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BULLETIN No. 9. DEPARTMENT OF CERAMICS

C. W. ROLFE, Director

A CHEAP ENAMEL FOR STONEWARE

By R. T. STULL

THE VISCOSITY OF CLAY SLIPS

By A. V. BLEININGER

NOTE ON SOME FUSION CURVES

By A. V. BLEININGER

1907-1908

PUBLISHED FORTNIGHTLY BY THE UNIVERSITY

A CHEAP ENAMEL FOR STONEWARE.

BY

R. T. STULL, Urbana, Ill.

The object of this work was to investigate some of the possibilities of producing a cheap enamel, primarily for the improvement of the better grades of stoneware, and secondly, cheap enamels for other purposes at temperatures lower than those required for the stoneware industry. The main object sought for was an enamel which would be whiter and better than the commercial stoneware glaze; something on the order of a tin enamel in appearance, though much cheaper.

No special field was plotted for investigation. The method first adopted for carrying out the work was to make up a series of glazes, then select the most promising one, after firing, for the upper member of the next series, and continuing thus.

The method of preparing and applying each glaze in this work was the same, so as to obtain an accurate comparison of results in so far as mechanical and physical application was concerned. The batch weight of each glaze was calculated from its respective formula, weighed separately, (500 gm.), ground wet for two hours in a small porcelain ball mill, passed through a 100 mesh sieve, placed in a Mason jar, allowed to settle and the water decanted. Sufficient water was then added in order to "set" the glaze at 1.5 B. & L. hydrometer (equal to about 48 to 52 Beaume). Each glaze was then ready for dipping.

Trials were made from ready prepared stoneware clay from Moumouth, Ill. A test showed that the clay when made into slip form readily passed a 60 mesh screen but left quite a residue of dark colored granular particles on the 100 mesh screen.



Trial pieces nested in "Fives" ready for the Sagger.

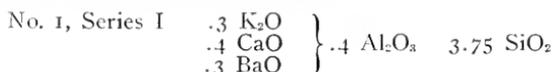
The trial pieces were made in the form of small "milk" crocks 3" across the top and 1½" high. These were made in plaster molds on a jigger and finished outside on a "potter's chum."

The trials were dipped in the bone dry condition, each piece being immersed in the glaze about 2½ seconds. The dipped pieces were then placed on a potter's wheel, the glaze turned off from the rim and shoulder and the pieces nested in "fives" for setting in saggars.

In order to determine the relative fusibility of the glazes, cones were made from each mixture and the bending points or softening temperatures determined by Seger cones and the Le Chatelier pyrometer.

All the glazes in this work were fired in the down-draft open fire kiln of the ceramic department at the University of Illinois. Series I was fired to cone 6 in 12 hours, coke being the fuel used.

For the starting point of this work, a glaze having the following formula* was selected:



This formula produces a clear bright glaze having a range of temperature from cone 4 to cone 8, within which it is a good glaze.

*Sprechsaal; 1905, No. 42.

SERIES I.

Formula.

Glaze	K ₂ O	CaO	BaO	ZnO	Al ₂ O ₃	SiO ₂
No. 1	.3	.4	.30	0	.4	3.75
No. 2	.3	.4	.20	.10	.4	3.75
No. 3	.3	.4	.15	.15	.4	3.75
No. 4	.3	.4	.10	.20	.4	3.75
No. 5	.3	.4	0	.30	.4	3.75

Percentage Batch Weights.

Brandswine Feldspar	Georgia Kaolin	Whiting	Barium Carbonat	Zinc Oxide	Flint	Temperature at which Glaze Softens.
42.09	6.50	10.07	14.89	26.45	1200°
43.36	6.70	10.38	10.22	27.24	1190°
44.02	6.86	10.54	7.78	3.20	27.66	1180°
44.70	6.00	10.70	5.27	4.33	28.10	1150°
46.14	7.12	11.04	6.71	28.99	1110°

SERIES II.

Formule.

Glaze	K ₂ O	CaO	ZnO	Al ₂ O ₃	SiO ₂	P ₂ O ₅
No. 5	.3	.4	.3	.4	3.75	0
No. 6	.3	.4	.3	.4	3.75	.03 ₃
No. 7	.3	.4	.3	.4	3.75	.06 ₂₃
No. 8	.3	.4	.3	.4	3.75	.10
No. 9	.3	.4	.3	.4	3.75	.13 ₃

Percentage Batch Weights.

Brandswine Feldspar	Whiting	Boneash	Zinc Oxide	Georgia Kaolin	Flint	Temperature at which Glaze Softens.
46.14	11.04	6.71	7.12	28.99	1110°
46.10	8.28	2.84	6.70	7.12	28.96	1160°
46.05	5.51	5.70	6.70	7.11	28.93	1200°
46.01	2.75	8.54	6.69	7.10	28.01	1230°
45.97	11.36	6.69	7.10	28.88	1250°

The first series was made for the purpose of determining the relative fusibility, brilliancy and opacity induced by BaO as against ZnO, by gradual replacement. The formulæ and batch weights of series I are:

Appearance of Trials.

The softening temperatures expressed in degrees centigrade are given in the column at the right of percentage batch weights above. When taken from the kiln, all five glazes were well matured, and showed no bad defects. After two days' time crazing began to appear in all glazes except No. 5. Crazing was worst in No. 1, gradually decreasing toward No. 5, which was free from crazing.

No. 5 is the most fusible glaze in the series and has the best brilliancy and opacity; opacity not very great.

SERIES II.

No. 5 from series I being the best glaze, was selected for the starting point of Series II, in which the CaO from whiting was gradually replaced by CaO from bone ash, all other members remaining constant. This, of course, increased the acidity by the amount of P_2O_5 brought in with the bone ash.

After series II was made, it was thought that the use of raw bone ash might cause defects such as blistering and curdling, consequently series III was made, keeping the same formulæ as in series II but having the bone ash calcined with flint. This calcine was marked "A."

Calcine A Formula:	Percent Batch Weights:
1. CaO, 1. SiO ₂ , $\frac{1}{3}$ P ₂ O ₅	Bone Ash63.19
Combining weight 163 $\frac{1}{3}$.	Flint36.81

This mix gave a friable mass when burned to cone 6.

SERIES III.

Percentage Batch Weights.

°C.

Glaze	Brandswine Feldspar	Whiting	Calcene A	Zinc Oxide	Georgia Kaolin	Flint	Softening Temperature
No. 6A	46.09	8.28	4.50	6.70	7.12	27.31	1160°
No. 7A	46.05	5.51	9.00	6.70	7.11	25.63	1200°
No. 8A	46.01	2.75	13.50	6.69	7.10	23.95	1230°
No. 9A	45.97	17.97	6.68	7.10	22.28	1250°

All glazes except No. 5 in series II flaked off after dipping. If the trial pieces were jarred after drying, the glazes shelled off leaving the trials bare. None of the trials in series III showed signs of flaking after dipping.

In order to test the relative effects of raw and calcined bone ash, 3% of dextrine was added to glazes 6, 7, 8 and 9 of series II after which they dipped very nicely, giving no trouble in flaking when dry, but later flaked some during burning.

Glazes from series II and III were placed in saggars and fired to cone 6 in 11 hours.

Appearance of Trials.

There is no appreciable difference in fusibility between those glazes which contained raw bone ash and the corresponding ones in which it had been calcined with flint.

Glazes No. 5, 6 and 7 of series II and 6A and 7A of series III were matured; Nos. 8 and 9, 8A and 9A were dull in appearance due to insufficient temperature to mature them.

Since the members in the two series highest in bone ash were immature, it was decided to make a second firing of series II and III to cone 8. The trials were set and the kiln finished in 14 hours. On opening the kiln, the cones showed evidence of a much higher temperature than cone

8, probably cone 9 at least. All the softer glazes showed evidence of overfiring, showing a sort of "pitted" or egg-shell surface. 8 and 8A, 9 and 9A were nicely matured. No blistering or serious crawling occurred. Nos. 9 and 9A were whitest, whiteness decreasing toward No. 5. Nos. 9 and 9A were crazed in fine meshes, the crazing gradually decreasing toward No. 5.

SERIES IV.

No. 9A is the best as to working qualities and whiteness, though crazed and too refractory for the best development of the stoneware body used. This glaze was selected for the upper members of series IV. The object undertaken was to "soften down" 9A and at the same time overcome the crazing and retain as much of its whiteness as possible.

In order to do this the most promising method seemed to be to introduce B_2O_3 in the form of calcium borate. By replacing part of the SiO_2 with B_2O_3 , the softening of the glaze and the overcoming of crazing might be accomplished. The introduction of calcium borate was looked upon as a possibility for retaining the whiteness, since it sometimes produces opalescence in glazes.

In order to save the time and trouble of making a fritt, the naturally occurring mineral "Colemanite" was used. This is the mineral from which our borax and boracic acid is largely obtained. Its formula is $2 CaO, 3 B_2O_3, 5 H_2O$. Molecular wt.=412. Combining wt. 1. RO=206. According to Dana* colemanite crystallizes in the monoclinic system, usually in short prisms. Cleavable, b—highly perfect; c—distinct. Before the blowpipe; decrepitates, exfoliates, sinters and fuses imperfectly.

Series IV was produced by gradually replacing part SiO_2 in glaze No. 9A, .2 equivalents at a time, by B_2O_3 in the form of colemanite, up to 1.0 equivalent of B_2O_3 . By introducing 1.0 B_2O_3 as colemanite, .62½ CaO was carried in. This replaced .3 ZnO and .32½ CaO from bone ash, giving glaze No. 14 in the table below:

*A Text Book of Mineralogy, pp. 519, 520.

SERIES IV.

Percentage Batch Weights.

°C.

Formula.

Glaze	K ₂ O	CaO	ZnO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	B ₂ O ₃	Brandwine Feldspar	Colemanite	Calcine A	Zinc Oxide	Georgia Kaolin	Flint	Solventing Temperature
No. 9A	.3	.40	.30	.4	3.75	.133	0	45.97	17.96	6.67	7.10	22.28	1250°
No. 10	.3	.46	.24	.4	3.55	.169	.2	45.60	7.50	14.55	5.30	7.01	20.04
No. 11	.3	.52	.18	.4	3.35	.084	.4	45.21	14.85	11.20	3.95	6.98	17.81
No. 12	.3	.58	.12	.4	3.15	.060	.6	41.85	22.11	7.90	2.60	6.92	15.62
No. 13	.3	.64	.06	.4	2.95	.035	.8	41.52	29.28	4.37	1.31	6.88	13.61
No. 14	.3	.70	0	.4	2.75	.011	1.0	44.15	36.26	1.42	6.82	11.35

The trials, and cones made from the glazes for the fusibility test, were set in the kiln in the same manner as in the previous burns. Before a dull red heat was reached, all the cones made from those glazes containing colemanite crumbled to a fine powder on the placque. No. 9A, containing no colemanite, remained standing. Powdered colemanite dehydrates at a very low heat, swelling to nearly twice its original volume without softening, which accounts for the "falling to powder" of the cones made from those glazes in which colemanite was present.

The first intention was to fire the trials to cone 6. From the behavior of the cones it was assumed that the colemanite glazes would crumble from the trials, hence the kiln was finished at cone 8 in order to mature No. 9A.

Appearance of the Trials.

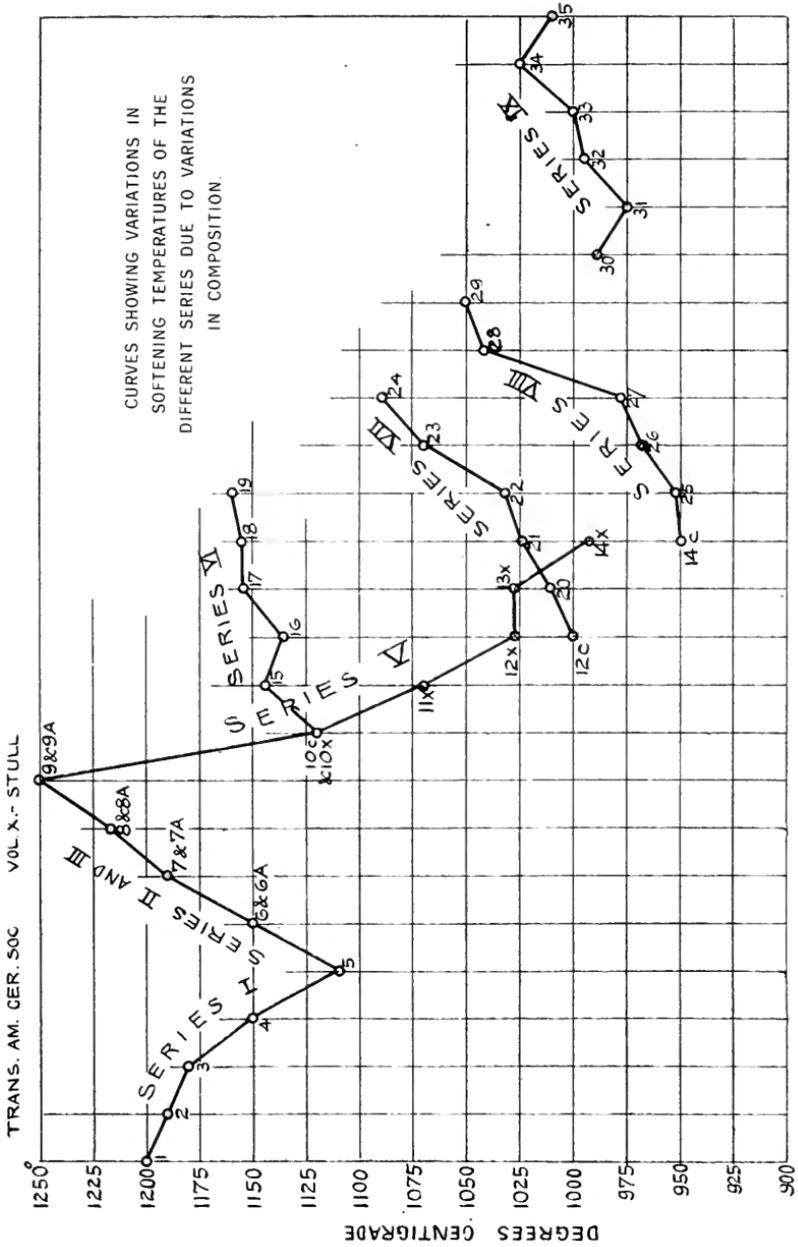
No. 9A softened at 1250°C the same as in the previous burn, going down with cone 6. It was impossible to determine the softening points of the other members of this series since their cones crumbled to powder in the early stage of the burning.

No. 9A matured nicely, is a little whiter than in the previous burn.

Nos. 10, 11, 12, 13 and 14 crumbled off from the outsides of the crocks leaving them bare, and fell to the bottom on the insides, fusing in thick layers. No accurate conclusions could be drawn from these except that they were much more fusible than No. 9A and overfired.

This is as far as the work has been carried. The next step will be to make a series in which the colemanite has been dehydrated after the same formulae as those in series IV.

According to the composition and low temperature of fusion of colemanite, it is undoubtedly a valuable flux for the introduction of B_2O_3 in an insoluble form. If it can be had in a reasonable state of purity in sufficient quantities, and at a moderate price, a large portion of the trouble



and expense of fritting can be eliminated. It will also be valuable in simplifying the leadless glaze problem, as well as a valuable flux for hardening terra cotta slips for low fire work.

DISCUSSION.

Mr. Purdy: I will ask Mr. Stull two questions. One is as to the calcium phosphate. We know that increased whiteness and opacity of the glaze can be secured by use of bone ash, but can you use it to any great extent without having crawling?

Mr. Stull: I have not experimented extensively in using bone ash for a glaze ingredient, but here used .4 CaO as bone ash and there was no evidence of crawling. Whether more could be used, I do not know. It materially increases the temperature necessary to mature it.

Mr. Purdy: Bone ash, according to my experience, is a very dangerous substance to use in a glaze of that kind. Another question I will ask, in harmony with Prof. Binns' paper; does B_2O_3 tend to increase the opacity of the glaze? Are you getting a whiter glaze with increased content of B_2O_3 ?

Mr. Stull: I believe it depends on the composition of the glaze whether it imparts whiteness or not. In itself, I do not believe it does. But colemanite is borate of lime, practically insoluble when cold, though it fuses by itself at a low heat, and the theory is advanced that whiteness can be obtained by incorporating calcium borate into the glaze, which precipitates or segregates within the glaze. That is what I intend to follow out. The line of inquiry will be changed from time to time as results from the kiln warrant. I do not believe that B_2O_3 can take the place of Al_2O_3 , or that it will act as Al_2O_3 does. In nature, in all the boracic acid minerals, B_2O_3 takes the part of an acid.

Mr. Humphreys: I will ask Mr. Stull what form of whitening he used. I think that is important in comparing with bone ash for temperature. In some recent experi-

ments I found as much as a difference of two cones in the different whittings.

Mr. Stull: I am not prepared to say where it was obtained. I found it in the laboratory there.

Mr. Purdy: The whiting was ordered from the Illinois Supply Co., East St. Louis.

Mr. Mayer: Mr. President, I was not present when all the paper was read, but I heard him mention colemanite. I made some experiments with some colemanite, the analysis of which was:

B ₂ O ₃	54.80
CaO	29.50
H ₂ O	13.50
Carbonate & Sulphate of Lime and Silica	2.00
	99.80

The experiments were made with a raw glaze of exactly the same composition of the fritted glaze we were using right along. The note I made was, "good glaze, apparently as good, as brilliant as the ordinary glaze, suits colors as well as our regular glaze." It was much better color than our own glaze. I have experimented largely with different forms of colemanite. There is only one discouraging feature and that is we cannot buy it cheaper than borax and boracic acid. Did Mr. Stull ask the price of this colemanite? I found that the trouble. We could get any amount of it and extremely pure, very little impurity in it—white as snow; but they want an unearthly price for it. I did not get mine from the Pacific Coast Borax Co. I got it from a firm that had just found some deposits of extreme purity. The only trouble is the high price the people want for it. In practice they make glazes identically the same. I have not seen any peculiarity about it.

Mr. Binns: I will ask Mr. Mayer if he dehydrated the colemanite?

Mr. Mayer: I just used the colemanite as it comes from the mines.

Mr. Binns: And you did not experience the trouble Mr. Stull had on account of the pulverizing of the glaze?

Mr. Mayer: I never had any such experience, and I used colemanite from three different firms. The last one I experimented with was an extremely pure sample. I never saw that difficulty in any of them. It is extremely hard, very difficult to grind. I broke several mortars before I learned how to break it up.

Mr. Binns: Although not germane to this discussion, Mr. Chairman, I want to speak concerning Mr. Stull's last statement. In mineralogy there is one case of boron as a base, as I said yesterday. Datolite is a silicate of boron and calcium. The claim which I made yesterday was for boron in the presence of silica, which must not be ignored. I granted that boron acted as an acid in the presence of bases.

I am interested in Mr. Stull's optimism when he thinks the difficulty of leadless glazes may be solved by the use of colemanite. I do not think the difficulty of a leadless glaze lies in the expense of fritting, rather in the manipulation of the process. I am interested in the development of the use of bone ash, and there are one or two points which seem to me need more light. My experience has been rather on the line of Mr. Purdy's', that bone ash is dangerous to use. If intended to be used on a large scale, on a large mass of ware, that is where the trouble would come in.

I will ask Mr. Stull in that connection what his experience was in calcining bone ash with flint; to what temperature he took it; what he tried to accomplish, and if he thinks he accomplished his purpose?

Mr. Stull: Answering Mr. Mayer's question, these experiments were carried out on the strength of the new deposit which has been discovered, and I am told that the Pacific Coast Borax Co. have carloads of it. The representative of this company said, in answer to a question, that they could furnish it, but I do not know at what price. I

have not gotten the price yet. As I stated, we will have to investigate the matter of price.

The mineral which Mr. Mayer speaks of as colemanite is not colemanite, but partially dehydrated colemanite. It is of the same composition as colemanite except that it is partially dehydrated. There is a deposit in Oregon of this dehydrated colemanite. That, of course, being partly dehydrated would not decrepitate and would not give the trouble I experienced with colemanite, the trouble of the true mineral colemanite which contains 21.90% H_2O .

Replying to Mr. Binns' inquiry in regard to the use of bone ash, my experience is limited to this case. I had no trouble at all. I made three burns, six, eight and nine, and in no case did I run into blistering. The great difficulty I had with the raw bone ash was that it flaked off badly. It fell off by itself in the drying. The idea of calcining it with flint was that in the manufacture of bone china it is often found necessary to recalcine bone ash to prevent blistering, etc., and I thought it best to recalcine it with a little flint and grind it and use it in that form. There was no special reason except to get a soft mass—not to burn too hard, though it would not harden by itself.

Mr. Binns: I was born and raised on bone china and that is news to me. The calcining of feldspar, without producing any chemical change produces a physical change; but I did not see how the calcining of bone ash could have any effect, as it has already been calcined.

Mr. Stull: I do not know anything about bone china, never saw it made; and as I said, my only experience in the use of bone ash was in this case. Professor Bleininger suggested calcining it for that reason, and I think the German literature has something about the calcining of bone ash as obtained from the dealer, and gives instances where the trouble was overcome by recalcining.

Mr. Mayer: Mr. Stull says the colemanite I got was not colemanite. I am not a mineralogist and do not pretend to know about these things, but I give you the name they call it. They sometimes call it "pandermite" and some-

times colemanite. I cannot tell the difference. I know it had 13.50% water in it. That I will vouch for. I think the man who gave me the sample is as much of a mineralogist as I am, and he sometimes calls it pandermite and sometimes colemanite. But it gave excellent results. The price made it simply out of the question to use.

Mr. Bleininger: The main object of Mr. Stull's experiments is twofold,—one practical and one more theoretical. The practical object is to obtain a cheap stoneware glaze for a certain type of stoneware. The second is to make large and extensive experiments which will tend to show the various opacifying agents. Therefore, he has undertaken the work to show how the various opacifying agents behave and produce workable glazes.

Mr. Binns: If Mr. Mayer's glazing was done on biscuit ware and Mr. Stull's on green ware, it may be possible that the combined water in the clay may have caused decrepitation.

Mr. Stull: It occurred before the kiln was red enough to see the cone. May I ask Mr. Mayer what percent of water was in the dehydrated colemanite he used?

Mr. Mayer: The composition of the glaze in which I used this colemanite was:

Feldspar	271.7
White Lead	105.0
Florida Clay	59.2
Colmanite	150.0
Whiting	44.8
Flint	139.6

Mr. Stull: In pure colmanite there is 21.90% H₂O. I will ask Mr. Mayer what was the percent of water in his variety?

Mr. Mayer: 13.50 of water.

Mr. Stull: It was probably half way between hydrated and dehydrated colmanite.

*CONTINUATION OF THE WORK.

The next step decided upon was to make up several glazes representing five different series, determine their softening temperatures in the form of cones, group these glazes according to their softening temperatures and fire them accordingly.

Since the glazes in series IV in which raw colemanite was used powdered from the trial pieces, series V was made according to the respective formulæ in series IV, the only difference being that the colemanite was dehydrated.

SERIES VI, VII AND VIII.

These series were constructed for the uprose of determining the effect of increasing Al_2O_3 and P_2O_5 by the use of aluminum phosphate in the presence of calcined bone ash and B_2O_3 .

Since the sample of colemanite at hand was exhausted in making series V, and since letters to two borax companies brought replies that they had no colemanite for sale as it was all absorbed by their plants in the refining of borax and boracic acid, it was decided to make a fritt after the dehydrated colemanite formula for use in the three following series. This was designated "Fritt B."

Fritt B.	Batch Weights:
Formula	Whiting50
2 CaO, 3 B ₂ O ₃	Boracic Acid93

A preliminary test in the Pelton electric furnace shows that this mixture swells at 700 °C to a porous sponge-like mass and melts to a clear, water-like fluid at 950 °C. An attempt to make a drop fritt of this mixture had to be abandoned on account of its great fluidity and corrosive action. It soaked through the fire clay crucible, draining

*This installment represents the work which has been done on "A Cheap Enamel for Stoneware," between the last convention and the date of publication.

SERIES V. Percentage Batch Weights. °C.

Formulae.

Glaze	Formulae							Feldspar	Calcine A	Dehydrated Colemanite	Zinc Oxide	Georgia Kaolin	Phim	Softening Temperature
	K ₂ O	CaO	ZnO	*Al ₂ O ₃	SiO ₂	P ₂ O ₅	B ₂ O ₃							
No. 10X	.3	.46	.24	.4	3.55	.109	.2	46.35	14.18	5.95	5.39	7.16	20.35	1120°
No. 11X	.3	.52	.18	.4	3.35	.084	.4	46.73	11.56	12.01	4.08	7.22	18.40	1077°
No. 12X	.3	.58	.12	.4	3.15	.060	.6	47.12	8.29	18.16	2.74	7.27	16.42	1027°
No. 13X	.3	.64	.06	.4	2.95	.035	.8	47.37	4.93	24.06	1.38	7.32	14.34	1027°
No. 14X	.3	.70	0	.4	2.75	.011	1.0	47.93	1.56	30.78	7.40	12.33	992°

SERIES VI.

Glaze	Formulae							Frit C	Brandywine	Calcine A	Zinc Oxide	Georgia Kaolin	Phim	Aluminum Phosphate	Softening Temperature
	K ₂ O	CaO	ZnO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	B ₂ O ₃								
No. 10C	.3	.46	.24	.40	3.55	.109	.2	15.40	37.08	14.50	5.39	7.16	20.47	1120°
No. 15	.3	.46	.24	.43	3.55	.139	.2	15.69	36.34	14.22	5.29	7.01	20.06	1145°
No. 16	.3	.46	.24	.46	3.55	.169	.2	14.80	35.63	13.94	5.18	6.88	19.67	1137°
No. 17	.3	.46	.24	.49	3.55	.199	.2	14.52	34.95	13.66	5.08	6.75	19.30	1155°
No. 18	.3	.46	.24	.52	3.55	.229	.2	14.24	34.30	13.41	4.99	6.62	18.93	1150°
No. 19	.3	.46	.24	.55	3.55	.259	.2	13.98	33.66	13.16	4.90	6.50	18.58	1161°

SERIES VII.

Formulae.

Glaze	K ₂ O	CaO	ZnO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	B ₂ O ₃	Frit C	Brands white Feldspar	Calcine A	Zinc Oxide	Georgia Kaolin	Flint	Aluminum Phosphate	Softening Temperature
No. 12C	.3	.58	.12	.49	3.15	.06	.6	46.99	18.86	7.37	2.74	7.28	16.76	1000°
No. 20	.3	.58	.12	.43	3.15	.09	.6	46.04	18.48	7.22	2.60	7.13	16.42	2.02	1010°
No. 21	.3	.58	.12	.43	3.15	.12	.6	45.13	18.11	7.08	2.63	6.99	16.00	3.97	1025°
No. 22	.3	.58	.12	.49	3.15	.15	.6	44.25	17.76	6.94	2.58	6.85	15.78	5.84	1033°
No. 23	.3	.58	.12	.52	3.15	.18	.6	43.41	17.42	6.81	2.53	6.72	15.48	7.63	1070°
No. 24	.3	.58	.12	.55	3.15	.21	.6	42.59	17.09	6.68	2.49	6.60	15.19	9.36	1090°

SERIES VIII.

Formulae.

Glaze	K ₂ O	CaO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	B ₂ O ₃	Frit C	Georgia Kaolin	Flint	Aluminum Phosphate	Softening Temperature
No. 14C	.3	.7	.46	2.75	.011	1.0	79.89	7.41	12.91	650°
No. 25	.3	.7	.43	2.75	.041	1.0	78.04	7.25	12.65	2.00	952°
No. 26	.3	.7	.46	2.75	.071	1.0	76.46	7.11	12.40	4.03	667°
No. 27	.3	.7	.49	2.75	.101	1.0	74.95	6.97	12.15	5.03	980°
No. 28	.3	.7	.52	2.75	.131	1.0	73.50	6.84	11.91	7.75	1049°
No. 29	.3	.7	.55	2.75	.161	1.0	72.10	6.70	11.69	9.51	1050°

Percentage Batch Weights.

Percentage Batch Weights.

SERIES IX.

Percentage Batch Weights.

Formulae.

°C.

Flint

Georgia Kaolin
Calined

Georgia Kaolin

Zinc Oxide

Calcine A

Flint F

B₂O₃

P₂O₅

SiO₂

Al₂O₃

ZnO

CaO

K₂O

Glaze

Softening
Temperature

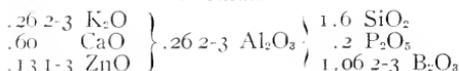
No. 30	.2	.6	.2	.40	2.5	.2	.2	63.85	7.05	2.33	7.43	6.30	12.05	901 ^c
No. 31	.2	.6	.2	.43	2.5	.2	.2	63.29	6.99	2.31	7.36	8.24	11.81	975 ^c
No. 32	.2	.6	.2	.46	2.5	.2	.2	62.74	6.93	2.29	7.30	10.05	10.69	990 ^c
No. 33	.2	.6	.2	.49	2.5	.2	.2	62.20	6.87	2.27	7.24	11.83	9.59	1001 ^c
No. 34	.2	.6	.2	.52	2.5	.2	.2	61.67	6.82	2.25	7.17	13.58	8.51	1020 ^c
No. 35	.2	.6	.2	.55	2.5	.2	.2	61.15	6.75	2.23	7.11	15.31	7.45	1011 ^c

chemical arrangement took place, except the formation of calcium borate with the free CaO from whitening.

On account of its solubility, fritt D could not be used. Instead, fritt E was made.

FRITT E.

Formula.



Combining weight=295.733

Batch Weights.

Brandywine Feldspar	42.01
Bone Ash	17.53
Zinc Oxide	3.05
Flaky Boracic Acid	37.41

This mixture makes a beautiful drop fritt; white and opaque, resembling a tin fritt in appearance, and is practically insoluble.

The softening temperatures of all the glazes in series V, VI, VII, VIII and IX were determined by the Le Chatelier pyrometer in the Pelton furnace, the softening points of each series being determined separately. These glazes were then arranged according to their bending temperatures for burning, No. 14c being softest and No. 19 the most refractory. As arranged in the table, all glazes softening between 950°C and 1027°C were fired at cones 03 and 01. Those softening between 1027°C and 1120°C were burned at cone 2, while those bending between 1033°C and 1161°C were fired at cones 4 and 6.

Length of Time of Burning.

Cone 03 was made in 9 hours.

Cone 01 was made in 11 hours.

Cone 2 was made in 18 hours.

Cone 4 was made in 17 hours.

Cone 6 was made in 19 hours.

Coke being used as fuel.

Glaze	°C	
No. 14c	950	Fired at Cones 03 & 01
No. 25	952	
No. 26	967	
No. 31	976	
No. 27	980	
No. 30	991	
No. 14X	992	
No. 32	996	
No. 12c	1000	
No. 33	1001	
No. 20	1010	
No. 35	1011	
No. 21	1025	
No. 34	1026	
No. 12X	1027	
No. 13X	1027	
No. 22	1033	
No. 28	1040	
No. 29	1050	Fired at Cones 4 & 6
No. 23	1070	
No. 11X	1077	
No. 24	1090	
No. 10c	1120	
No. 10X	1120	
No. 16	1137	
No. 17	1155	
No. 15	1145	
No. 18	1155	
No. 19	1161	

In each case after firing was finished, the fire was drawn, the fire box door and damper left open and the kiln allowed to cool quickly.

APPEARANCE OF THE TRIALS.

Series V.

No. 10X. Cone 2: White, smooth, no beading; under-fired.

Cone 4—Quite white and opaque, slightly beaded, crazed some.

Cone 6—Opalescent, blisters where thick, crazed. No beading.

No. 11X. Cone 2: Opalescent, crazed, otherwise a good glaze.

Cone 4—A good clear bright glaze. Opalescent where thick, no crazing.

Cone 6—Appearance same as at cone 4 except runs more.

No. 12X. Cone 03: Glaze somewhat immature, smooth.

Cone 01—Bright and opalescent, slightly beads, crazed.

Cone 2—Bright, less opalescent, smooth, no crazing.

No. 13X. Cone 03: Underfired, matt.

Cone 01—Bright, opalescent, beads a little, crazes.

Cone 2—Bright, opalescent, beads a little, no crazing.

No. 14X. Cone 03: Opalescent, bright, crazed some, no beading.

Cone 01—Clear, bright, a few craze marks, no beading.

Series VI.

In both the cone 4 and 6 burns there is a gradual gradation in this series from 10C to 19. All are whiter in the cone 4 burn than in the cone 6 burn. 10C is opalescent, blisters, crawls very slightly, has no crazing. Whiteness, opacity, crawling and crazing increase and blistering decreases from 10C to 19. No. 19 is as white as a tin enamel, though some beaded and crazed.

Series VII.

No. 12C. Cone 03: White, opalescent, tendency to bead, crazed.

Cone 01—Bright, opalescent. Beads some, crazes some.

No. 20. Cone 03: White, opaque, crawls some, blisters some, crazed a little.

Cone 01—Translucent, crawls less, blisters some, crazes less.

No. 21. Cone 03: White, opaque, beaded badly, crazes badly.

Cone 01—White, opaque, beads, crazes, no blistering.

No. 22. Cone 2: White, opaque, beaded, crazed and blistered.

Cone 4—Blisters, no crazing or beading; translucent.

Cone 6—Opalescent, blisters, no beading, no crazing.

No. 23. Cone 2: Blisters slightly, beads and crazes.

Cone 4—Blisters slightly, white, no crazing; beads some.

Cone 6—Opalescent, blisters, no crazing or beading.

No. 24. Cone 2: Badly blistered, slight beading, badly crazed.

Cone 4—Whitest glaze of series. Blisters, no crazing or beading.

Cone 6—Appearance same as in cone 4 burn.

Series VIII.

No. 14C. Cone 03: Opalescent, crazes, otherwise good.

Cone 01—Same as the cone 03 burn.

No. 25. Cone 03: Translucent, crazes, otherwise good.

Cone 01—Opalescent, smooth, bright and crazed.

No. 26. Cone 03: Translucent, whiter than 25, blisters, crazes.

Cone 01—Opalescent, smooth, bright and crazed. Slight beading.

No. 27. Cone 03: Translucent, whiter than 26, crazes, no beading.

Cone 01—Translucent, crazed and beaded.

No. 28. Cone 2: White, opaque, beaded some, crazed, no blistering.

Cone 4—Blistered, crazed, no beading.

Cone 6—Opalescent, blistered, no crazing or beading.

No. 29. Cone 2: White, opaque, beaded, blistered some, crazed.

Cone 4—Blistered badly, crazed, no beading.

Cone 6—Blistered, translucent, no crazing or beading.

Series IX.

Cone 03—This series has given the best results so far. All are white, opaque, though crazed. No. 30 quite badly crazing decreases toward No. 35 which has only three or four craze marks. There is very little difference in this series in whiteness, opacity and brilliancy. The glazes, however, are not at their best, a little higher temperature would improve them. None are blistered. There is a very slight tendency to crawl in No. 30 which appears less in 35.

Cone 01—All the members of the series are beautiful opaque glossy enamels. The appearance is a light ivory in color and texture. No evidence of blistering. No. 30 is crazed slightly; crazing decreases toward 35. Nos. 34 and 35 are not crazed though the body is very soft. Trials were also taken from the kiln quite hot. There is a very slight



tendency in all members to crawl a little in one or two small spots on the outside rim near the shoulder. The insides of the crocks are smooth and practically flawless, aside from those members (Nos. 30, 31, 32, and 33) which crazed. Brilliancy and opacity equal to a good tin enamel, color very nearly as good. An interesting point noticed both in the cone 03 and 01 burns is that the tendency to crawl in No. 35 containing .55 Al_2O_3 is less than in No. 30 containing .40 Al_2O_3 , all other members in both remaining constant. The crawling tendency, however, is no greater than that of many good stoneware glazes and tin enamels in commercial use in which raw borax is used to overcome that trouble. Therefore, the enamels of Series IX are very promising, since they contain no raw borax or other soluble salts.

CONCLUSIONS.

Series I.

In this series ZnO causes greater fusibility than BaO. Replacement of BaO by ZnO reduces crazing, increases brilliancy and opacity.

Series II and III.

The use of bone ash direct causes flaking of the glaze after dipping. Flaking is overcome by calcining the bone ash with flint.

Replacement of CaO from whiting by CaO from bone ash increases refractoriness materially and induces crazing.

.13 $\frac{1}{3}$ P_2O_5 introduced as bone ash with .3 ZnO and .4 Al_2O_3 caused no beading and materially increased whiteness.

Series IV and V.

The use of raw colemanite* in glazes causes "powdering" before a dull red heat is reached. Powdering is overcome by using dehydrated colemanite.

Replacement of bone ash, ZnO and SiO₂ by dehydrated colemanite, materially decreases the temperature of fusion, decreases opacity and increases brilliancy.

The opacifying effect of colemanite is very small, producing at best only a small degree of opalescence.

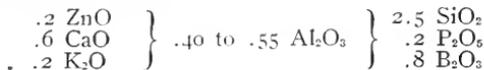
Series VI, VII and VIII.

It is difficult to determine the action of aluminum phosphate in these three series. Increasing Al₂O₃ and P₂O₅ by aluminum phosphate increases the temperature of fusion a little, increases whiteness and opacity.

The blistering which occurs has the appearance of being what "single fire" enamel brick men call "steamed glazes." Opacity, whiteness, blistering, beading and crazing generally seem to be lessened by increase in temperature of firing, though not borne out in all cases. None of the glazes containing aluminum phosphate are good enough for commercial use. It is probable that more consistent results would have been obtained if the aluminum phosphate had been calcined or fritted.

Series IX.

This series shows that good glossy enamels are possible as low as cone 01 with:



*A distinction should be made between the three most general forms of calcium borate, viz., colemanite, priceite, and pandermite, which have different physical properties such as hardness, specific gravity, action before the blow pipe, etc., as well as differences in their respective chemical analyses. However, both priceite and pandermite are classed as "varieties" under colemanite by mineralogists.

in which bone ash has been calcined or fritted and in which the excess above .3 Al_2O_3 is added as calcined clay.

Experiments with bone ash as an opacifier in glazes are not new, yet the author fails to find any work in ceramic literature pertaining to its use purely as a glaze ingredient aside from its uses in glass and bone china. The question has been raised that bone ash is dangerous to use in glazes on account of its liability to cause beading.

It is a well known fact that an excess of the opacifiers, alumina, tin oxide, zinc oxide and bone ash causes beading. The observations of the writer lead him to believe that bone ash has no greater tendency to cause beading than ZnO , SnO_2 or Al_2O_3 . If we attempt to add bone ash to a glaze of the Bristol type, which is already loaded up with an opacifier, bringing it close to the danger point of beading, then of course beading will occur.

This concludes the work to date. The next step will be to test the members of series IX for range of temperature to determine whether they are suitable for commercial use. If so, then the best one of the series will be selected for the starting point of series X, in which the B_2O_3 is to be gradually replaced by SiO_2 in an endeavor to produce suitable "tinless" enamels for higher temperatures.

THE VISCOSITY OF CLAY SLIPS.

BY

A. V. BLEININGER, Champaign, Illinois.

The plasticity of clays is still a quality whose physical definition has not yet been established, though many attempts have been made to do so. All we can do at present is to continue the search for some criterion which bears some relation to this elusive property. Up to the present such properties of the clay as the tensile strength, both green and dry, its deformation, crushing strength in the green state, and also recently the viscosity imparted to suspensions of clay particles have been studied. This last property is promising inasmuch as it produces a phenomenon which is clearly not shared by non-plastic materials. *In fact, it might be said that clays are rocks which when pulverized and suspended in water produce a decided increase in viscosity.*¹

This viscosity is capable of being expressed numerically with a degree of accuracy which is superior to that of any of the methods mentioned above.

Simonis¹ has measured the viscosity by determining the volume of a clay-slip flowing through an aperture of 2 mm., under constant pressure, in a given time. A marriott flask was used to maintain constant pressure in the burette containing the slip. The burette was first standardized with water and the relation existing between the volume flowing from the tube and the pressure obtained. This relation is evidently $v=kp$, where v =volume of liquid discharged in a given time, p =pressure of the liquid or its height in the vessel, and k =coefficient of fluidity or the viscosity. Solving for k we obtain $k=\frac{v}{p}$. If now the pressures are plotted along the abscissa and the volumes es-

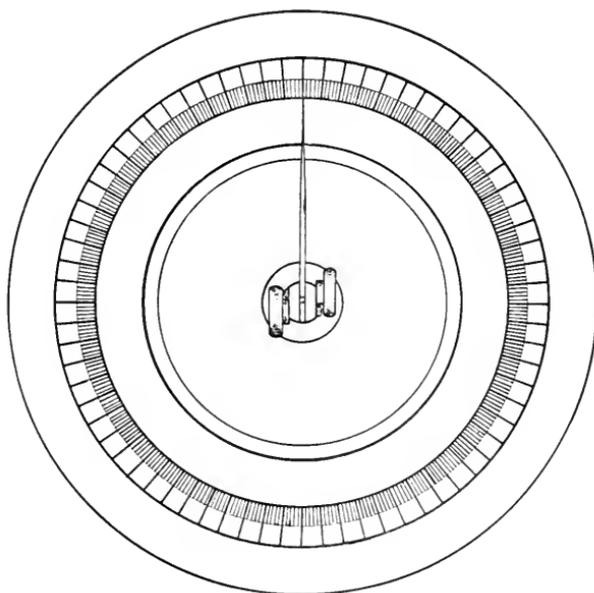
¹Sprechsaal, 1905, 597.

caping, along the ordinate we obtain for ideal liquids straight lines where $\frac{dv}{dp}$ is equal to the coefficient of fluidity represented by the tangent of the angle made by the line to the x axis.

In the case of clay slips Simonis found the relation to be not so simple and he obtained as the functions of volume and pressure, curves of higher degree. The same investigator, having found considerable difficulty with this method in working thick slips, devised a second method for determining the cohesion of clay slips by measuring the weight necessary to pull away a 5 cm. glass plate from the surface of the liquid. For this purpose he arranged a balance, having the glass disc attached at one end of the beam and a scale pan at the other. He caused very fine shot to pour onto the pan, arranging at the same time an automatic shut-off device. Before working with clay slips he determined the weight necessary to pull away the disc from water. Applying the test to clay suspensions and noting the weight necessary to release the disc he subtracted from this load the weight required to pull away the glass from water, which is a constant factor. In this way the cohesion value of the clay for the concentration employed was determined. This method is applicable to thick slips which fail to give results by the flow method.

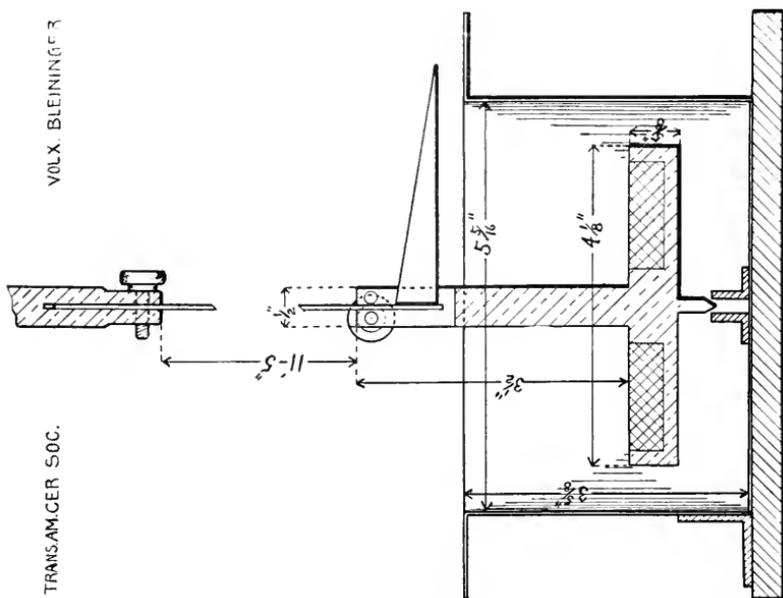
There are faults inherent with both of these methods, the flow method being subject to irregularities caused by the roughening of the walls of the vessel, the irregular stopping up of the aperture and other difficulties while the disc method is faulty as soon as even the slightest settling takes place.

In looking about for a method which perhaps might overcome some of these difficulties the Coulomb method, as employed in the determination of the viscosity of oils, was considered and adopted. Although the apparatus as designed is not suitable for thick slips, some interesting results were obtained, and the ease and accuracy with which the viscosity of thin slips could be determined makes



Viscosity Apparatus.

Fig. 1.



it quite suitable for certain investigations. It has afforded the writer a delicate means of distinguishing clays of different plasticity, or of following the effects of electrolytes and organic substances upon the physical character of the clay suspensions.

The apparatus itself is exceedingly simple, the disc being suspended from a steel wire 11 ft. 6 in. long, and allowed to rotate within a vessel filled with the clay slip, figure 1. At the center of the brass disc filled with lead, a rod projects, provided with a clamp for gripping the wire firmly. An aluminum pointer is fastened to the rod which swings over a circular scale around the rim of the receptacle, graduated in degrees. This scale is made out of paper and varnished. The weight of the disc is 1333 grams and the thickness of the wire 0.85 mm.

In making the test the slip is first thoroughly stirred up and poured into the vessel. The disc is then turned about 180° by means of the pointer and released. The number of degrees is read off at the turning point of the vibration so that the amplitude of each swing in the same direction is observed. This is continued until several readings have been taken. The disc is then stopped and the slip stirred up for another set of check readings. The ratio of the amplitude of two successive swings is obtained by dividing the first reading into the second, the second into the third, and so on. This ratio is a constant for the same slip at the same temperature. It is important to make note of the temperature, or better to keep it constant, since the viscosity of the water itself changes with change in temperature.

The time of periodic vibration is obtained by taking the total of, say, ten complete vibrations and dividing by 20. It may also be calculated from the length of the wire. For the apparatus in question the periodic time of vibration was found to be 3.6 seconds, thus enabling two observers to make the readings quite readily.

Knowing the period of oscillation and the ratio of the amplitudes we can calculate the viscosity of water and that

of the slips to be compared with it. In this work the viscosity of water is always used as the standard, and hence the viscosities obtained are in terms of the viscosity of water.

Several methods of calculation might be employed, based upon the laws of the dampening of vibrations. Assuming for instance that the ratio of the amplitudes is 0.8 and the time is 1.5 seconds for each vibration, we obtain as an expression for the viscosity of the liquid:

$$\begin{aligned} 0.8 &= e^{-1.5k} \quad \text{or} \\ -\log 0.8 &= 1.5k \log e. \\ \text{Then } K &= \frac{-\log 0.8}{1.5 \log e} = 0.15 \end{aligned}$$

If in another slip the ratio of the amplitudes is found to be equal to 0.7 and solving again for K we obtain 0.24. The viscosities of the two liquids, therefore, are to each other in the ratio of 15:24. A somewhat simpler relation might be used for determining the relative though, of course, not the absolute viscosities, in which

v_1 = coefficient of viscosity of one liquid;

r_1 = ratio of the amplitudes of any two successive oscillations in the same direction in the same liquid;

T_1 = period of oscillation;

d_1 = dampening constant.

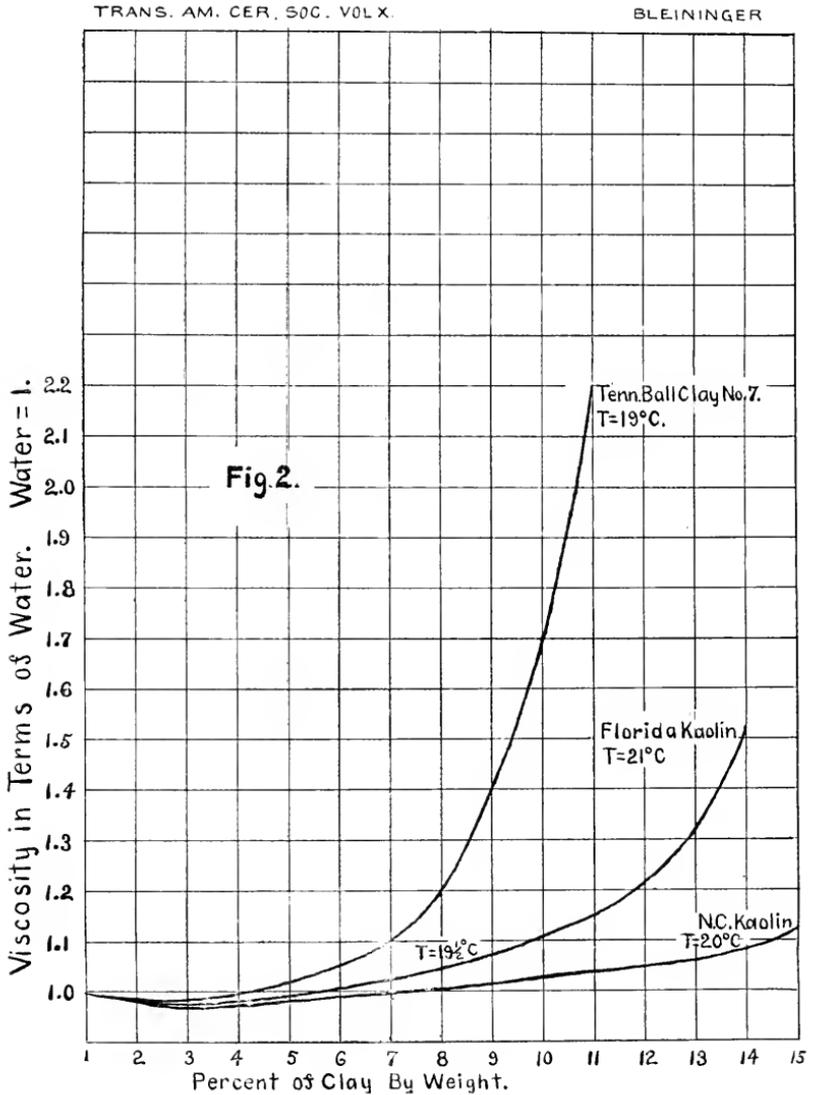
Similarly let v_2 , r_2 , T_2 and d_2 be the corresponding values for the second liquid. We have then the relation

$$\frac{d_1}{d_2} = \frac{T_2 \log r_1}{T_1 \log r_2} = \frac{v_1}{v_2} = K$$

This gives us K , a coefficient of specific viscosity. The standard employed is distilled water whose ratio is determined at the same temperature as the ratio of the slips to be tested. The constant for water, of course, differs with different apparatus. In the experiments carried on in the apparatus described it was 0.89.

It was decided to make viscosity determinations with three kaolins which differ widely in their physical behavior.

The method obviously is not intended for clays which are coarse-grained, and if they are to be tested in this way the coarser portion must be screened off. The three clays se-



lected were the North Carolina kaolin, Florida kaolin, and the Tennessee ball clay, No. 7.

The clays were weighed out, the moisture factor being allowed for, and mixed with a weighed amount of distilled water. The mixing was done in a small gallon porcelain ball mill, it being run for forty minutes in each case. The heaviest slip employed consisted of 85%, by weight, of water and 15% of clay, the apparatus not being able to vibrate in thicker slips. The 15% slip was at first blended with a 1% suspension for the intermediate compositions, but later the heavier slip was diluted with the distilled water alone. The blending was accomplished by simply stirring in a pitcher. In Fig. 2 will be found the viscosity curves of the three clays for different concentrations. They were the average of 30 determinations in each case, and the readings showed a variation of about $\pm 2\%$. It will be observed that at first the viscosity was decreased, a fact which is quite interesting. The Florida kaolin occupies a position midway between the North Carolina kaolin and the ball clay, but nearer to the kaolin than to the latter.

Might it not be possible that by this or other viscosity measurements we shall be enabled to correlate or classify our plastic clays? The practical potters constantly call our attention to the fact that we disregard the physical properties of kaolins, and that in our ceramic schools we do not properly discriminate in their use in our experiments. It is our duty therefore to employ such means as these to help them, as well as to push forward our knowledge of the elusive properties of clays.

As has been said, this apparatus is very delicate, and such things as the use of hydrant water in place of the distilled will cause marked changes in the behavior of the clays. This naturally leads to the action of salts like sodium carbonate, sodium silicate, etc., which opens up a large field of experimentation.

The work is to be continued with a modified apparatus which it is intended to operate in heavier slips.

NOTE ON SOME FUSION CURVES.

BY

A. V. BLEININGER, Champaign, Ill.

In the study of the chemistry and physics of ceramics the mechanism of the fusion processes is of vital importance. Yet our knowledge of these fundamental processes is exceedingly limited, which is due to the fact that we have continued to investigate complex conditions with so many variables that our equations have been far too few to solve them. It is high time that we modestly begin the study of the simpler relations and, after mastering them, proceed to the investigation of the more complicated systems. There is no reason why in this way we should not obtain in time a fairly accurate knowledge of the chemistry of clays.

We must expect to meet discouragement, however, for only by the most painstaking care in keeping the conditions constant will we be enabled to obtain consistent results. We must at all times remember that the reactions taking place in our clays are never completed but will always remain far from the end-point, and that they are dependent upon the conditions of the partial equilibrium reached. Evidently important factors are,—slow or rapid heating, crystalline or amorphous substances, etc.

Though we may not reach the most exact knowledge of the subject, by keeping as many conditions constant as possible we shall learn much from the correlation of melting-points and here, fortunately, the differentiation of melting point series is of greater help to us than the knowledge of isolated melting points. In this way the maximum and minimum points are detected, and we are enabled to fix the temperatures at which chemical reactions take place, the fusion temperatures of the eutectic mixtures

and the compositions corresponding to these critical points. Usually, maximum points correspond to compounds and the minimum points to eutectic mixtures.

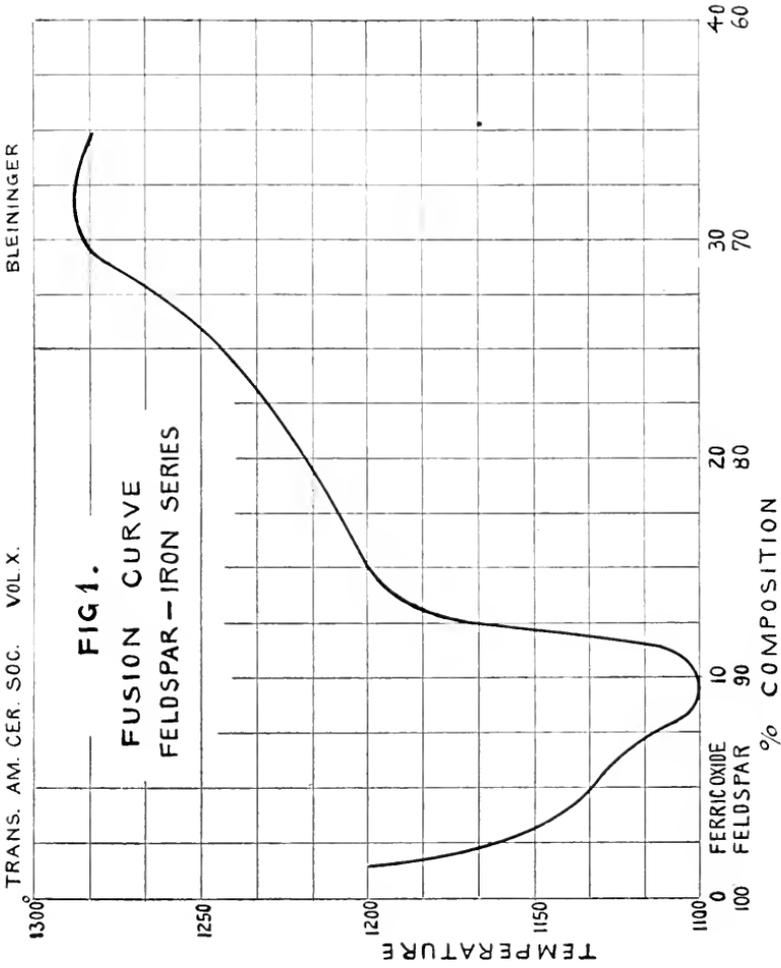
In the preliminary study of the fusion curves begun at the University of Illinois, the first work comprised mixtures of feldspar, ferric oxide and whiting, since these mixtures are of interest in the study of some of the Illinois clays. Commercial materials were used and it was found that, though the whiting appeared to be of good color and was bought as high-grade material it proved quite impure.

The composition of the feldspar, whiting and ferric oxide was found to be as follows:

	Silica	Alumina	Ferric Oxide	Lime	Magnesia	Potash	Soda
Feldspar	68.22	17.83	trace	0.30	0.11	12.13	1.90
Whiting	1.62	3.50	trace	52.65	trace
Iron Oxide	99.00

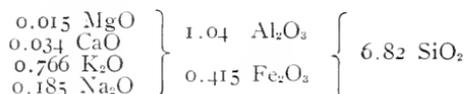
These three constituents were ground together, dry, in a small ball mill, in various proportions, from 100% feldspar to 40% ferric oxide, 100% spar to 40% whiting and 100% spar to 40% ferric oxide plus whiting, in intervals from 1-5%. The interval was made small, close to 100% of feldspar, so as to bring out the effect of smaller amounts of either material upon feldspar. From 1% the interval was increased to 2, 3 and 5% as the feldspar diminished in amount.

The mixtures were then made up with dextrine water into a dough and molded into cones in a metal mold. After drying, the cones were placed on clay slabs, in duplicate rows and melted down in a coke-fired test kiln, having been protected from the flame by means of a high tile sagger which was partially open on the back and front. A thermocouple was placed as close to the cones as possible. Correction was made for the cold junction temperature. Through a thin piece of glass inserted in the spy hole the cones were watched with ease, and their going down ob-



served by means of an opera glass. Unfortunately some of the cones were cracked in drying due to an excess of dextrine and broke off in the kiln. These were not considered in the result.

In Fig. 1 we have shown the fusion curve of the feldspar-ferric oxide series, and a eutectic is clearly indicated with 91% feldspar and 9% of iron oxide. This mixture corresponds to the formula:

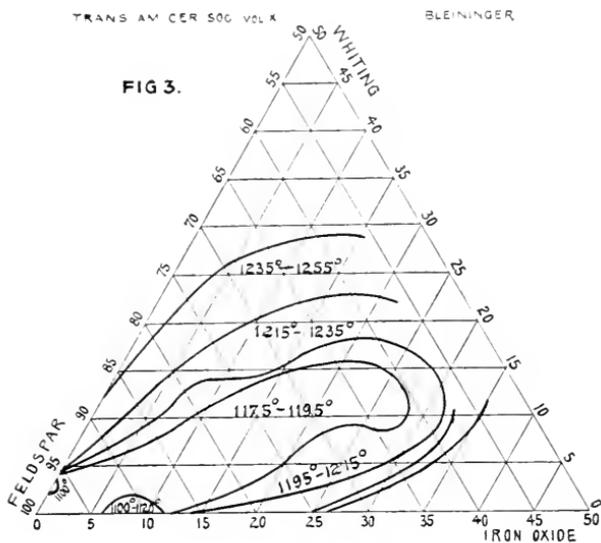
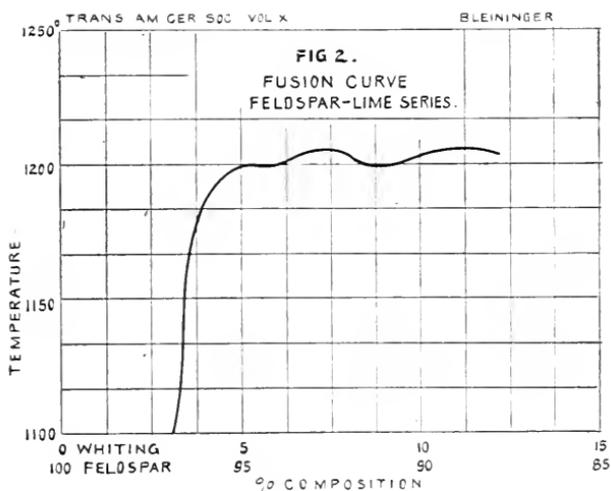


The feldspar formula is represented by the same formula but without the iron. This mixture therefore fuses at a lower temperature than the feldspar itself or any other mixture of the series.

In Fig. 2 the feldspar-line series is not complete owing to the breaking of some of the cones, and hence the eutectic point is not definitely established, but it is quite evident that it is close to 97% feldspar, 3% whiting. This would correspond to a slight enriching of the spar in line corresponding to about 0.03 equivalent. Since this point was not determined with certainty no interest is attached to its formula.

In Fig. 3 there are plotted the compositions of the mixtures in terms of the percentages of feldspar, iron-oxide and whiting, and it is evident that the isothermal compositions are connected by some definite law. Each curve represents a temperature interval of 20°, corresponding to the range of one cone. Thus every mixture in the inner area melted between 1175° and 1195°C. The increase in area indicates the enriching of the fusion by the solution of other materials on raising the temperature, and we learn thus in what direction this takes place.

Similar curves carried on very carefully with pure lead silicates, fusing the cones in an electric furnace, showed some disturbing factor owing to volatilization of the lead, and hence are not produced. This trouble was



encountered even with fritted glasses, and the work is to be repeated under special precautions.

DISCUSSION.

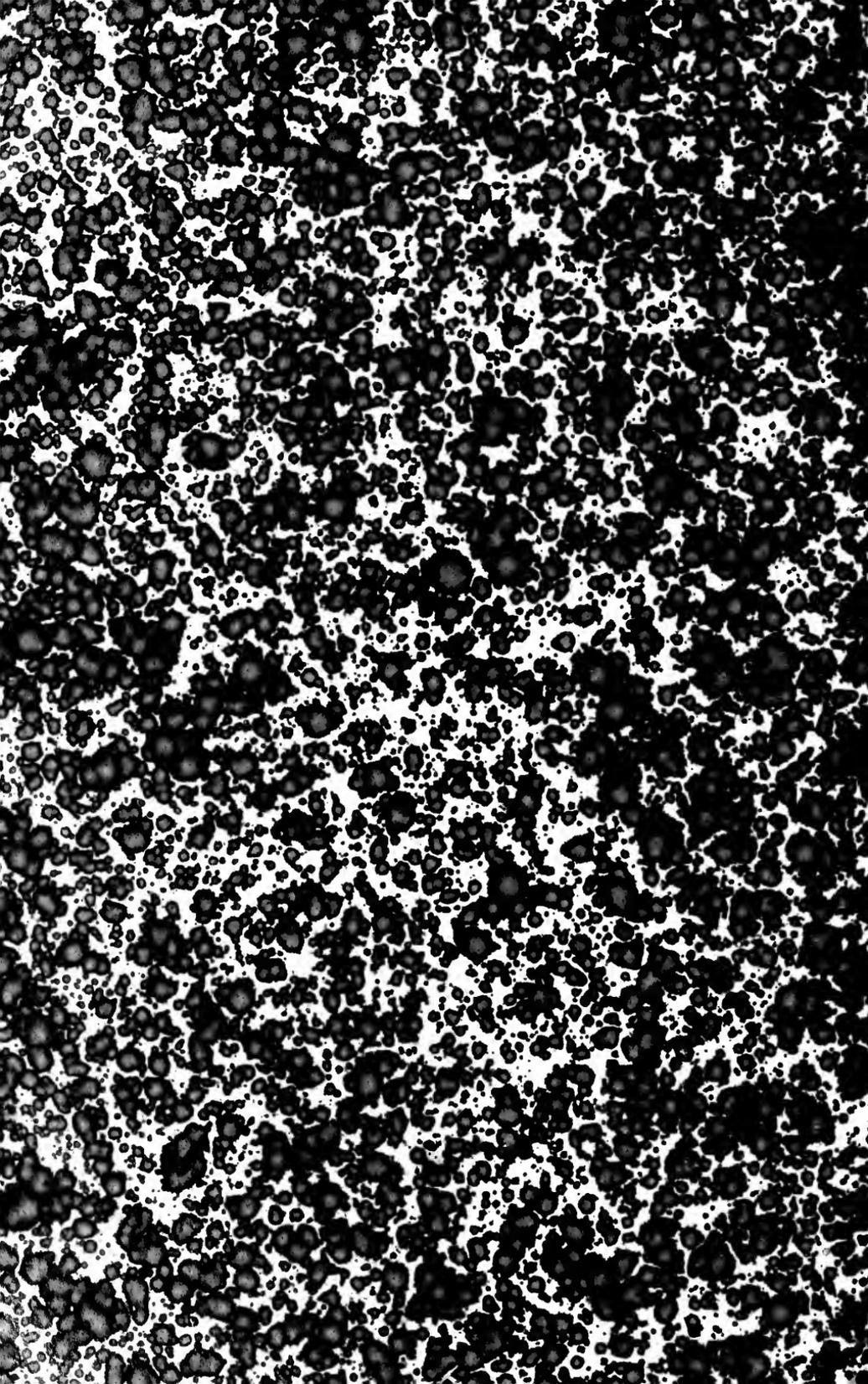
The Chair: I think Professor Bleininger has given us a splendid paper, and I am very glad to know that he is getting near to the practical man. I will call on Mr. Stover, as a practical man, to discuss the paper.

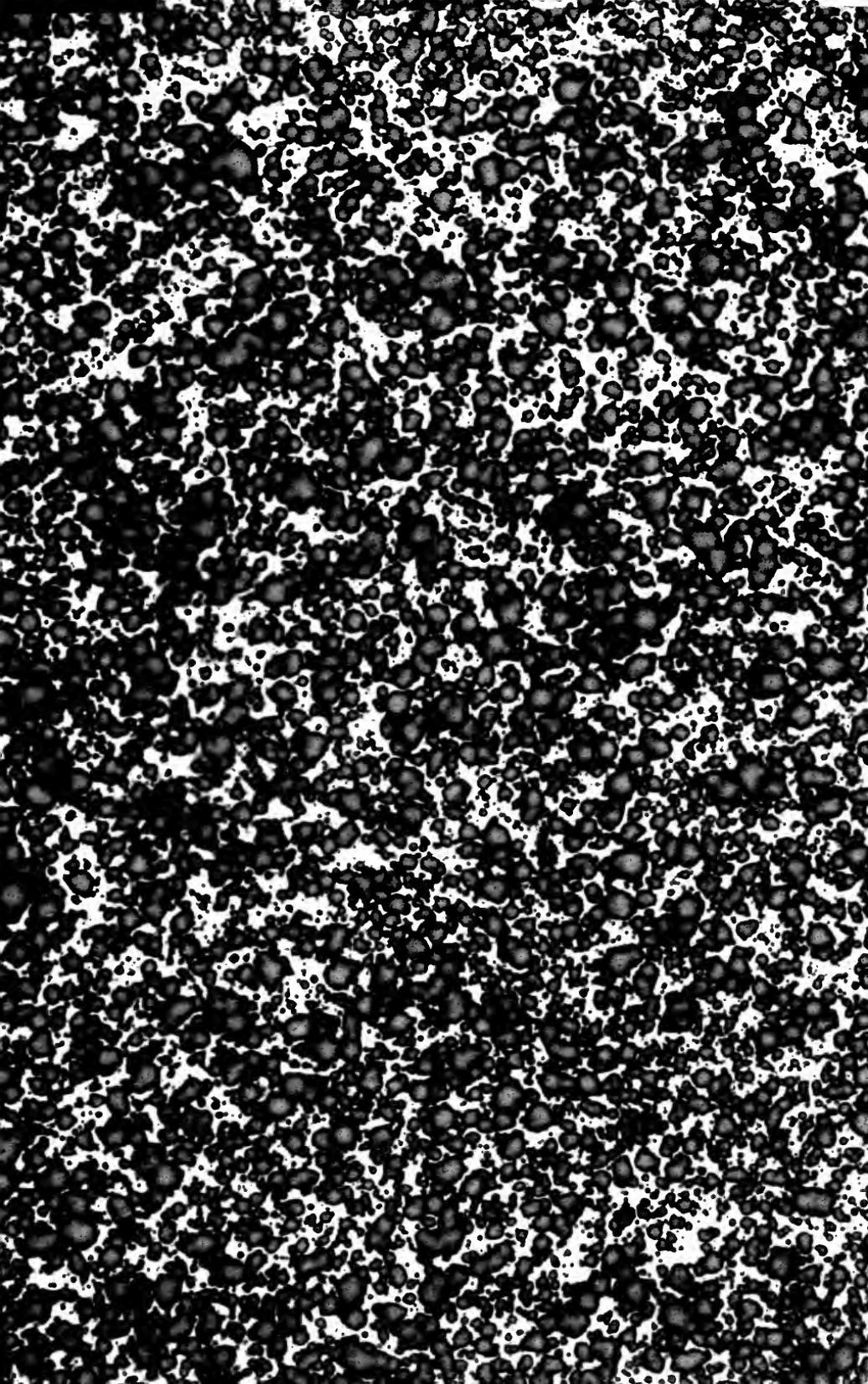
Mr. Storer: I would like to ask Professor Bleininger why he expressed the data in percentage weights?

Mr. Bleininger: Because it was the simplest way.

Mr. Storer: I don't want to say a word against the use of technical formulæ, for I sat up many nights in order to master it so I could figure them out by myself; but it is a fact that I have been arguing along the line of this sort of expression without much result until at this meeting. I have contended it would make it more simple, and if we could have the kind of data Mr. Bleininger has been trying to give us lying around in the mixing rooms of the practical potters in Trenton and East Liverpool, we would have something here in the pages of the proceedings which would be of practical value. But for the most part, we are soaring around over their heads and they cannot understand us. I am glad Professor Bleininger has put his data in plain percentages, for I believe it will be productive of practical results.

The Chair: I want to say, as a practical man, that I am impressed with the fact that we are getting closer and closer to the practical man in our work. There are many things about the technical part of the American Ceramic Society's work which the practical man cannot understand; but he realizes the meat is there even though he cannot always digest it. I think this is a simple method and that the practical man will by and by conclude that after all there is something in pottery worth knowing that he does not understand.





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