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MELTING POINTS OF FIRE BRICKS

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MELTING POINTS OF FIRE BRICKS

By C. W. Kanolt

We are accustomed to thinking of a melting point as a temperature at which a substance changes from a condition in which it does not flow perceptibly under its own weight to a condition in which it flows readily. However, no rational definition of the melting point can be based upon this conception. For example, when glass is heated, the transition from its ordinary condition to a distinctly fluid condition is a very gradual one and occupies a range of several hundred degrees. Some other substances which possess perfectly definite temperatures of transition to a fluid phase undergo changes resembling fusion at lower temperatures. Solids in general become softer when heated; they may become so soft as to yield to their own surface tension, with the result that sharp corners are rounded off. Rutile, in its natural, slightly impure condition, melts at about 1700° C. The melting point is very definite, there being a sharp transition from a rather soft solid to a liquid of low viscosity, with a considerable absorption of heat. Yet at temperatures 100° or more below the melting point, corners are rounded off and small particles become sintered together. The sintering of clay is probably a similar phenomenon of surface tension. Clay exists generally in the form of extremely fine particles, its unusually slight solubility in water accounting for this condition. Fine particles are more readily united by surface tension than large ones.

A melting point can be precisely and rationally defined only as the temperature at which a crystalline or anisotropic phase and an amorphous or isotropic phase of the same composition can exist in contact in equilibrium. In harmony with this, one might define a solid as a crystalline substance, and a fluid as a substance not crystalline; however, these definitions of solid and fluid would lead to results quite different from our ordinary conceptions, as they would require us to consider glass, even silica glass, as a fluid, and certain "anisotropic liquids" whose viscosities are even less than that of water, as solids. Perhaps it is better to leave solid and liquid or fluid as general terms without precise definitions.

While the above definition of melting point is satisfactory for the case of a pure substance, so complex a mixture as an ordinary fire brick usually has no single definite melting point according to this definition, since several anisotropic phases may be present, all differing in composition from the isotropic phase produced by fusion. We can, then, only select the temperature at which the transition from a rigid to a fluid state seems most distinct, and can call this the melting point only by apology.

In the case of fire bricks the transition temperatures so found are fortunately sufficiently definite to make their determination of practical value. I have taken as the melting point the lowest temperature at which a small piece of the brick could be distinctly seen to flow. Experiments showing the degree of definiteness of this temperature will be described.

The melting point of a brick is, of course, not the only property to be considered in determining its fitness for a given purpose. Its crushing strength cold, its behavior under load conditions when heated, and its resistance to fluxes may all be important. The present paper deals only with melting points.

A large number of the brick samples tested were kindly furnished by Dr. A. V. Bleininger, being identical with samples tested by Bleininger and Brown[1] under load conditions at 1300° C, this work being carried out at the Pittsburgh testing station of this bureau, which station was at that time the technologic branch of the United States Geological Survey. The chemical composition, crushing strength cold, porosity and density of these samples are given by Bleininger and Brown. They also determined the Seger

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[1] Bureau of Standards Technologic Papers, No. 7; 1912.

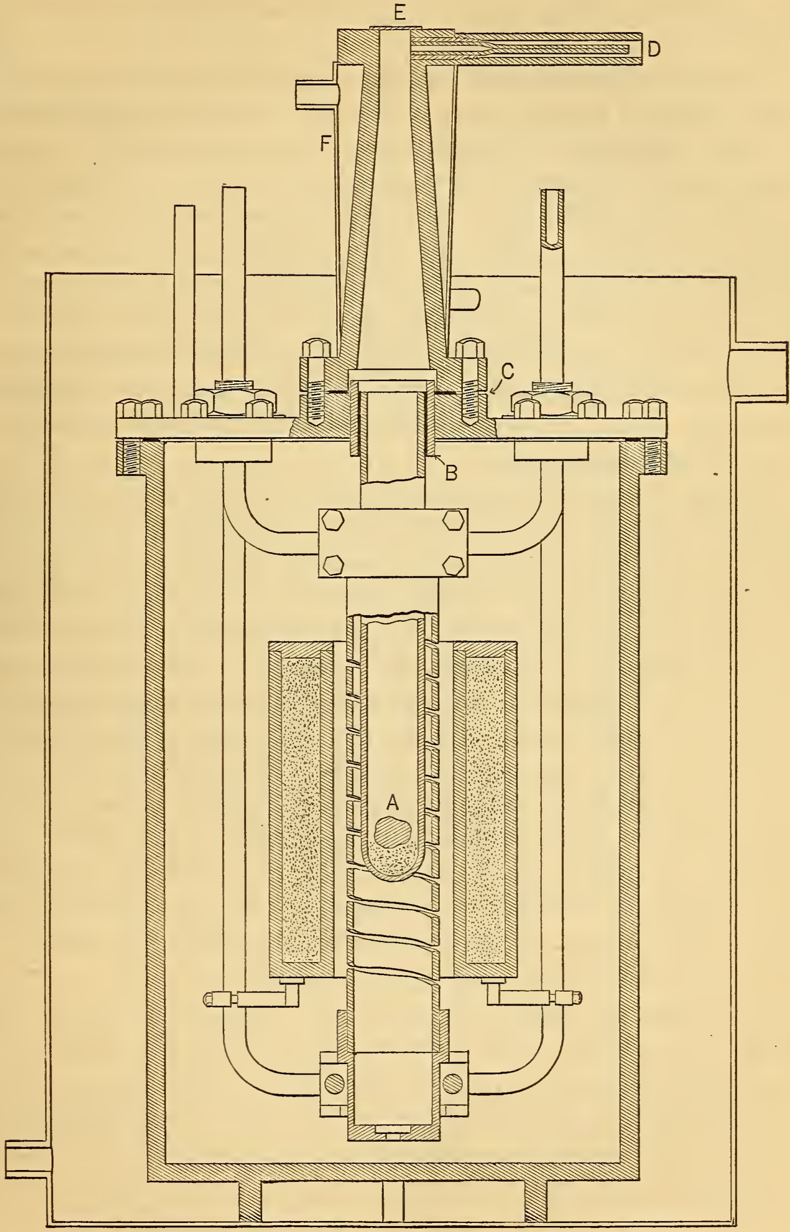

Fig. 1

cone numbers corresponding to the softening temperatures of the bricks.[2]

It is difficult to interpret results obtained with Seger cones in terms of actual temperatures, for it has been shown by Simonis [3] that the falling over of Seger cones, of certain numbers at least, depends not only upon the temperature, but also to a large extent upon the rate of heating. This observation has been confirmed in this laboratory, using the cone numbers having softening temperatures in the same range as fire brick, and it has appeared that in many cases the melting point of a fire brick, defined as stated above, is more definite than the softening temperature of the corresponding Seger cone. It has, therefore, appeared preferable to use a more direct and more accurate method of determining temperatures.

The experiments were conducted in an Arsem graphite resistance vacuum furnace [4] (Fig. 1). In some preliminary experiments upon fire bricks samples were heated in graphite crucibles and exposed to such gases as might remain in the furnace. Although the pressure was kept as low as 2 mm of mercury, there was still a slight reducing action upon the brick, and some samples were blackened superficially. Subsequent work has shown that the action was insufficient to produce any very great difference in the results, but, as a matter of precaution, the specimens were protected by a refractory tube (*A*, Fig. 1) being placed upon a bed of white alundum (fused alumina) in the bottom of the tube. This tube was supported by the brass collar, *B*, in which it was made to fit tightly by winding with asbestos string, or otherwise, and which was soldered to the lead gasket by which the joint *C* in the top of the furnace was made air tight.

The refractory tubes were made of a mixture of 72 per cent kaolin and 28 per cent alumina, these being the proportions

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[2] See also Hofman and Demond: Trans. Am. Inst. Mining Eng., **24**, p. 42; 1894. Hofman: Trans. Am. Inst. Mining Eng., **25**, p. 3, 1895; **28**, p. 435; 1898. Hofman and Stroughton: Trans. Am. Inst. Mining Eng., **28**, p. 440; 1898. Gary: Mitt. k. tech. Versuchsanstalten, **14**, p. 63; 1896. Jochum: Thonindustrie Zs., **27**, p. 764; 1903. Weber: Trans. Am. Inst. Mining Eng., **35**, p. 637; 1904.

[3] Simonis: Thonindustrie Zs., **32**, p. 1764; 1908.

[4] Arsem: J. Am. Chem. Soc., **28**, p. 921, 1906; Trans. Am. Electroch. Soc., **9**, p. 153, 1906.

for the formation of sillimanite, Al_2O_3,SiO_2. According to Shepherd and Rankin[5] pure sillimanite melts at 1811°. The sillimanite tubes were found to melt at about this temperature. Kaolin tubes were used for some samples of relatively low melting points. Either sillimanite or kaolin tubes were used with all fire bricks melting below 1800°. The chromite and magnesia bricks were melted in graphite crucibles without protecting tubes. The experiments were made rapidly and the graphite had little action on the samples until after fusion had occurred and the melting point had been determined. Magnesia tubes may be used in place of sillimanite tubes, and they have the advantage of a higher melting point; but all magnesia tubes that have been tried have been found to be very easily cracked by changes of temperature, and to be so porous as to afford little protection from the gases of the furnace. When a sillimanite or kaolin tube was used, 20 to 30 minutes was consumed in heating the furnace to a little below the melting point of the sample. This initial heating could be performed much more quickly, so far as the furnace is concerned, but more rapid heating would be likely to crack the tube. When the temperature had nearly reached the melting point it was raised much more slowly. When these protecting tubes were used the samples showed only very slight signs of reduction. In certain experiments made with this apparatus—for example, the determination of the melting point of platinum as a check upon the work—it has been desirable to still further diminish the chances of the presence of reducing gases. This has been accomplished by the device shown at *D* in Fig. 1, which consists of a glass tube drawn out into a fine capillary and leading into the furnace. An outer brass tube protects the capillary. This capillary admits a slow current of air to the inside of the refractory tube, and the air passes out through the pores of the tube into the interior of the furnace. By working the vacuum pump continually the pressure in the furnace was kept down to a few millimeters of mercury, in spite of the influx of air. This process was, of course, injurious to

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[5] Shepherd and Rankin: Am. Jour. Sci., 28, p. 301; 1909.

the graphite heater, but several such experiments could be made with one heater before it was burned out.

While the sample was being heated it was observed through the window *E* at the top of the furnace. A glass window was used here in place of the mica window provided by the makers of the furnace. The glass was attached by a mixture of rosin and beeswax. To keep this material cool an additional water jacket, *F*, was applied to the part of the furnace projecting above the water of the tank.

The temperatures were determined by means of a Morse optical pyrometer of the Holborn-Kurlbaum type,[6] which was sighted vertically downward through the furnace window. The current in the pyrometer lamp was measured with a Siemens and Halske milliammeter with a shunt. This instrument was calibrated several times, and its very small temperature coefficient was taken into account. Its readings are certainly reliable to 0.001 ampere and probably to 0.0005 ampere.

The carbon-filament pyrometer lamp was one of the standard lamps of the bureau, and had been thoroughly aged, and had been accurately calibrated by several observers before the beginning of the investigation, and again, by a different method, toward its close. In the first calibration it was sighted into a platinum-resistance furnace in which black-body conditions were obtained, and the temperature of which was measured by platinum, platinum-rhodium thermocouples. These thermocouples had been calibrated against the freezing points of pure metals. In the second calibration the lamp was calibrated against the freezing points of metals directly, without the intermediation of thermocouples. The metals used were copper, silver, and the copper-silver eutectic, which freeze at 1083°, 961°, and 779°, respectively. These were placed in Acheson graphite crucibles of the form shown in Fig. 2, about 100 grams of metal being used in each. The pyrometer was sighted downward into the central graphite tube, of which the inside diameter was 6 mm and the wall thickness 1

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[6] Holborn and Kurlbaum: Sitzber. d. k. Akad. d. Wissensch. zu Berlin, June 13, p. 712, 1901; Ann. d. Phys., **10**, p. 225; 1902. Waidner and Burgess: Bull. Bureau of Standards, **1**, No. 2; 1904. Mendenhall: Phys. Rev., **33**, p. 74; 1911. Henning: Zs. f. Instrumentenkunde, **30**, p. 61; 1910.

mm. The crucibles were heated or cooled gradually, while observations were made every 10 to 20 seconds, and the resulting heating or cooling curves were plotted. A typical curve is shown in Fig. 3. In this diagram a change of temperature of 1° corresponds to a change of current of about 0.0005 ampere. By means of the three known temperatures, the relation between the current in the lamp and the temperature was expressed in the form, $C = a + bt + ct^2$.

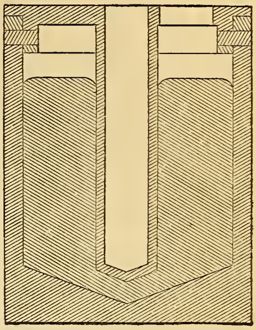

Fig. 2

This method presented two possible sources of error to be especially considered, namely, the conduction of heat along the inner graphite tube, and the departure of the interior of this tube from black-body conditions. If the conduction of heat along the tube introduced appreciable error, the observed temperatures would be too high with rising temperature, when the furnace heater would be hotter than the metal, and too low with falling temperature, when the reverse would be true. The fact that the results from the heating curves obtained with copper and silver were in very close agreement with those from the cooling curves shows that the errors from conduction are negligible; and these and

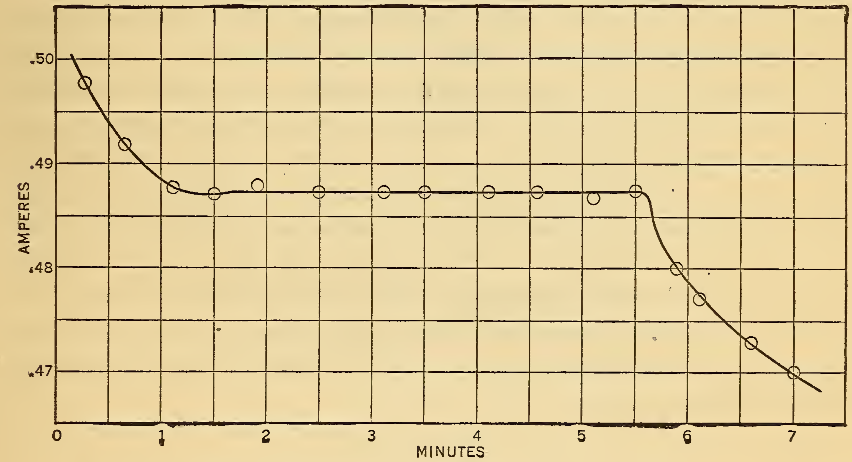

Fig 3.—*Calibration of lamp at freezing point of silver*

numerous other observations have indicated that such a tube when made of graphite, which is itself nearly perfectly black, is a very good black body. Using the copper-silver eutectic somewhat unsatisfactory results were obtained with heating curves, the trouble appearing to be in the behavior of the alloy rather than in the temperature measurement. In this case only cooling curves were used for the calibration. The calibration of the lamp at this temperature is relatively unimportant, since none of the melting points here given correspond to a lamp temperature below the melting point of silver. The results of this calibration agreed with those obtained by the first method within 1°, from 700° to 1300°, which includes all lamp temperatures used in this work. Near the close of the investigation the pyrometer lamp was accidentally overheated, and it was necessary to recalibrate it. This time, only the cooling-curve method was used.

As the melting points to be measured were above the working limit of the pyrometer lamp, an absorption glass was interposed between the pyrometer and the furnace. This glass was calibrated as follows: In the Arsem furnace was placed a graphite crucible, the interior of which, when the furnace was hot, furnished a field of very uniform brightness. A steady current was passed through the furnace and the temperature allowed to become stationary. The temperature was then measured with the pyrometer, and also the apparent temperature as observed through the absorption glass. If T_1 is the absolute temperature, observed without the glass, and T_2 is the apparent absolute temperature observed with the glass, we have the following relation, which is easily deduced from the Wien radiation law:

$$\frac{1}{T_2}-\frac{1}{T_1}=A$$

The constant, A, was determined in this way with several different furnace temperatures, and the results were in close agreement. The mean value of A was 0.0002249. The values of the constants of Wien's law do not enter into the work.

The glass furnace window absorbed and reflected a small amount of light and it was necessary to apply a correction for this. The constant of this glass was determined in the same way as that of the absorption glass, and was found to be 0.0000040. The correction in degrees depends, of course, upon the temperature; it is about 15° at the melting point of ordinary fire brick.

As a check upon the whole apparatus, a determination of the melting point of platinum was made. A piece of platinum foil was placed within two magnesia tubes, one within the other, in the place of the single tube shown at *A* (Fig. 1), and a slow current of air was admitted at *D*. Fairly good black-body conditions were obtained, but the platinum was faintly visible. The observed melting point was 1750°. The value accepted by the Bureau of Standards as the melting point of platinum is 1755°. After the pyrometer lamp had been overheated and recalibrated, this test was repeated. In place of magnesia tubes, tubes of a mixture of magnesia and alumina,[7] obtained from the Königliche Porzellan-Manufactur, Berlin, were used. These were found to be more satisfactory than magnesia tubes, being much less porous and less easily cracked. The material softens at a little above the platinum point. Using a single tube the value 1746° was obtained, with two tubes 1750°. Tests were also made in an iridium tube furnace, where there was no possibility of a reducing atmosphere. So good black-body conditions were obtained that the platinum was quite invisible. A piece heated to 1747° and removed was found not melted; one heated to 1759° and removed was found melted.

The use of the optical pyrometer depends upon the assumption that the object sighted upon emits black-body radiation. This is the case if the object is within a vessel at uniform temperature, and is viewed through a relatively small opening in the vessel. This can readily be accomplished in the apparatus used, but under such conditions it would be impossible to distinguish the sample, owing to the uniform brightness of the sample and its environs. In practice, the sample was placed slightly below the hottest part of the furnace, in such a position as to be barely distinguishable. This requires only a temperature difference of about 5°. When the temperature of a sample had almost reached the melting point, the temperature was raised more slowly and the sample was

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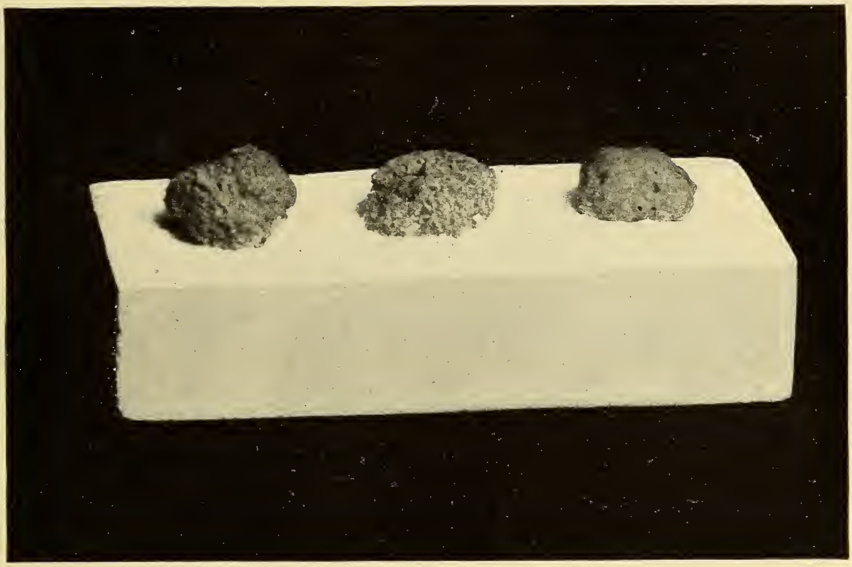
[7] Heinecke, Zs. angew. Ch., **21**, p. 687; 1908.

observed continually. When it was seen to melt, a final temperature measurement was made and the heating current was immediately stopped, thus preventing the temperature from rising any higher. The observation was then verified by an examination of the sample after its removal from the furnace.

An idea of the degree of definiteness of the melting points may be obtained from Fig. 4 and Fig. 5, in which are shown samples which have been heated to successively higher temperatures. Fig. 4 may be taken to represent the usual degree of definiteness. The sample heated to 1680° C appears unaffected; that heated to 1695° shows very slight signs of fusion; that heated to 1705° is completely fused; 1705° was taken as the melting point. Fig. 5 shows perhaps the least definite melting point found. Complete fusion occurred at 1620°, but there was partial fusion at considerably lower temperatures; 1600° was taken as the melting point.

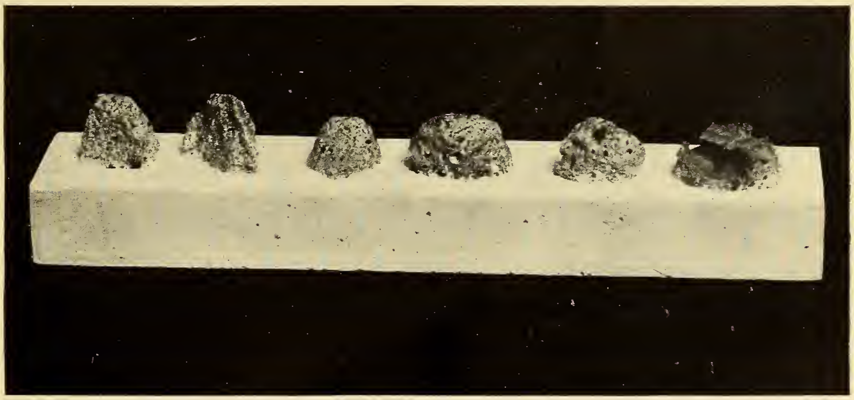
Near the melting point samples were heated at the rate of about 10° per minute. This rate of heating was quite slow enough, as a temperature measurement can be made in a few seconds, and a slower rate of heating near the melting point was found to give the same results. The entire time required for heating a sample from room temperature to its melting point was 30 to 40 minutes. Experiments were made to determine whether samples could undergo any change in melting point as the result of heating in the vacuum furnace. Samples of various kinds were heated in the furnace to about 1550° for about six hours, and their melting points were then determined. The results are given in Table I.

It appears from the table that Nos. 14 and 27, which are bricks of low melting point, melted at a somewhat higher temperature after long heating, while the other bricks were unaffected. The melting point of No. 27 is less definite than that of most bricks. This increase of melting point might be accounted for upon the assumption that the brick consisted of two materials existing in distinct particles, one sort having a lower melting point than the other, these two materials becoming run together after long heating near the melting point, producing a material of higher melting point than that of the more fusible of the original materials. The appearance of samples of No. 14 and No. 27 after heating for



1680° 1695° 1705°

Fig. 4



1570° 1580° 1590° 1600° 1610° 1620°

Fig. 5

TABLE I

Sample	Material	Determinations of melting point without previous heading	Determinations of melting point after six hours heating
		deg	deg
14	Fire clay	1635	1655
		1630	1655
27	do	1600	1635
		1610	1630
		1600	1635
		1585	1640
		1595	....
48	do	1710	1705
		1705	1695
		1705	....
		1700	....
		1705	....
20	Bauxite	1740	1740
		1745	1730
41	Silica	1705	1695
		1700	1700
		1695	....
		1695	....

a short time to near the melting point supports this explanation. Sample No. 27 is shown in Fig. 5. After the six hours heating they appeared more uniform. Long heating in a vacuum would tend to vaporize the more volatile constituents of the brick, and this might produce an appreciable increase of melting point.

The most common fire brick are those made of clay, of which the essential ingredient is kaolin, $Al_2O_3,2SiO_2,2H_2O$. Shepherd and Rankin[8] found that the only compound of alumina and silica that can exist in the neighborhood of the melting points is sillimanite, Al_2O_3,SiO_2. It follows that clay when near its melting point is no longer a single compound but a mixture of Al_2O_3,SiO_2 and SiO_2. Hence, the melting point of pure kaolin, like that of fire brick, can not be expected upon theoretical grounds to be perfectly definite, but fairly definite results are obtained when the

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[8] Loc. cit.

visible flow is taken as the criterion of fusion, as in the case of fire brick. Two samples of white kaolin, one English, the other probably English also, both melted at 1740°. A sample of brownish white German kaolin melted at 1735°. Therefore, the highest melting point that can be attained in brick containing nothing more refractory than kaolin is 1740°. When an excess of alumina is present, as in bauxite brick, the melting point may be higher.

A few brands of silica brick were tested. Pure silica melts at about 1600°.[9] However, the fused silica possesses such extreme viscosity near the melting point that it does not flow or change shape distinctly until considerably higher temperatures are reached. I have obtained 1750° as the *apparent* melting point of pure silica, i. e., the temperature at which it flows distinctly. This temperature, however, is naturally a very indefinite one. The temperature at which silica bricks flow distinctly is more definite.

Bauxite brick are made from bauxite, $Al_2O(OH)_4$, containing usually considerable quantities of other material. Bauxite, of course, becomes converted to alumina, Al_2O_3, when heated. Pure alumina melts at about 2010°.[10] None of the bauxite bricks examined approached this melting point. A single sample of bauxite was tested and found to melt completely at 1820°, the centers of the nodules melting at 1790°. A sample of bauxite clay melted at 1795°.

One brand of magnesia brick was examined and found to melt at 2165°. The melting point of pure magnesia is more or less in doubt at present. It is to be expected that it would be higher than that of the impure magnesia brick. This brick contained a large quantity of iron and was of a dark brown color. When heated for a few minutes to near its melting point it became white. This occurred both in the electric vacuum furnace and in an oxy-hydrogen flame. It might result either from the vaporization of the iron, or from the formation of a colorless compound of iron and magnesia. An analysis of the specimens has shown that the heated samples contain much less iron.

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[9] Day and Shepherd: J. Am. Chem. Soc., 28, p. 1089; 1906.

[10] A paper on the melting points of pure refractory oxides is in preparation.

One brand of brick made from chromite, FeO,Cr_2O_3, was examined. It was found to melt at 2050°. A sample of natural chromite from a different source melted at 2180°.

Silicon carbide, SiC, has been used as a refractory material. At very high temperatures it decomposes without melting. Tucker and Lampen[11] state that it decomposes at 2220°. Results in close agreement with this have been obtained by Gillett[12] and by Saunders.[13] This is undoubtedly correct for decomposition under ordinary conditions, but silicon carbide can be heated for a short time to much higher temperatures without complete decomposition. It was thought that by very rapid heating it might be possible to reach the melting point of the silicon carbide before decomposition was complete, although such a melting point would probably have only theoretical interest. With this object, a few grams of silicon carbide was placed in a graphite crucible, which was placed in a second larger graphite crucible to give more uniform temperature, and the whole was heated in an arc furnace at atmospheric pressure, the temperature being measured by sighting the optical pyrometer through a narrow graphite tube inserted in the top of the crucible. A temperature of 2700° or more was reached and was maintained for a few minutes. The silicon carbide was about half decomposed, leaving a residue of carbon, but showed no indication of fusion.

The results for fire brick will be found in Table II. Samples 1 to 26 are identical with the samples of the same numbers studied by Bleininger and Brown.[14] All the bricks tested were made in the United States. Each melting point given is the mean of at least two determinations. The mean of the melting points of the 41 samples of fire-clay bricks is 1649°.

The analyses of a large number of the fire-clay bricks studied are available,[15] but it does not seem possible to establish any very definite relation between composition and melting point. Since

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[11] Tucker and Lampen: Jour. Am. Chem. Soc., 28, p. 853; 1906.

[12] Gillett: Jour. Phys. Chem., 15, p. 213; 1911.

[13] Paper presented before the American Electrochemical Society, May, 1912.

[14] Loc. cit.

[15] Bleininger and Brown, loc. cit.

TABLE II

Melting Point of Various Bricks

Sample	Melting point	Sample	Melting point
Fire-clay brick:	Deg	**Fire-clay brick—Continued,**	Deg
1	1630	34	1570
2	1635	35	1650
3	1605	36	1590
4	1605	37	1650
5	1705	38	1660
6	1705	47	1725
7	1700	48	1705
8	1700	49	1715
9	1675	[16] 53	1635
10	1710	[16] 54	1635
11	1660	[16] 55	1685
12	1555	[16] 56	1605
13	1635	**Bauxite brick:**	
14	1630	19	1760
15	1655	20	1740
17	1650	44	1720
21	1615	45	1785
22	1640	46	1720
23	1660	50	1665
24	1660	51	1565
25	1715	52	1590
26	1695	**Silica brick:**	
27	1600	39	1700
28	1695	40	1705
29	1595	41	1700
30	1560	**Chromite brick:**	
31	1600	42	2050
32	1650	**Magnesia brick:**	
33	1655	43	2165

[16] Samples 53, 54, 55, and 56 are of the same brands as 4, 9, 7, and 17, respectively, but from different lots.

the bricks contain about eight different constituents in quantities sufficient to affect the melting point, and since the melting point may also be affected by lack of homogeneity in the material, it is obvious that a prediction of the melting point upon the basis of a chemical analysis would be uncertain. If the melting points are compared with the results of tests under load conditions at 1300°[17], it is evident that there is little relation between the two.

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[17] Bleininger and Brown, loc. cit.

SUMMARY

1. The melting points of 54 samples of fire brick, including fireclay, bauxite, silica, magnesia, and chromite brick, have been determined in an electric vacuum furnace, the temperature being measured with an optical pyrometer (Table II).

2. The following melting points of materials important in the manufacture of fire brick were determined:

	Deg.		Deg.
Kaolin	1740	Bauxite	1820
Pure alumina	2010	Bauxite clay	1795
Pure silica	[18] 1750	Chromite	2180

3. An improved method of calibrating the Holborn-Kurlbaum optical pyrometer is described.

Mr. H. P. Greenwald and Mr. S. E. Moore have rendered efficient assistance in carrying out this work.

WASHINGTON, June 15, 1912.

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[18] This is not the true melting point but represents approximately the temperature at which silica flows distinctly.