

b) Density of Solution: 3.0-3.1 g./cm<sup>3</sup>.  
 Temp. Coeff. of Density: -0.00147 g./cm.<sup>3</sup>/°C.  
 Samples: 11, Observations: 10.

Heating Rate (°C/min.)	0.1	1.0
$\sigma$ mean (°C)	0.102	0.281
$\sigma$ max. (")	0.155	0.488
$\sigma$ min. (")	0.062	0.151

The standard deviations of measurements with the water solution also increase approximately linearly with the heating rate, but its increase is far less than that of the bromoform-mixture. Then it can be said that, the water solution is more convenient than organic heavy liquids for rapid determinations at some expense of precision in density measurements.

As the densities of pure *s*-tetrabromoethane and bromoform are less than 2.9 g./cm<sup>3</sup>, these organic bromo-compounds are not applicable to density determinations of most lead-containing glasses of industrial uses (density: 3.0-3.1 g./cm.<sup>3</sup>). The water solution of the complex salt, on the other hand, having the maximum density of ca. 3.2 g./cm.<sup>3</sup>, is available for measurements almost all glasses for industrial purpose, by adjusting the density of the solution simply by evaporation or dilution. However, the solution of high concentration, having high viscosity, makes the measurements appreciably, inaccurate even if the same rate of heating of the bath is secured. The precision of measurements with a concentrated solution (density: 3.0-3.1 g./cm.<sup>3</sup>) is indicated in Table 2-b). Moreover, as the water solution is more stable to light, moisture and impurities than the organic bromo-compounds, it can be concluded that, the former is more favourable for factory practice of glass industry in this country.

### 13. Heat Resisting Enamel Coatings to be Applied on Nickel-Chrome Stainless Steel

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As stated before (*This Bulletin*, 23, 6 (1952)) the heat resisting enamel to be applied on nickel-chrome stainless steel should give the coating of high expansion, while, at the same time, it should be difficult fusible.

Obviously, however, it would be difficult to find out the composition satisfying simultaneously the both seemingly contradicting characteristics. Although, for example, it is easy to get a large size in expansion by substituting silica with alkali etc., the fusibility of frit decreases as the necessary result.

The authors, in an attempt to obtain some basic data which might prove of service to the production of good heat resisting enamels, have tried to make clear the effects of barium oxide, a conventional component in some high temperature enamels, on the two important properties mentioned above. For this purpose the effects of the successive substitution of other components with BaO in frit on some physical properties have been studied. The comparative studies on the effect of the different kind of mill addition on the same properties have also been carried out.

(1) **The relation between the amount of barium oxide and the properties of coating.** In Table 1 are given the compositions calculated from batch of the frits, Ba-20~Ba-60, prepared by substituting successively SiO<sub>2</sub>, Na<sub>2</sub>O and K<sub>2</sub>O with BaO in the basic frit Ba-O.

No. 331 is the frit known in the market as a heat resisting type and No. 331' is the same other than the elimination of BeO.

In Table 2 are cited the values of the coefficient of thermal expansion and softening and transformation temperatures. The expansion was measured with rod samples prepared by drawing the rods from the remolten frits. The firing temperatures, taken provisionally as the comparative values of fusibility, are the temperature at which the surface of coating applied on metal by the slip, prepared by mixing 100 frit, 5 clay and 0.2 MgSO<sub>4</sub> with the proper amount of water, becomes glossy during the predetermined interval of 3 minutes. The softening and transformation temperatures were determined in an ordinary way from the characteristic points in the expansion curves.

The figures in the table indicate clearly that the addition of BaO leads to the remarkable result of increasing expansion along with the decreasing fusibility. Especially the profound effects were observed by the addition more than 40% BaO. The frit No. 331 as well as No. 331' have shown the similar characteristics as Ba-40 and Ba-50.

Table 1

	SiO <sub>2</sub>	B <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	CaF <sub>2</sub>	CaO	BaO	ZnO	BeO
Ba-0	73.0	4.0	11.0	5.0	2.0	—	—	5.0	—
Ba-20	55.0	4.0	8.0	4.0	2.0	—	20.0	5.0	—
Ba-40	44.0	4.0	—	5.0	2.0	—	40.0	5.0	—
Ba-50	39.0	4.0	—	—	2.0	—	50.0	5.0	—
Ba-60	29.0	4.0	—	—	2.0	—	60.0	5.0	—
331	38.0	6.5	—	—	—	4.0	44.0	5.0	2.5
331'	39.0	6.7	—	—	—	4.1	45.1	5.1	—

Table 2.

	Ba-0	Ba-20	Ba-40	Ba-50	Ba-60	331	331'
Linear thermal expansion coefficient ( $\times 10^{-7}$ ), (20~350°C)	86	86	87	89	97	82	81
Firing temp. (°C)	770	765	960	965	970	910	900
Softening temp. (°C)	545	595	670	690	665	710	700
Transformation temp. (°C)	487	525	605	640	625	670	650

(2) **The influence of mill addition.** For the purpose of investigating the effect of mill addition, 10 parts of feldspar, alumina, silica *etc.* were respectively added to 100 parts of the frit No. 331' at the milling. The similar characteristic properties given in the preceding table have been measured, whose results are summarized in Table 3. In this case, however, the samples for measuring the expansion were prepared by the rod method given by Andrews (J. Am. Cer. Soc., 16, 338 (1933)). From the data it can be concluded that such mill addition as chromium oxide may be effective for raising the firing temperature without decreasing the expansion coefficient.

Table 3.

	CuO	NiO	CoO	Feldspar	Cr <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>
Linear thermal expansion coefficient ( $\times 10^7$ ), (20~350°C)	83	81	82	81	82	79	74	75
Firing temp. (°C)	895	895	900	920	925	920	945	975
Softening temp. (°C)	625	680	675	695	710	710	720	710
Transformation temp. (°C)	575	630	615	635	650	665	650	650

#### 14. Studies on the Joint Reaction Concerning Acetylene

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We have examined the joint reaction concerning acetylene. In the present paper, the results of the direct joint reaction and related discussions are reported.

Diethylamine and acetylene were jointed by CH<sub>2</sub>O and propargyl amine was produced. On the other hand, dimethylamine hydrochloride did not react under the same condition of reaction. Ammonium chloride and