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FLOUR MATURING AND BLEACHING WITH ACYCLIC ACETONE PEROXIDES[1]

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ABSTRACT

The preparation, properties, and chemical nature of the acyclic acetone peroxides are described. Carotene values of commercially bleached flour are given as a basis for evaluating the bleaching effect of the new product. Maturing may be accomplished by treatment with 0.0015% to 0.008% hydrogen peroxide equivalents of the acyclic acetone peroxides, the quantity depending largely on flour grade. When the amount required for optimum maturing does not remove color adequately, as often occurs, relatively small quantities of benzoyl peroxide may be added to attain the desired bleaching effect.

Data for carotene values and baking tests illustrate the effect of flour treatment with acyclic acetone peroxides, alone and in combination with benzoyl peroxide and chlorine dioxide. Stability of the starch-acetone peroxide composition is excellent at 50° and good at 70°F., but diminishes as temperature of storage is increased above room temperature. Enrichment vitamins are unaffected by the acyclic acetone peroxides used for maturing and bleaching flour. Studies by a radioactive tracer technique show a residue of 0.5 p.p.m. in bread, calculated as acetone equivalents.

The Definitions and Standards of Identity for wheat flour were amended in 1961[3] to include an acetone peroxide composition as an optional flour maturing and bleaching agent. The purpose of this paper is to describe the product and to provide information about its use. Research leading to the development of acetone peroxides was initiated some years ago by H. O. Renner[4], to whom credit is due for discovering their useful properties for maturing and bleaching wheat flour.

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[2]Address: J. R. Short Milling Co., Research Laboratories, Chicago, Ill.
[3]Federal Register, June 28, 1961, p. 5751.
[4]Patent applications in the name of H. O. Renner are pending.

Materials and Methods

Preparation and Description of Acyclic Acetone Peroxides. In general terms, acyclic acetone peroxides are prepared by controlling the reaction between acetone and hydrogen peroxide of 35 to 50% strength, although stronger or weaker solutions may be employed with appropriate modifications. The reaction can take place at room temperature, but may be accelerated by heat, acid catalysts, or both. Also, the proportion of acetone to hydrogen peroxide may be varied over a wide range. The nature and ratio of the peroxides formed depend on the conditions used.

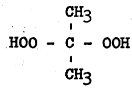
While it is possible to treat flour directly with the liquid peroxide reaction mixture, a more convenient procedure is to prepare a starch concentrate by adding the liquid reaction mixture to starch and drying. The resulting product may be used as a dry, free-flowing agent which can be fed into flour. The concentration of acyclic acetone peroxides may be adjusted accurately to a convenient peroxide activity. This may range from 10 to 15% hydrogen peroxide equivalents.

The following procedure[5] will illustrate the preparation of a highly satisfactory product on a laboratory scale:

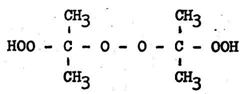
> To 57 ml. (68 g.) of 50% hydrogen peroxide in a beaker, add 2 ml. of normal hydrochloric acid, followed by dropwise addition of 37 ml. acetone. (The molar ratio of the above is 2:1.) Keep the reaction mixture cool, say from 0° to 10°C. To ensure completion of the reaction, let stand 1 hour. Add 2.3 ml. normal sodium hydroxide to neutralize. The pH can be from 6.8 to 7.0. At this point the reaction mixture will have a volume of about 100 ml.
>
> Add the entire reaction mixture to 160 g. food-grade corn starch and mix thoroughly. Spread out in a thin layer and dry in air at room temperature or by gentle heating. After drying, grind. Once the liquid reaction mixture has been combined with starch and dried, the product is not hazardous in an explosive sense and may be ground safely without using special precautions. However, in preparing and handling the liquid reaction mixture and the starch concentrate, care must be exercised, as the peroxide concentration is high and must be treated accordingly. Contact with skin and eyes should be avoided.

Prepared as above, the starch concentrate will have a peroxide strength of 12 to 13% hydrogen peroxide equivalents of organic peroxide. (Commercial products will contain approximately 10%.) The organic peroxides in the starch concentrate will be comprised of acyclic acetone peroxide monomer principally (85–95%), some acyclic

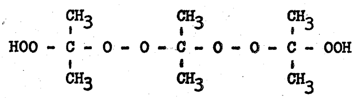
[5] Preparation of these peroxides is hazardous and great care must be exercised to control reaction conditions.



MONOMERIC ACETONE PEROXIDE
2, 2- DIHYDROPEROXY PROPANE



LINEAR DIMER OF ACETONE PEROXIDE
BIS - (1,1' HYDROPEROXY 1, 1′ METHYL)
DIETHYL PEROXIDE



LINEAR TRIMER OF
ACETONE PEROXIDE

Fig. 1. Acetone peroxides.

dimer (5–15%), from none to a trace of acyclic trimer, no cyclic trimer, and some residual hydrogen peroxide.

The structure of the peroxides formed is shown in Fig. 1. The reaction conditions chosen are such that the liquid mixture consists chiefly of the dihydroperoxy monomer (I), with a much smaller proportion of linear dimer (acyclic) (II) and a still smaller proportion of linear trimer (acyclic) (III). The cyclic trimer (IV) is not a flour maturing and bleaching agent. Thus its formation is not desired. The liquid product contains practically no cyclic trimer, and the starch composition also contains none.

The monomer (I) is a viscous liquid at room temperature; the acyclic dimer (II) is a crystalline solid. When a starch concentrate is prepared by adding the liquid reaction mixture to starch and drying, water, any residual acetone, and some hydrogen peroxide volatilize, as well as some linear dimer which is quite volatile. Hydrogen peroxide volatilizes more readily than the monomer (I). Thus, the dry starch concentrate contains chiefly the monomer, a variable amount of linear dimer, and some unreacted hydrogen peroxide.

Flour Treatment Calculations. The following example will serve to illustrate the calculation of the amount of a 10% hydrogen-peroxide-equivalent-starch-concentrate of acetone peroxides needed to treat flour at the rate of 0.0025% hydrogen peroxide equivalents:

$$0.0025/0.1 = 0.025\% \text{ (0.025 lb./cwt. flour or 0.4 oz.)}$$

Also, 1 lb. of this concentrate (10%) will treat 100/0.025 = 4,000 lb. flour.

Baking Test Procedure. All laboratory baking tests were made by a sponge-dough procedure using the following formula:

Sponge g	*Ingredients*	*Dough* g	*Total* %
450 (65%)	Flour	250	100.0
275–300	Water	variable	variable
17.5	Yeast	......	2.5
2.0–3.5	Yeast food (bromate type)	......	0.28–0.5
......	Milk solids	21	3.0
......	Salt	14	2.0
......	Sugar	35	5.0
......	Lard	21	3.0

Doughs were mixed in a Hobart mixer equipped with a McDuffee bowl. Two loaves from each dough were scaled at 520 g. Sponge time was 4 hours, and the dough was rested for 25 minutes. Bread was baked for 25 minutes at 430°F. Loaf volumes reported are the average of two.

Results and Discussion

Carotene Values of Commercially Bleached Flours. Before results of flour maturing and bleaching tests with acetone peroxides are presented, it may be useful to consider the extent to which color removal is practiced commercially in the United States and Canada. These data will provide a basis for evaluating the carotene values

TABLE I

Carotene[a] Values of Commercially Bleached Flours

Carotene	U.S. Flours		Canadian Flours		Total		Empirical Appraisal of Bleaching Treatment
	No.	Percent	No.	Percent	No.	Percent	
Below 0.60	2	19.1	0	15.8	2	18.3	overbleached
0.61–0.70	10		3		13		
0.71–0.80	8	27.0	2	10.5	10	23.2	heavily bleached
0.81–0.90	9		0		9		
0.91–1.00	13	47.6	2	42.1	15	46.4	moderately bleached
1.01–1.10	7		1		8		
1.11–1.20	5		1		6		
1.21–1.30	5		4		9		
1.31–1.40	2	4.7	1	26.3	3	9.7	lightly bleached
1.41–1.50	1		4		5		
1.51–1.60	0	1.6	0	5.3	0	2.4	underbleached
1.61–1.70	1		1		2		
	63	100.0	19	100.0	82	100.0	

[a] Water-saturated butanol extract; spectrophotometric procedure.

that will be reported. Flour samples were obtained representative of different wheat-growing areas, and carotene values were measured by the official AOAC method (1), except that a centrifuge was used to clarify the extract rather than filter paper, and a 5-cm. cell was employed for spectrophotometric measurements rather than a 1-cm. cell. Both modifications greatly improved accuracy. Bleaching treatment was evaluated empirically by visual appraisal of the crumb color of bread. The data are shown in Table I.

Some samples appeared overbleached in that the extreme chalky whiteness of the bread was not so pleasing to the eye as less heavily bleached flours. It would appear that carotene values between 0.91 and 1.30 p.p.m. (46% of the total number of samples examined) represent satisfactory color removal as judged by bread crumb color. Above 1.30 p.p.m. carotene, bread crumb color left something to be desired.

Flour Maturing and Bleaching Experiments with Acyclic Acetone Peroxides Alone. Extensive experimental work extending over many crop years has shown that the starch-acetone peroxide composition is an effective flour maturing agent. The amount required depends on flour grade, as is true also of other maturing agents. In general it has been found that patent flours require from 0.001 to 0.003% hydrogen peroxide equivalents. Clear flours may need 0.006% or more. Excessive amounts damage baking properties, but the product has shown good tolerance.

Treatment of a southwestern winter and a northwestern spring wheat flour with 0.003, 0.0035, and 0.004% hydrogen peroxide equivalents of acyclic acetone peroxides resulted in the data shown in Table II. The winter wheat flour showed good maturing at 0.003%, as evidenced by improved loaf volume, grain, and texture compared with

TABLE II
TREATMENT OF WINTER AND SPRING WHEAT PATENT FLOURS WITH ACETONE PEROXIDES

Flour	H_2O_2 Equiv. Acetone Peroxides	Carotene	Volume	Grain (10)	Texture (15)	Color (10)
	%	ppm	cc			
S. W. Patent	none	2.17	2,750	6.0	12.0	6.0
	0.0030 B[a]	1.33	2,825	9.0	13.5	9.0
	0.0035	1.25	2,725	8.5	12.7	8.5
	0.0040	1.23	2,650	8.0	12.0	8.0
N. W. Patent	none	1.66	2,640	7.5	10.0	6.5
	0.0030	1.21	2,700	8.0	11.5	7.5
	0.0035 B	1.21	2,725	8.0	12.5	8.5
	0.0040	1.14	2,600	7.0	10.5	6.5

[a] B, best over-all bread quality.

the control. Carotene removal was barely adequate, the crumb color score being high, no doubt because of excellent grain structure. Higher levels of treatment were not as good as 0.003%.

The unbleached northwestern spring wheat patent flour had a lower than average carotene value. In this test 0.0035% hydrogen peroxide equivalents gave the best bread. Marked loaf volume response and improved grain were obtained by the treatment. The 0.004% level proved excessive; carotene removal was best at 0.004%, but crumb color suffered because the crumb became coarse.

The results of treating an air-classified patent flour are shown in Table III. With this flour, 0.003% hydrogen peroxide equivalents

TABLE III
FLOUR[a] TREATMENT WITH ACETONE PEROXIDES

H_2O_2 Equiv. Acetone Peroxides	Loaf Volume	Grain (10)	Texture (15)	Crumb Color (10)	Carotene	
					24 hours	72 hours
%	cc					
None	2,250	6.0	12.0	6.0	2.30	2.23
0.003	2,525	7.0	12.0	8.0	1.97	1.58
0.0045	2,525	8.5	12.0	8.5	...	1.29
0.006	2,340	8.0	12.0	8.0	1.63	1.25
0.002[b]	2,450	8.5	12.0	7.5	1.49	1.29

[a] Patent flour, air-classified.
[b] Plus 0.001% benzoyl peroxide.

did not give quite as good bread as 0.0045%. Perhaps the optimum is in between. A level of 0.006% was excessive, as indicated by a falling-off in loaf volume. When 0.001% benzoyl peroxide was superimposed on a 0.002% acetone peroxide treatment, the carotene value was 1.29 p.p.m., the same as obtained with 0.0045% acetone peroxides. This illustrates how effective a small amount of benzoyl peroxide is in conjunction with a low level of acetone peroxides.

Flour Treatment with Acyclic Acetone Peroxides and Benzoyl Peroxide. The data in Table IV afford a comparison between the bleaching effect of acyclic acetone peroxides, benzoyl peroxide, and a combination of them. The flour used was a southwestern winter wheat flour of high extraction and normal carotene value. None of the treatments with acetone peroxides at the levels shown provided adequate color removal, and additional color removal with benzoyl peroxide was required. Adequate color removal, 0.93 p.p.m. carotene, was obtained by 0.003% benzoyl peroxide alone. A level of 0.002% hydrogen peroxide equivalents alone gave a carotene value of 1.68

TABLE IV

FLOUR[a] TREATMENT WITH ACETONE PEROXIDES AND BENZOYL PEROXIDE

H_2O_2 Equiv. Acetone Peroxides	Benzoyl Peroxide	Carotene 21 hours	Carotene 9 days
%	%	ppm	ppm
None	....	2.25	...
0.002	....	...	1.68
0.003	....	...	1.51
0.004	....	...	1.47
0.005	....	...	1.40
0.002	0.0015	...	0.97
0.003	0.0015	...	0.91
0.004	0.0015	...	0.88
...	0.0015	1.44	1.42[b]
...	0.0030	0.99	0.93[b]
...	0.0060	0.65	0.58[b]

[a] Southwestern high-extraction patent.
[b] 4 days.

p.p.m.; 0.0015% benzoyl peroxide alone gave a carotene value of 1.42 p.p.m. — both inadequately bleached. However, 0.0015% benzoyl peroxide and 0.002% acyclic acetone peroxides together resulted in a carotene value of 0.97 p.p.m. These data illustrate again the effectiveness of small amounts of benzoyl peroxide as a bleaching agent when used in conjunction with acyclic acetone peroxides.

Effect of Bromate in Baking Test with Flours Treated with Acyclic Acetone Peroxides. All flour maturing treatments pose a question about the additional need for bromate or other oxidizing agents in the bread formula. In general, commercial bakers find oxidizing agents desirable. Many baking tests with the acyclic acetone peroxide composition have shown that it does not eliminate the need for oxidizers in the bread formula. The data in Table V illustrate

TABLE V

EFFECT OF BROMATE IN BAKING TEST ON FLOUR MATURED WITH ACETONE PEROXIDE COMPOSITION

Flour[a] Treatment	Carotene	Bread: Volume	Bread: Grain (10)	Bread: Texture (15)	Bread: Crumb Color (10)
	ppm	cc			
Control, unbleached	2.38	2,425	6.0	10.5	6.0
0.25% Yeast food (6.8 p.p.m. $KBrO_3$)	...	2,605	7.0	11.0	7.0
0.50% Yeast food (13.5 p.p.m. $KBrO_3$)	...	2,655	6.5	11.5	7.0
0.003% H_2O_2 equiv.	1.70	2,470	6.0	10.5	8.0
Same, with 0.25% yeast food	...	2,740	9.0	13.0	9.0
Same, with 0.5% yeast food	...	2,755	8.0	12.0	8.7

[a] S.W. winter wheat patent.

this. An unbleached southwestern winter wheat patent flour was baked with and without two levels of a complete bromate-type yeast food and compared with a similar series of tests on flour treated with 0.003% hydrogen peroxide equivalents of acetone peroxides. The unbleached control gave a loaf volume of 2,425 cc. Addition to the control of 0.25 and 0.50% of bromate yeast food gave a volume response of 2,605 and 2,655 cc. respectively.

Treatment alone with 0.003% hydrogen peroxide equivalents of acyclic acetone peroxides showed a small maturing effect, which was greatly enhanced by the use of 0.25 and 0.5% bromate yeast food. Loaf volumes increased to 2,740 and 2,755 cc. respectively, and bread characteristics were greatly improved. This particular flour had a relatively high requirement for oxidation.

An illustration is given in Table VI of the use of bromate in the

TABLE VI

TREATMENT OF HIGH-EXTRACTION WINTER WHEAT FLOUR WITH ACETONE PEROXIDES
(Baked with and without bromate)

Hydrogen Peroxide Equiv.	Loaf Volume		Grain		Crumb Color		Carotene
	BrO_3[a] (a)	No BrO_3 (b)	BrO_3 (a)	No BrO_3 (b)	BrO_3 (a)	No BrO_3 (b)	
%	*cc*	*cc*					*ppm*
Control	2,600	2,400	6.5	6.5	6.0	5.0	2.27
0.002	2,650	2,500	8.0	7.3	8.0	7.0	1.68
0.003	2,650	2,535	8.0	7.5	8.0	7.5	1.51
0.004	2,700	2,550	8.5	7.0	8.5	7.5	1.47
0.005	2,725	2,575	8.5	7.5	8.0	8.0	1.40
0.003[b]	....	2,625	..	9.0	..	7.5	2.27

[a] Yeast food, 0.5%; a) complete with bromate; b) without bromate.
[b] Acetone peroxide added to flour at time of mixing.

baking formula with a high-extraction winter wheat flour treated with 0.002 to 0.005% hydrogen peroxide equivalents of acyclic acetone peroxides. One series of baking tests was run with 0.5% of a complete, bromate-type yeast food and a second series with the yeast food components without bromate.

The untreated control flour had a pronounced volume response to bromate. Increments of acetone peroxides without bromate in the baking formula also showed good volume response and improved bread characteristics. When bromate was used in the baking formula with various levels of acetone peroxides, still greater volume response was obtained along with improved grain.

An additional interesting effect of acyclic acetone peroxides is illustrated by the data on the last line of Table VI. In this experiment

0.003% hydrogen peroxide equivalents was added to the flour at the time of mixing, a procedure which provided very little time for the peroxides to act on the dry flour before a sponge or dough was prepared. Nevertheless, an excellent improver effect was obtained, and dough-handling properties were greatly improved. This characteristic of acyclic acetone peroxides may be of interest to millers who must deliver matured flour to a near-by baker under circumstances which do not permit flour to age somewhat before it is used. While the maturing effect of acetone peroxides is obtained under these circumstances, there is insufficient time for maximum bleaching.

Comparative Maturing and Bleaching Effects of Pure Monomer and Acyclic Dimer. It has been stated earlier that the acetone peroxides of the starch concentrate are chiefly the monomer and some acyclic linear dimer. A question which naturally arises concerns the effectiveness of the separate components. It has been found that both monomer and acyclic dimer are maturing and bleaching agents. A comparison of the effect of 0.003% hydrogen peroxide equivalents of pure monomer alone and acyclic dimer alone is illustrated by the data in Table VII.

TABLE VII

TREATMENT OF WINTER AND SPRING WHEAT PATENT FLOURS WITH MONOMERIC AND DIMERIC ACETONE PEROXIDE

	CONTROL	0.003% H_2O_2 EQUIV. OF MONOMER	0.003% H_2O_2 EQUIV. OF LINEAR DIMER
Winter wheat flour			
Dough consistency	good	sl. drier	sl. drier
Loaf volume (cc.)	2,550	2,750	2,710
Crumb color (10)	6.5	9.0	8.0
Grain (10)	8.5	7.5	8.0
Texture (15)	11.0	11.0	11.0
Carotene (p.p.m.), third day	2.73	1.66	2.02
Carotene (p.p.m.), tenth day	2.73	1.66	2.08
Spring wheat flour			
Dough consistency	good	dry	dry
Loaf volume (cc.)	2,750	2,775	2,750
Crumb color (10)	7.0	9.5	8.0
Grain (10)	7.0	8.0	8.0
Texture (15)	11.5	12.5	11.5
Carotene (p.p.m.), third day	1.91	1.20	1.44
Carotene (p.p.m.), twenty-third day	1.91	0.98	1.40

With winter wheat patent flour, baking results were improved by both monomer and acyclic dimer. There was little, if any, significant difference in grain and texture. Volume response was somewhat better for the monomer. Color removal by the monomer was much better than by the dimer. Thus, 0.003% monomer removed about 39% of the carotene, whereas the same amount of acyclic dimer removed 26%.

For spring wheat flour, loaf volume response favored the monomer and, again, superior color removal was obtained with the monomer as compared with the acyclic dimer. Carotene removal was 49% for monomer and 26% for acyclic dimer.

In general the monomer has proved to be a superior bleaching agent to the acyclic dimer, but only small differences in maturing effect have been found. Altogether, the presence of a small proportion of acyclic dimer with monomer has no measurable effect on the over-all bleaching and maturing action of the acetone peroxide mixture, and nothing would be gained by separating the acyclic dimer from the monomer. Because the starch concentrate is a mixture of the two components (about 85–95% monomer and 5–15% dimer), and since both are effective, it has appeared best to express peroxide concentration in terms of total hydrogen peroxide equivalents of total acetone peroxides. This has been done throughout the text.

Flour Treatments with Acyclic Acetone Peroxides and Chlorine Dioxide. A mixture of 60% southwestern winter wheat flour and 40% clear flour was made with the objective of preparing a flour sensitive to maturing treatment. Acetone peroxides were used at levels from 0.001 to 0.005% hydrogen peroxide equivalents in combination with a fixed amount of chlorine dioxide – 0.25 g./cwt.[6] Results of treatment are given in Table VIII.

Acetone peroxides alone, 0.001%, were inadequate, as expected, and reduced carotene from 2.64 p.p.m. in the control to 2.27 p.p.m. Increased amounts of peroxides alone caused a gradual reduction in carotene to 1.92 p.p.m. for 0.005% – the highest level applied.

Chlorine dioxide alone, 0.25 g./cwt., dropped the carotene from 2.46 to 2.27 p.p.m., a value exactly the same as that obtained with 0.001% acetone peroxides. However, chlorine dioxide at 0.25 g./cwt. gave a volume response not obtained with 0.001% acetone peroxides. As more peroxide was superimposed on chlorine dioxide, carotene values were further reduced, the lowest achieved being 1.81 p.p.m. with 0.004% acetone peroxides.

The bleaching effect of all treatments as reflected by the carotene values was poor, as might be expected from the inclusion of 40% clear in the flour mixture. Clear flours are much harder to bleach than patent flours.

[6] Flour prepared and treated in the laboratory of Sterwin Chemicals, Inc.

TABLE VIII

FLOUR[a] TREATMENT WITH ACETONE PEROXIDES AND CHLORINE DIOXIDE

Flour Treatment: H_2O_2 Equiv. Acetone Peroxides	Flour Treatment: ClO_2	Carotene	Loaf Volume	Grain (10)
%	g/cwt	ppm	cc	
Control	. . .	2.46	2,440	8.0
	0.25	2.27	2,500	7.0
0.001	. . .	2.27	2,440	8.0
0.001	0.25	2.11	2,490	9.0
0.002	. . .	2.09	2,380	7.5
0.002	0.25	1.97	2,530	8.0
0.003	. . .	2.00	2,415	8.5
0.003	0.25	1.86	2,505	7.5
0.004	. . .	1.92	2,515	9.0
0.004	0.25	1.81	2,505	7.5
0.005	. . .	1.92	2,500	9.0
0.005	0.25	1.86	2,515	7.5

[a] A mixture of southwestern winter wheat patent flour + 40% clear. Flour prepared and treated in the laboratory of Sterwin Chemicals, Inc.

Combinations of chlorine dioxide and acyclic acetone peroxides gave the best maturing with 0.002–0.003% acetone peroxides and 0.25 g. chlorine dioxide. In these tests the best bread was obtained by a flour treatment with 0.004–0.005% hydrogen peroxide equivalents of acetone peroxides alone. Bread volume response was from 2,440 cc. for the control to 2,515 cc., and grain improvement at these levels was excellent.

Treatment of first clear flour with acyclic acetone peroxides has given interesting results. An example is provided by the data in Table IX, which concern maturing a southwestern hard winter wheat clear flour with acetone peroxides alone. A comparison was made with chlorine dioxide alone. Clear flours are normally heavily

TABLE IX

TREATMENT OF FIRST CLEAR FLOUR[a] WITH ACETONE PEROXIDES AND CHLORINE DIOXIDE

Flour Treatment: H_2O_2 Equiv. Acetone Peroxides	Flour Treatment: ClO_2	Carotene	Bread: Volume	Bread: Grain (10)	Bread: Texture (15)	Bread: Crumb Color (10)
%	g/cwt	ppm	cc			
Control	. . .	3.06	2,500	6.5	10.0	5.0
0.003	. . .	3.02	2,550	7.0	10.0	5.5
0.006	. . .	2.78	2,625	7.5	11.0	6.3
. . . .	0.75	2.78	2,525	7.0	10.0	6.0
. . . .	1.25	1.29	2,625	8.0	12.0	7.0

[a] Southwestern hard winter.

pigmented. This one contained 3.06 p.p.m. carotene. While 0.003% hydrogen peroxide equivalents had a noticeable improver effect, it was inadequate. However, 0.006% gave good loaf volume response and improved the grain as compared with the control.

It happened that chlorine dioxide, 0.75 g./cwt., caused the same color removal as 0.006% acetone peroxides, but loaf volume response to chlorine dioxide at this level was not so good, nor were bread characteristics.

When chlorine dioxide alone (1.25 g./cwt.) was applied, color removal was excellent for a clear flour, and loaf volume response equal to 0.006% acetone peroxides. This bread was the best in this series.

Stability of Starch Concentrate. In the drying process which follows the incorporation of the liquid reaction mixture with starch, water, some residual hydrogen peroxide, acetone, and acyclic dimer are vaporized. The acyclic dimer is very volatile, whereas the monomer is not. For this reason, the organic peroxides in the starch product may consist of 85–95% monomer, 5–15% acyclic dimer, and fractional percentages, if any, of the acyclic trimer. Thus, stability is chiefly a function of the monomer and acyclic dimer.

The starch peroxide composition showed excellent stability under storage at 50°F. (10°C.) for an almost unlimited time. There was no appreciable loss in activity in 12 months. At 70°–75°F. stability was also good for many months; a loss of 0–10% may occur in 90–100 days. For commercial applications this loss would not be significant. As the temperature of storage is increased above room temperature, stability decreases. For this reason, cool storage is recommended for the starch concentrate, about 70°F. or below.

Effect of Acyclic Acetone Peroxides on Enrichment Vitamins. Because some flours are enriched, it is of importance to examine the effect of the acetone peroxide composition on natural vitamins of flour and those supplied by enrichment. Unbleached, unenriched flour and unbleached, enriched flour were treated with acyclic acetone peroxides at the rate of 0.003 and 0.03% hydrogen peroxide equivalents. The lower level represents an average treatment; the higher, an exaggerated level of ten times normal dosage giving unsalable bread.

Samples were assayed[7] immediately after treatment and after 30, 90, and 180 days' storage at 25°C. for thiamine, riboflavin, niacin, pantothenate, and pyridoxine. It was concluded that "the new process for the maturation of bread flour is without effect on its vitamin

[7] Assays performed by Food and Drug Research Laboratories, Inc., Maspeth, N.Y.

content or stability over a period of at least 180 days at room-temperature storage."

Residue Studies.[8] The acyclic acetone peroxide product was synthesized at Tracerlab, Inc., with radioactive acetone bearing C^{14} distributed in both the carbonyl and methyl positions. Flour was treated with it and then bread was baked. The bread was sliced, air-dried, ground into crumbs, and analyzed for C^{14} by wet combustion to carbon dioxide and subsequent measurement of radioactivity in standard ionization chambers. The average C^{14} activity found in six bread samples was 0.6 p.p.m. of acetone equivalents on the air-dry basis and 0.5 p.p.m. calculated to the usual 62% solids basis for bread.

Literature Cited

1. ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS. Official methods of analysis (9th ed.), p. 168. The Association: Washington, D.C. (1960).

[8] Work conducted at Tracerlab, Inc., under the supervision of Food and Drug Research Laboratories, Inc., and J. R. Short Milling Co.