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DIFFERENCE IN PYROMETRIC CONE
EQUIVALENT BETWEEN RAW AND CALCINED CLAY

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SUBMITTED AS A PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR DEGREE OF BACHELOR OF SCIENCE IN CER-
AMIC ENGINEERING AT
MISSOURI SCHOOL OF MINES AND METALLURGY

MAY 23, 1928.

The writer wishes to acknowledge the many helpful suggestions and aid given by Dr. M.E. Holmes in the preparation of the work and in writing of this paper. It is due to his efforts that the paper is possible.

**INFORMATION CONCERNING THE DIFFERENCE IN
PYROMETRIC CONE EQUIVALENT BETWEEN RAW
AND CALCINED CLAY.**

In a recent survey of the refractories field with particular respect to the Pyrometric Cone Equivalent of different clays it was suggested that a difference in the P. C. E. value of clays might be found due to different stages of heat treatment that these clays had previously undergone.

At present there is considerable apparent lack of information regarding this problem. It is evidenced largely in the samples of clay sent into commercial testing and research laboratories by manufacturers who are attempting to develop definite properties from a given clay or clay body. There is a large difference in the type of samples sent into these laboratories. Whether it is lack of more definite information or disregard for the errors that may result from this practice is not known however standardization of test samples is not practiced, but should be if the errors introduced are sufficient to make any difference in the P. C. E. value of the samples submitted. Some manufacturers wishing a cone fusion test run on their product or on a natural clay will send in a sample of the clay or the product that has already been burned, while others will submit samples of the raw clay or body for the test. It is possible that there may be a great difference in the P. C. E. value of the same clay or body under the two conditions given.

It was with the hope of finding more information regarding this subject that the following efforts were prompted.

To study this problem it was decided to select three clays which would give a more or less representation of the field of clays.

With this in view a high refractory flint clay, a ball clay and a low grade plastic fire clay were selected for study.

The object was to calcine a portion of the samples of each clay and to use the other portion of the sample raw and to determine the P. C. E. of each clay under each condition and determine the difference, if any, in the amount of heat treatment they would stand as evidenced in the standard cone fusion test.

The three different clays selected were, one a low fusing fire clay of the Venitian Fire Clay Company of Kansas City, Missouri, and one a flint fire clay refractory from a diaspore pit north of Rolla and the third a plastic ball clay being a # 9 Tennessee Ball clay of Mandel Porter Company, Paris, Tennessee.

The selected samples were put through a primary crusher of the Blake type, the jaws and the receiving pan of the crusher being thoroughly cleaned to prevent contamination from impurities in the crusher. After being crushed to about one-eighth inch maximum size the samples were put through a set of Stustevant laboratory rolls having eight inch crushing rolls traveling at 150 R.P.M. The ball clay and flint clay were found a little damp so that several passes were made on each of these clays. From the roll crusher the samples were transferred to a Braun Pulvizer where the grinding discs were placed as far apart as possible to avoid extreme fine grinding of the material. After the pulverization the whole of each sample was quartered and half of the original sample was reserved for future reference.

After the samples had been quartered and half the original sample reserved the remaining half was quartered until about 800 grams of each sample was left. The 800 grams was to be the portion of each clay that was to be calcined.

The samples to be calcined were then prepared as follows:

Each clay was tempered with water until a plastic state of the mass was obtained. Bars were then rolled in a hand press mold twelve inches long and one inch square. These bars were then cut in two making four bars 6" x 1" x 1". A dirt block was used in forcing the clay in the molds and it is believed that little specks of foreign clay (possibly feldspar) may have been become imbedded in the top surface of the samples. This was evidenced by seeing some white specks on the surface of the dark burning fire clay after calcination. The samples to be calcined were dried rather hurriedly in order to start calcination as rapidly as possible. The bars were placed in the bottom tray of a thermostat dryer whose temperature was set at 100° F and were allowed to stand half an hour. The bars were then placed near the top of the dryer and allowed to dry at 100° F for five hours. The temperature of the dryer was then increased to 275° F and the bars permitted to dry at this temperature for nine and one-half hours. No ill effect of the rapid drying was evidenced and the flint clay while it developed small hair cracks did not crumble but kept the shape of the bar.

The firing of the bars was done in a standard Bureau of Mines experiment kiln of the down draft type. The bars were placed in the kiln with a rather open bottom to facilitate the creation of a draft in the firing chamber. Considerable trouble was experienced in the beginning stages of the firing due to lack of draft, the atmosphere being highly reducing for some time.

The preliminary heating was done with gas until the kiln had obtained sufficient temperature to ignite the oil which was forced from a storage tank by air under fifty-five pounds pressure gauge. The oil was

atomized by air from a centrifugal fan in a Denver Fire Clay Company burner.

The gas was started at seven thirty A. M. and at eleven forty-five the oil was turned on and both oil and gas were then used until the temperature of the kiln was sufficient to support combustion of the oil alone. The oil was fed into the kiln as rapidly as it was possible to fire without producing reducing conditions.

At three-fifteen cone ten was just down and cone eleven was practically down there being very little difference in the time interval between the two standard cones.

At this point the bars of the venitian fire clay were withdrawn because previous experience with this clay had shown it to be of very low fusing properties.

The four bars of venitian fire clay were then allowed to cool in air until reaching room temperature. The bars were examined and two of them which were of regular size had split along the longer axis showing an unoxidized core and a considerable accumulation of fused iron on the face that had split open. Two of the bars were of much smaller size being about three-eighth inch thick. These two bars were found to be oxidized clear through and were apparently well calcined samples. The two larger bars were discarded as being of little value and the two small bars used as the samples for the problem.

The firing of the other two clays was continued as rapidly as possible until cone sixteen was flat on the plaque. Cone sixteen was down at five-fifteen P. M. and the kiln was then allowed to soak until five forty-five when the oil was shut off and the kiln allowed to cool over night. Upon examination of the flint and ball clay bars it was found that there were apparently well calcined and made good samples.

The sample of raw clay not used for calcination was quartered

until the remaining sample was of a good size to determine a screen analysis. Each of the raw clays were quartered in this manner and the screen analysis was determined. After determination of the screen analysis the sample was considered representative of the whole and was then discarded.

The screen analysis of the clays follows:

Venetian Fire Clay

200 Gram Sample.

Thru 60 on 80	20.16
" 80 " 100	23.38
" 100 " 150	35.80
" 150 " 200	22.06
" 200	97.53
	<hr/> 198.73

Tennessee Ball Clay

Thru 60 on 80	33.23 grms.
" 80 " 100	38.27
" 100 " 150	27.26
" 150 " 200	38.18
" 200 "	40.11
	<hr/> 197.05

Flint Fire Clay

Thru 60 on 80	26.26
" 80 " 100	68.62
" 100 " 150	38.56
" 150 " 200	24.15
" 200	40.17
	<hr/> 197.76

The calcined samples of each of the clays were then ground in

the same manner was used for the raw clay. The crusher and rolls etc. were thoroughly cleaned between each sample to avoid contamination. The calcined samples of each of the samples were ground until approximately the same amount of fineness were in the calcined as were in the raw clay.

The analysis of the calcined clay was as follows:

Venitian Fire Clay

36% thru 200 mesh.

Tennessee Ball Clay 33% thru 200 mesh.

33% thru 200 mesh

Flint Fire Clay

25% thru 200 mesh.

This closely approximated the screen analysis of the raw material and was considered sufficient to eliminate any errors that might arise due to difference of grain size.

Cones were then made of the different samples of clay and as it was necessary to use a dextrine solution to bond the calcined material it was also used in making cones of the raw material to eliminate possible errors due to any fluxing action that might have been caused by the dextrine solution binder.

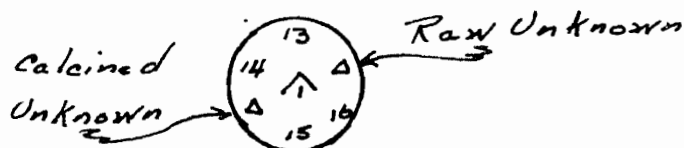
After the cones were molded and dried thoroughly plaques were made in which a calcined cone and a raw cone of the same sample were placed with several standard cones in order to observe the difference, if any in the heat treatment which the cones would stand.

The firing was done in a granular carbon resistance electric furnace as shown in the accompanying blue print. This furnace is run on single phase A. C. 110 volt current supplied from a synchronous converter, which converts from three phase which is the current supplied in the lines to one phase which is supplied to the furnace. The procedure of the firings and the currents showing the rate of heating follows:

FIRE RUN ON INDIAN FIRE CLAY

The furnace was started at 10:07 and the voltage increased as rapidly as possible to arid heat.

The plaque was set with six cones, four being standard cones and one being a raw unknown and one being a calcined unknown. The standard cones in the plaque were 13, 14, 15 and 16 and were placed in the following manner:



At 11:00 the walls of the alundun tube had reached a red heat. At 11:15 the high series voltage was thrown in and the voltages set at 37volts-.75 amperes. At this time the temperature was 1652 degrees F. At 11:30 the temperature was up to 1760 degrees F. and had an ammeter reading of .380. At 11:35 the temperature was 1745 degrees F. with an ammeter reading of .378. The voltage was then increased to 40 volts and .8 amp. At 11:45 the temperature of the cone plaque was 1988 degrees F. with an ammeter reading of 416. At 11: 52 the temperature was 2070 degrees F. This heating was a little fast as the voltage was retarded a little to 38 volts. At 12:00 the temper ture was 1966 degrees F. The voltage was slightly increased to 39 volts. At 12:05 the temperature was still a little down being 1951 degrees F. so the voltage was again increased to 41 volts and .8 amp.

At 12:10 the temperature was 1966^oF.

At 12: 11 the voltage was increased to 42 volts.

At 12:15 the temperature was 2020^oF.

At 12:20 " " " 2030^o "

The voltage was increased to 44 volts and .9 amperes.

At 12:25 the temperature was 2050^oF.

The voltage was increased to 47 volts and 1 ampere.

At 12:30 the temperature was 2187^oF.

At 12:38 the temperature was 2306^oF.

The voltage was cut down to 44 volts.

At 12:47 the temperature was 2306^o at 48 volts.

At 12:50 the voltage was increased to 48 volts.

At 12:55 the temperature was 2384^o F.

At 1:00 the temperature was 2305^oF.

The voltage was increased to 50 volts.

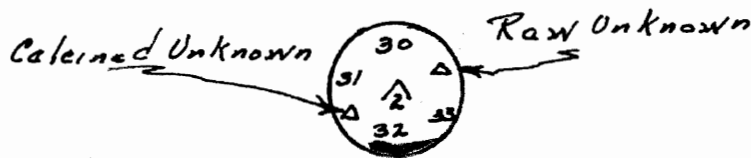
At 1:28 the furnance was shut down.

FIRST RUN ON FUSION POINT OF
VENETIAN FIRE CLAY

Temperature	Time
1988	11:45
2070 ^o F	11:52
1966 ^o F	12:00
1931 ^o F	12:05
1966 ^o F	12:10
2020 ^o F	12:15
2030 ^o F	12:20
2050 ^o F	12:25
2187 ^o F	12:30
2306 ^o F	12:38
2290 ^o F	12:40
2306 ^o F	12:48
2346 ^o F	12:50
2384 ^o F	12:55
2405 ^o F	1:00
2468 ^o F	1:05
2520 ^o F	1:10
2555 ^o F	1:15
2609 ^o F	1:20
2691 ^o F	1:27

FIRST RUN ON TENN. BALL CLAY

The plaque consisted of four standard cones and two unknowns. one unknown a raw sample and one a calcined sample. The standard cones were 30, 31, 32, and 33. The placing of the cones in the plaque was as follows:



The current was turned on at 11:30, but due to a loose connection in one of the terminals of the carbon elements it was 2:00 before the furnace became red.

At 12:10 the temperature of the wall of the furnace was 1669°F

At 2:20 " " " " " " " " 1745°F

The voltage was then adjusted to 45 volts and .85 amps.

At 2:30 the temperature was 1816°F.

The voltage was then increased to 48 volts and 1 amp.

At 2:40 the temperature was 1988°F

At 2:50 the voltage was increased to 49 volts.

At 3:00 the voltage was increased to 53 volts.

At 3:12 the voltage was increased to 57 volts and 1.15 amps.

At 3:20 it was found that the temperature was lagging and the voltage was increased to 60 volts and 1.25 amperes.

At 3:25 a loose connection was found in one of the terminals which probably caused the trouble in heating.

The temperature rose rapidly after firing terminal so that the voltage was cut down at 3:30 to 57 volts.

At 3:57 the voltage was raised to 63 volts 1.3 amperes.

At 4:25 the calcined unknown is tipping.

FIRST RUN ON TENN. BALL CLAY.

Temperature	Time
1669 ^o F.	2:10
1745 ^o F	2:20
1816 ^o F	2:30
1988 ^o F	2:40
2030 ^o F	2:50
2080 ^o F	3:00
2140 ^o F	3:10
2160 ^o F	3:20
2398 ^o F	3:28
2435 ^o F	3:32
2470 ^o F	3:40
2570 ^o F	3:52
2731 ^o F	4:02
2809 ^o F	4:10
2861 ^o F	4:15
2991 ^o F	4:20
3106 ^o F	4:25

FIRST RUN ON FLINT CLAY.

Furnace started 1:00 P. M. with voltage of 20 volts.

At 1:15 voltage was increased to 23 volts.

At 1:24 " " " " 25 "

At 1:35 " " " " 28 "

At 1:54 " " " " 30.5 "

At 2:12 " " " " 34 "

At 2:15 the sides of the alundun tube were red hot and had a temperature of 1656°F -905°C.

At 2:22 the voltage was increased to 35 volts

At 2:37 the voltage was increased to 38 volts

At 2:40 the temperature was 1734°F-945°C

At 2:50 the temperature was 2035°F-1112°C

At 2:52 the voltage was reduced to 35 volts

At 2:57 the temperature was 2045°F-1120°C

The voltage was again increased to 38 volts.

At 3:05 the temperature was 2055°F and the voltage was increased to 42 volts.

At 3:10 the temperature was 2105°F-1150°C.

At 3:20 " " " 2161°F-1185°C.

At 3:25 the voltage was increased to 45 volts.

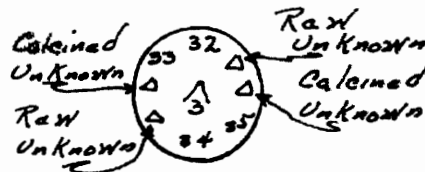
At 3:30 the temperature was 2263°F-1240°C

At 3:40 the temperature was 2327°F-1275°C

At 3:40 the voltage was increased to 48 volts.

At 3:50 the temperature was 2576°F-1450°C.

Voltage cut down to 45 volts.



At 3:55 the temperature was 2642° F-1450° C.
Voltage cut to 43 volts.

At 4:00 the temperature was 2603° F-1430° C.

At 4:10 the temperature was 2642° F-1450° C.

At 4:20 the temperature was 2797° F-1535° C.

At 4:30 the temperature was 2881° F-1585° C.

At 4:40 " " " 2941° F-1618° C.

At 4:49 " " " 3009° F-1655° C.

At 4:55 " " " 3032° F-1670° C.

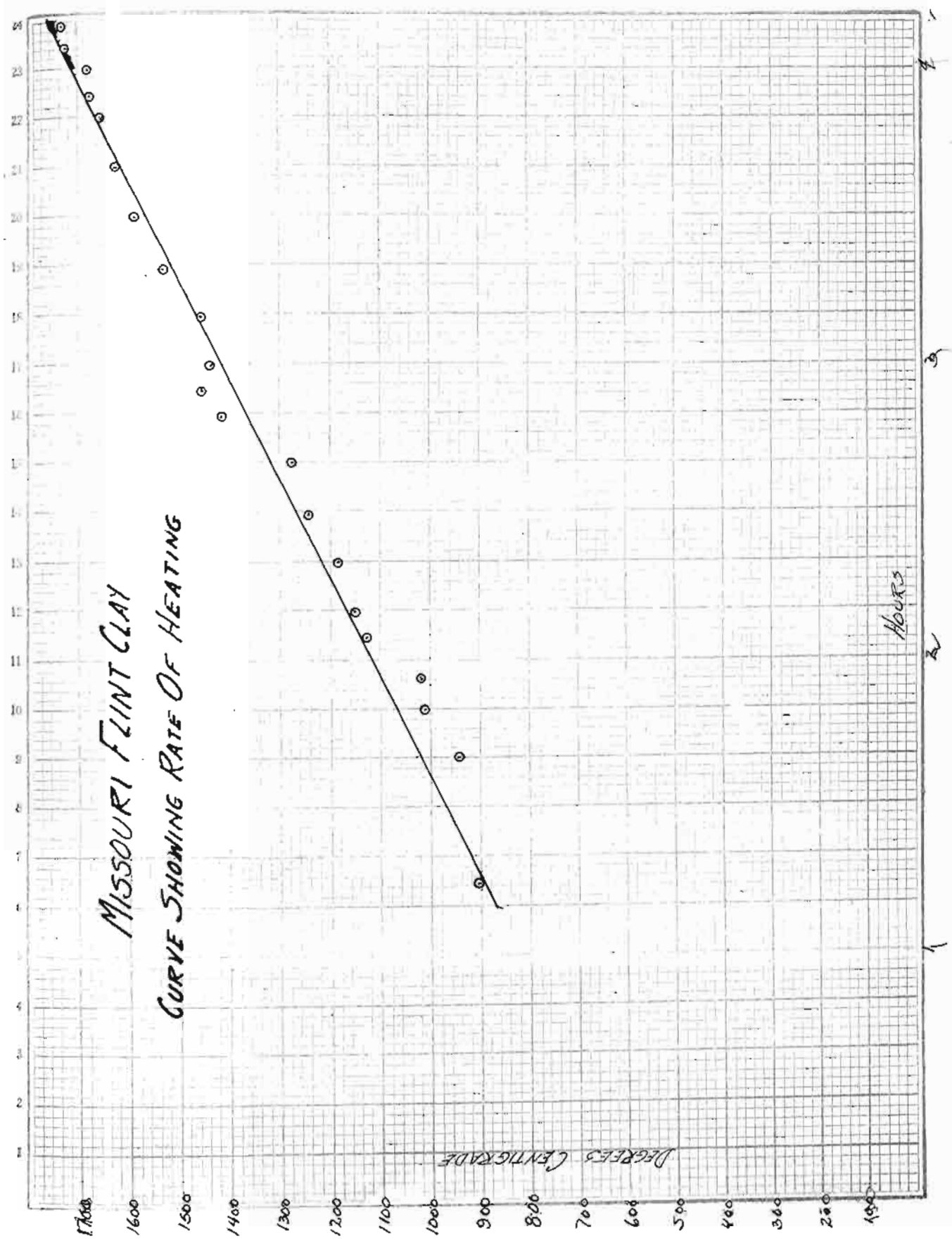
At 5:00 " " " 3032° F-1670° C.

At 5:05 " " " 3124° F-1720° C.

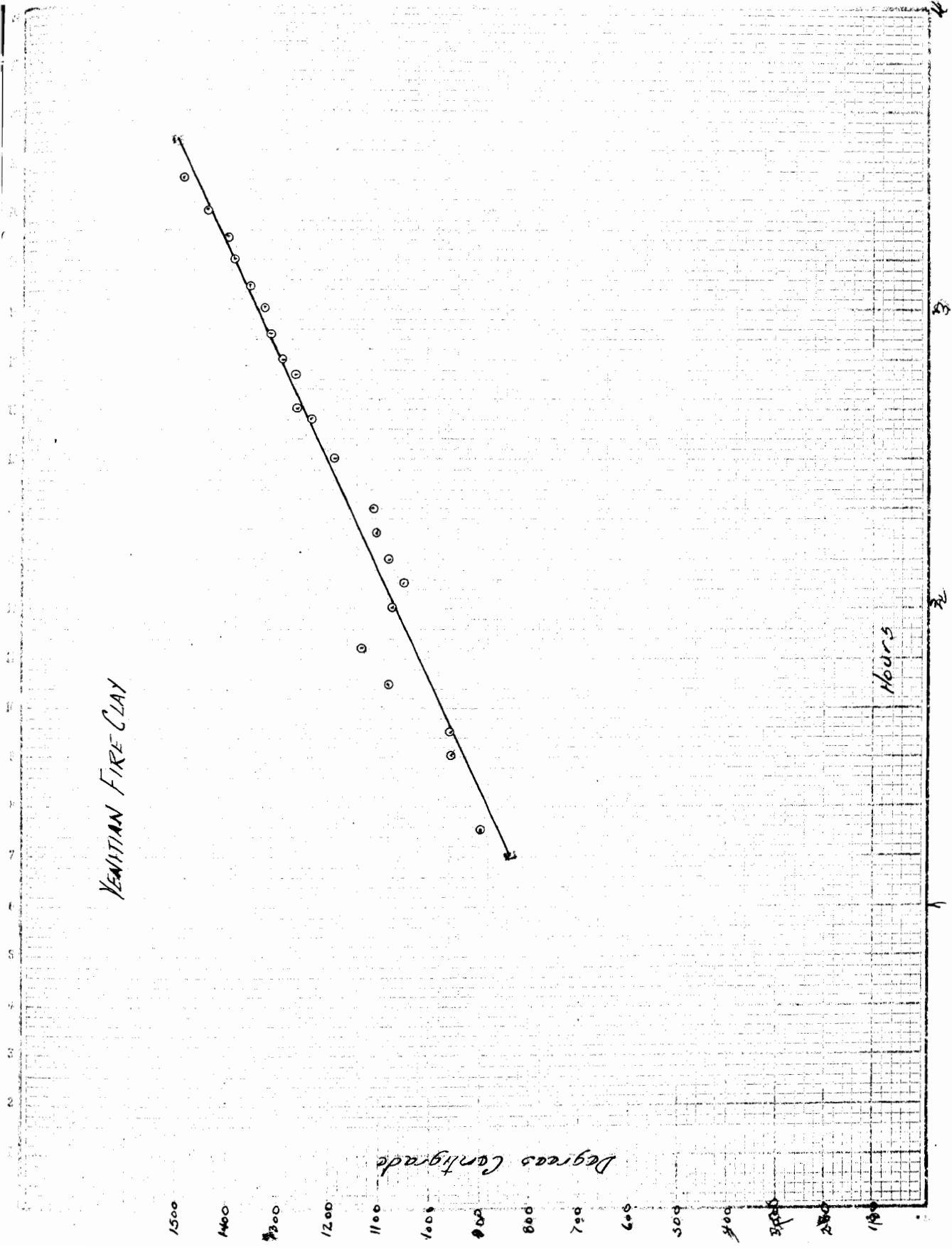
At 5:10 " " " 3125° F-

At 5:15 " " " End of Run.

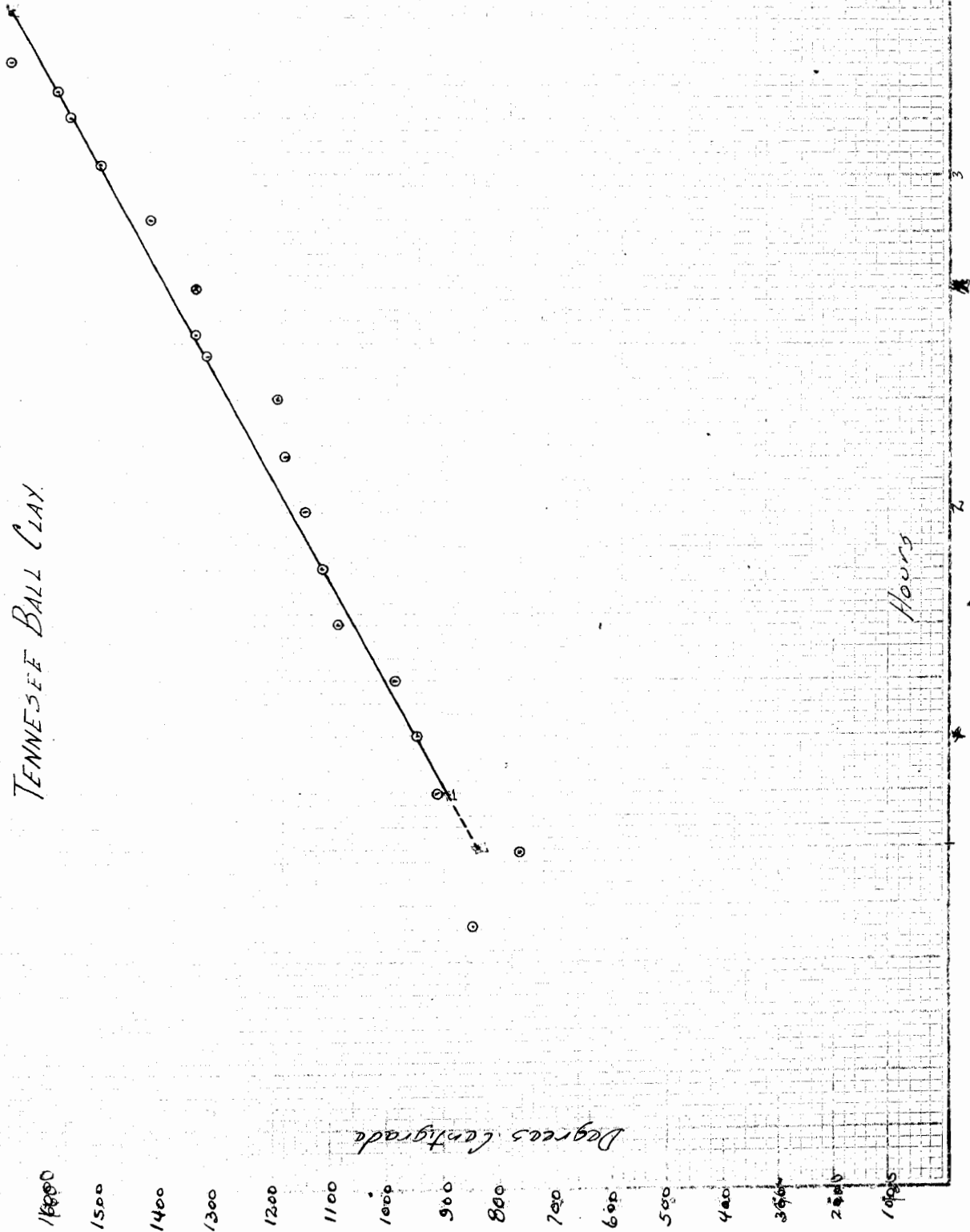
MISSOURI FLINT CLAY
CURVE SHOWING RATE OF HEATING



VENTILATION FIRE CLAY



TENNESSEE BALL CLAY



RESULTS:

In all samples and trials run in which several of the runs were duplicated it was found that there was about one half cones difference in the heat treatment that the unknown cones would stand. It was found in every case that the calcined cone came down before the raw cone although it was difficult to tell exactly what the difference was, but it was mostly found to be about the one half cone as stated above. The most representative result obtained from the runs was that gotten from the Venetian fire clay plaque. The calcined cone was clear down at the end of the run while the raw cone was in a two o'clock position. While in the case of the Tennessee ball clay not as sharp a difference was observed it could be seen that there was technically speaking a difference in the P. C. E. value of the two cones and likewise similar results were obtained from the flint cones which were fired to a much higher temperature. There was perhaps no commercial difference found in the two samples, but for laboratory and scientific purposes the difference found can be significant. *A sketch of the cone plaques is given on separate page.*

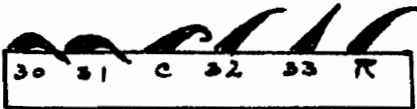
CONCLUSIONS:

It can be distinctly seen from the results obtained that there is a difference in the P. C. E. value of the two samples of the same material. In trying to explain and account for this difference it seems that it can most logically be based upon the fact that a given clay will stand a certain amount of heat treatment. This treatment may be administered at one time



VENITIAN FIRE CLAY

R = Raw Clay Cones
 c = Calcined Clay Cones



TENN. BALL CLAY



MISSOURI FLINT CLAY

NOTE:

This sketch shows one side of furnace to be hotter than other. Hot side is on side of which raw samples were placed which would tend to show that there is even greater difference in P.C.E. value than shown in other two sketches above

or extended over a period of firings as is characterized by the clacining period of those samples which were calcined. If the sample in question is receiving its first fire, then from these results it is permissable to assume that it will stand a slightly higher temperature for a short time than will a sample that has had an initial firing and is receiving subsequent heat treatment.

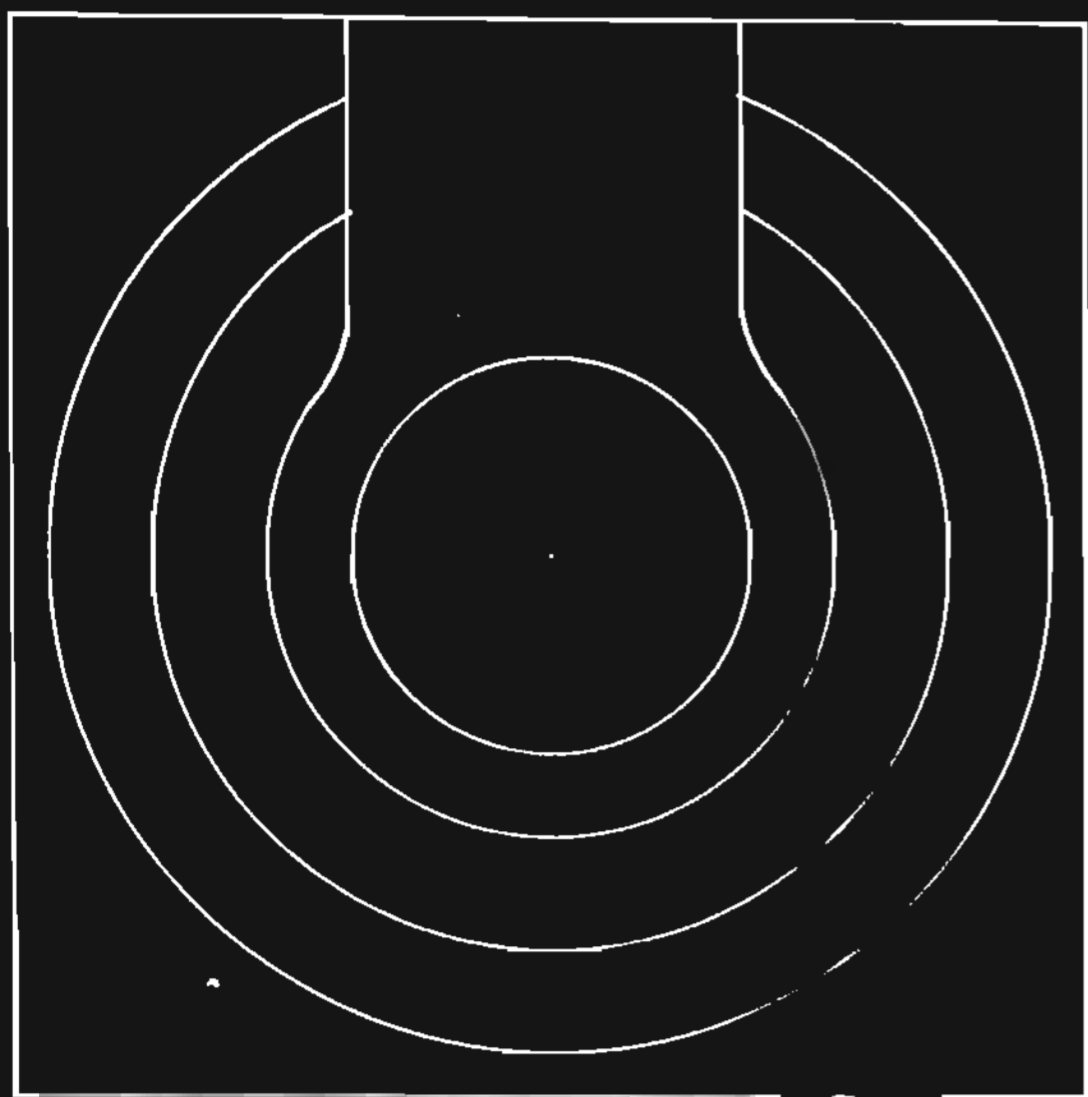
This is perhaps caused by the fact that there are certain chemical changes and reactions which occur within the clay due to the heat treatment and the more advanced these reactions the less resistant to heat the body becomes. From this it might be fair to assume that eutectics are formed which give a slightly lower P. C. E. value than the uncombined constituents of the raw clay. The longer the sample has been subject to the heat treatment the more advanced are these reactions and the less able is the clay to withstand the action of heat. There is a limit to this however for it is evident that the P. C. E. value would not continue to become lower and lower but would more nearly approach a constant point somewhat lower than the original raw clay.

It may be that there is even a greater difference in the P. C. E. value of the two samples than was shown in the results of this problem. This may be proven and would make an excellent problem to be worked upon at some later time, but due to lack of time the writer did not find it possible to go into this phase of the problem. It was noticed that the size of the raw cone after drying was considerably less than that of the calcined cone due to high shrinkage of the plastic portions

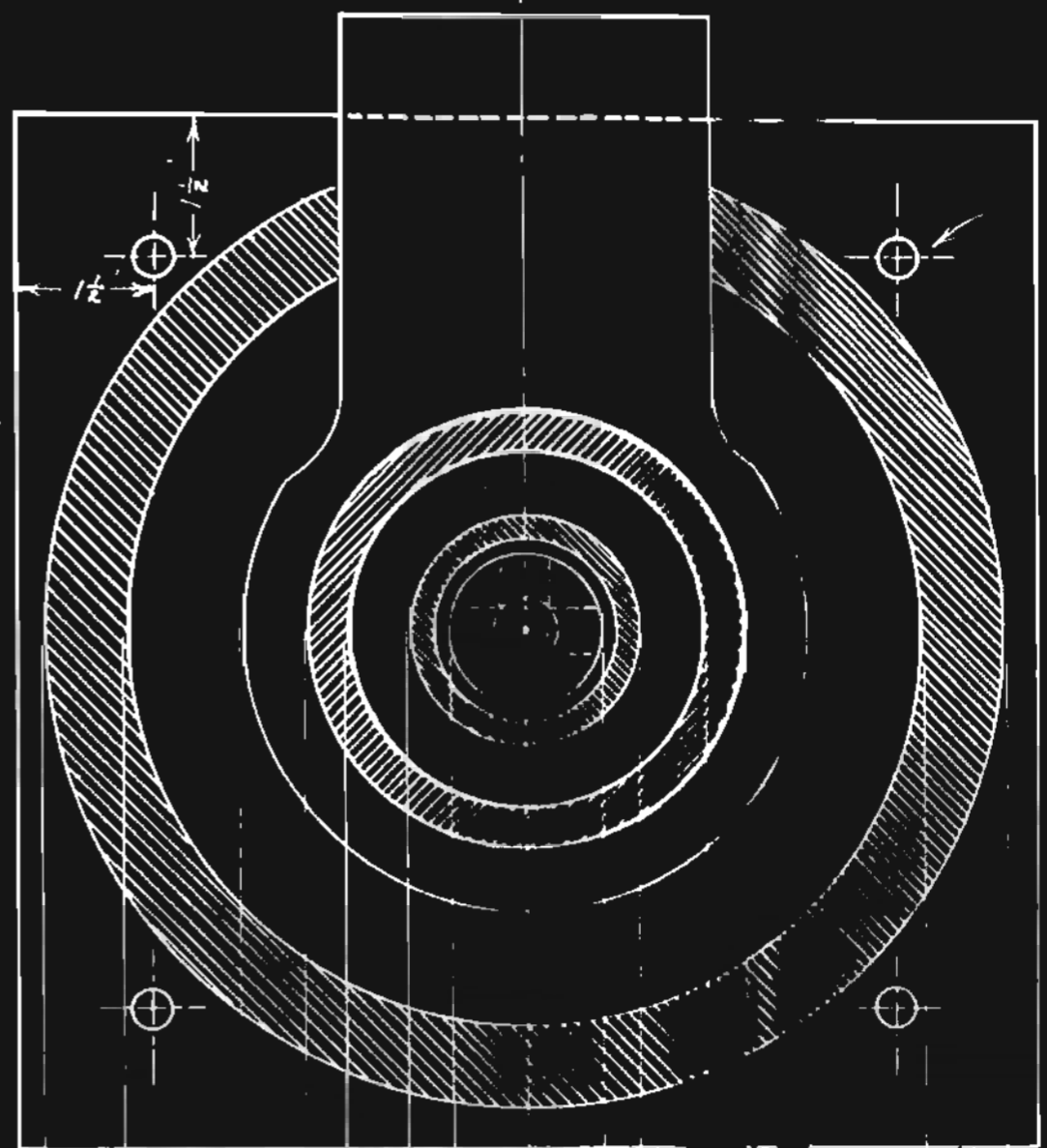
of the raw clay which was considerably more than that of the calcined clay. Both cones were molded in the plastic state from the same mold and after drying, the thickness of the calcined cone was considerably greater than that of the raw. If the raw cone were as thick at the time of firing as the calcined cone it might possibly stand a slightly higher temperature than the small cones would and thus make an even greater difference in the P. C. E. value of the two types of clays.

This is a very possible action and it is recommended that some student investigate the problem to find what facts may be obtained.

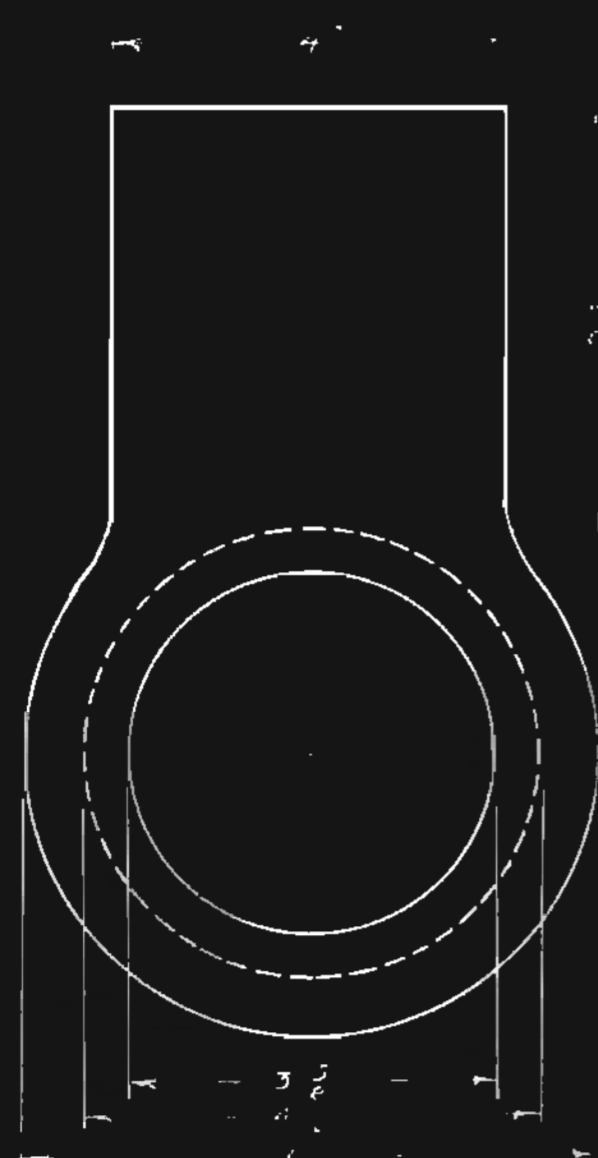
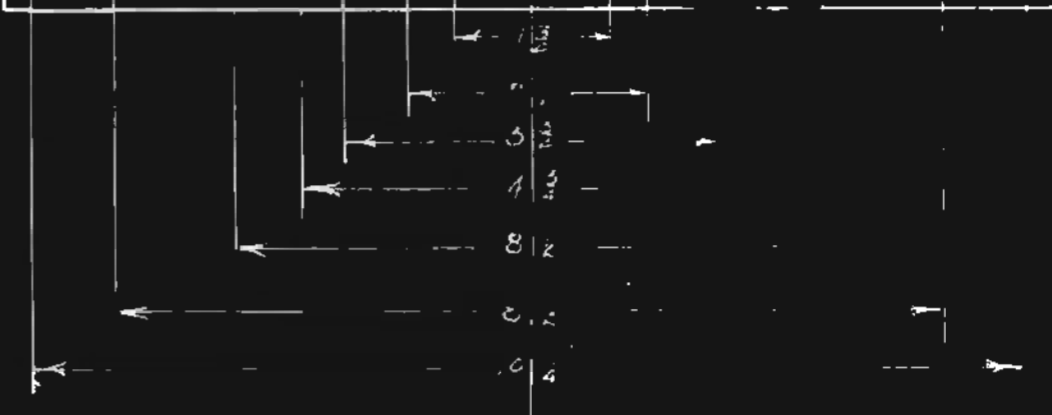
Another possible point of attack in which additional information might be obtained and which lack of time now prohibits is the obtaining of a mineralogical analysis of the clays before and after calcination and obtain such information concerning the transitions of the different mineral fragments as may be obtained and additional light may be shown upon the subject and the true cause for the difference in the P. C. E. value obtained.



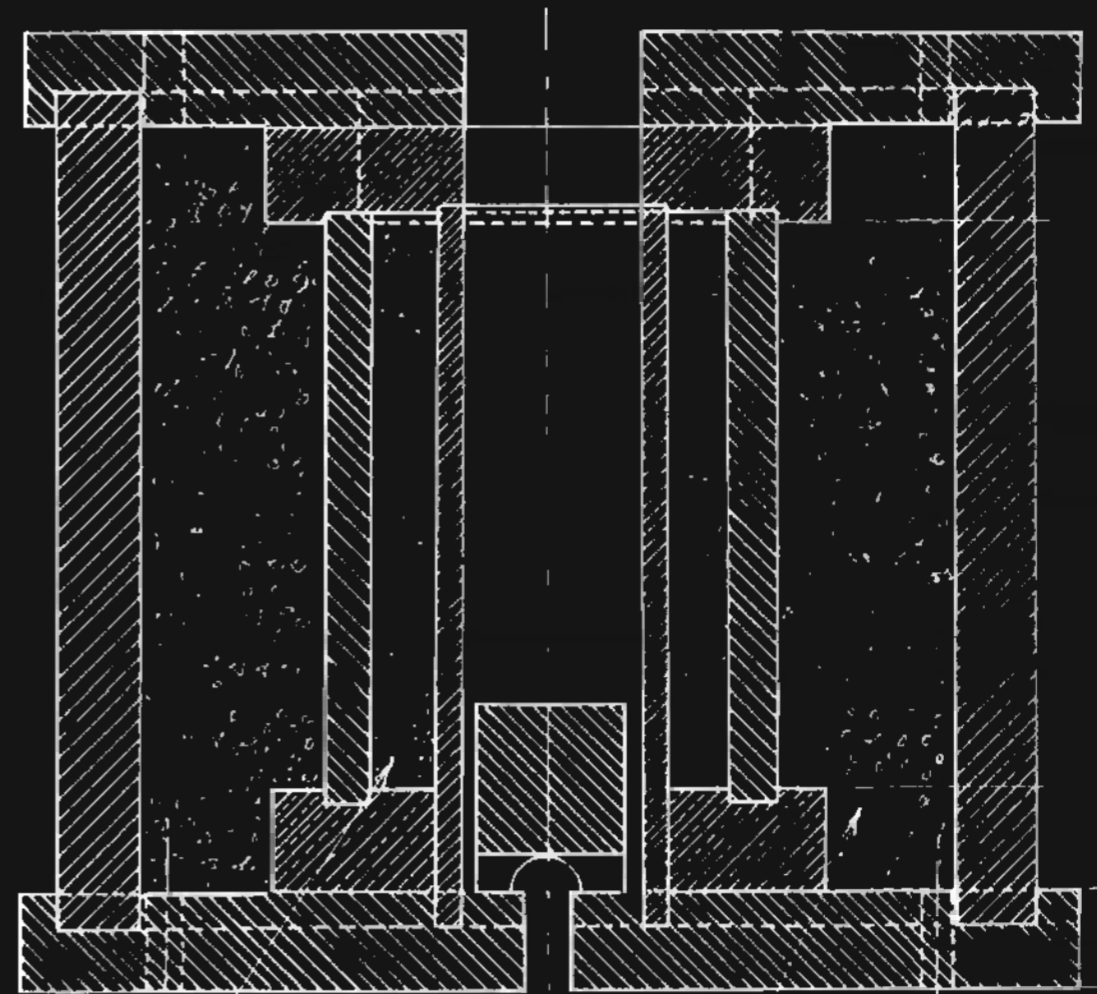
TOP AND BOT. M.V.



1/8" Hole



REAR VIEW



FRONT VIEW

REAR VIEW

ENGR. J. M. ...

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