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THE THERMAL EXPANSION OF FIRECLAY BRICKS

BY

ALBERT E. R. WESTMAN

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ENGINEERING EXPERIMENT STATION

BULLETIN No. 181 SEPTEMBER, 1928

THE THERMAL EXPANSION OF FIRECLAY BRICKS

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ALBERT E. R. WESTMAN

RESEARCH ASSOCIATE IN CERAMIC ENGINEERING

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

THE THERMAL EXPANSION OF FIRECLAY BRICKS

I. Introduction

1. *Preliminary Statement.*—This bulletin is the report of an investigation of the thermal expansion and correlated properties of twenty brands of fireclay brick. The accumulation of a large part of the data which are here presented was made possible by the generosity of the Utilities Research Committee, and was incidental to a more comprehensive study of checkerbricks for carbureters of water-gas machines. The Utilities Research Committee consisted of representatives of the following companies: the Commonwealth Edison Company, the Public Service Company of Northern Illinois, The Peoples Gas, Light and Coke Company, the Middle West Utilities Company, the Chicago Rapid Transit Company, and the Chicago, North Shore and Milwaukee Railroad Company.

2. *Importance of Thermal Expansion Data.*—Thermal expansion data for fireclay brick have important uses both in commerce and in the development of ceramic theory. In commercial work such data are used in the design of structures for operation at high temperatures and in the development of structural units which will withstand repeated temperature changes. From the standpoint of theory, the thermal expansion of a burned-clay product is one of its fundamental physical properties. It is of particular scientific interest because, as will be shown later, different brands of fireclay brick expand to different degrees when heated; the expansions are seldom proportional to the increase in temperature, but are subject to very marked and characteristic irregularities; and silica and its various allotropic modifications, which are present in varying concentrations in fireclay bricks, have very high and irregular, but characteristic, expansions when heated.

3. *Work of Previous Investigators.*—Table 1 indicates briefly the nature and extent of the more important investigations of the thermal expansion of refractories which have been made in the past. In the reports of these investigations thermal expansion curves are given for only a relatively small number of brands of fireclay brick, as a large number of other refractory materials were usually studied at the same time.

4. *Scope of Investigation.*—It was the purpose of the present investigation to determine the thermal expansion behavior of twenty brands of fireclay brick for the temperature range from room temper-

TABLE I
SUMMARY OF PREVIOUS INVESTIGATIONS

Investigators	Temperature Range Investigated	Materials Tested	Data Reported	Method Used
(1) Booze and Phelps	0°-1100° C	Eleven fireclay mixes	Coefficients and curves	Direct* Micrometer microscope
(2) F. A. Kohlmeyer	0°-1370° C	Firebrick	None	Direct Transit and scale
(3) F. H. Norton	0°-1700° C	4 brands of fireclay brick, 3 of zircon, 2 of magnesite, 1 each of silicon carbide, mullite, kaolin, alumina, insulating brick and silica brick.	Coefficients and curves	Direct Micrometer microscope
(4) H. S. Houldsworth and T. W. Cobb	15°-1000° C	Kaolin, siliceous & aluminous fireclays, quartzite, alumina magnesia, silicon carbide and silica brick.	Coefficients and curves	Indirect† Micrometer microscope
(5) B. Bogitch	0°-1600° C	Fused bauxite, clay, silica, chromite, magnesia	Curves only	Not stated
(6) R. F. Geller and R. A. Heindl	0°-1000° C	49 sagger clay mixes	Coefficients and curves	Indirect† Similar to writers

(1) Jour. Amer. Cer. Soc. 8, 361, 1925
(2) Jour. Amer. Cer. Soc. 8, 313, 1925
(3) Jour. Amer. Cer. Soc. 8, 799-815, 1925
(4) Jour. Amer. Cer. Soc. 6, 645-62, 1923
(5) Comp. rend. 173, 1358-60, 1921. Cer. Abs. 1, 163, 1922
(6) Jour. Amer. Cer. Soc. 9, 555, 1926

*Direct—Measurements made by sighting directly on specimen
†Indirect—Measurements made on end pieces in contact with specimen

ature (25 deg. C.) to approximately 900 deg. C. (1652 deg. F.), along with other properties which would be of interest in connection with these data. The twenty brands of brick had been chosen so as to include a wide range of properties and examples of the principal types manufactured in the United States. A list of these brands and the companies from which they were obtained, is given on page 10. Information regarding method of manufacture and chemical analysis was obtained from the manufacturers; determinations of the percentages of free silica which were present were made by Mr. T. N. McVay of the Department of Ceramic Engineering, as part of this investigation; and data regarding the physical properties were obtained as part of the checkerbrick investigation which was mentioned in the preliminary statement.

TABLE I (Concluded)
SUMMARY OF PREVIOUS INVESTIGATIONS

Investigators	Temperature Range Investigated	Materials Tested	Data Reported	Method Used
(7) M. L. Freed	0°-1000° C	Calcined cyanite	Mean per cent expansion	Indirect† Similar to writers
(8) Y. Tadokoro	0°-1300° C	Magnesia, silica, chrome, grog and red brick	Coefficients and curves	Indirect Optical lever
(9) W. Steger	15°-750° C	5 fireclay bodies, Silesian zinc retort clay, Gross-Almerode clay	Coefficients only	Indirect Mechanical lever
(10) J. Kratzert	25°-1470° C	Silica brick, clay and mullite refractory, corundum, carborundum, zirconia and chromite brick	Curves only	Indirect† Mechanical lever
(11) R. F. Geller	0°-1000° C	6 brands fireclay brick	Curves and coefficients	Not stated
(12) Bureau of Standards	0°-1000° C	13 brands of firebrick 3 brands of silica brick	Mean per cent expansion	Not stated
(13) F. H. Norton	Values given for 500° C	6 brands fireclay brick, 1 each of sillimanite, silicon carbide, bauxite, kaolin, spinel and silica	Coefficients only	Direct‡ Micrometer microscope

(7) Jour. Amer. Cer. Soc. 9, 249, 1926
(8) Rept. of the Research Lab. of the Imperial Iron Works, 2, 41 pp., 1922
(9) Ceramist 6, 453, 1925
(10) Tonindustrie Zeitung, 51 (26) 417-22, 1927
(11) Amer. Refrac. Inst. Tech. paper No. 4
(12) Cer. Ind. 8, (6) 696, 1927
(13) Jour. Amer. Cer. Soc. 8, 34, 1925

†Indirect—Measurements made on end pieces in contact with specimen
‡Direct—Measurements made by sighting on specimen

5. *Manufacturing Data.*—The more important manufacturing data for the twenty brands of brick are tabulated in Table 2, where the brands are designated by the numbers which will be used in referring to them throughout this report, and which were used in a previous bulletin.* It will be seen that the different brands differed widely in raw materials and method of manufacture.

6. *Chemical Data.*—The analyses of the brands of brick which were included in the investigation are given in Table 3, essentially as they were submitted by the manufacturers. It will be readily seen from an examination of this table that a considerable range of composition was included in the investigation.

7. *Physical Data.*—In Table 4, the pyrometric cone equivalents, absorptions, porosities, and apparent specific gravities of the different

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*"Investigation of Checkerbrick for Carbureters of Water-gas Machines," Univ. of Ill. Eng. Exp. Sta. Bul. 179.

TABLE 2
DATA PERTAINING TO MANUFACTURE

Brand	Location of Plant	Method of Manufacture*	Burning Temperatures	Raw Materials
I	California	H. M.	17-18	lean, "bone" clay, like bauxite
II	Missouri	D. P.	7†	2nd. grade semi-flint and plastic clays
III	Missouri	S. M. R.	7†	2nd. grade semi-flint and plastic clays
IV	Pennsylvania	D. P.	10-15	Penn. semi-flint clay
V	Illinois	S. M. R.	9-11	plastic clay
VI	Pennsylvania	H. M.	10-12	flint and plastic clays
VII	Missouri	S. M. R.	8	plastic clay, flint clay and grog
VIII	Missouri	S. M. R.	10-12	semi-flint clays
IX	Kentucky	H. M.	11	flint and semi-flint clay
X	Kentucky	S. M. R.	11	flint and semi-flint clay
XI	New Jersey	H. M.	12	plastic clays
XII	Missouri	D. P.	8-10	plastic clays
XIII	Kentucky	S. D. P.	8	flint, semi-flint, and plastic clays
XV	Missouri	D. P.	7	flint, and semi-flint clay
XVI	New Jersey	S. M. R.	15‡	calcined raw material and plastic clay
XVII	Missouri	S. M. R.	10	high grade plastic clay
XVIII	Ohio	A-B	31	Ga. kaolin, flint, semi-flint and plastic clays
XIX	Illinois	S. M. R.	11-12	Mo. flint and brand V plastic clay
XXIII	Missouri	H. M.	14	Mo. diaspore, high grade plastic clay
XXIV	Missouri	S. M. R.	10	Mo. semi-flint clay

*S. M. R. stiff-mud repress; D. P. dry press; S. D. P. semi-dry press; A-B auto-brick; H. M. hand made.

†Reported as 2200-2400 deg. F.

‡Reported as 2600 deg. F.

TABLE 3
ANALYSES OF FIRECLAY BRICKS

Brand	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Alkalis	Total
	per cent							
I	51.00	44.00	2.10	1.10	←	1.80	→	100.00
II	60.40	34.04	2.28	1.84	0.59	0.54	1.19	100.88
III	60.40	34.04	2.28	1.84	0.59	0.54	1.19	100.88
IV	52.28	41.10	2.43	2.35	0.15	0.23	1.27	99.81
V	64.60	30.13	2.73	1.52	0.44	0.45	0.63	100.50
VI	59.20	35.60	2.60	....	0.40	0.40	1.80	100.00
VII	52.53	41.12	2.56	1.64	0.23	1.05	0.87	100.00
VIII	55.20	38.87	2.21	1.48	0.52	0.42	0.86	99.54
IX	53.32	41.51	1.17	2.46	0.28	0.44	0.66	99.84
X	53.32	41.51	1.17	2.46	0.28	0.44	0.66	99.84
XI	63.60	31.38	....	....	....	....	....	....
XII	60.48	32.68	2.12	1.32	0.27	0.68	0.28	97.83
XIII	54.10	41.20	2.02	1.83	0.30	0.29	0.70	100.44
XV	59.10	36.00	1.10	2.80	....	....	0.80	99.80
XVI	73.46	22.08	1.50	1.94	0.14	0.20	0.58	99.90
XVII	59.76	35.90	2.63	0.00	0.22	0.64	0.84	99.99
XVIII	51.96	45.38	0.57	1.73	0.10	0.22	(0.04SO_3)	100.00
XIX	53.78	40.76	2.12	1.80	0.52	0.28	0.95	100.21
XXIII	19.66	75.03	1.01	3.40	0.21	0.38	0.49	100.18
XXIV	55.03	41.07	2.42	0.00	0.40	0.40	0.66	99.98

brands are reported. The data on pyrometric cone equivalents (formerly called softening points) were obtained from the manufacturers; the absorptions were determined in accordance with the American Society for Testing Materials method for building brick, C **21-20**, five standard straight bricks of each brand being tested; the porosities and

TABLE 4
PHYSICAL PROPERTIES

Brand	P. C. E.† cones	Absorption per cent	Porosity per cent	Apparent Spec. Gravity‡ gm/cc.
I	30–31	16.6	30.9	2.72
II	30–31	11.8	23.5	2.63
III	30–31	6.4	14.1	2.55
IV	33	10.3	20.6	2.54
V	29–30	7.7	16.7	2.59
VI	30–31	11.7	23.5	2.63
VII	32	7.8	17.1	2.63
VIII	32	7.5	16.8	2.67
IX	32	10.1	21.9	2.59
X	32	7.5	16.5	2.66
XI	34–35*	18.0	32.7	2.71
XII	30	11.4	23.0	2.63
XIII	32–33	6.5	14.8	2.67
XV	32*	10.3	21.3	2.62
XVI	.....	16.1	29.6	2.60
XVII	32	6.8	15.2	2.64
XVIII	34	....	....	....
XIX	32–33	9.8	20.1	2.57
XXIII	38–40	16.6	34.0	3.11
XXIV	33–34	6.5	15.0	2.68

* Reported in degrees Fahrenheit.
† Pyrometric cone equivalent, formerly called "softening point."
‡ Apparent specific gravity, the ratio between the weight in grams and the apparent volume in cubic centimeters, the apparent volume being equal to the bulk or overall volume diminished by the volume of the pores into which water can penetrate.

apparent specific gravities were calculated from the absorption data and from bulk volume data obtained by means of a mercury balance.* Further information regarding the properties of these brands of brick may be obtained by consulting Bulletin No. 179.†

8. *Acknowledgments.*—This investigation has been a part of the work of the Engineering Experiment Station of the University of Illinois, of which DEAN M. S. KETCHUM is the director, and of the Department of Ceramic Engineering of which PROF. C. W. PARMELEE is the head.

The writer wishes to express his appreciation of the interest and help of PROFESSOR PARMELEE; MR. THOMAS N. MCVAY, Associate, Department of Ceramic Engineering; the members of the Sub-committee on Refractories of the Utilities Research Committee; and the manufacturers of the brands of fireclay brick which were included in the investigation. The members of the Sub-committee on Refractories were: MR. ROBERT B. HARPER, Chief Testing Engineer, The Peoples Gas, Light and Coke Company, chairman; MR. ALEXANDER D. BAILEY, Superintendent of Generating Stations, Commonwealth Edison Company; MR. DWIGHT F. BURRITT, Vice-president, Middle West Utilities Company; MR. GEORGE W. HAMILTON, Vice-president, Middle West Utilities

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*Westman, A. E. R., "The Mercury Balance—An Apparatus for Measuring the Bulk Volume of Brick." Jour. Amer. Ceram. Soc., 9, 5, pp. 311-18, May, 1926.
†Loc. cit.

Company; Mr. Julius Hecht, Vice-President, Public Service Company of Northern Illinois; and Mr. John M. Lee, Construction Superintendent, Public Service Company of Northern Illinois. A list of the manufacturers of fireclay bricks, their addresses, and the brands of bricks they submitted, are given below:

LIST OF MANUFACTURERS AND OF THE BRANDS OF BRICK THEY SUBMITTED

Firm & Address	Brand
Ashland Fire Brick Co., Ashland, Ky.	1. Ashland crown 2. Ashland Empire crown
Chicago Retort and Fire Brick Co., Ottawa, Ill.	1. Ajax 2. Superior
Crescent Refractories Co., Curwensville, Pa.	1. Reliance
Evens and Howard Fire Brick Co., St. Louis, Mo.	1. Evens-Howard Checker
J. H. Gautier and Co., Jersey City, N. J.	1. J. H. Gautier and Co. No. 1 Jersey City, N. J
A. P. Green Fire Brick Co., Mexico, Mo.	1. A. P. Green F. B. Co. Crown S. M. 2. A. P. Green F. B. Co. Empire S. M. 3. A. P. Green F. B. Co. Kruzite
Harbison-Walker Refractories Co., Pittsburgh, Pa.	1. Widemire
Laclede Christy Clay Products Co., St. Louis, Mo.	1. Laclede St. Louis
Parker-Russel Mining and Manufacturing Co., St. Louis, Mo.	1. P. R. M. and M. Co. Checker★
Louisville Fire Brick Works, Louisville, Ky.	1. L. F. B. Wks. Louisville
Seaboard Refractories Co., Perth Amboy, N. J.	1. Seaboard Middlesex Reg. U. S. Pat. Off.
The Babcock and Wilcox Co., East Liverpool, O.	1. No. 80 (Kaolin) 2. No. 80 (Kaolin 4 mesh)
The Vitrefax Co., Vernon, California.	1. Argon, A. A.
Walsh Fire Clay Products Co., Vandalia, Mo.	1. Salamander (D. P.) 2. Salamander (S. M.)

II. Thermal Expansion Measurements

9. *Preparation of Specimens.*—In the thermal expansion measurements, cylindrical specimens approximately nine inches (22.9 cm.) long, and one inch (2.54 cm.) in diameter were used. These were prepared by cutting pieces 9 in. by 1 in. by 1 in. (22.9 cm. by 2.54 cm. by 2.54 cm.) from standard straight firebricks by means of thin grinding

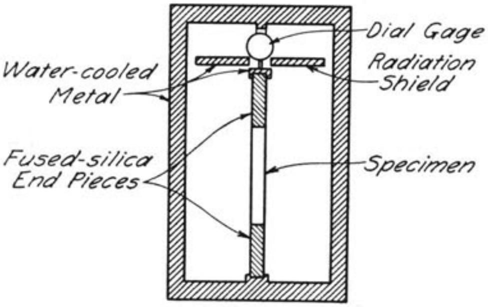

FIG. 1. PRINCIPLE OF THERMAL EXPANSION APPARATUS

wheels, removing the edges by means of an ordinary grinding wheel and then truing the ends on a polishing lapp. Notches for thermocouple insertion were cut at one inch (2.54 cm.) from each end and at the middle of each specimen.

10. *Principle of Method.*—The operating principle of the apparatus which was used in the thermal expansion measurements is shown diagrammatically in Fig. 1. The apparatus consisted essentially of a rigid, water-cooled, metal framework; fused-silica end-pieces; a dial gage; a water-cooled radiation shield; and a specimen. If the specimen were heated and the water-cooled metal parts maintained at a constant temperature, it is evident that the dial gage would register the increase in length of the specimen and the fused-silica end-pieces, the dial itself being protected from excessive temperature changes by the radiation shield and the water-cooled cap on the upper fused-silica end-piece. By using a specimen of known expansion, such as a specimen of fused silica, the thermal expansion of the end-pieces, which in the experiments of this investigation amounted to about ten per cent of the change in length registered by the dial, could be obtained and applied as a correction in experiments with other specimens. In the same way corrections could be applied for variations in the temperature of the circulating water, which seldom exceeded 2 deg. C. in these experiments, using data obtained in tests in which the specimen was not heated but the temperature of the circulating water was varied.

11. *Description of Apparatus.*—Details of the construction of the apparatus are shown in Fig. 2. The actual length of specimen *A* is nine inches (22.9 cm.), and the remainder of the apparatus is drawn to the same scale. An electric furnace of a simple type with nichrome

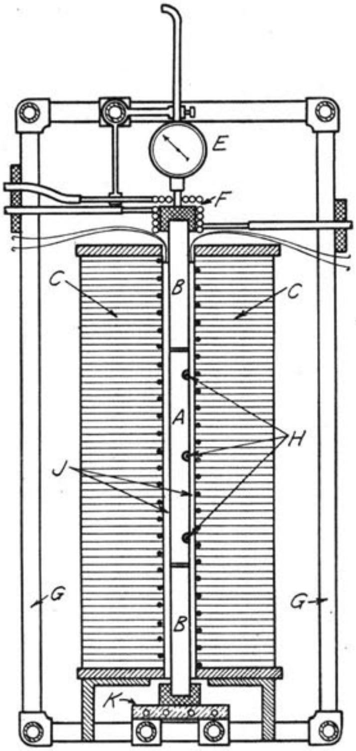

FIG. 2. DETAILED CONSTRUCTION OF THERMAL EXPANSION CURVES

winding and heat insulation *C* was used to heat specimen *A,* whose temperature was measured at three points by means of a multiple, base-metal thermocouple having three hot junctions *H* and a common return, the hot junctions being placed in notches cut in the specimen. End-pieces *BB* were made by sealing fused-silica discs to the ends of tubes of the same material, which were filled with Sil-o-cel, and were in turn cemented to water-cooled metal caps. Radiation shield *F* consisted of a flat coil of copper tubing through which water circulated. Dial gage *E,* which could be read to 0.0001 inch (0.00025 cm.), was fastened securely to water-cooled frame *G,* which was a rectangular framework of ordinary three-quarter inch water pipe. The furnace had no mechanical connection to the rest of the apparatus, the specimen, end-pieces, and caps being supported on a water-cooled brass plate *K,* which was clamped to the lower part of framework *G.* Space *J* between the specimen and end-pieces and the furnace was filled with pure asbestos tape to prevent convection currents.

12. *Operation.*—The length of the specimen was measured at room temperature by calipers reading to 0.01 inch (0.0254 cm.), the multiple couple was bound in place by a winding of pure asbestos tape, and then the whole was inserted in the furnace by removing the dial gage and upper end-piece. These were then replaced and the circulation of water through the water-cooled system was started. In two or three minutes, when the water-cooled parts had reached a steady temperature, the dial gage was adjusted to zero and the furnace circuit closed. The temperature of the specimen was regulated by means of a rheostat and wattmeter; it was raised at an average rate of about 250 deg. C. (450 deg. F.) per hour during the run and held constant at intervals of 50 deg. C. and sometimes less for two or three minutes, while readings of the dial gage and of the temperatures of the three hot junctions, the cold junction, and the circulating water were made. Further experiments have shown that this procedure can be varied quite radically without noticeable influence on the final result, no appreciable error being incurred by raising the temperature continuously and at more rapid rates. This was due to the small thermal lag of the end-pieces and the practically constant temperature of their water-cooled caps. The dial readings were corrected when necessary for changes in length of the water-cooled frame, which were nearly always negligible, and for the expansion of the fused-silica end-pieces, obtained as described in Section 10. They were then reduced to percentages of the length of the specimen at room temperature. The temperature of the specimen was taken to be the weighted average of the temperatures indicated by the multiple couple, the temperature of the junction at the middle of the specimen being assigned twice the weight of the temperatures of the other junctions. Corrections were applied for the temperature of the cold junction. The data in its final form, therefore, consisted of a series of average temperatures and the corresponding percentage expansions.

13. *Calibration.*—By the use of Johannsen blocks, it was found that the dial gage was subject to random errors averaging about 0.0002 inch (0.00051 cm.), with a maximum of 0.0007 inch (0.00178 cm). Since these errors were relatively small and were not systematic but cancelled at each complete revolution of the pointer, they could be largely eliminated by drawing smooth curves when the final data were plotted. The thermocouple circuit, which consisted of the multiple couple described in Section 10, an indicator, and a cold junction box, was found by direct comparison with a standard circuit to give readings correct to within 3 deg C. The difference in temperature be-

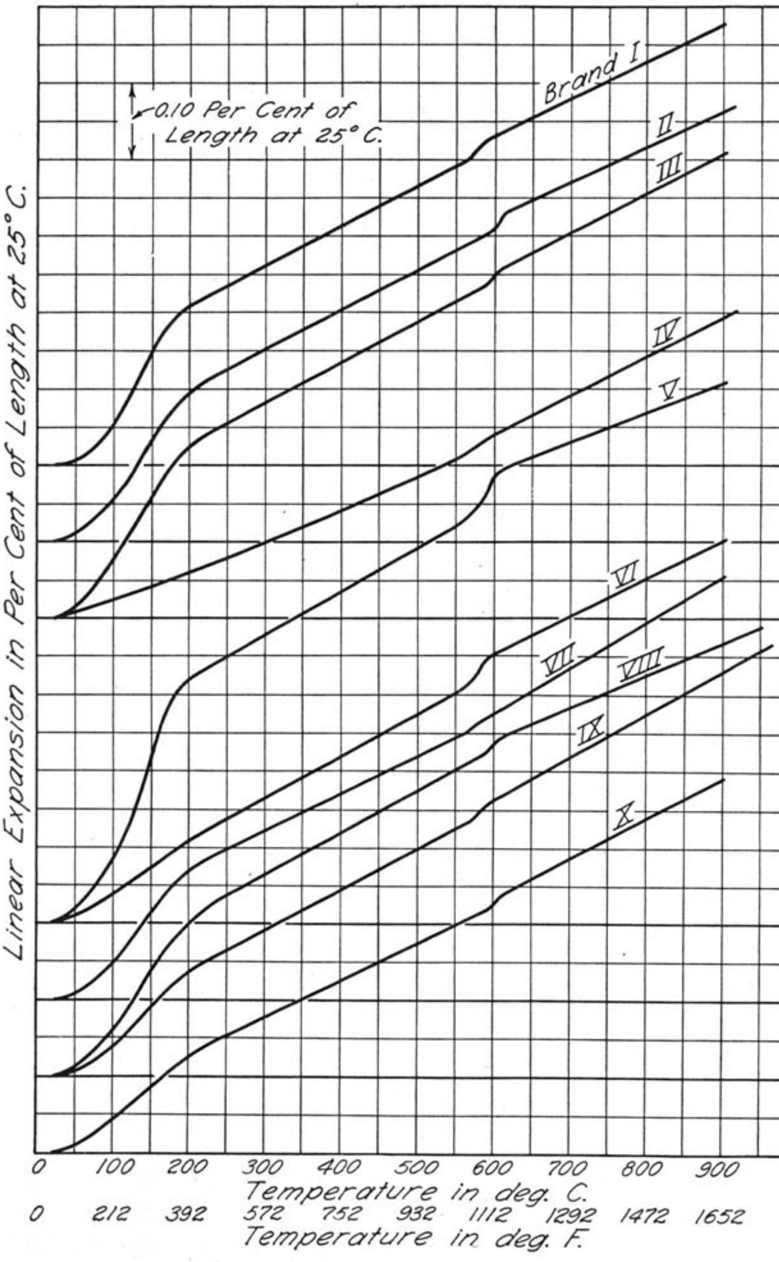

FIG. 3. THERMAL EXPANSION CURVES

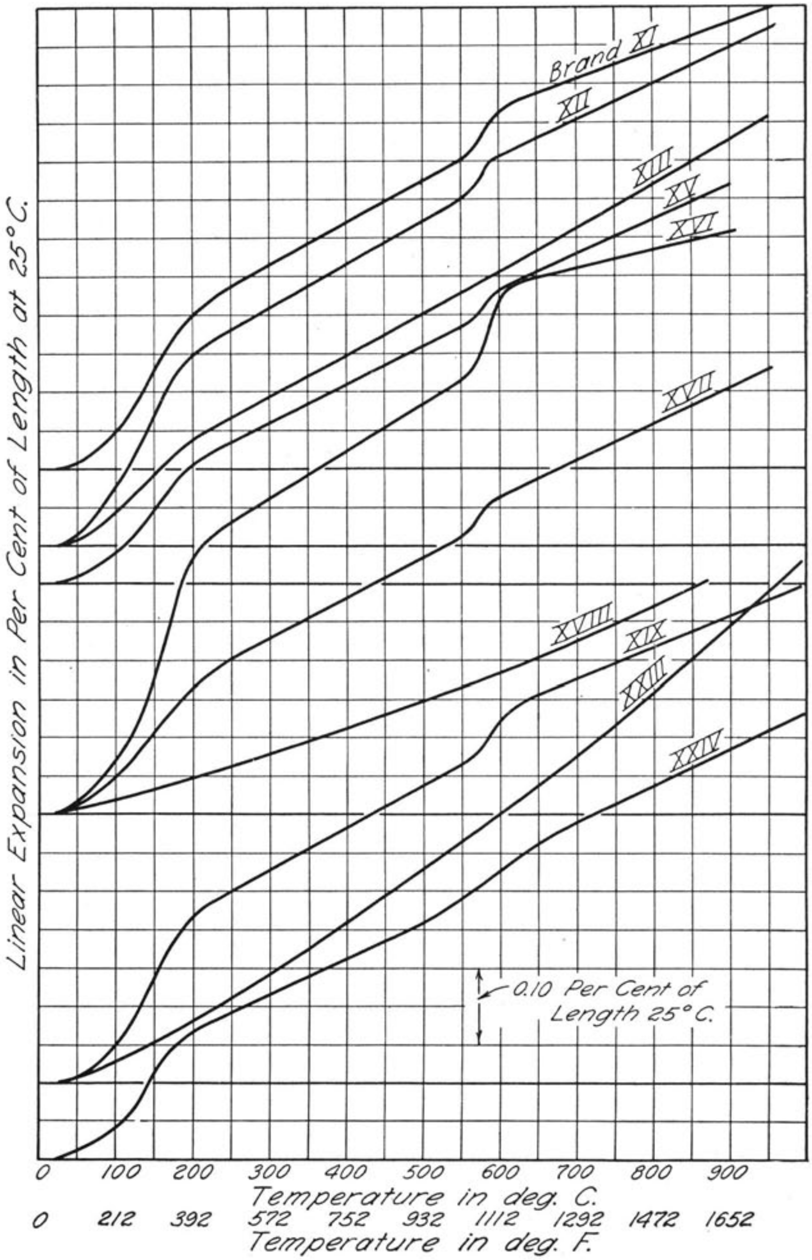

Fig. 3 (continued). Thermal Expansion Curves tween the middle and end junctions was found to be usually less than 3 deg. C., and to seldom exceed 10 deg. C. The average temperatures were therefore sufficiently accurate for this investigation, the only uncertainty being that due to possible radial temperature gradients in the specimen, which could not be very large.

14. *Thermal Expansion Curves.*—The data obtained, as described in Section 12, were used in plotting thermal expansion curves for each specimen which was measured, the average temperatures being plotted as abscissas and the percentage expansions as ordinates. Two curves were obtained for most of the specimens and were averaged graphically to give the curves which are shown in Fig. 3. For most of the twenty brands only one specimen was used but in the case of brand XI, five specimens were used, two of these being cut from one brick, and one from each of three other bricks. This enabled an estimate to be made of the sampling errors involved in the thermal expansion measurements.

15. *Precision of Thermal Expansion Curves.*—An estimate of the errors of measurement was formed by an examination of the duplicate curves obtained for fifteen specimens. In five cases the curves were identical, and in the other cases the curves were parallel to each other for temperatures above 200 deg. C. (392 deg. F.), and differed by less than 0.015 on the scale of per cent expansion. That these differences were due largely to changes in the specimen brought about by the first heating was shown by the fact that for the five pairs of duplicate curves obtained for brand XI, the curve for the second measurement was higher by about 0.015 per cent expansion in every case.

The sampling errors were determined by an examination of the average curves for the five specimens of brand XI, which are shown in Fig. 4. The expansion curves for different specimens differed quite appreciably, although the curves were to a considerable extent parallel to each other, variations in the expansion below 200 deg. C. (392 deg. F.) being responsible for most of the lack of agreement observed.

16. *Classification of the Curves.*—The thermal expansion curves for commonly used engineering materials are, for the most part, remarkably linear over the temperature range of this investigation. An examination of the curves for the twenty brands of brick, however, shows that, in the majority of cases, although the curves are linear over part of the range, they have very pronounced inflections, one of which occurs usually between room temperature and 200 deg. C. (392 deg. F.) and the other in the neighborhood of 575 deg. C. (1067 deg. F.). A convenient and useful classification of the curves can be made

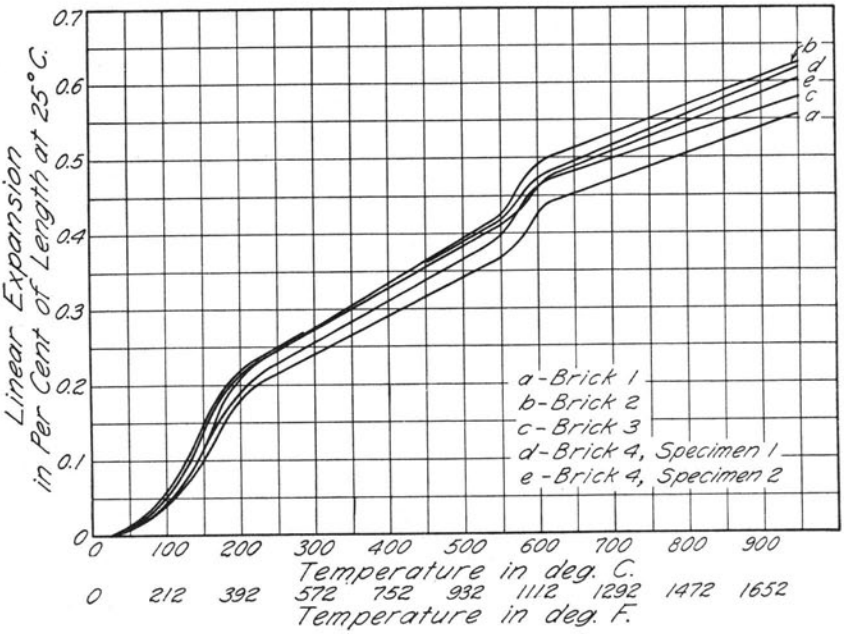

FIG. 4. NATURE AND EXTENT OF SAMPLING ERRORS OF THERMAL EXPANSION CURVES

on the basis of the presence or absence of one or more of these characteristic departures from linearity. On this basis the curves which were obtained could be divided into the following types:

Type	Inflection Below 200° C.	Inflection near 575° C.	Remainder of Curve	Brands Included
I	Present	Present	Linear	I, II, III, V, VIII, IX, X, XI, XII, XV, XVI, XVII, XIX, XXIV
II	Present	Absent	Linear	VII, XIII
III	Absent	Present	Linear	VI
IV	Absent	Absent	Small positive curvature	IV, XVIII, XXIII

Examples of these four types are shown in Fig. 5.

17. *Mean Curve.*—In general design and for comparison with materials which differ greatly from burned fireclay in thermal expansion, a curve giving the average expansion of a number of brands of fireclay brick is frequently of use. The curve shown in Fig. 6 gives

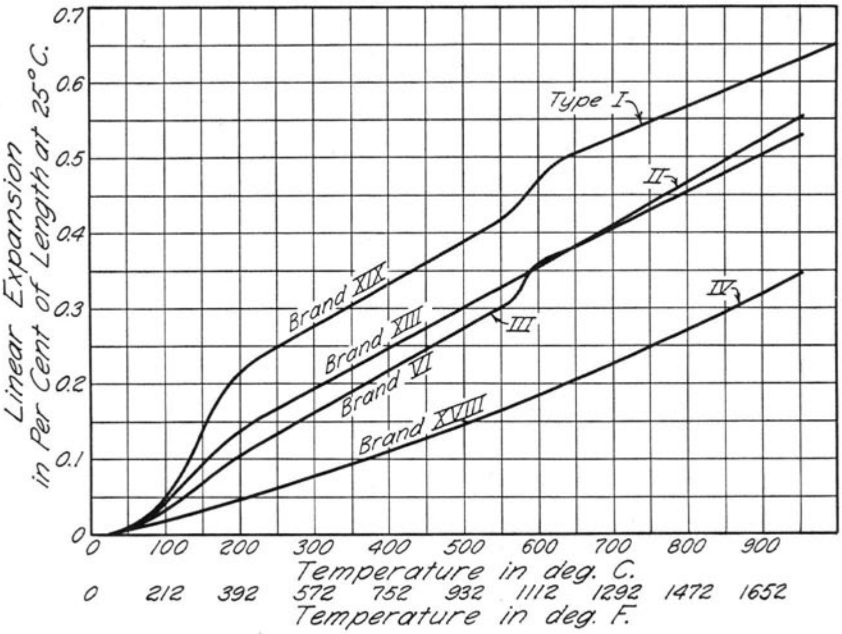

FIG. 5. CURVES ILLUSTRATING THE FOUR TYPES OF THERMAL EXPANSION CURVES

the average expansion of the brands of fireclay brick included in this investigation, each of the seventeen fireclays being given equal weight. When no information regarding the properties of the bricks to be used in an installation is available, design may properly be based on this curve; when the composition or other pertinent properties of the bricks is known, allowance should be made for exceptionally high or low silica content, etc., in accordance with the relations given in Chapter V of this bulletin. Above the thermal expansion curve in Fig. 6 the probable error range is plotted. This gives the range within which one-half of a large number of similarly obtained mean curves may be expected to lie.

III. PETROGRAPHIC MEASUREMENTS

18. *Preparation and Examination of Slides.*—For each brand of brick a number of fragments of about 30 grams total weight were broken from a specimen and crushed to pass through a 200-mesh sieve. The powder, so obtained, was screened, and the portion which

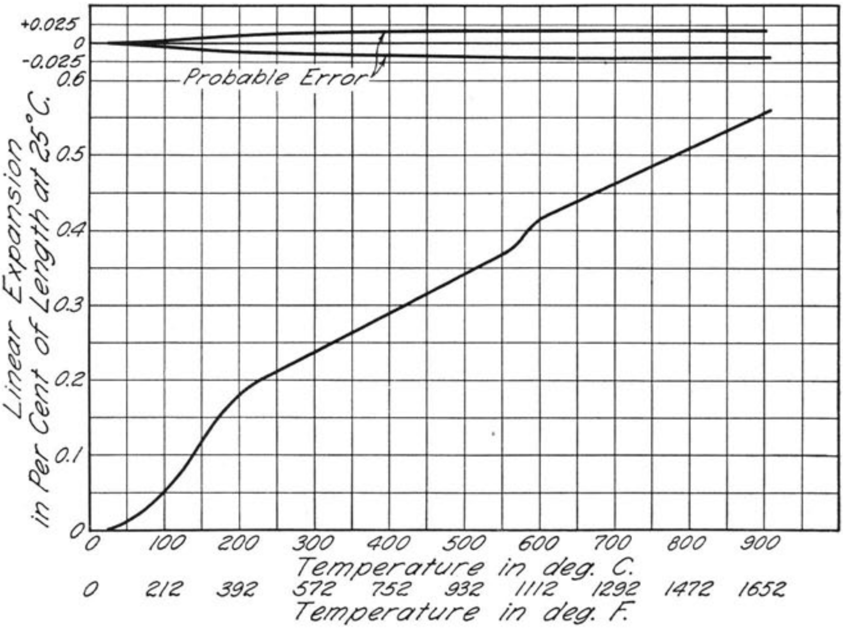

FIG. 6. MEAN THERMAL EXPANSION CURVE FOR FIRECLAY BRICKS

passed through a 200-mesh sieve and was retained on a 300-mesh sieve was taken for examination. By means of a small spoon a representative sample was taken, immersed in oil of refractive index 1.52 on a glass slide and under a circular cover-glass 18 mm. in diameter and stirred to give a uniform dispersion. With slides prepared in this way the grains did not touch each other and covered about one-third of the area under the cover-glass. The slide, so prepared, was examined under a petrographic microscope using a magnification of 240 diameters. Under the microscope the particles could readily be recognized as either fragments of quartz which had been produced by crushing the quartz grains in the firebrick, or fragments of burned clay. In some cases, the quartz fragments showed evidence of partial inversion to cristobalite, an allotropic modification of quartz, at their borders; and in many of the burned clay fragments there were very small fragments of cristobalite or quartz. It was not possible to estimate the amount of cristobalite and quartz present in this form but it was possible to make a fairly accurate determination of the relative volumes of the quartz and clay fragments which were present.

TABLE 5

MINERALOGIC COMPOSITION OF BRICKS

Brand	Calculated from Thermal Expansion Data			Calculated from Petrographic Data	
	Cristobalite volume per cent	Quartz volume per cent	Free Silica volume per cent	Quartz* volume per cent	Probable† Error
I	12.2	3.1	15.3	10.2	±1.5
II	11.4	5.2	16.7	4.4	0.5
III	13.7	2.1	15.8	5.1	0.2
IV	0	1.9	1.9	5.4	0.6
V	22.7	11.0	33.6	12.4	0.5
VI	1.0	6.2	7.2	6.5	1.0
VII	8.8	0.7	9.5	0.6	0.1
VIII	11.1	3.1	14.2	5.4	0.9
IX	4.6	3.1	7.7	4.5	0.9
X	4.1	2.4	6.5	3.6	0.2
XI	11.8	11.0	22.7	25.9	1.0
XII	15.9	7.1	23.0	7.6	0.5
XIII	4.8	1.0	5.8	0.6	0.3
XV	7.2	6.2	13.4	15.0	2.0
XVI	24.7	23.8	48.6	33.0	1.0
XVII	8.7	6.7	15.3	7.2	0.8
XVIII	0	0	0	0	...
XIX	12.8	10.0	22.8	12.4	1.7
XXIII	0	0	0	0	...
XXIV	8.8	8.6	17.3	1.8	0.4

*The borders of some of the quartz grains showed evidence of partial inversion to cristobalite.

†Calculated from the values obtained from three slides of each material using "Student's" small ample theory.

19. *Determination of Free Quartz Content.*—By means of a Wentworth Recording Micrometer* the total lengths of the intercepts formed by a straight line drawn at random across a slide and the images of the quartz and the clay grains could be determined. Four such measurements were made on each slide, the distance traversed being equal to about 4.5 cm. of clay for each determination. The sum of the quartz intercepts were divided by the sums of the quartz and clay intercepts and multiplied by 100 to give the percentage free quartz. Three slides were prepared for each brand and the percentages obtained averaged. The average values are reported in Table 5.

20. *Precision.*—Since the percentage of free quartz was calculated for each slide, and since three slides were measured for each brand, the probable errors of the average values could be calculated,† and are reported in the sixth column of Table 5. These values show that consistent results could be obtained. Nevertheless, the use of this method involves assumptions which are never completely satisfied, and consequently the data are not so accurate as the probable errors indicate. Some of these assumptions are (1) that the portion of the powder passing 200 mesh and retained on 300 mesh is representative

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*The American Mineralogist, 9, 9, pp. 190-193, Sept., 1924.

†Small sample theory, "Student," Biometrika, Vol. VI, pp. 1-15, 1908. *Ibid.*, XI, pp. 416-17, 1917.

TABLE 6
MINERALOGIC COMPOSITION OF BRICKS

Brand	Calculated from Thermal Expansion Data	Calculated from Chemical Analyses	
	Free Silica weight per cent	Free* Silica weight per cent	Cristobalite possible† weight per cent
I	13.4	(0.7 excess Al_2O_3)	33.7
II	15.4	20.3	27.0
III	14.7	20.3	27.0
IV	2.0	3.9	32.7
V	31.5	29.1	24.0
VI	7.2	17.3	28.5
VII	8.4	4.1	32.9
VIII	12.8	9.4	31.1
IX	7.3	4.4	33.2
X	6.0	4.4	33.2
XI	20.8	26.6	26.1
XII	21.2	22.0	26.1
XIII	5.2	5.6	33.0
XV	12.7	16.7	28.8
XVI	46.3	47.5	17.6
XVII	14.3	17.5	28.7
XVIII	0	(1.3 excess Al_2O_3)	34.1
XIX	21.8	5.7	32.6
XXIII	0	(58.4 excess Al_2O_3)	0.0
XXIV	16.1	6.7	32.8

*In excess of that required by formula $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$.
†Cristobalite which could be set free by the maximum formation of mullite $3\,Al_2O_3 \cdot 2\,SiO_2$.

of the whole brick, (2) that the volumes of the quartz grains bear the same relation to their linear intercepts as the volumes of the clay grains do to theirs, and do not vary greatly, and (3) that the quartz grains are composed entirely of quartz, and that the clay grains contain no quartz, or that only compensating errors are introduced by departures from these conditions. That assumptions (1) and (2) were satisfied to a considerable degree was shown by further experiments in which (a) some of the material passing 300 mesh was examined and (b) the average intercept for the clay and the quartz grains was found to be practically the same, 0.0675 mm. being the average intercept for 23 quartz grains, and 0.066 mm. being the corresponding value for a similar number of clay grains. In none of the slides was there any noticeable difference between the shapes of the quartz and the clay grains.

IV. CALCULATIONS FROM CHEMICAL ANALYSES

21. *Free Silica Content.*—The free silica content in percentage by weight was calculated for each brand from its chemical composition using the data given in Table 3. In making this calculation it was assumed that all of the alumina present was combined with silica in the proportions of one reacting weight of alumina to two of silica and

that the amount of silica which was combined with the alkalis was negligible. This was equivalent to assuming that the fireclays which were used as raw materials consisted essentially of kaolinite ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) and free silica. The values obtained by this method are reported in Table 6. For three of the brands, viz., I, XVIII, and XXIII, the calculations indicated a negative amount of silica, or that excess alumina was present. The excess alumina was negligible in the case of brands I and XVIII but not for brand XXIII, which, as shown in Table 2, contained a large proportion of diaspore ($Al_2O_3 \cdot H_2O$), and would not ordinarily be classified as a brand of fireclay brick.

22. *Possible Additional Cristobalite.*—Products formed by heating clays at high temperatures frequently contain a considerable proportion of mullite ($3Al_2O_3 \cdot 2SiO_2$), a compound which crystallizes in long, thin crystals. If we consider that this is formed from the decomposition products of kaolinite, the complete reaction can be represented by the equation:

$$3(Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O) \rightarrow 3Al_2O_3 \cdot 2SiO_2 + 4SiO_2 + 6H_2O$$

This shows that if mullite were formed in this way, silica would be set free, presumably as cristobalite, the modification of silica which is usually produced in the temperature range of the reaction. The possible additional cristobalite for each brand of brick was calculated from its analysis on the assumption that this reaction went to completion, and that as much as possible of any free alumina which might be present combined with silica to form mullite. The data, which for this reason are really maximum values, are given in Table 6. They indicate that a large percentage of cristobalite might conceivably be formed in the manner described.

V. RELATION OF THERMAL EXPANSION TO MINERALOGIC COMPOSITION

23. *Thermal Expansion of Modifications of Silica.*—The free silica in a fireclay brick might conceivably be present as quartz, cristobalite, tridymite or mixtures formed from them. The thermal expansion behaviors of these three forms of silica are quite different and have been the subject of a number of investigations. A critical summary of these investigations is given by Sosman.* The thermal expansion curves which Le Chatelier† obtained are shown by the full line curves in

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*Sosman, Robert B., "The Properties of Silica," Amer. Chem. Soc. Monograph Series, The Chemical Catalog Co., New York, 1927.

†Le Chatelier, Henry, "La Silice et les Silicates." p. 495. A. Hermann et Fils, Paris, 1914.

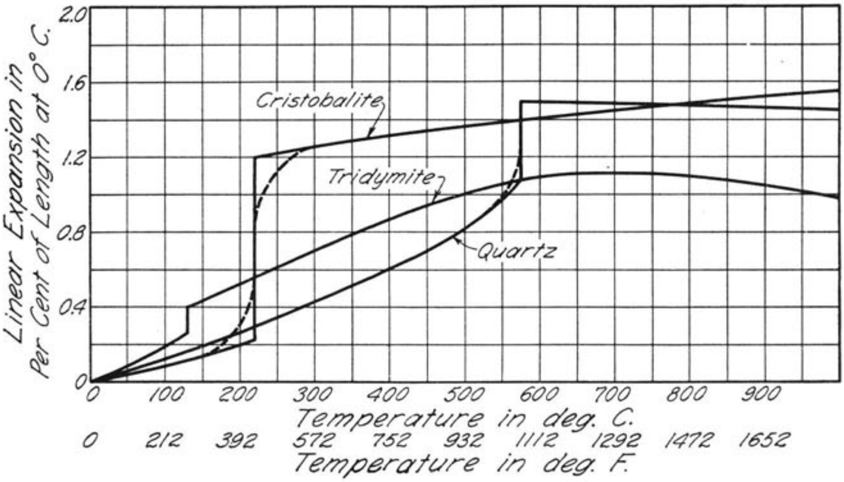

Fig. 7. Le Chatelier's Thermal Expansion Curves for Modifications of Silica

Fig. 7; later work indicates that some of the inflections are not so abrupt but are more like those shown by the broken lines in the figure. The curve for cristobalite has a very pronounced inflection at 220 deg. C. (428 deg. F.) and that for quartz a less pronounced inflection at 575 deg. C. (1067 deg. F.).

Many of the thermal expansion curves for fireclay bricks, Fig. 3, contain pronounced inflections at these temperatures and it is customary to ascribe them to the presence of cristobalite and quartz in the bricks. In the present investigation sufficient data were obtained to enable this explanation to be tested quantitatively. This was done by calculating the percentages of quartz and cristobalite which would account for the inflections found in the thermal expansion curves of the fireclay bricks, and comparing these percentages with the data obtained by petrographic examination and by calculation from the chemical analyses.

24. *Calculations Based on Thermal Expansion Curves.*—The amount of expansion which could be ascribed to inversion in the neighborhood of 200 deg. C. (392 deg. F.) was determined for each brand of brick as a percentage of the length at 25 deg. C. (77 deg. F.) by producing the linear portion of its expansion curve between the lower and upper inflection and finding the vertical distance, measured on the percentage expansion scale, from this line to a parallel line cutting the temperature axis at 25 deg. C. The value, so obtained, was

divided by 0.97, which was the percentage expansion of cristobalite at 220 deg. C. (428 deg. F.) determined by measurements on Le Chatelier's curves, Fig. 7, and then multiplied by 100 to give the volume percentage of cristobalite in the brick. These calculations were based on the assumption that the lower inflection in the expansion curve of the brick was due to cristobalite and was proportional to the volume percentage of cristobalite present. The data, so obtained, are reported in the first column of Table 5.

The same procedure was followed in calculating the volume percentage of free quartz from the magnitudes of the upper inflections in the thermal expansion curves, the value 0.42 per cent, obtained from Le Chatelier's curves, being used for the expansion of quartz due to inversion at 575 deg. C. These data are reported in the second column of Table 5.

The value for the expansion of cristobalite at the inversion temperature (0.97 per cent), calculated from Le Chatelier's curves, agreed fairly well with those calculated from data obtained by more recent investigators, the latter values being: Braesco,[*] 1.01 per cent; Day, Sosman and Hostetter,[†] 0.94 per cent; and Washburn and Navias,[‡] 0.94 per cent. This was not the case for the expansion of quartz at the inversion temperature of 575 deg. C. (1067 deg. F.), the different values being: Braesco, 0.74 per cent; Washburn and Navias, 0.72 per cent; Le Chatelier, 0.42 per cent; and Day, Sosman, and Hostetter, 0.29 per cent. An examination of the original papers, however, showed that Le Chatelier's value was more applicable to the case under consideration than those of the other investigators, because it could be calculated definitely from the curves which he published and because he made direct linear measurements on quartz aggregates. The alpha quartz to beta quartz inversion is of such a nature that *linear* factors cannot be estimated satisfactorily from measurements of *volume* changes. Sosman[§] discusses in detail the reasons for the discrepancies between the values for the expansion of quartz which were obtained by the different investigators.

25. *Comparison of Thermal Expansion, Petrographic and Chemical Data.*—Tables 5 and 6 afford a comparison of the values obtained for the percentages of the different forms of silica calculated from thermal expansion, petrographic, and chemical data. In making this

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[*] Braesco, P., "Etudes des Points de Transformation par une Methode Dilatometrique." Ann. Physique, 14, pp. 5-75, 1920.

[†] Day, A. L., Sosman, R. B., and Hostetter, J. C., "The Determination of Mineral and Rock Densities at High Temperatures," Amer. Jour. of Science, 37, pp. 1-39, 1914.

[‡] Washburn, Edward W., and Navias, Louis, "The Products of the Calcination of Flint and Chalcedony," Jour. Amer. Ceram. Soc., 5, 9, pp. 565-585, 1922.

[§] Loc. cit.

comparison, it should be borne in mind that the data are subject to quite appreciable errors. In particular, the estimation of small percentages of quartz from the thermal expansion curves is subject to large percentage errors. The object of the comparison is to determine whether the available data are in semi-quantitative agreement with the explanation of the inflections in the expansion curves which is based on the presence of cristobalite and quartz. In Table 5 it will be observed that the volume percentages of free quartz calculated from the petrographic data do not exceed the volume percentages of total free silica calculated from the thermal expansion data, within the limits of error of the values given. It will also be observed that, with the exception of brands I, XI, XV, and XXIV, the volume percentages of quartz agree reasonably well with those calculated from the thermal expansion curves. The discrepancies shown by brands I, XI, and XV were probably due to the partial conversion of the quartz grains to cristobalite, the thermal expansion data indicating that considerable quantities of this form were present.

By using the apparent specific gravity data given in Table 4 and assuming that none of the modifications of silica contained pores, the volume percentages of free silica calculated from the thermal expansion data and reported in the fourth column of Table 5, were converted to weight percentages. These are given in the second column of Table 6, where a comparison is afforded with the weight percentages calculated from the chemical analyses. An examination of this table will show that, with the exception of brands I, XIX, and XXIV, the chemical analyses indicate enough free silica to account for the thermal expansion data within limits of error and, in fact, the percentages of silica calculated by the two methods are in fair agreement. The petrographic data, Table 5, indicate that for brands I and XIX calculations based on the chemical analyses do not indicate the true mineralogic composition, probably due to the presence of bauxite in the raw clays. For brand XXIV, both the petrographic data and chemical analysis indicate less free silica than that calculated from the thermal expansion curves. This indicates that the data for this brand were subject to unusual errors, or that the different assumptions made in the calculations were not satisfied.

The data in the fourth column of Table 6, show that quite large percentages of cristobalite could conceivably be formed by the decomposition of kaolinite and subsequent formation of mullite for practically all of the brands. With the possible exception of brands XIX and XXIV, however, it is not necessary to consider this source of cristobalite in order to explain the thermal expansion data.

VI. SUMMARY

26. *Scope of Investigation.*—Samples of twenty brands of fireclay bricks which included a rather wide range of physical and chemical properties were obtained from the manufacturers. The thermal expansion behavior of these bricks in the temperature range 25 deg. C. (77 deg. F.) to 950 deg. C. (1742 deg. F.) was determined by means of an apparatus developed for the purpose. Information concerning the mineralogic composition was obtained by petrographic methods and from chemical analyses submitted by the manufacturers. The thermal expansion curves obtained were classified on the basis of the presence or absence of certain characteristic inflections in the curves. The customary explanation for these inflections, viz., that they were due to the inversion of cristobalite and quartz present in the bricks, was subjected to a quantitative test by calculations based on the curves, the petrographic data, and the chemical analyses.

27. *Data Reported.*—A tabular summary of previous investigations of the thermal expansion of refractories is given in Table 1. For the twenty brands of brick the following data are reported: plant location, method of manufacture, and raw materials (Table 2); pyrometric cone equivalent, absorption, porosity, and apparent specific gravity, (Table 4); thermal expansion curves between 25 deg. C. (77 deg. F.) and 950 deg. C. (1742 deg. F.) and a mean curve for seventeen fireclays (Figs. 3 to 6); volume percentages of cristobalite, quartz, and free silica calculated from thermal expansion data, and of quartz, calculated from petrographic data (Table 5); weight percentages of free silica calculated from thermal expansion data and chemical analysis, and the possible weight percentage of cristobalite resulting from mullite formation (Table 6).

28. *Analysis of Data.*—An analysis of the data pertaining to the mineralogic composition of the different brands of brick calculated from the thermal expansion, petrographic, and chemical data led to the following conclusions:

(1) Within the experimental error, the volume percentages of free quartz calculated from the petrographic data obtained by the powder-immersion method were either equal to or less than the volume percentages of free silica calculated from the thermal expansion data, and approximately equal to the volume percentages of free quartz.

(2) With a few exceptions, calculations based on the chemical analyses indicated a free silica content high enough to account for the inflections in the thermal expansion curves.

(3) With the exception of possibly two of the twenty brands investigated, it was not necessary to postulate the presence of cristobalite set free as a result of the decomposition of kaolinite and subsequent formation of mullite in order to account for the thermal expansion data.

(4) For at least eighteen of the twenty brands investigated, the data obtained from thermal expansion measurements, petrographic examination by the powder immersion method, and chemical analysis were in reasonably quantitative agreement with the assumption that the lower inflections in the thermal expansion curves were due to the inversion of cristobalite and the upper inflections to the inversion of quartz, the cristobalite and quartz being originally present in the raw materials as uncombined silica. It could reasonably be expected, therefore, that with more accurate methods for determining percentage expansions due to inversions and more reliable factors for calculating percentages of cristobalite and quartz from such data, useful analyses of fireclay bricks could be made.

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The College of Agriculture (Curricula: General Agriculture; Floriculture; Home Economics; Landscape Architecture; Smith-Hughes—in conjunction with the College of Education)

The College of Education (Curricula: Two year, prescribing junior standing for admission—General Education, Smith-Hughes Agriculture, Smith-Hughes Home Economics, Public School Music; Four year, admitting from the high school—Industrial Education, Athletic Coaching, Physical Education. The University High School is the practice school of the College of Education)

The School of Music (four-year curriculum)

The College of Law (three-year curriculum based on two years of college work. For requirements after January 1, 1929, address the Registrar)

The Library School (two-year curriculum for college graduates)

The School of Journalism (two-year curriculum based on two years of college work)

The College of Medicine (in Chicago)

The College of Dentistry (in Chicago)

The School of Pharmacy (in Chicago)

The Summer Session (eight weeks)

Experiment Stations and Scientific Bureaus: U. S. Agricultural Experiment Station; Engineering Experiment Station; State Natural History Survey; State Water Survey; State Geological Survey; Bureau of Educational Research.

The Library collections contain (June 1, 1927) 733,580 volumes and 162,783 pamphlets.

For catalogs and information address

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