"MOISTURE EXPANSION" OF CERAMIC WHITE WARE

By R. F. Geller and A. S. Creamer

ABSTRACT

This report supplements the reports of a number of investigations of "moisture expansion." The relative susceptibility of various materials, individually and as consitituents of bodies, was studied, as was also the effect of the temperature to which the specimens had been heated. The relative susceptibility to moisture expansion of the principal ingredients of ceramic bodies appears to be approximately as follows: Fused feldspar, 2.4; calcined clay, 0.40; unfused feldspar, 0.20; calcined flint, 0.06. The expansion caused by reaction with moisture may be decreased at temperatures as low as 120° C. The removal of the moisture is relatively sluggish at 120° C., proceeds faster as the temperature is raised, and apparently may be completed at 250° C. The marked difference is susceptibility to moisture expansion of fused and unfused feldspar has an important effect on ceramic bodies. The data indicate that at temperatures below approximately "cone 8" and absorptions above 10 per cent the ratio of fused to unfused feldspar is the predominating factor determining the resistance to moisture expansion while at higher temperatures and lesser absorptions the relative absorption is the predominating factor. The data obtained offer no evidence regarding the nature of the reaction involved.

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I. INTRODUCTION

This report supplements the reports of many investigations [1] which have been made during the past six years on the general subject of moisture expansion or expansion due to the action of water. It was undertaken primarily to determine which of the usual constituents of white-ware bodies are most susceptible to moisture expansion.

[1] G. E. Merritt and C. G. Peters, J. Am. Ceram. Soc., vol. 9 (6), p. 332, 1926; H. G. Schurecht, vol. 11 (5), p. 271, 1928, and vol. 12 (2), p. 118, 1929; H. G. Schurecht and G. R. Pole, vol. 12 (9), p. 596, 1929; R. G. Mills, vol. 13 (12), p. 903, 1930; H. H. Holscher, vol. 14 (3), p. 207, 1931; H. G. Schurecht and G. R. Pole, vol. 14 (4), p. 313, 1931, and B. S. Jour. Research, vol. 3 (RP98), August, 1929; O. S. U. Eng. Exp. Sta. Cir. Nos. 18, J. Otis Everhart, and 22, H. H. Holscher.

II. MATERIALS AND METHODS

1. COMMERCIAL BODIES

Dinner plates of the same quality as supplied regularly to the trade, and representing the product of 16 manufacturers of earthenware, were available for the study of commercial bodies. The specimens for water-absorption determinations were irregularly shaped pieces broken from the plates and tested without removing the glaze, using three specimens of each brand. The determinations were made by the so-called 5-hour boiling test in accordance with the method prescribed in Federal specification No. 243a for vitrified chinaware. Indirect moisture-expansion measurements were made by means of the interferometer according to the method described by Schurecht.[2] These indirect measurements are based on the assumption that the difference in total thermal expansion obtained at 400° C. (or at a higher temperature) in the first and second tests of specimens which have had the opportunity of reacting with moisture, either during prolonged storage or in an autoclave, is a measure of the moisture expansion.

2. EXPERIMENTAL BODIES

The compositions of the experimental bodies, together with the heat treatment received, are given in Table 1 and in part A, Table 2. The potter's flint and the feldspar (No. 3)[3] were composed of particles varying in size from 10μ to 80μ, the separation from coarser and finer particles having been made by means of a No. 325 United States standard sieve and an air elutriator.[4] The clay (Georgia kaolin) was not sized in the elutriator because it "balled" in the apparatus and made the separation of any particular range of particle sizes impracticable.

The mixtures were made into specimens 6 inches long and one-half inch in diameter. The extrusion method was used to form the specimens of bodies containing clay, while the flint-feldspar series, containing gum tragacanth as a binder, were tamped. The specimens which had been heated as described in section B, Table 1, were dried at 115° ± 5° C., weighed, measured, and autoclaved at 150 lbs./in.^{2} steam pressure (183° C.) for one and one-half hours while submerged in water. They were then removed from the autoclave, reweighed and remeasured before and after drying (constant in weight to 0.01 g) at 115° ± 5° C. and at 270° ± 5° C. to determine changes in weight and length due to these drying treatments. The absorption was taken as the percentage gain in weight during the autoclave treatment.[5] The changes in length resulting from the autoclave treatment and subsequent drying were measured by means of a comparator using a graduated invar bar as reference.[6] Reference points in the specimens were obtained by cementing capillary Pyrex glass tubes in small holes approximately 12 cm apart, using a hydraulic high alumina cement. The precision in these length measurements was found to be approximately ± 0.01 per cent of the length measured. Room temper-

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[2] H. G. Schurecht, J. Am. Cer. Soc., vol. 11 (5), p. 271, 1928.
[3] H. Insley, J. Am. Cer. Soc., vol. 10 (9), p. 651, 1927.
[4] J. C. Pearson and W. H. Sligh, B. S. Tech. Paper No. 48.
[5] This method of determining water absorption is believed to be fully as effective as the 5-hour boiling method because it has been shown by the authors that a 10-hour autoclave treatment at 150 lbs./in.^{2} steam pressure is as effective as 240 hours boiling. See B. S. Tech. News Bull. No. 167, March, 1931.
[6] J. C. Pearson, J. Am. Concrete Inst., 1921.

atures were noted at all readings, but did not vary sufficiently to justify correcting the measurements for this factor.

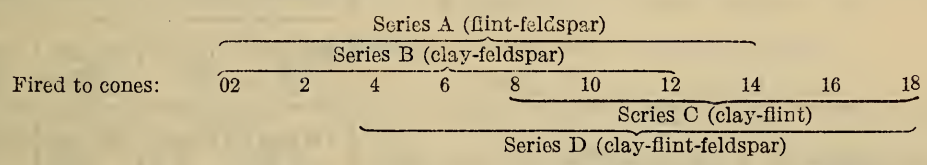
TABLE 1.—*Experimental bodies and heat treatments*

A. BODIES

[Three specimens of each mixture for each heat treatment]

Series A			Series B			Series C			Series D			
Body No.	Flint	No. 3 Feldspar	Body No.	Clay	No. 3 Feldspar	Body No.	Clay	Flint	Body No.	Clay	Flint	No. 3 Feldspar
	Per cent	*Per cent*		*Per cent*	*Per cent*		*Per cent*	*Per cent*		*Per cent*	*Per cent*	*Per cent*
A1	80	20	B1	80	20	C1	80	20	D1	20	40	40
A2	60	40	B2	60	40	C2	60	40	D2	40	30	30
A3	50	50	B3	50	50	C3	50	50	D3	50	25	25
A4	40	60	B4	40	60	C4	40	60	D4	60	20	20
A5	20	80	B5	20	80	C5	20	80	D5	80	10	10

B. HEAT TREATMENT



3. INDIVIDUAL MATERIALS

The individual raw materials included: (*a*) Feldspars Nos. 3 and 18; (*b*) pulverized quartz obtained by crushing a sample of standard Ottawa sand; (*c*) washed kaolin from Georgia containing about 4 per cent of impurities, such as mica and quartz; (*d*) two samples of lepidolite;[7] (*e*) Cornwall stone as supplied to the pottery trade; and (*f*) Lemoor china clay. Three samples of the china clay were available: (*a*) The original clay; (*b*) a granular separate containing particles larger than 1μ in diameter; and (*c*) a "colloidal" separate containing particles less than 1μ in diameter.[8] With the exception of the feldspar and the Lemoor china clay used in the determination of the effect of particle size, each of the individual materials tested as powders was heated at approximately 1,225° C.[9] in a small gas-fired furnace, cooled rapidly, and crushed to pass a No. 120 United States standard sieve. It is realized that differences in particle size (or ratio of surface to unit weight) introduce an unmeasured variable which may lessen appreciably the significance of the values. For this reason the materials were ground with a mortar and pestle and sieved repeatedly as the grinding progressed so as to produce a minimum of fines.

[7] The following chemical compositions were obtained from the concerns furnishing the lepidolite:

	SiO_2	Al_2O_3	Fe_2O_3	MnO	K_2O	Li_2O	Na_2O	F	H_2O
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
Sample A	49	27	0.3	0.5	8–10	4–5	2	--------	1.5
Sample B	63	24	.05	.35	6.8	2.9	1.5	1.5	.2

[8] The fractions were obtained by K. Langenbeck during the course of an investigation at the Bureau of Standards, the results of which have not been published.

[9] This temperature was chosen because it lies between the temperatures used by earthenware and vitrified chinaware manufacturers to mature their ware.

The determination of the relative degree of reaction of individual materials with moisture was based on weight change, assuming that expansion (or volume change) would be proportional to weight change. The crushed samples were dried to constant weight [10] at 115° ± 5° C., exposed to water vapor at 150 lbs./in.^{2} steam pressure for five hours (precautions being taken to prevent condensation of moisture on the samples), weighed immediately on removal from the autoclave and again after drying at 115° ± 5° C. and at 250° ± 10° C.

TABLE 2.—*Effect of autoclave treatment on individual materials*

A. 1 BY 1 BY 5 INCH BARS (THREE SPECIMENS OF EACH BODY) AUTOCLAVED FOR THREE HOURS AT 150 LBS./IN.^{2} STEAM PRESSURE (183° C.)

	Temperature of heating			
	Cone 5 to 6 (approximately 1,180° C.)		Cone 8 to 9 (approximately 1,230° C.)	
	Expansion	Absorption	Expansion	Absorption
	Per cent	*Per cent*	*Per cent*	*Per cent*
50 per cent feldspar No. 18—50 per cent flint	0.03	6.0	0.00	1.4
50 per cent feldspar No. 3—50 per cent flint	.15	15.1	.03	3.9
Florida kaolin [1]	.04	14.8	.02	13.3
Georgia kaolin [1]	.03	20.7	.02	17.8

B. POWDERED SPECIMENS HEATED AT 1,225° C., RECRUSHED AND AUTOCLAVED FOR FIVE HOURS AT 150 LBS./IN.^{2} STEAM PRESSURE

		Weight gain during autoclave treatment	Percentage of water absorbed during autoclave treatment which was removed by drying at—	
			115°± 5° C.	250°± 10° C.
		Per cent	*Per cent*	*Per cent*
Feldspar No. 3	1	2.4	32	84
	2	2.4	30	95
	3	2.5	30	93
	4	2.5	----------	----------
Quartz	1	.06	67	92
	2	.06	69	86
	3	.05	17	81
	4	.05	15	78
Kaolin	1	.46	64	100
	2	.34	37	91
Lepidolite	A 1	1.7	35	84
	A 2	1.6	33	83
	B 1	1.3	27	78
	B 2	2.0	29	77
Cornwall stone	1	1.4	23	76
	2	1.4	22	74
	3	1.3	20	73

[1] Taken from Table 7 of H. H. Holscher's paper. (See footnote 12, p. 299.)

[10] In this phase of the work the specimens were considered to have reached constant weight when the weight difference after 2-day intervals of drying did not exceed 0.5 mg.

III. RESULTS

1. COMMERCIAL BODIES

(a) MOISTURE EXPANSION OF UNAUTOCLAVED SPECIMENS

As a preliminary study, specimens of five brands (Nos. 1, 5, 6, 14, and 16) were taken from glazed plates which had been stored in the laboratory for several months. The moisture expansion was found to vary from 0.03 to 0.06 per cent for four brands which had water absorptions of from 9.4 to 12.5 per cent; the other brand (No. 1) showed no moisture expansion and the water absorption was 2.0 per cent. Typical curves are given in Figure 1 and the values in Table 3.

Since the moisture expansion tests of unautoclaved glazed plates were made on specimens of the body immediately after being taken from the plates, it is indicated that the expansion noted had taken place regardless of the supposedly impervious coating of glaze.[11] The data, therefore, justify the conclusion that glazed earthenware may be subject to crazing caused by penetration of moisture through the glazed and the resultant moisture expansion of the body.

(b) MOISTURE EXPANSION OF AUTOCLAVED SPECIMENS

One specimen of each of the 16 brands was autoclaved at 150 lbs./in.^{2} steam pressure for one hour, the pieces being immersed. Typical results are shown by the curves in Figure 1. For any one specimen the expansions during the second and third heatings were practically identical. The curves in Figure 2 show also that the contraction during the cooling, following the first heating of an autoclaved specimen, is practically along the same curve as the expansion during the second heating. The data indicate that the first heating and the first cooling are sufficient to give the desired information. Pertinent data obtained in this phase of the investigation are summarized in Table 3 and the calculated moisture expansions are plotted against percentage water absorption in Figure 3. Although the bodies are all of one type and probably had been matured at about the same temperature, there are undoubtedly sufficient differences in composition and structure to affect the porosity-expansion relation. As shown by data given in this paper, feldspar alone may be an important factor.

TABLE 3.—*Effect of moisture on specimens either stored in laboratory air or autoclaved*

Brand No.	Water absorption	Moisture expansion after storage [1]	Moisture expansion after autoclave [2]	Brand No.	Water absorption	Moisture expansion after storage [1]	Moisture expansion after autoclave [2]
	Per cent	*Per cent*	*Per cent*		*Per cent*	*Per cent*	*Per cent*
1	2.	0	0.05	9	11	----------	0.12
2	7.5	----------	.07	10	11	----------	.11
3	9.	----------	.07	11	11	----------	.14
4	9.5	----------	.08	12	11	----------	.09
5	9.5	0.03	.11	13	12	----------	.08
6	10	[3] .04	.10	14	12	0.06	.12
7	10	----------	.10	15	12	----------	.09
8	10	----------	.08	16	12	.03	.09

[1] Difference in total expansion at 400° C. between first and second heating of unautoclaved specimen which had been taken from a whole plate stored in the laboratory under normal atmospheric conditions for several months.

[2] Difference in total expansion at 400° C. between first and second heating of specimen autoclaved for one hour in steam at 150 lbs./in.^{2} pressure.

[3] Especially selected and tested for unbroken glaze before specimen was taken from plate.

[11] It was shown by Schurecht (footnote 1, p. 291) that glazes may undergo moisture expansion. This is further evidence that they are not impervious to moisture.

(c) EFFECT OF VARIOUS DRYING TEMPERATURES ON THE LENGTH OF AUTOCLAVED SPECIMENS

A number of observations were made by holding autoclaved specimens at several temperatures (maintaining the temperatures as nearly constant as possible) and observing length changes. Seventeen tests

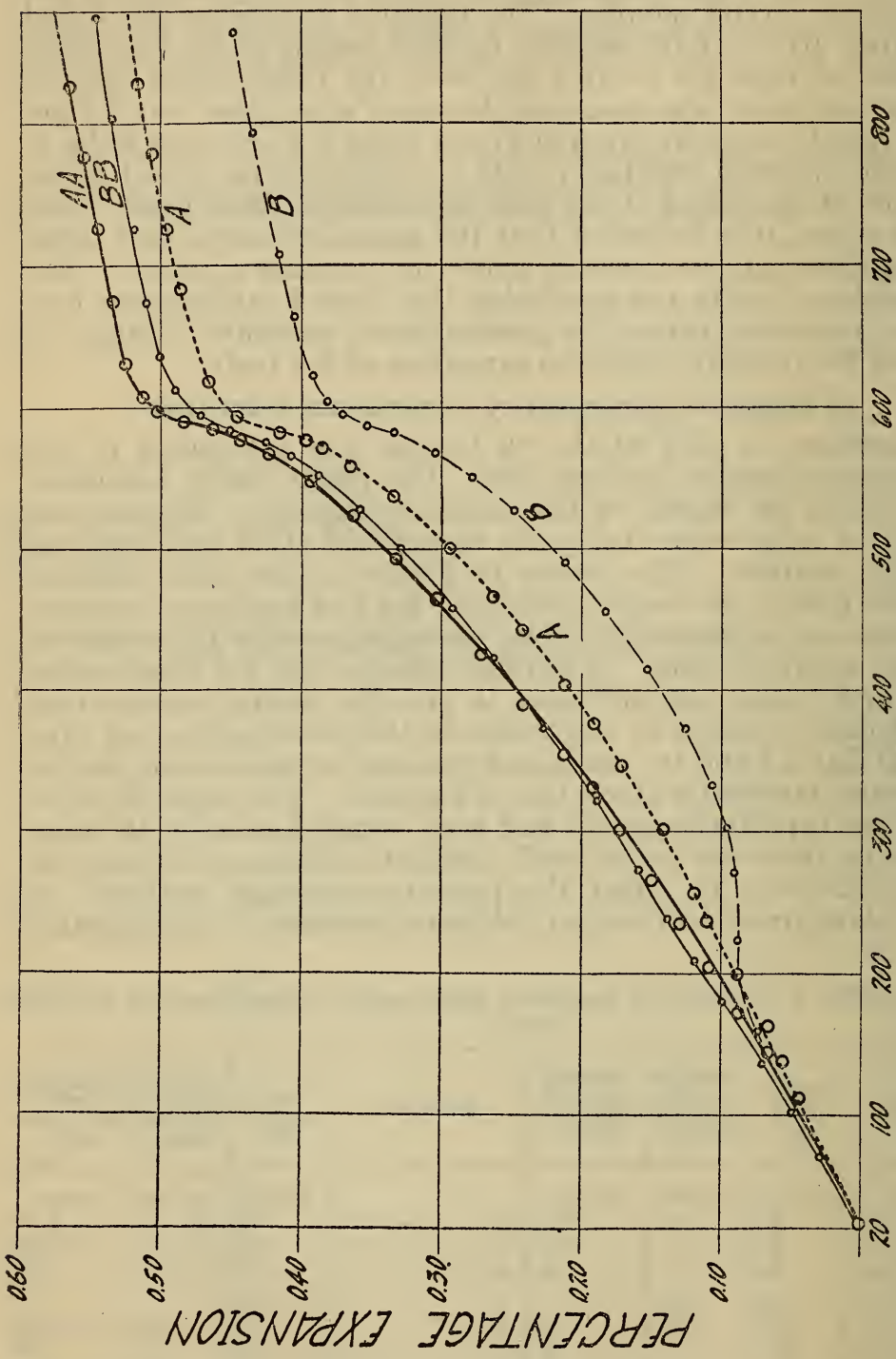


FIGURE 1.—*Thermal expansion determinations on specimens of two pieces of Brand No. 6*

Plate A had been stored in the laboratory for several months and plate B had been autoclaved for one hour at 150 pounds per square inch steam pressure. Curves *A* and *B*, first heating; curves *AA* and *BB*, second and third heating. Estimated percentage moisture expansions are given in Table 3.

were made on three different bodies. These tests were modifications of the following, the results of which are typical and are discussed in detail:

1. A specimen of brand No. 5 was "autoclaved" for two hours at 150 lbs./in.^{2} steam pressure while immersed in water. Length changes

were then observed while the specimen was heated in a furnace to approximately 118° C. and held for 5½ hours, reheated to approximately 180° C. and held for 1½ hours and again heated to 246° C. and held for 1½ hours and finally heated to 600° C. The specimen was permitted to cool to room temperature after each heat treatment.

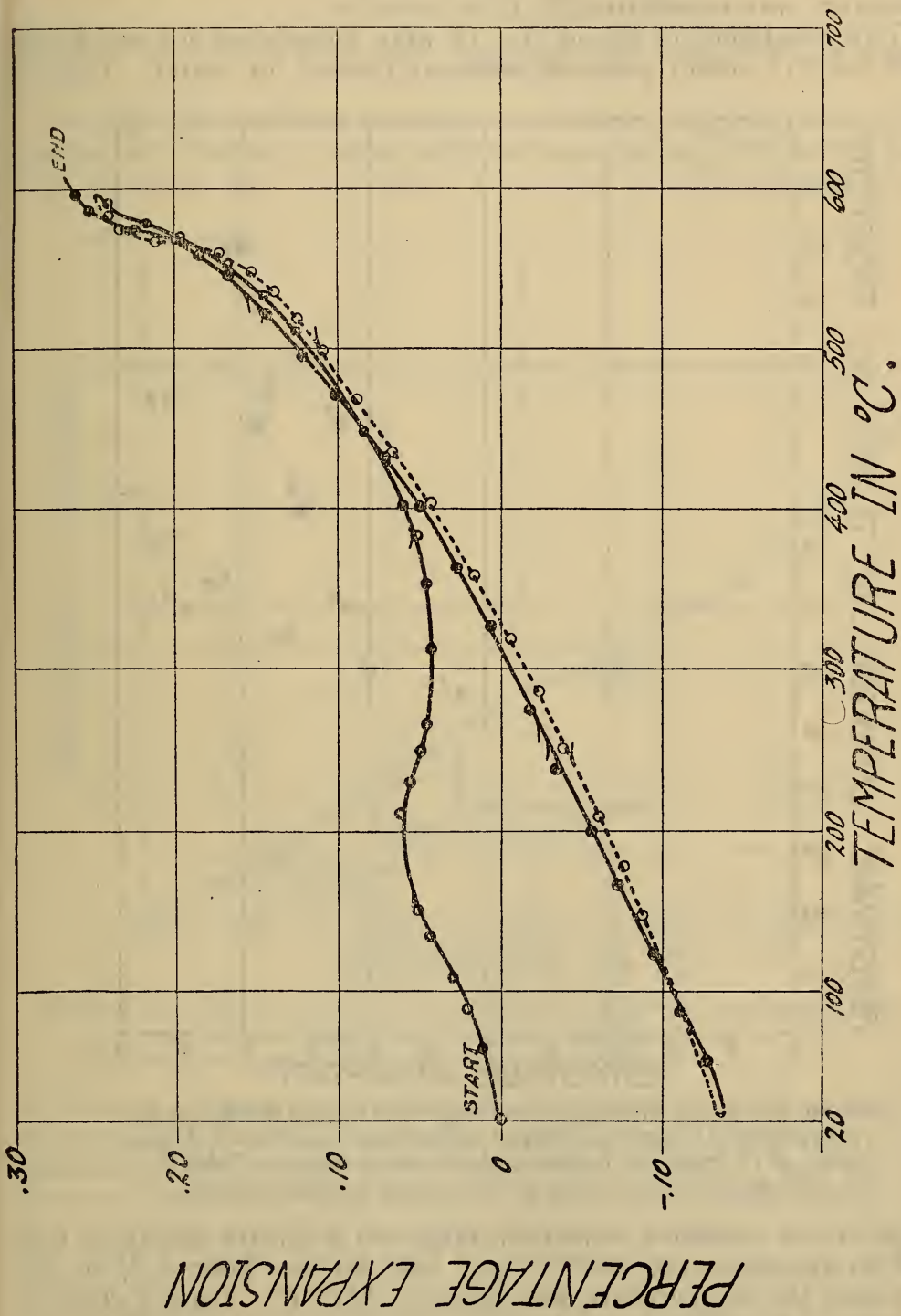


FIGURE 2.—*Thermal expansion curves showing that the contraction of an autoclaved specimen when cooled after the first heating is practically the same as the expansion during the second and third heating*

Test made on a specimen of commercial body No. 3 autoclaved for two hours at 150 pounds per square inch steam pressure.

The results obtained are shown by the curves in Figure 4. The specimens were held at each temperature until further length changes were negligible.

2. Length changes of a second specimen of brand No. 5 were observed while the specimen was held at approximately 245° C. for

five hours. As is evident from the curve in Figure 5, the rate of contraction had decreased considerably at the end of the 5-hour period, but conditions had not reached equilibrium. The specimen, after cooling to room temperature, was again heated to approximately 245° C. and held for four and one-half hours, after which the temperature was raised to 325° C.

3. Two specimens of brand No. 15 were autoclaved for two hours at 150 lbs./in.^{2} steam pressure while immersed in water. Length

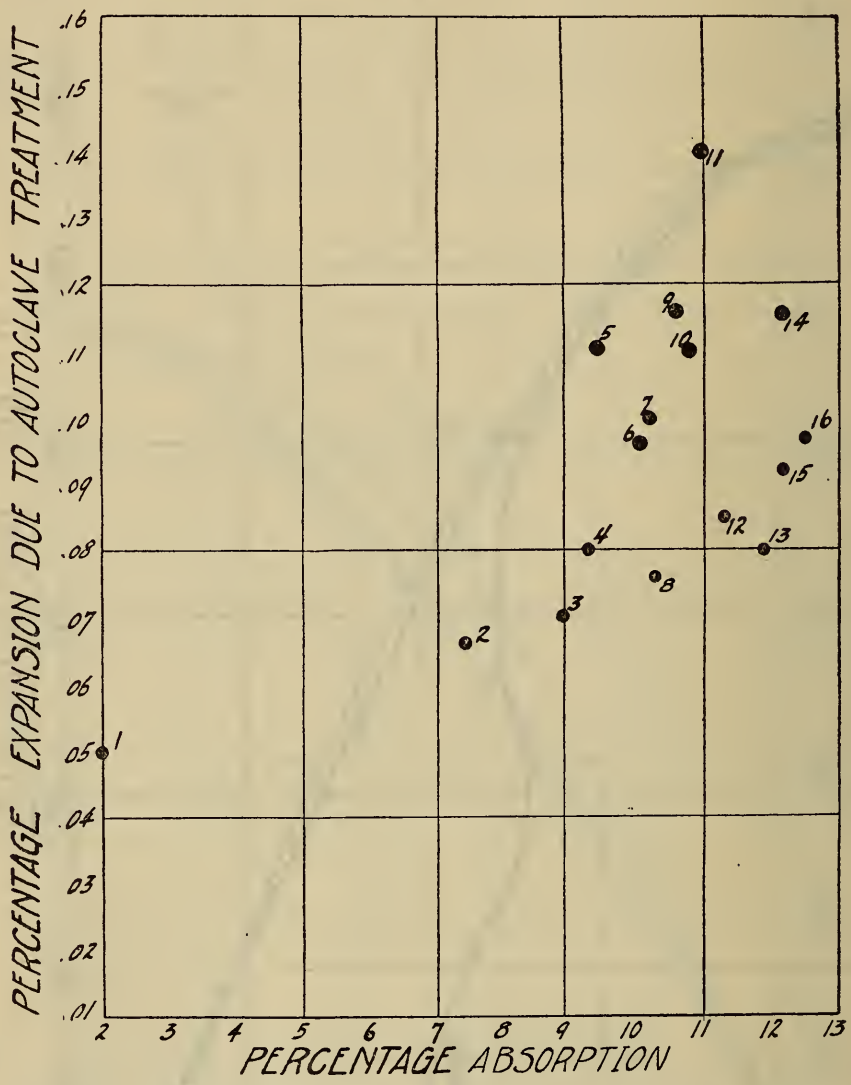


FIGURE 3.—*Water absorption and difference in total thermal expansion at 400° C. (during first and second tests) of autoclaved specimens of 16 brands of commercial whiteware (data in Table 3)*

The difference in total expansion at 400° is considered as moisture expansion.

changes of one specimen were then observed while the specimen was heated to approximately 260° C. and held for two hours (*A*, fig. 6), after which the temperature was raised to 380° C. Length changes of the second specimen were observed while heating it to 680° C. at a constant rate of temperature increase (*B*, fig. 6) permitting it to cool to room temperature, and again heating to 680° C. at a constant rate of 3° C. per minute.

The information given in Figures 4, 5, and 6 indicates that the reactions induced by heating autoclaved specimens, and evidenced by contraction, are relatively sluggish at 120° C., proceed faster as the temperature is raised, and are markedly accelerated at temperatures above 230° C.

2. EXPERIMENTAL BODIES

(a) EFFECT OF COMPOSITION, HEAT TREATMENT AND ABSORPTION ON MOISTURE EXPANSION

As originally outlined this phase of the investigation included the making and testing of 435 specimens representing 20 different bodies, each of which was to be heated at several temperatures as specified

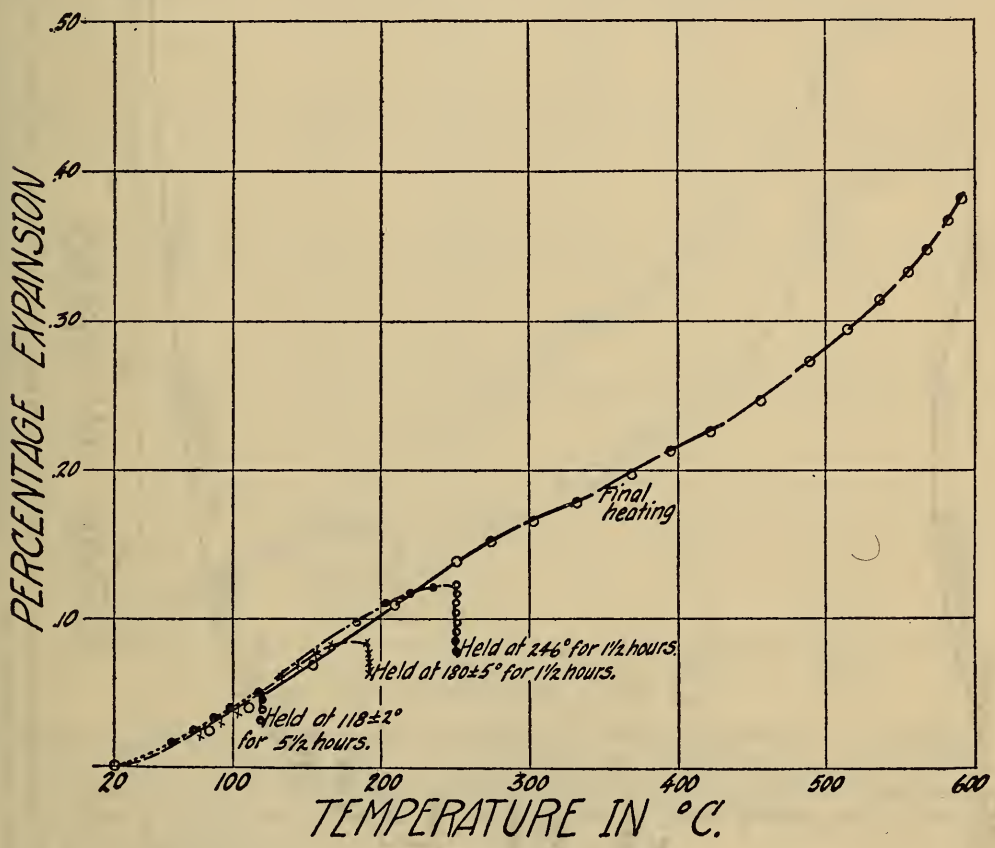


FIGURE 4.—*Length changes of an autoclaved specimen of commercial body No. 5 when held at various temperatures*

All tests made on the same specimen after one autoclave treatment.

in Table 1. Three specimens were prepared for each heat treatment of each body.[12] Because of the extreme weakness of many of the specimens, particularly the *A* series (flint feldspar), and the specimens of the *B* series (clay feldspar) heated at the lower temperatures, only 215 were available for the entire series of tests. Had this been anticipated, fewer bodies and more specimens of each would have

[12] It has been shown by previous investigators (footnote 1, p. 291) that the relative porosity is an important factor in determining the extent of the reaction of ceramic bodies with moisture. Obviously the relative moisture expansions of two bodies varying in composition are not comparable unless the porosity is the same both in extent and in nature. Not only is the nature of the pore space difficult of determination (ratio of fine to coarse pores, etc.), but it would be practically impossible to develop the same porosity in two bodies of the whiteware type, varying in composition, without using a different heat treatment for each. This introduces a third variable which, it was anticipated, would be a significant factor. It was this practical difficulty of changing only one variable at a time that prompted the study of such a large number of specimens in the hope that the data could be studied as group averages which might indicate the effect to be expected by changing either composition, porosity, or heat treatment.

been made. However, such values as could be determined are presented in Table 4. This table gives the average percentage moisture expansion and water absorbed for each body during the autoclave treatment, but only the group averages of each series of bodies for the determinations which were made after the drying treatments.

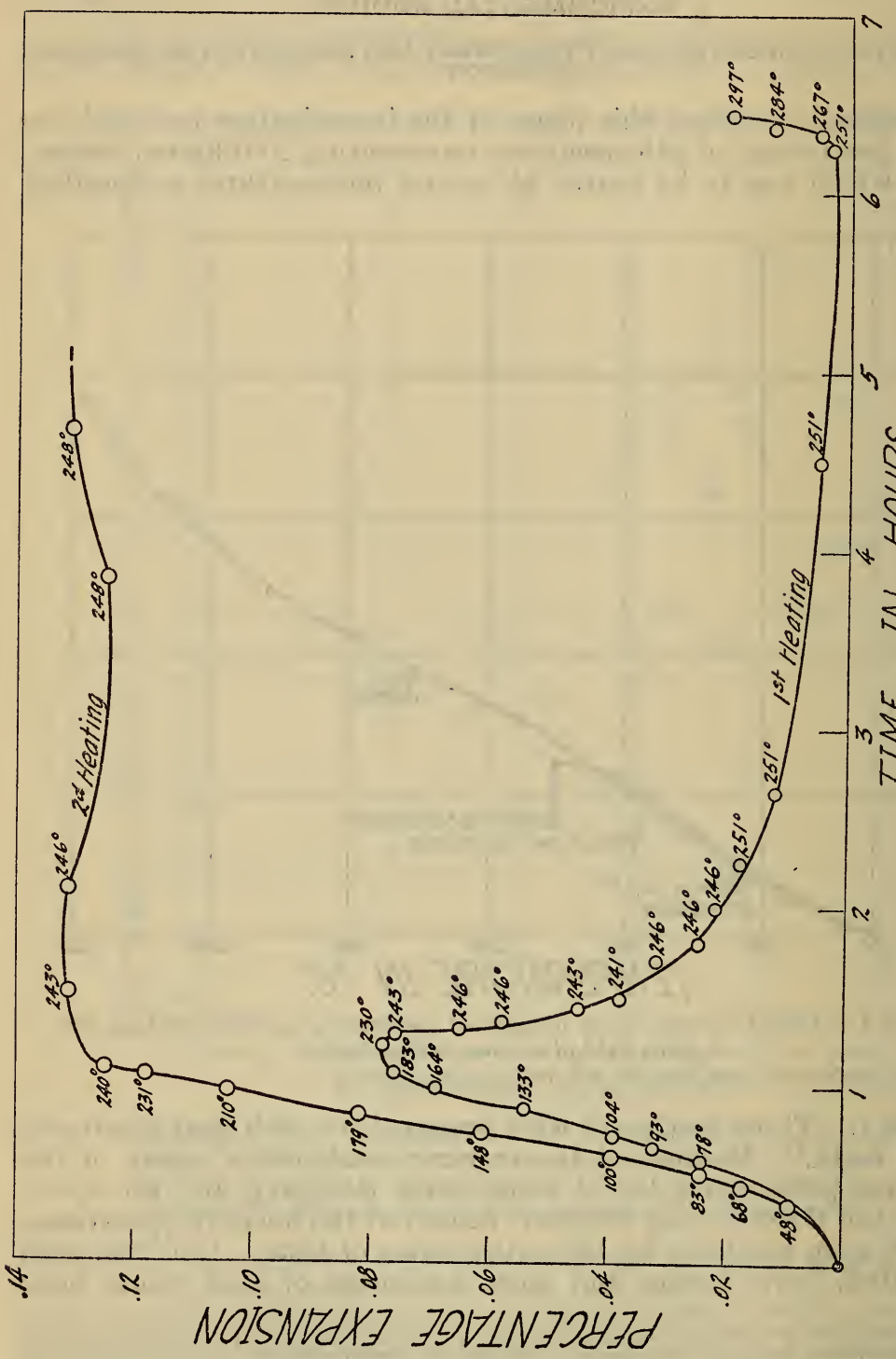


FIGURE 5.—*Thermal expansions of an autoclaved specimen of commercial body No. 5*

The curve obtained during the first heating indicates that the vapor pressure or dissociation pressure of that water in an autoclaved specimen which causes moisture expansion (the removal of which is accompanied by contraction) increases sharply as the temperature is raised above 180° C.

As stated in footnote 12, the analysis of data, obtained by heating a number of different ceramic bodies to a series of temperatures and subjecting them to moisture in an autoclave at elevated temperatures, is made difficult by the fact that one factor can not be changed without changing others. In studying the data (Table 4) it should

be remembered that only one clay, one feldspar, and one flint were investigated. Also, little significance can be attached to certain irregular absorption-temperature relations because the bodies were heated by the draw-trial method, which is comparatively rapid and not suitable for a study of absorption-temperature relations.

The length measurements show that autoclaved specimens of earthenware which have expanded due to reaction with moisture apparently return to their original length when held for a reasonably short time at 250° to 270° C. in air and under atmospheric pressure. However, the most noticeable development is the unique behavior of the *C* (or clay-flint) series of bodies. Although the compositions

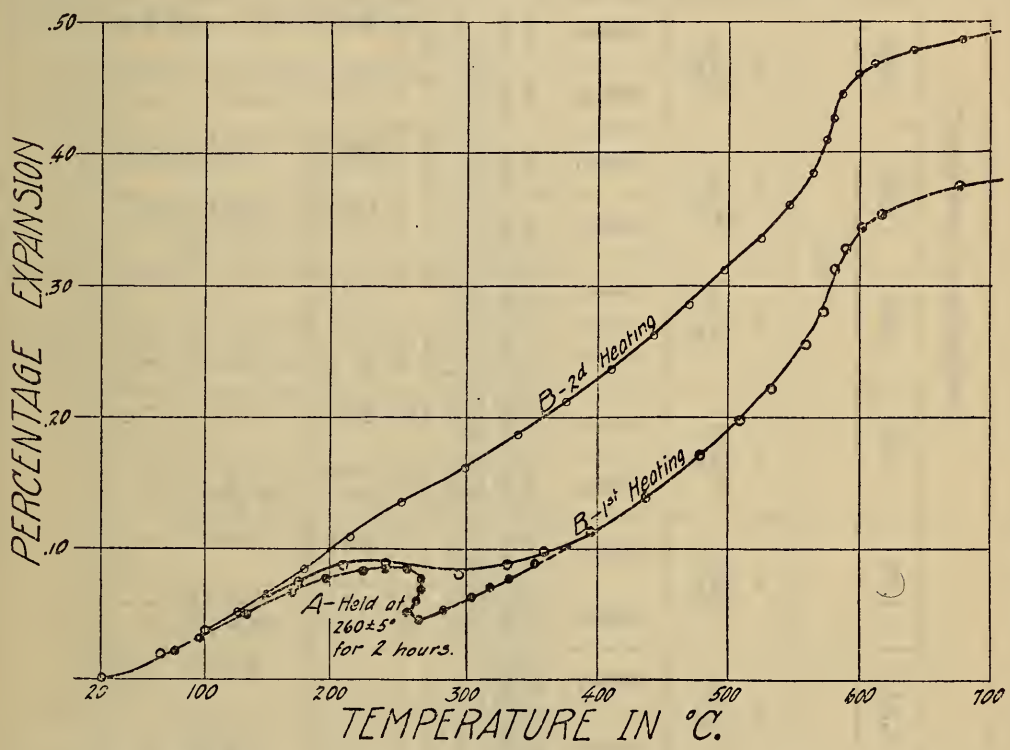


FIGURE 6.—*These thermal expansion curves for commercial body No. 15 (together with the curves given in figs. 4 and 5) show that subjection to a temperature of approximately 250° C. for a relatively short time is sufficient to overcome the effect of autoclave treatment on length change, while prolonged treatment at lower temperatures is not*

are very refractory, and the specimens highly porous, even after heating to cone 18, the only specimens showing a significant length change, or moisture expansion, after autoclaving are those heated to cone 8. This indicates that a body composed of the clay used in this investigation (Georgia kaolin) and of flint would be very resistant to moisture expansion even though the porosity was relatively high. The data obtained by Holscher [13] would indicate that this might not be true for all clays.

[13] J. Am. Cer. Soc., vol. 14 (3), p. 207, 1931.

TABLE 4.—*Percentage change in weight and length after autoclave treatment and also after subsequent drying*

Body	Heat treatment (approximate °C.)																			
	1,100		1,125		1,170		1,190		1,215		1,250		1,300		1,380		1,440		1,490	
	Cone																			
	02		2		4		6		8		10		12		14		16		18	
	Change [1] in—		Change [1] in—		Change [1] in—		Change [1] in—		Change [1] in—		Change [1] in—		Change [1] in—		Change [1] in—		Change [1] in—		Change [1] in—	
	Weight	Length	Weight	Length	Weight	Length	Weight	Length	Weight	Length	Weight	Length	Weight	Length	Weight	Length	Weight	Length	Weight	Length
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
Flint-feldspar:																				
A1	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------
A2	------	------	------	------	29.5*	0.04*	28.0*	0.06*	21.5*	0.05*	------	------	------	------	------	------	------	------	------	------
A3	------	------	------	------	27.5*	.07*	27.5	.07	17.0	.08	------	------	------	------	------	------	------	------	------	------
A4	------	------	------	------	------	------	29.0	.05	15.0*	.14*	7.5*	0.06*	0.0*	0.00*	------	------	------	------	------	------
A5	------	------	------	------	25.5*	.10*	30.0*	.05*	8.0	.12	------	------	.0	.02	0.0	0.00				
Average after autoclave [2]	------	------	------	------	27.5	.07	28.6	.06	15.4	.10	7.5*	.06*	.0	.01	------	------	------	------	------	------
Average after first drying [3]	------	------	------	------	−.3	.04	−.2	.04	.1	.06	.0*	.05*	.0	.00	------	------	------	------	------	------
Average after second drying [4]	------	------	------	------	−.5	.02	−.4	.01	−.1	.02	−.3*	−.02*	−.1	.00	------	------	------	------	------	------
Clay-feldspar:																				
B1	25.0*	0.10*	------	------	------	------	19.5	.11	------	------	------	------	7.0*	.08*	------	------	------	------	------	------
B2	25.0	.08	21.5*	.13*	12.0	.17	------	------	7.0	.12	6.5	.12	4.0	.10	------	------	------	------	------	------
B3	20.5	.09	20.0	.14	9.5	.18	11.0	.20	4.5*	------	4.0*	.05*	------	------	------	------	------	------	------	------
B4	20.0	.06	15.0	.15	10.0	.18	9.0	.18	5.0	.14	4.0*	.05*	.0	.00	------	------	------	------	------	------
B5	23.5*	.10*	22.0	.11	15.0*	.14*	16.0	.13	3.0*	.01*	0.0	.00	.0	.00	------	------	------	------	------	------
Average after autoclave [2]	22.8	.09	20.0	.13	11.5	.17	14.0	.16	5.0	.09	3.5	.06	3.0	.09	------	------	------	------	------	------
Average after first drying [3]	.0	.03	.2	.05	.3	.07	.2	.07	.3	.05	.1	.04	.1	.02	------	------	------	------	------	------
Average after second drying [4]	−.2	.00	−.2	.00	−.2	.01	−.1	.00	−.1	−.01	−.2	.00	−.1	.00	------	------	------	------	------	------

Clay-flint:																				
C1	------	------	------	------	------	------	------	------	22.0	.08	17.5	.02	15.0	.00	13.5	.02	12.0	0.01	8.5	0.00
C2	------	------	------	------	------	------	------	------	22.0	.07	------	------	20.0	.00	17.5	.00	17.0*	.00*	13.5*	*.00
C3	------	------	------	------	------	------	------	------	22.5	.04	21.0	.00	19.5	.02	18.9	.00	18.5	.01	14.5	.00
C4	------	------	------	------	------	------	------	------	22.5	.09	21.5*	.00*	20.5	.00	20.0	.00	20.0	.00	16.5	.00
C5	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	22.5*	.03*	23.5	.00
Average after autoclave [2]	------	------	------	------	------	------	------	------	22.5	.07	19.0	.01	19.0	.00	23.5	.00	18.0	.01	15.5	.00
Average after first drying [3]	------	------	------	------	------	------	------	------	−.1	.03	−.1	.01	.0	.00	−.1	.00	−.1	.00	.0	.00
Average after second drying [4]	------	------	------	------	------	------	------	------	−.2	.00	−.2	.00	−.1	.00	−.1	.00	−.1	.00	−.1	.00
Clay-flint-feldspar:																				
D1	------	------	------	------	19.5	.17	20.0*	.27*	16.0	.13	11.0	.07	3.0	------	1.0	.06	------	------	------	------
D2	------	------	------	------	17.0	.11	17.0*	.21*	14.0	.14	11.5*	.06	8.5	.08	5.0	.09	------	------	------	------
D3	------	------	------	------	17.0	.17	15.5	.15	13.0	.12	11.5	.09	7.0	.09	5.5	.10*	.3	.00	.1	.00
D4	------	------	------	------	15.5*	.13*	16.0*	.15*	13.0	.13	12.0	.10	8.5	.04	6.0	.02	.2	.03	.2	.00
D5	------	------	------	------	23.0	.09	------	------	13.5	.09	12.0	.06	9.0	.05	6.5	.04	.1	.02	.0	.00
Average after autoclave [2]	------	------	------	------	18.5	.13	17.0	.19	14.0	.12	11.5	.08	7.0	.06	5.0	.06	.2	.02	.1	.00
Average after first drying [3]	------	------	------	------	.1	.06	.1	.09	.0	.04	------	.02	.2	.03	.0	.03	.0	.00	.0	.00
Average after second drying [4]	------	------	------	------	−.3	.00	−.2	.02	−.2	−.02	------	−.02	−.1	.00	−.2	.01	−.1	.00	.0	.00

[1] The negative values for averages 3 and 4 indicate that the specimens were lighter, or shorter, than after the preliminary drying (115° ±5° C.) before autoclave treatment.
[2] The individual values in the table of which these values are the average, represent determinations on from 1 to 3 specimens of each body and each heat treatment after autoclaving for 1½ hours at 150 lbs./in.^{2} steam pressure (183° C.). The asterisk (*) identifies values representing determinations on 1 specimen only.
[3] Average difference in weight and length, from weight and length before autoclaving, after drying at 115°±5° C.
[4] Average difference in weight and length, from weight and length before autoclaving, after drying at 270°±5° C.

A point of interest concerning the B series is the progressively decreasing resistance to expansion which was shown by the bodies fired to cones 02, 2, and 4 even though the percentage absorption also decreased. (Table 4 and fig. 7.) This led to the supposition that crystalline or unaltered feldspar is less susceptible to moisture expansion than feldspar glass. Therefore, a specimen of raw feldspar No. 3 and one of the same sample fused at 1,225° C., both ground to pass a No. 325 United States standard sieve (<80μ), were subjected to identical autoclave treatment. Also, specimens of body B4 heated to cones 02, 6, and 10 were examined petrographically.[14] It was found that the body heated to cone 02 was composed of two phases—unmelted feldspar (index of refraction=1.52) and dehydrated clay

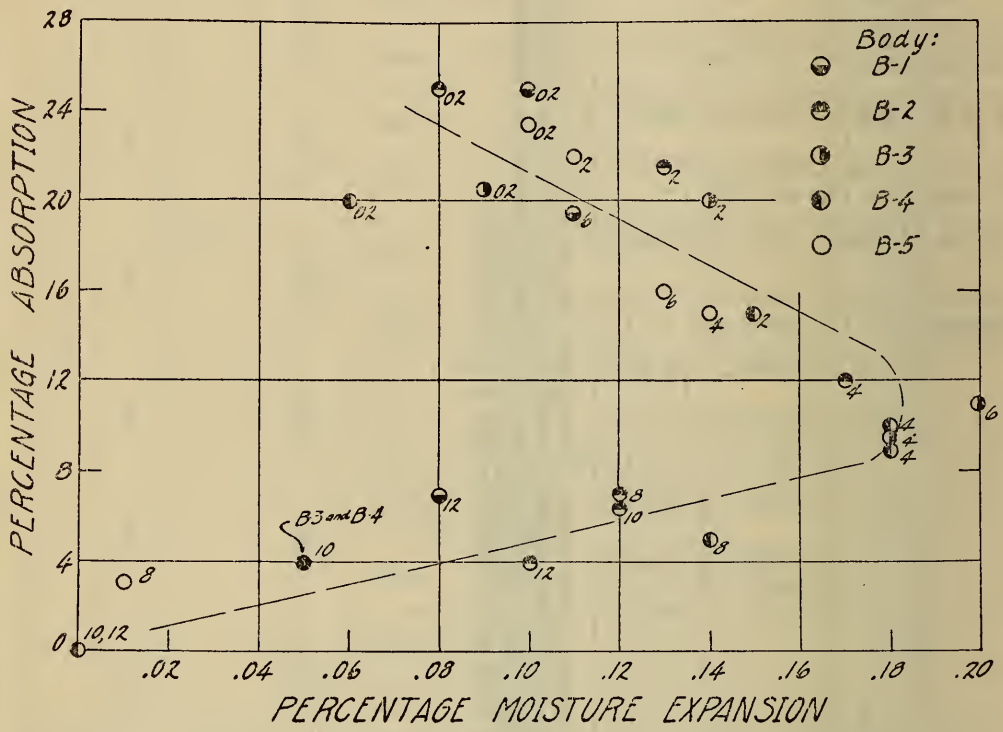


FIGURE 7.—*Relation between water absorption and moisture expansion of the clay-feldspar series of bodies (B, Table 4)*

The numbers indicate the temperatures, in terms of cones, to which the specimens were heated.

(index of refraction=1.53 to 1.54). The specimen fired to cone 6 contained three phases—melted feldspar (index of refraction=1.49), unmelted feldspar (index of refraction=1.52), and dehydrated clay (index of refraction=1.53 to 1.54). The cone 10 specimen contained the same phases as that fired to cone 6, but much more of the feldspar had melted. The absorption values obtained for raw and fused feldspar, which are given in Table 5 and supported by the other values for fused feldspar in Table 2 when considered in connection with the phase compositions of the B bodies examined may be accepted as supporting the supposition. Therefore, commercial earthenware, which is matured at about cone 6 and has an absorption averaging 10 per cent, may have a structure peculiarly susceptible to moisture expansion.

[14] Examinations made by R. H. Ewell.

3. INDIVIDUAL MATERIALS

(a) RELATIVE REACTION TO MOISTURE

Holscher [15] found that a body composed of feldspar, clay, and flint showed greater length changes after autoclave treatment than the pure clay although the absorptions were lower. This indicates that the other constituents of the bodies he investigated, namely, the feldspar or flint, may be even more reactive to moisture than the clay. Schurecht [16] investigated a series of bodies in which ball clay, feldspar and flint were varied between wide limits. He found that a mixture of 40 per cent feldspar and 60 per cent flint showed the greatest percentage length change in the series (0.188 per cent), while 60 per cent feldspar and 40 per cent flint showed only 0.100 per cent. The absorptions, however, were 13.4 per cent for the former and 5.0 per cent for the latter mixture. Whether the relatively high moisture expansion of the former mixture was induced by a reaction between the flint and feldspar, as Schurecht surmised, or because the presence of the flint produced sufficient porosity to permit the water vapors to react with a greater portion of the body had not been proven. It is worthy of note that the three bodies of Schurecht's series which showed unusually low expansions, considering their high absorptions, concontained no feldspar. The data indicate feldspar to be a material highly reactive to water vapor. This indication was strengthened by the results of preliminary tests given in part A of Table 2. The values given in part B of Table 2 are for individual powdered materials heated, recrushed and autoclaved as described in Part II.

TABLE 5.—*Effect of particle size on percentage gain in weight during autoclave treatment (samples exposed to vapor only)*

Material	Particle size	Gain in weight		
		Sample 1	Sample 2	Sample 3
Feldspar No. 3: [1]	μ	*Per cent*	*Per cent*	*Per cent*
Fused	<80	2.8	----------	----------
Raw	<80	.18	0.22	----------
	80–10	.27	.28	0.28
	<10	1.7	1.2	1.2
Lemoor china clay [2]	Original.	2.3	2.2	----------
	>1	1.7	1.7	----------
	<1	3.2	3.3	----------

[1] Autoclaved at 500 lbs/in.² steam pressure for 1 hour (250° C.).
[2] Autoclaved at 320 to 350 lbs./in.² steam pressure for 16 hours (225°±5° C.).

The relative reactions of individual materials with moisture (Table 2) lead to conclusions similar to those indicated by the results of the much longer series of tests with experimental bodies. The weight increase of the feldspar glass investigated was the greatest, that of dehydrated clay was considerably less, and that of the calcined flint almost negligible. This is in accord with the observed fact that the bodies containing no feldspar (series C, Table 4) showed the least expansion. No lepidolite or Cornwall stone having been used in the experimental bodies, the data obtained with them are of general

[15] See footnote 13, p. 301. [16] J. Am. Ceram. Soc., vol. 12 (9), p. 596, 1929.

interest only. However, they are of possible value, when compared with those for feldspar, to those concerns using lepidolite or Cornwall stone in their bodies and glazes.

(b) EFFECT OF PARTICLE SIZE

The values obtained by autoclave treatment of fractions of one feldspar and of one clay, the fractions differing in the range of particle sizes comprised, are given in Table 5. They may be taken as further evidence that reactions with moisture in an autoclave will increase with increase in the surface exposed per unit mass. This is to be expected since there is no breaking down of the structures and the reaction is confined to the original surfaces or must proceed from these surfaces.

(c) PETROGRAPHIC EXAMINATIONS

These petrographic examinations and the following report were made by Dr. H. Insley.

Georgia clay (heated to 1,225° C., recrushed to pass a No. 120 sieve, and autoclaved at 760 to 800 lbs./in.^{2} steam pressure or at approxmiately 275° C., for 22 hours). Mean index of refraction of grains about 1.57. No difference between autoclaved and unautoclaved samples.

Cornwall stone (same treatment as Georgia kaolin). No apparent difference between autoclaved and unautoclaved samples.

Mica and quartz (same treatment as Georgia kaolin). No perceptible effect of autoclaving.

Lepidolite (same treatment as Georgia kaolin). Some etching marks on surfaces of grains, but otherwise indistinguishable from lepidolite which has been heated but not autoclaved.

Raw feldspar No. 3 ($<10\ \mu$; autoclaved at 800 lbs./in.^{2} steam pressure or 275° C., for 16 hours). Identical with untreated material.

Raw feldspar No. 3 (10 to 80 μ; same treatment as other raw feldspar). Identical with untreated feldspar.

Feldspar No. 3 (heated at 1,225° C.; recrushed to pass a No. 120 sieve and autoclaved for 24 hours at 700 to 1,000 lbs./in.^{2} steam pressure or approximately 265° to 290° C.). The feldspar is fused. Index of grains is slightly but unmistakably higher than the unautoclaved sample. (Unautoclaved=1.493, autoclaved=1.496.) Autoclaved sample shows a slight etching of the surface of the grains. The unautoclaved sample shows an extremely narrow layer on each grain somewhat higher in index than the body of the grain. The higher index of the autoclaved sample may be due to the annealing action during the autoclave treatment.

4. NATURE OF REACTION TERMED MOISTURE EXPANSION

While the results obtained in this investigation throw further light on the factors affecting the resistance of ceramic whiteware to moisture expansion, they offer no evidence regarding the nature of the reaction involved. Any of the observed facts can apply equally well, whether this expansion is caused by adsorption, absorption, or solid solutions. It is possible that the nature of the reaction could be proven using the method described by Washburn [17] for studying the dissociation equilibrium of autoclaved material at different temperatures. This method has been used by Schwarz [18] and Huttig [19] in a study of silica and its hydrates.

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[17] J. Franklin Inst., p. 753, June, 1922.
[18] Ztschr. Elektrochem., vol. 32, p. 415, 1926.
[19] Ztschr. anor. Chem., vol. 121, p. 243, 1922.

IV. CONCLUSIONS

The most important conclusions justified by the data obtained in this investigation may be summarized briefly as follows:

1. Fused feldspar is the most susceptible to moisture expansion of the materials investigated.
2. The increasing content of fused feldspar and the decreasing absorption of whiteware bodies heated to progressively higher temperatures are counteracting influences which apparently result in least resistance to moisture expansion for those bodies matured at cone 6 to 8 and having an absorption of about 10 per cent.
3. Glazed earthenware may be subject to crazing caused by penetration of moisture through the glaze and the resultant moisture expansion of the body.
4. Reactions induced by heating autoclaved specimens and evidenced by contraction are relatively sluggish at 120° C., proceed faster as the temperature is raised, and are markedly accelerated at temperatures above 230° C.
5. Autoclaved specimens of whiteware which have expanded due to reaction with moisture apparently return to their original length when cooled to room temperature after holding for a reasonably short time at 250° to 270° C. in air and under atmospheric pressure.

WASHINGTON, June 21, 1932.