—methyl bromide, a mixture of ethylene dichloride and CCl_4 , and phosphine—were used in this research. Methyl bromide was applied at the rate of 2.5 lb. per 1,000 cu. ft. of storage. A liquid fumigant mixture of ethylene dichloride (75%) and CCl_4 (25%) was used at the rate of 6 gal. per 1,000 bu. Both bins were treated by the recirculation method. The phosphine-fumigated lot of wheat was treated at the rate of six aluminum phosphide tablets per ton, probed into the grain mass. A fourth bin of wheat was held at ambient temperatures with no fumigation or any other insecticide treatment. A fifth lot, or second control sample, was held in 32° F. storage for the duration of the project.

Annually, in September, the ambient-temperature control wheat was cleaned and screened to remove live and dead insects. The refrigerated control wheat was held in multiwall bags inside 12 tightly sealed 55-gal. drums. The schedule for fumigation and sampling of the wheat is given in Table I.

For each test period, a 200-lb. (3.3-bu.) sample of wheat was collected from each fumigation and storage variable, placed in multiwall paper bags inside 55-gal. drums, and shipped directly to the Field Crops and Animal Products Branch Laboratory of the Market Quality Research Division in Beltsville, Maryland, for milling.

Milling of Wheat

Detailed procedures of milling are given here because an understanding of results of flour and baked-product measurements and nutrient analyses of the wheat and various milling fractions depends on the specific procedures used.

TABLE I. SCHEDULE FOR FUMIGATION AND SAMPLING

Fumigation Date	Test Period and Sampling Dates
1965 Apr. 15	1) Apr. 22, 1965; 2) July 15, 1965
1965 Oct. 15	3) Oct. 22, 1965; 4) Jan. 15, 1966; 5) Apr. 1, 1966
1966 May 15	6) July 14, 1966
1966 July 15
1966 Sept. 15	7) Nov. 15, 1966; 8) Mar. 15, 1967; 9) May 14, 1967
1967 May 15	10) July 14, 1967
1967 July 15
1967 Sept. 15	11) Nov. 15, 1967; 12) Mar. 15, 1968

Each lot of wheat was cleaned with the use of a Carter dockage machine to remove all types of foreign material. Wheats were tempered to 14.5% moisture and allowed to stand for 72 hr. at room temperature before being milled. Tempered wheats were scoured in a laboratory-size scouring machine to remove the brush ends of the wheat, the bee's-wing, and to loosen and remove other foreign material. Approximately 1 hr. before milling, 1% more water was added to the wheat to toughen the bran coat so as to help reduce breaking-up of bran particles.

Millings were made on a Buhler pneumatic automatic laboratory mill. Samples were milled under controlled millroom conditions of 75° to 80°F. and 60 to 65% r.h. The mill was equipped with three break rolls and three reduction rolls which produced six flour streams, one bran, and one shorts stream. Each lot of wheat was milled to produce the highest possible yield of flour without overgrinding of the bran and shorts. The flour used in this research was composed of the first- and second-break flours and the first- and second-reduction flours, plus a necessary portion of the third-reduction flour to make a 90% patent flour. The long-patent flour approximated a commercial family-type flour.

The other milling fractions included low-grade flour, shorts, and bran. The low-grade flour consisted of all of the third-break flour, the remainder of the third-reduction flour, and flour that was recovered from sifting of bran and shorts. The shorts milling fraction included the germ and the portion of the endosperm from the reduction side of the mill that commercially would produce red dog flour. The bran included from 1 to 2% of flour. Bran particles were kept as large as possible to prevent the flour from becoming dark and ash content from increasing.

For each test period, flour samples were held in multiwall bags inside tightly closed containers for 3 weeks at room temperature. No bleach or other chemical treatment was used.

Physical Measurements

Physical measurements were determined on the 60 flour samples (12 test periods \times 5 treatments). Measurements included amylograph, farinograph, mixograph, and Hunter color (L , a_L , and b_L). Except for Hunter color, determinations were made according to the AACC methods (6).

Sampling for Residue Analysis

Samples of wheat and each milling fraction from each test period and treatment were sealed in cans lined with aluminum foil and placed in storage at -40°F. Cans

were packed in dry ice and shipped via air express to the laboratory in Savannah, Georgia, for residue analyses.

The sensitivity of the methods for detection of inorganic bromide residue (7,8) was 2 p.p.m.; for ethylene dichloride- CCl_4 (9), 0.5 p.p.m.; and for phosphine (10), 0.001 p.p.m., respectively.

Insect Fragment Count

Insect fragments in flour were counted according to the acid hydrolysis method of the AOAC (11).

RESULTS AND DISCUSSION

Yield and Condition of Samples

The flour samples milled from the 60 lots of wheat varied in yield between 65.3 and 69.5%. Ash content ranged between 0.41 and 0.48%.

Repeated fumigations throughout the 3 years' storage usually kept the wheat in good condition as shown by the relatively low insect fragment counts of the flour (Table II). Fragment counts of flour from the phosphine-fumigated wheat samples were often higher than those of flours from the other fumigated wheats. As expected, flours from wheat held at ambient temperatures without fumigation had high insect-fragment counts after the first year of storage of the wheat. Because the second control sample of wheat was held in refrigeration at 32°F., no increase in insect-fragment count was expected.

Flours from the methyl bromide-fumigated wheat gradually increased in inorganic bromide residue content—particularly after the second and third years of storage and treatment (Table III). No organic bromide residue was detected. The ethylene dichloride- CCl_4 -fumigated sample was almost stationary in residue content after the first year. Residue content of flour from the phosphine-fumigated wheat varied slightly from 0.001 to 0.009 p.p.m. during the first 2 years and six

TABLE II. INSECT-FRAGMENT COUNT^a OF 90% PATENT FLOURS FROM WHEATS FUMIGATED EIGHT TIMES DURING 3 YEARS' STORAGE

Test Period	Treatment of Wheat				
	Methyl Bromide	Ethylene Dichloride, CCl_4	Phosphine	Control, Refrigerated	Control, Ambient Temperature
1	1	2	1	4	2
2	13	4	25	6	2
3	17	7	4	3	22
4	8	5	9	7	6
5	11	19	26	7	14
6	5	11	20	2	82
7	7	10	44	4	299
8	16	10	53	10	337
9	5	17	37	6	106
10	7	1	8	0	86
11	13	8	36	0	168
12	0	5	29	1	143

^aOfficial method of AOAC, acid hydrolysis (11).

TABLE III. FUMIGANT RESIDUE CONTENT OF 90% PATENT FLOURS FROM WHEATS FUMIGATED EIGHT TIMES DURING 3 YEARS' STORAGE

Test Period	Methyl Bromide as Inorganic Bromide ^a p.p.m.	Ethylene Dichloride ^b CCI ₄ (3:1)		Phosphine ^c as Phosphine p.p.m.
		Ethylene Dichloride p.p.m.	CCI ₄ p.p.m.	
1	16.2	4.1	0.2	0.007
2	19.0	1.4	0.1	0.001
3	40.0	1.4	0.5	0.002
4	44.0	1.0	0.4	0.005
5	43.0	0.6	1.0	0.004
6	47.0	1.0	1.0	0.005
7	66.0	1.0	1.0	0.004
8	69.0	1.0	1.0	0.009
9	66.0	1.0	1.0	0.002
10	69.0	1.0	1.0	0.002
11	94.0	1.0	1.0	0.021
12	99.0	1.0	1.0	0.022

^aSensitivity 2 p.p.m.; method, refs. 7,8.

^bSensitivity 0.5 p.p.m.; method, ref. 9.

^cSensitivity 0.001 p.p.m.; method, ref. 10.

fumigation treatments. Two more phosphine fumigation treatments caused increases in residue content to 0.021 and 0.022 p.p.m. in the flour.

Physical Measurements

Viscosity of flour suspensions varied according to the length of storage and treatment of the wheat. For most test periods of the wheat, flours from the phosphine fumigation were among the highest in paste viscosity and flour from the methyl bromide and refrigerated control wheats were among the lowest in paste viscosity (Fig. 1). Although phosphine residue content did not build up significantly during the course of this research, phosphates may have accumulated in the milled products, causing the high paste viscosity. This measurement of viscosity of the flour suspensions may reflect amylase activity in the stored wheat samples or changes in starch-gelatinization properties of the flours.

The farinograph tests showed that mixing tolerance varied considerably among the treatments and the storage periods of the wheat (Table IV). Of the five treatments, the flour from phosphine-fumigated wheat had the highest mixing tolerance in more test periods than any other sample. Flours from methyl bromide-fumigated wheat tended to be less tolerant to mixing in the last two test periods.

Maximum height of the mixograph curve rose dramatically for all flours after the first 6 months' storage of the wheat and again after 1½ and 2 years' storage (Fig. 2). Strength of the doughs seemed to be more an influence of storage time than of treatment of the wheat. As shown previously (12), quality characteristics of flours change during storage of wheats without any fumigation treatment.

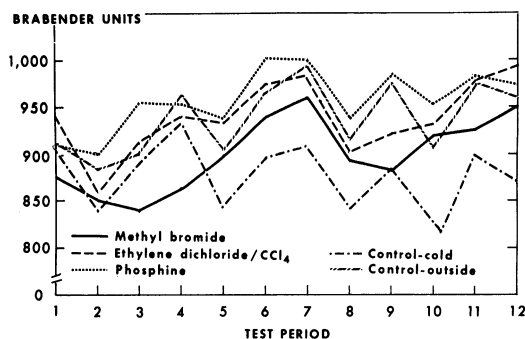


Fig. 1. Maximum amylograph readings of 90% patent flours from wheats fumigated eight times during 3 years' storage.

TABLE IV. MIXING TOLERANCE^a OF 90% PATENT FLOURS FROM WHEATS FUMIGATED EIGHT TIMES DURING 3 YEARS' STORAGE

Test Period	Treatment of Wheat				
	Methyl Bromide min.	Ethylene Dichloride, CCl ₄ min.	Phosphine min.	Control, Refrigerated min.	Control, Ambient Temperature min.
1	6.50	6.00	7.00	8.50	6.00
2	7.50	7.00	8.50	6.50	6.75
3	8.00	7.25	7.00	6.25	7.75
4	10.25	8.00	10.50	7.75	7.25
5	10.75	8.75	9.00	9.50	9.00
6	9.25	11.00	9.50	7.25	8.50
7	7.00	9.00	10.25	7.00	7.50
8	6.50	8.25	11.00	5.75	8.25
9	7.50	7.50	7.00	6.50	7.50
10	7.00	7.50	7.25	7.00	7.25
11	6.00	7.50	7.75	6.25	7.25
12	6.75	8.50	9.75	7.00	8.25

^aFarinograph tests, AACC method (ref. 6).

The L color values (lightness) of the flour increased with storage of the wheat to the maximum after approximately 2 years' storage. The a_L values of the flours showed some greenness during the first year of wheat storage. The b_L color values of the flours from wheats stored for 6 months, 1 year, and 2 years were highest (most yellow). Lightness, greenness, or yellowness of the flours showed little relation to the fumigation treatment of the wheat.

CONCLUSIONS

Fumigation treatment with methyl bromide held wheats in the best condition, as shown by low insect-fragment counts in the milled flours. Inorganic bromide residue content, however, built up considerably during the 3 years' storage and eight treatments. Flour from the phosphine-treated wheat usually had highest paste viscosity and most often had the highest mixing tolerance. Mixograph and Hunter

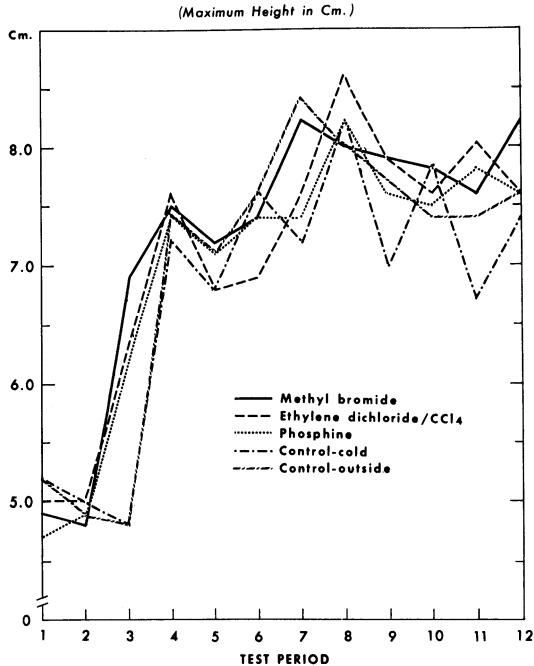


Fig. 2. Maximum height of mixograph curve of 90% patent flours from wheats fumigated eight times during 3 years' storage.

color readings of flours showed changes during the study and were influenced more by length of storage than by treatment of the wheats.

Acknowledgments

The authors wish to thank Elinora J. Sharpe (retired) and Willa M. Clark of the Human Nutrition Research Division and Dorothy Humphrey of the Market Quality Research Division for carrying out the physical measurements of the flours.

Literature Cited

1. DEAN, G. A., and SWANSON, C. O. Effect of common mill fumigants on the baking qualities of wheat flour. Kan. State Exp. Sta. Bull. No. 178, pp. 155-208 (1911).
2. SMITH, H. S., and STATEN, H. W. The effects of carbon disulphide upon germination and baking quality of wheat. Okla. Agr. Mech. Coll. Agr. Exp. Sta. Tech. Bull. No. T.14 (1942).
3. YOUNG, H. D., and BAYFIELD, E. G. Effect of hydrocyanic acid on the baking quality of flour. Cereal Chem. 21: 149 (1944).
4. HERMITTE, R. J. J., and SHELLENBERGER, J. A. Effect of excessive methyl bromide fumigation on flour. Cereal Chem. 24: 449 (1947).
5. LINDGREN, D. L., VINCENT, L. E., and STRONG, R. G. Studies on hydrogen phosphide as a fumigant. J. Econ. Entomol. 51: 900 (1958).
6. AMERICAN ASSOCIATION OF CEREAL CHEMISTS. AACC Approved methods (formerly Cereal laboratory methods, 7th ed.). The Association: St. Paul, Minn. (1962).
7. SCHRADER, S. A., BESHGETOOR, A. W., and STENGER, V. A. Determination of total and inorganic bromide in foods fumigated with methyl bromide. Ind. Eng. Chem., Anal. Ed. 14: 1 (1942).

8. HEUSER, S. G. Residues in wheat and wheat products after fumigation with ethylene dibromide. *J. Sci. Food Agr.* 12: 103 (1961).
9. RAMSEY, L. L., CONROY, H. W., and MUNSEY, V. E. B. Colorimetric determination of carbon tetrachloride in fumigated cereal products. *J. Assoc. Offic. Agr. Chemists* 40: 115 (1957).
10. BRUCE, R. B., ROBBINS, A. J., and TUFT, T. O. Phosphine residues from Phostoxin-treated grain. *J. Agr. Food Chem.* 10: 18 (1962).
11. ASSOCIATION OF OFFICIAL ANALYTICAL CHEMISTS. *Methods of analysis* (10th ed.). The Association: Washington, D.C. (1965).
12. MATTHEWS, RUTH H., FIFIELD, C. C., and HARTSING, T. F. Assessing wheat quality with breads prepared by two formulations. *Cereal Chem.* 45: 133 (1968).

[Received October 20, 1969. Accepted February 27, 1970]