1927

Effect of the use of old retorts as grog and of zinc oxide upon the properties of bodies for the manufacture of retorts

Elmer Gammeter

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EFFECT OF THE USE OF OLD METHODS AT COPPER AND OF ZINC OXIDE
UPON THE PROPERTIES OF BOLTS FOR THE MANUFACTURE OF RETORTS.

By

Elmer Sommerer.

A

THESIS

submitted to the Faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

in partial fulfillment of the work required for the

DEGREE OF

MASTER OF SCIENCE IN METALLURGY.

Rolla, Missouri,

May, 1927.

Approved:

[Signature]

Acting Superintendent of the Mississippi
Valley Experiment Station of the U. S.
Bureau of Mines.

33079
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INTRODUCTION.

In the fall of 1924 the Mississippi Valley Experiment Station of the United States Bureau of Mines, Department of Commerce, in cooperation with the Missouri School of Mines and Metallurgy began an investigation of the properties of raw materials used in the manufacture of retorts for the zinc industry. The investigation proceeded cooperatively until December, 1925, when the Bureau of Mines withdrew from ceramic research. The School then took over and is completing the problem as outlined.

The first phase of the investigation had for its object the determination of the properties of the fireclays and body mixtures now in use in the manufacture of zinc retorts.

The second phase deals with the comparative value of various grog materials presenting possibilities of use in the manufacture of zinc retorts.

The third phase of the investigation is the comparison of the properties of bodies made from various clays which present possibilities of utilization for the manufacture of retorts for the distillation of zinc.

This thesis contains a description of a part of the general Refractories Investigation, of which it is a supplementary phase. The methods of procedure and study in this thesis are in close harmony with the work done in the investigation as a whole.
REASONS FOR THE INVESTIGATION.

The cost of the retort in the smelting of zinc is a decided-
ly important item. The life of retorts is comparatively short, vary-
ing from twenty-five to fifty days. From a comparison of costs a
typical smelter in the Tri-State District reports an average life of
twenty-nine days for the retort, which however, is probably a little
low. The same smelter states that the cost of the retorts is roughly
five percent of the smelting cost. When all other items are consid-
ered, this immediately shows the effect of the service secured from
the retorts upon the smelting costs.

In addition the amount of zinc absorbed by a retort is quite
large, and in consequence, the loss of zinc by absorption is propor-
tional to the life of the retort.

Since retorts are subjected to severe treatment they must
be of high quality. To solve the matter of retort expense two me-
thods are apparent, namely, increase their life by less severe treat-
ment—which is impossible due to the process itself—or, to make
them of higher quality to render better service—which must be the
means of solving the problem.

From an investigation of the materials used in the manufac-
ture of retorts at a number of representative smelters, it was found
that some of the smelters were using old, or reclaimed retorts as a
part, at least, of their grog material. One serious handicap in
using old retorts is the effect of the slag that they carry. Although
this is usually chipped off before re-use, it is not done any too
well, and more or less slag, usually of very low fusibility, finds its way into the new retorts. In some plants, after removal of the slag, old retort material is crushed and added to the mixture. In other plants the reclaimed retort material, which forms a part of the new body, is the reject from concentrating processes that recover a portion of the zinc absorbed by retorts during smelting.

However, several reasons favor the use of old retorts for greg material. Probably the most evident is their relatively low cost, for in most cases they are a direct loss. Another reason is that the zinc present is thought to be in the form of zinc spinel, a highly refractory material, and if old retorts be used, this will increase the refractoriness. In the previous part of the investigation several some deformation values of old retort materials were determined, and in all cases the values were much lower than the raw clays. This may be attributed to a slag content, which despite careful picking, may be high due to inclusions or such. At any rate, zinc spinel, even if present, may have its refractoriness overcome by a slag content.
A BRIEF DISCUSSION OF ZINC RETORTS AND
THE ZINC RETORT PROCESS OF ZINC DISTILLATION.

Although a few generalities of zinc retorts and the retort process were discussed in the previous paragraphs, it does not seem out of place to include in this thesis considerably more of these governing generalities.

So it is that a discussion of the fundamentals of the retort process of zinc distillation, the manufacturing of zinc retorts, and the qualifications and service demanded of retorts, is included in the following. The discussion, of course, is very brief, but serves in a measure, to bring to light the fundamentals underlying the investigation.

The distillation of zinc after a reduction with carbon is commonly called "smelting." The process is quite suitable in theory but the practical execution is accompanied with a large amount of difficulty, so that the development in zinc smelting has been slow. The capacity and labor saving devices, which have been so largely and beneficially developed in other metallurgical processes, have been quite lacking in the zinc industry. This is due, in a large measure, to the conditions prevalent in the smelting. That is, in the reduction there must be present a strong reducing atmosphere of carbon monoxide, and a very small amount of carbon dioxide; the latter unfortunately has a strong tendency to oxidize the zinc vapor formed in the reduction. The favorable conditions can be easily obtained on a small scale—as the retort method—but all attempts to make a success—
ful reduction on a large scale, as a blast furnace or reverberatory
furnace, have failed. In either of the latter methods the failure
is mainly due to the poor control of the carbon dioxide content and
the resulting oxidation of the zinc vapors, particularly during cool-
ing of the gases to condensing temperatures. As a general result the
smelting of zinc has practically remained where it started and is
still a small scale process.

The reduction as carried out in the retorts is caused by the
heating of the roasted ore (chiefly zinc oxide) well mixed with fine
coal. The zinc formed is in the vapor state. This zinc vapor ex-
pells itself from the retorts and passes into condensers, which are
placed in contact with the open end of the retort, yet away from the
intense heat necessary for the reduction. The hot zinc vapors, upon
reaching this cooler region, condense as molten zinc.

The distillation furnaces are of numerous styles and designs,
but they may be roughly classified as follows: (1) those using natural
gas as fuel, and (2) those using coal as fuel. The latter may be sub-
divided into (a) those having direct firing, and (b) those using pro-
ducer gas made from coal. Direct firing, however, finds very little
use in this country.

Natural gas firing is very good, depending of course on the
supply on hand. It has a use limited to the life of the local field,
which in general, is comparatively short. Producer gas, on the other
hand, has only a dependency on the coal fields, which are much more
stabilized. Producer gas is made either by partially burning the
lower bed of the coal and thus distilling the upper coal and obtaining a gas rich in carbon monoxide and hydrocarbons, or, by the action of steam on hot coke previously burned and distilled of its rich gas content, as in the previous method. Although this latter method gives a gas richer in hydrogen (the action of steam on hot coke is: 
\[ \text{H}_2\text{O} + \text{C} \rightarrow \text{H}_2 + \text{CO} \]), nevertheless it requires more heat for the disintegration of the steam than the direct distillation. In either case there is a direct loss of heat by the burning necessary in the producer. This loss is decreased somewhat by placing the gas producer close to the furnace to take advantage of preheating, or other methods of heat conservation. It is usually assumed that simple firing with producer gas has a saving of about twenty per cent over direct firing. In any case of recuperative or regenerative methods of heat-saving the advantages are proportionally increased. A method of regeneration of both air and gas is in common use and is found very economical.

The furnaces, as has been said, are varied in design. In general they are heated vertically or horizontally. In the vertical method the flames are introduced below the lower retorts and rise up and around the upper retorts. In the horizontal method the flames are introduced at the sides and sent in somewhat of a longitudinal way under and over the rows of retorts. In either case the retorts are usually set in horizontal rows about four to six rows high, the whole set, roughly 600 to 1000 in number, is called a "block."
The retorts are, in all cases, bridged across and supported at their ends. The furnaces are usually double having two sets of retorts, which may be called a front set and a rear set, for they are arranged with butt end against butt end at the center wall of the furnace. They slope, at a slight angle, outward toward what may be called the front and rear respective parts of the furnace, where their open ends are supported. The angle of slope is to facilitate loading and to allow the hot slags formed to flow away from the intense heat of the center of the furnace, where the corrosive effect of the slags would be greatest.

The process of distillation may be briefly outlined in four steps, occurring every twenty-four hours, as follows:

1. The condensers are removed and the retorts cleaned of spent charge while still at a bright red heat. The residue is partially raked out, after which a stream of water is sent through a pipe imbedded in the remaining residue. The pipe extends to the rear of the retort and the steam, which at the water's injection is immediately formed, blows out the remaining residue of spent charge. Broken retorts are then replaced and all retorts are re-charged, usually by hand. The charge consists of a rather wet mixture of roasted ore (about 6 mesh size) and fine coal, thoroughly mixed and wetted with water. This first stage requires from three to five hours.
2. This stage consists of preliminary reduction, in which the water is driven off, the hydrocarbons expelled, and the oxides of the metals other than zinc are partially reduced. The gases are expelled from the retort going directly into the condenser, which is a frustrum-shaped vessel some two feet long, having its larger end fitted into and sealed against the open end of the retort. The smaller end is closed with a slotted plug through which the gases pass out. The gases are ignited and burn with a yellow flame at first, changing to a purple color as the carbon monoxide content increases. As the zinc distillation begins the flame changes to a green color.

This stage lasts from three to five hours.

3. This stage is the period of active distillation. The zinc distillation becomes very active and the indicative flame lengthens and becomes decidedly green.

This stage requires about thirteen hours.

4. This is the stage of waning distillation in which the flame gradually turns to a purplish carbon monoxide and loses its green of zinc. In order to get as much as possible of the remaining zinc, the furnaces are pressed hard with higher temperatures, and the retorts get severely tested. This stage lasts about four hours, the process then repeats itself going through a single distillation every twenty-four hours.
Zinc distillation as outlined above is, to say the least, a tedious process. It probably requires more labor than the extraction of any other metal of similar value. The losses are relatively numerous and large. Some zinc oxide is always formed and constitutes a rather large loss. In addition, some zinc remains in the residues, some fails to condense, and some is absorbed by the retort. Since a new retort may absorb a very large amount of zinc during its early use, this latter loss is quite marked. In many cases reground retorts, and even spent residues, are concentrated to recover their zinc value, and this, to some extent, overcomes some of the losses.

The zinc retorts themselves are of three different types: Silesian, American and Belgian. Since American smelters almost completely use the Belgian type it will be considered and explained in detail. This retort is of cylindrical or elliptical shape, the cylindrical probably more common. The retorts vary from 5 to 64 inches in length with an inside diameter of about eight inches and an outside general diameter of about 10 to 12 inches.

In the manufacturing of these retorts there is desired a very strong and highly refractory product. But the difficulty arises in that the very refractory fireclays are of great shrinkage when burned. This shrinkage is inclined to cause check marks or rents in the finished product and thus weaken it. It is standard practice to mix a proportion of previously burned material with this plastic fireclay; since this previously burned material has already shrunk it will reduce the shrinkage of the finished product. Mixtures of
forty to fifty per cent plastic clay, called the bond clay, and the balance to be of the burned material, called the grog, are in common use. The proportions vary, of course, with the properties of the bond clay and grog used. But even with a mixture of the grog certain clays, although highly refractory, are still too plastic and are, of consequence, mixed with the denser or the sandy clays. There are, of course, in several localities deposits of plastic clay (the St. Louis district is a good example) where a clay of suitable properties occurs alone in nature.

In the manufacturing of the retorts various problems are encountered. The manufacturing practically must be carried on at the smelter as the retorts must be put into the furnace while at a red heat, since the processes as outlined must go on with but slight time for installation and tempering. The retorts are also bulky and fragile and shipment would be difficult.

At the potteries, which the individual smelters have, the clay is received in lump form direct from the mines. The clay is allowed to remain exposed to weather conditions for about two weeks. This exposure, called "weathering", is peculiarly a benefit in developing the plasticity of the clay.

The clay is then ground to pass a 6 mesh sieve and the grog to pass an 8 mesh sieve. The size of the grog is a particularly important feature and much work has been done towards attaining the proper size. The materials are now thoroughly mixed dry and then run into a pug or mixing machine, where water is added to the extent of about
ten per cent. The clay, in a semi-plastic state, is taken from the
mill in large blocks and then sent to the rotting pits—large brick
chambers with no ventilation. Here these blocks are covered with
damp burlap and allowed to remain for about three weeks, in which
time they "rot" or "age", which develops their full plasticity. The
material is then repugged to insure the proper mixture and water con-
tent. It then leaves this second pug machine in ballots, the correct
size and shape to fit the presses.

The material comes to the machine for making the retorts in
a rather stiff condition. A stiff mixture yields a retort of high
strength, resistance to slag action, and a low porosity, and in con-
sequence, is desired. Many of the machines used are hydraulic
presses, although some sugar machines are still in use. The retorts
are pressed in these machines, which are equipped with steel molds—
shaped to form the retorts.

The retorts then are measured, trimmed and sent to the
drier, a room heated by steam coils to about 52 to 54° C. and equip-
ped with ventilating flues at the roof to carry off the moisture.
The retorts are placed upright on their butt ends and remain in the
room from fifty days to four months, depending on the operating
conditions. The retorts are then brought to a storage room ready
for the tempering kilns.

The retorts are placed in the kilns (of either up or down-
draft type) and firing starts about noon. Heating continues until
at 6 A. M. a temperature of around 800° C. has been reached. Since
the furnaces are usually being overhauled for defective retorts, re-
charging, etc., from about 6 A.M. to 10 or 11 A.M., the annealed re-

torts are taken hot from the kilns as needed and set in the places made 

vacant for them. The kilns are, in all cases, located close to the 

furnaces.

It is well to consider again, and in more detail, the ser-

vices rendered and expected of these retorts. They must be very 

highly refractive to withstand a temperature of 1350 to 1400° C. 

The retort must be a compact mass and this can be accomplished by 

a plasticity of the bonding clay, which closely cements the hard 

dense particles of the pre-burned greg. The retorts should have con-

siderable mechanical strength to withstand handling, and they must 

support a load of about one hundred pounds of charge per retort at 

the high temperatures to which they are subjected. They must be 

dense when in use and of corresponding low porosity so as to have 

maximum imperviousness to the action of the zinc vapors, and to with-

stand the chemical corrosive effect of the hot slags in contact. 

They must have thin walls to permit the high conductivity necessary 

to maintain the heat flow, and they must withstand the sudden change 

of temperature that the clearing out with a stream of water, while 

still red hot, and the loading with a damp charge, requires.

These requirements are dependent primarily on the raw mater-

ials, the plastic clay being first in importance. The chemical 

analysis of clays does not give any definite conclusion as to the 

properties a clay may have. Clays must be tested for their physical 

properties, such as shrinkage, sag, porosity and spalling, but even 

these tests are inadequate to their actual use.
As has been said a high refractoriness and a moderately high plasticity is essential to a good bond clay. The chemical analysis, however, does, to a certain extent, determine the properties. The presence of some oxides, chiefly iron, lime and alkalies, causes a low fusion, and consequently, these oxides should be low. The total of these oxides should be very little over three per cent. of the clay content, preferably below that figure.

MATERIALS USED.

The materials that were used in this investigation may be classified in outline form as follows:

I. Clay.
   1. Milled Cheltenham fireclay from the St. Louis District.

II. Grog.
   1. A Missouri flint clay, calcined at Orton Cone No. 16.
   2. Reclaimed retorts.
   3. Zine oxide, C. F.

III. Body Mixtures.
    From the above material the following eight body mixtures were made:

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The bond clay was a very good grade of Cheltenham clay. The flint clay was obtained locally and was carefully calcined in the test kiln to Cone 16, at a rate of about 300° C. per hour until 1000° C. was reached, after which the rate of heating decreased to about 100° C. per hour. The calcination was held just at Cone 16 (1450° C.) for two hours. The calcine was hand-picked to remove dark brown aggregates of iron that appeared after burning, the remainder crushed in the laboratory jaw crusher, and finally screened through a 6 mesh sieve.

The reclaimed retorts used were a mixture of two laboratory samples of ground reclaimed retorts on hand. They were as follows:

1. Grasselli recovered retorts - 66.5 pounds.

2. Athletic Mining & Smelting Co. old retorts - 51.0 pounds.

The above two samples were carefully mixed, and screened through a 6 mesh sieve.
The zinc oxide used was the chemically pure amorphous powder. Since it was deemed advisable to study the effect of the addition of zinc in retorts there was some discussion as to the form in which it should be introduced. It is uncertain as to what form zinc is present in retorts. It is often considered as a spinel—zinc aluminate (ZnAl₂O₄) and again as a silicate (Zn₂SiO₄). In either case the zinc may be primarily considered as an oxide. Thus in adding zinc into a retort mixture it was deemed advisable to add it in the form that it would most probably have when present in a used retort; this was thought to be the oxide.

TESTS MADE.

All of the tests were made according to the standard procedure of the American Society for Testing Materials and the American Ceramic Society, with however, some changes made necessary by local conditions.

Chemical Analysis.

The materials that were analyzed were carefully coned and quartered and cut on a Jones riffle sampler to the required size. This was then ground in an agate mortar; put through a 65 mesh sieve and cut in half. One-half was used directly for cone deformation value determinations, while the other half was further ground to pass a 100 mesh sieve for chemical analysis. In all cases careful rolling and general sampling requirements were observed. The experimental results, Figure 5, show the determinations that were made.
Cone Deformation Value.

As noted under Chemical Analysis, cones for this test were prepared from samples of bond clay, flint clay and old retorts, ground to pass a 65 mesh sieve. These samples were mixed with gum arabic and worked to a firm, yet plastic, consistency. From the body mixtures samples were taken and ground to pass a 65 mesh sieve; gum was used for a binder. All cones were formed in steel molds, the size and shape of the Standard Pyrometric Cones as prepared by Edward Orton, Jr.

The test cones were mounted separately on circular plaques along with the standard cones with which they were to be compared. These circular plaques (about one and one-half inches in diameter and one-half inch thick) were prepared from the following ingredients:

Fifty per cent St. Louis plastic fireclay.

Twenty-five per cent calcined flint clay.

Twenty-five per cent alumnum cement, and sufficient water to form a stiff mixture.

A small furnace of Fulton's granular resistance type, Figure 1, suitably arranged, was used for the deformation value determinations. This furnace was operated by a motor generator set, producing accurate voltage control to 125 volts.

The cone plaque was set in the center of the inner tube on a cylindrical alumnum support, about one and one-half inches in diameter. This support was cross-notched at the base and placed over a small hole in the bottom of the furnace. Through this opening air entered and circulated about the plaque, thus maintaining an oxidizing atmosphere without appreciable cooling of the furnace.
Figure 1.

Fulton's Granular Resistance Furnace.
The furnace was heated rather rapidly to about 800°C, after which a rise of ten degrees per minute was maintained. When the tip of the unknown cone bent over and just touched the plaque the deformation value was indicated. It was recorded as the serial number of the standard cone that concurrently deformed. Since these standard cones run about 20°C apart, differentiation between cones was sometimes necessary. The values determined are shown in Figure 6.

Preparation of the Bodies.

The eight body mixtures were made up, consisting of fifty pounds of ingredients for each mixture, and proportioned as previously noted. These bodies were mixed with water in a small laboratory pug mill. The mixtures were then set aside in tubs to 'age', or season, for a period of from ten to fourteen days. The plasticity is said to be increased by this treatment, in which the presence of water is the controlling factor. If proper water content is added it can be so arranged that the mix will be in shape to be molded at the end of the aging period. However, the water content varied considerably in the eight mixtures, and along with some mechanical drying and other difficulties, the aging period was increased in some cases. The period for each mixture follows:

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<td>14.5 days</td>
<td>7</td>
<td>19 days</td>
</tr>
<tr>
<td>4</td>
<td>12.5 days</td>
<td>8</td>
<td>21 days</td>
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Figure 2.

Detail Drawing of Mercury Volumeter.
Figure 3.
Photographic Views of Mercury Volumeter.
It was decided that the small discrepancy in aging period would not materially affect the tests, although a small error may be attributed to this cause.

From each mixture all of the specimens needed—firing behavior, transverse strength, sag and spall—were made as rapidly as possible so that any errors due to differences in water content would be avoided.

Drying Shrinkage.

The specimens for drying shrinkage determinations were made in steel molds, and were one and one-eighth by one and one-eighth by one and seven-eighths inches in size. The mixtures were well pounded into the molds with a steel hammer and the specimens made as uniformly smooth as possible.

The specimens, immediately after being formed, were weighed on a balance to an accuracy of .01 gram and this weight recorded as the plastic weight. The volumes were then determined in a mercury volumeter, Figure 2 and Figure 3.

This volumeter was designed and developed especially for such measurements, but it can also be used to secure the volumes of other materials of similar size. The volumeter has been tried out very carefully in actual work as well as in comparative tests using standard specimens. In all cases it has given very satisfactory results.

Calibration of the volumeter with mercury indicated a slight compression of the rubber packing ring, the amount of compression depending on the height of the mercury column. The correction varies,
but is only 0.13 cc. for volumes of 40 cc., and is correspondingly
less for smaller volumes. A steel test specimen with a volume of
35.78 cc., as calibrated by the Bureau of Standards, when measured in
the volumeter, indicated a volume of 35.65 cc. To this is added
0.11 cc., the correction for the compression mentioned above at 35 cc.,
or the volume as given by the instrument is 35.78 cc. The difference
between the true and measured volumes is only 0.02 cc. and is negli-
gible.

The principle of operation of the volumeter is simple. The
specimen, or other object whose volume is to be measured, is placed
in the reservoir or well, containing a suitable amount of mercury.
A piston, to which a measuring burette is attached, fits into this
well, making a mercury-tight sliding contact with the walls of the
well. The lowering of the piston into place submerges the specimen,
forcing the displaced mercury up into the burette, where the volume
is read. The test piece is then removed and a zero reading taken.
This zero reading is taken after the reading with the specimen includ-
ed to eliminate errors due to possible absorption of mercury into the
pores of the specimen. The difference in the two readings is then the
volume of the specimen.

After the plastic volume of the specimens had been taken,
they were lightly brushed to remove adhering particles of mercury,
and set aside to air dry. They were turned over every twelve hours
to permit as uniform drying as possible. This drying period lasted
about two weeks. At the end of that time they were placed in a drying
oven and heated slowly to about 108° C. (just under 110° C.) and held at that temperature for five hours.

The specimens were then allowed to cool in a desiccator, re-weighed as before, and the weight recorded as the dry weight. The test pieces were then immersed in kerosene and permitted to soak for twelve hours; this was done to fill the pores and prevent errors due to the absorption of mercury. After removal from the kerosene the specimens were dried lightly with a cloth to remove the film of adhering kerosene. The volumes were then taken and these volumes recorded as their dry volumes.

The drying shrinkage, or the per cent volume change in drying, was then calculated as follows:

\[
\text{Per cent drying shrinkage} = \frac{\text{Plastic volume} - \text{dry volume}}{\text{dry volume}} \times 100. \\
\]

The values were then averaged and plotted as shown in Figure 7.

Water of Plasticity.

The same specimens were used for the determination of the water of plasticity as were used for the determination of the drying shrinkage. The method, run simultaneously with the drying shrinkage determination, has been previously outlined. The calculations were made according to the following relation:

\[
\text{Per cent Water of Plasticity} = \frac{\text{Plastic weight} - \text{dry weight}}{\text{dry weight}} \times 100. \\
\]

The plotted results are shown in Figure 7.
Firing Behavior.

After drying shrinkage and water of plasticity had been determined, the specimens were placed in six equal files, according to the temperatures to which they were to be burned. Of the twelve test pieces made from each mixture, two were burned at each of the following temperatures: 1000° C., 1100° C., 1200° C., 1300° C., 1350° C., and 1400° C.

The specimens were arranged in the test kiln, Figure 4, with those to be burned at the lower temperatures in front.

The test kiln is arranged for both gas and oil firing. The gas was ordinarily used for temperatures up to 700° C., after which oil was used. The oil burning was accomplished with burners using forced fan draft. The flames were sent over the two-side baffle walls and down through the hearth and into the flue. Careful manipulation of the burners gave a sufficiently close regulation of temperatures. The measurement of the temperatures was accomplished by both Orton cones and noble-metal thermocouples. The temperature was brought up at a uniform rate of fifty degrees per hour.

At the first temperature of drawing (1000° C.), the specimens for this burn were removed through the wicket. They were immediately covered with hot sand to prevent rapid cooling. Then cool enough to handle they were placed in desiccators and permitted to cool further to room temperatures.

This operation repeated at each of the succeeding higher temperatures. At 1400° C., however, the furnace was shut down, and the final lot of specimens permitted to remain in the furnace until cool.
Figure 4.

Laboratory Test Kiln.
After cooling to room temperatures the fired weight was obtained to an accuracy of 0.01 gram. The specimens were then boiled in distilled water for two hours, allowed to cool in the water to room temperatures, removed from the water and lightly dried with a cloth, and finally weighed on a balance to the same accuracy as before; this weight was recorded as the saturated fired weight. The fired volumes were then determined in the mercury volumeter, as previously described. Data was then calculated according to the following relations:

\[ \text{Per cent volume change in firing, or firing shrinkage} = \frac{\text{Dry volume} - \text{fired volume}}{\text{dry volume}} \times 100 \]

\[ \text{Per cent porosity} = \frac{\text{Saturated fired weight} - \text{fired weight}}{\text{fired volume}} \times 100 \]

\[ \text{Per cent absorption} = \frac{\text{Saturated fired weight} - \text{fired weight}}{\text{fired weight}} \times 100 \]

The averaged data and curves plotted for the above determinations are shown on Figures 6, 9 and 10, respectively.

Transverse Strength Test.

Twenty specimens for each mixture were made in steel molds, each specimen measuring one inch by one inch by seven inches. They were permitted to dry in the room, but were covered with damp towels to prevent too rapid drying. The specimens were turned over every twelve hours to prevent warping, and the towels were re-dampened at these times. Thorough drying took from three to four weeks.
After drying, five specimens of each mixture were burned at the following temperatures: 600° C., 1000° C., and 1400° C. The test pieces were arranged in two rows in the test kiln.

The test pieces were then broken on a machine as shown in Figure 5. The specimens were supported on two knife edges set five inches apart, and were broken by a load applied on the end of a beam; the latter carried a knife edge resting on the center of the supported test pieces. Increments of the load were made by adding shot automatically to a container on the end of the beam. After the break, the load applied, the breadth and depth of the specimen at the break (measured with a micrometer) were recorded.

The modulus of rupture for each test piece was then calculated according to the following formula:

\[
\text{Modulus of Rupture} = \frac{3P}{bd^2}
\]

Where

- \( P \) = breaking load in pounds
- \( b \) = breadth in inches
- \( d \) = depth in inches.

Averages of the specimens for each mixture at each burning temperature were then calculated. After individual values, which deviated more than fifteen per cent from their averages were discarded, the remaining values of the set were re-averaged. The results were then plotted as shown in Figure 11.
Figure 5.

Transverse Strength Testing Machine.
Sag Test.

Twelve specimens, each measuring one inch by one inch by thirteen inches, were made for each of the eight body mixtures. They were formed and dried as were the drying shrinkage specimens previously described. Two specimens from each mixture were set on bricks with a span of ten inches, and fired to each of the following temperatures: 1050° C., 1100° C., 1200° C., 1300° C., 1400° C., and 1450° C.

After cooling to room temperatures the amount of sag was measured by outlining the specimen on paper, and the amount of deflection for the ten inches was measured and recorded in inches. The sag data appears in tabular and graphical form in Figure 12.

Spalling Test.

Six specimens of each mixture were made for this test; each measured one and one-eighth by two and one-half by six inches. The specimens were air dried in the usual manner, probed to 1400° C., held at that temperature for five hours, and then cooled and weighed.

Four specimens of each body mixture were fired to a temperature of 1270° C. The test pieces were arranged in two rows in the wicket of the furnace, care being taken to expose only the one and one-eighth by two and one-half inch end to the heat of the furnace. After one hour of heating the specimens were removed from the furnace and the heated end immersed in running water (22° C.); approximately one-half of the test piece was immersed.
After three minutes the specimen was withdrawn from the wa-
ter, allowed to steam for five minutes, weighed, and then returned
to the furnace to re-heat. Two specimens were withdrawn at a time,
and the schedule was so arranged that two were in the water and two
others steaming at the same time. This cycle required forty minutes,
and then thirty minutes was allowed for more complete re-heating to
the required temperature, making a total cycle of one hour and ten
minutes. Each specimen was carried through twenty similar cycles.
The test pieces were carefully observed and any cracking or spalling
noted. After the test the specimens were allowed to air dry, and
were then weighed.

The number of dips before spalling commenced was determined
from averages of the four specimens of each mixture. The average
per cent decrease in weight was determined according to the following:

\[
\text{Per cent decrease in weight} = \frac{\text{Original weight} - \text{Fired dry weight}}{\text{Original dry weight}} \times 100.
\]

This test, however, has many variables, as for instance, ob-
taining the exact degree of water exposure, permitting accurate and
uniform heating conditions, proper timing for the period in the water
and in steaming, and many others. The method cannot be accepted as an
accurate method of determining spalling action, and only rough compar-
sions can therefore be made. Figure 13 shows a chart of the spalling
test data. It contains all of the information concerning the test that
was recorded. Figure 14 shows the two curves that were plotted.
EXPERIMENTAL RESULTS.

On the following pages will be found the experimental results; both the averaged data and the curves for each test are included on the same sheet.

The chart of the chemical analysis and deformation values, Figure 6, has been made up in this concise form, so that its information would be readily available.
## Chemical Analyses and Deformation Values of Clay, Groggs, and Body Mixtures

<table>
<thead>
<tr>
<th>Material</th>
<th>$\text{Al}_2\text{O}_3$</th>
<th>$\text{SiO}_2$</th>
<th>$\text{Zn}$</th>
<th>$\text{Fe}_2\text{O}_3$</th>
<th>$\text{CaO}$</th>
<th>$\text{MgO}$</th>
<th>$\text{TiO}_2$</th>
<th>$\text{Na}_2\text{O}$</th>
<th>$\text{K}_2\text{O}$</th>
<th>Ignition Loss</th>
<th>Deformation Value</th>
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<tbody>
<tr>
<td>Fire Clay</td>
<td>29.96</td>
<td>54.00</td>
<td>2.33</td>
<td>0.67</td>
<td>0.13</td>
<td>1.49</td>
<td>0.55</td>
<td>0.34</td>
<td>11.52</td>
<td>29</td>
<td></td>
</tr>
<tr>
<td>Old Retorts</td>
<td>30.84</td>
<td>50.60</td>
<td>9.10</td>
<td>6.79</td>
<td>Trace</td>
<td>1.47</td>
<td>0.47</td>
<td>0.81</td>
<td></td>
<td>17</td>
<td></td>
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<tr>
<td>Flint Clay</td>
<td>45.54</td>
<td>50.60</td>
<td>1.88</td>
<td>0.10</td>
<td>Trace</td>
<td>2.06</td>
<td>0.39</td>
<td>0.18</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Body No.1</td>
<td>37.75</td>
<td>52.30</td>
<td>2.105</td>
<td>0.385</td>
<td>0.065</td>
<td>1.775</td>
<td>0.47</td>
<td>0.260</td>
<td>5.76</td>
<td>31-32</td>
<td></td>
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<tr>
<td>Body No.2</td>
<td>35.92</td>
<td>52.31</td>
<td>1.14</td>
<td>2.73</td>
<td>0.463</td>
<td>0.065</td>
<td>1.698</td>
<td>0.481</td>
<td>0.338</td>
<td>5.76</td>
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<tr>
<td>Body No.3</td>
<td>34.08</td>
<td>52.30</td>
<td>2.28</td>
<td>3.335</td>
<td>0.530</td>
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<td>52.31</td>
<td>3.41</td>
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<tr>
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<td>2.01</td>
<td>2.055</td>
<td>0.383</td>
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<td>1.720</td>
<td>0.460</td>
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</tr>
<tr>
<td>Body No.7</td>
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<td>49.77</td>
<td>4.01</td>
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<td>0.251</td>
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<td>Body No.8</td>
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<td>0.441</td>
<td>0.247</td>
<td>5.76</td>
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</tbody>
</table>

**Note:** Chemical analyses of bodies are calculated. Deformation value expressed as Orton cone number.
Figure 6.

Chemical Analysis and Deformation Values of Clays, Grags and Body Mixtures.
Figure 7.

Water of Plasticity and Drying Shrinkage of Body Mixtures.
<table>
<thead>
<tr>
<th>Body No.</th>
<th>Composition</th>
<th>Volume Change</th>
<th>Degrees Centigrade</th>
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</thead>
<tbody>
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<td></td>
<td>Fire Clay</td>
<td>Old Refers</td>
<td>Flint Clay</td>
</tr>
<tr>
<td>1</td>
<td>50.0</td>
<td>0.0</td>
<td>50.0</td>
</tr>
<tr>
<td>2</td>
<td>50.0</td>
<td>12.5</td>
<td>37.5</td>
</tr>
<tr>
<td>3</td>
<td>50.0</td>
<td>25.0</td>
<td>25.0</td>
</tr>
<tr>
<td>4</td>
<td>50.0</td>
<td>37.5</td>
<td>12.5</td>
</tr>
<tr>
<td>5</td>
<td>50.0</td>
<td>50.0</td>
<td>0.0</td>
</tr>
<tr>
<td>6</td>
<td>50.0</td>
<td>0.0</td>
<td>47.5</td>
</tr>
<tr>
<td>7</td>
<td>50.0</td>
<td>0.0</td>
<td>45.0</td>
</tr>
<tr>
<td>8</td>
<td>50.0</td>
<td>0.0</td>
<td>42.5</td>
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</tbody>
</table>

**KEY**

- No.1
- No.2
- No.3
- No.4
- No.5
- No.6
- No.7
- No.8

**Percent Volume Change**

- 0.0
- 1.0
- 2.0
- 3.0
- 4.0
- 5.0
Figure 8.

Volume Change of Body Mixtures.
Figure 9.

Porosity of Body Mixtures.
<table>
<thead>
<tr>
<th>Body No.</th>
<th>Composition</th>
<th>Absorption</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fire Clay</td>
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<td>1.</td>
<td>50.0</td>
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<tr>
<td>2.</td>
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<tr>
<td>3.</td>
<td>50.0</td>
<td>25.0</td>
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<td>4.</td>
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</tr>
<tr>
<td>5.</td>
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<td>50.0</td>
</tr>
<tr>
<td>6.</td>
<td>50.0</td>
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<tr>
<td>7.</td>
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</tr>
<tr>
<td>8.</td>
<td>50.0</td>
<td>0.0</td>
</tr>
</tbody>
</table>

**Key**

No. 1, No. 2, No. 3, No. 4, No. 5, No. 6, No. 7, No. 8
Figure 10.
Absorption of Body Mixtures.

- 36 -
Figure 11.

Modulus of Rupture of Body Mixtures.

- 57 -
## SAG TEST

| Body No. | Composition | Deflection in Inches at 1300° | 1200° | 1100° | 1000° | 900° | 800° | 700° | 600° | 500° | 400° | 300° | 200° | 100° | 100° |
|----------|-------------|-------------------------------|-------|-------|-------|------|------|------|------|------|------|------|------|------|------|------|
|          | Fire Clay   |                               |       |       |       |      |      |      |      |      |      |      |      |      |      |      |
|          | Old Flint   |                               |       |       |       |      |      |      |      |      |      |      |      |      |      |      |
| 1        | 50.00       | 0.00                          | 0.00  | 0.00  | 0.00  | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 2        | 50.00       | 12.50                         | 37.50 | 0.00  | 0.00  | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 3        | 50.00       | 25.00                         | 0.00  | 0.00  | 0.00  | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 4        | 50.00       | 37.50                         | 0.00  | 0.00  | 0.00  | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 5        | 50.00       | 50.00                         | 0.00  | 0.00  | 0.00  | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 6        | 50.00       | 62.50                         | 0.00  | 0.00  | 0.00  | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 7        | 50.00       | 75.00                         | 0.00  | 0.00  | 0.00  | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 8        | 50.00       | 87.50                         | 0.00  | 0.00  | 0.00  | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |

**KEY**
- No. 1
- No. 2
- No. 3
- No. 4
- No. 5
- No. 6
- No. 7
- No. 8
Figure 12.
Sag of Body Mixtures.
Figure 15.

Spelling Test Data.
Figure 14.

Spalling of Body Mixtures.
DISCUSSION OF RESULTS.

It will be noted from the materials used that a good grade of plastic fireclay was used for the bond clay, and that fifty per cent of all body mixtures consisted of this fireclay. It will also be noted that the grog constituents varied between a good grade of calcined flint clay and old retorts, with three mixtures using the flint clay and small amounts of zinc oxide.

No. 1 body mixture, containing fifty per cent fireclay as bond and fifty per cent flint clay as grog, was adopted as a standard of comparison. Previous work in the investigation has shown this mixture to be of high quality.

The cone deformation values of the materials and also the body mixtures are shown in Figure 6. These values are quite low for the body mixtures containing old retorts. The old retort material has a very low value itself (Cone 17). It was thought that the fluxing action of zinc present contributed to the low deformation value of old retort material in general, but some of the mixtures, namely 6, 7 and 8, have more zinc present than the mixtures containing old retorts, and yet mixtures 6, 7 and 8 have much higher deformation values—in fact almost equaling the value of the standard No. 1 mixture.

However, the iron content of the mixes containing old retorts was relatively high. The fluxing action of iron is in general, marked, and in this case the iron content may affect the cone deformation values of the mixture more than does the zinc.
Figure 7 shows the drying shrinkage and water of plasticity of the eight mixtures. The water of plasticity varied from about 11.5 per cent to about 14.5 per cent with mixture No. 1 having the least. The drying shrinkage, however, varied from about 6 to 9 per cent, with the standard mixture having about an average. The three lowest are those containing zinc oxide, while the highest is the mix containing the largest amount of old retort material.

In the porosity curves, shown on Figure 3, the standard No. 1 again shows a very uniform curve and is among the lowest in porosity. It is somewhat grouped with Nos. 2, 3 and 4, while the other mixes are higher in porosity and less uniform.

Figure 9 shows the absorption values. They are very similar to the porosity values, of which they are a function.

In the volume change or firing shrinkage value (Figure 10), mixes Nos. 7 and 6 appear strikingly high and variable as compared to the others. Mixture No. 1 again shows a uniform curve and is practically an average of the other mixes.

Figure 11 shows the modulus of rupture of the eight mixtures. No. 1 is again noted as being quite uniform. It is closely followed by Nos. 2, 3, 4 and 5, while 6, 7 and 8 are comparatively low. The relatively high values of 2, 3, 4 and 5 in this test may be explained by the cementing action of the fluxes present, and the small particles of slag which these mixtures contain from their content of reclaimed retort material. In this test it should be remembered that the specimens are broken in the cold, after having been fired to the temperatures...
indicated, and that the test is not a measure of the strength of the bodies at elevated temperatures.

In the sag test, shown in Figure 12, it must be remembered that low sag values are desirable. If, as reasoned above for the modulus of rupture, fluxes increase the strength of the specimens in the cold state, similarly, fluxes may cause the test pieces to soften and bend when hot. This reasoning would explain the relatively high sag values of mixtures Nos. 2, 3, 4 and 5 at 1450° C. Test pieces for mixtures Nos. 7 and 8, containing five and seven and one-half per cent zinc oxide respectively, failed above 1300° C; similarly, specimens for body No. 5 failed above 1400° C. In these mixtures rupture by sagging or bending occurred at temperatures to which they would be subjected in practice; this indicates their unsuitability for use in zinc retorts.

In the spalling test, as shown in Figure 14, the dotted line, indicative of the number of dips that the specimen withstood before spalling action, is probably the better curve. However, in both curves No. 1 again stands out as decidedly the better. No. 2 also seems very good. Nos. 6, 7 and 5 appear next, decreasing with increase of zinc content. Here again the effect of adding zinc oxide appears detrimental. The remaining mixtures, Nos. 3, 4 and 5 were quite poor in regard to resistance to spalling action.
SUMMARY.

The object of this investigation has been the determination of properties of body mixtures using old retorts and zinc oxide for greg constituents, and comparing these properties with those of a mixture of purer clay material.

This study is only a part of a much larger investigation not as yet completed. To attempt to draw definite conclusions without more available material for comparison would be far-fetched. It must be realized that, even in this definite phase of the investigation, to state conclusions clearly as to the effects of old retorts and zinc oxide as greg would require a very detailed study. The study for such information must include not only extensive laboratory investigation of properties but comparisons using practical smelting conditions.

However, this investigation, previously outlined and described, brings forth several ideas which may be stated and explained. In short, the effect of the use of old retort material and of zinc oxide as greg upon the properties of bodies for the manufacture of retorts, may be set forth in outline form as follows:

1. In general, reclaimed retorts are unsuitable for any appreciable proportion of the greg material, for several reasons. They are usually of low deformation value and therefore decrease the refactoriness of the body. The flux and slag content—often relatively
high—although, perhaps, increasing the strength in the cold state, tends to cause softening in the hot state; on this account retorts made using reclaimed material as grog would not hold up at high temperatures.

2. The effect of the use of zinc oxide (as a grog constituent) indicates an inferior product, especially if used in quantities exceeding five per cent of the grog. The firing shrinkage is increased, the transverse strength low, and the sag high; a good product would indicate reversed conditions for those three properties.

3. In conclusion, it may be stated that when selecting clay materials for retort mixtures all desirable qualities are rarely present in any one clay. In general the properties of mixture No. 1, using only good clay materials, are superior to those of the other bodies, and the refractoriness of the mixture has been increased above that of the bond clay by the use of calcined flint clay as grog. For the bond clay some decrease of refractoriness may have to be accepted to secure binding power, plasticity, and moderate shrinkage. For grog, on the other hand, burned material is always used, and, if originally made from high quality clays, may benefit the properties of the re-
sultant mixture. While initial costs may favor the use of old retorts for a part, at least, of the grog, yet this material adds little and often detracts from the desired properties, and in service may prove less economical.

ACKNOWLEDGMENTS.

The writer is indebted to Messrs. E. S. Wheeler and A. H. Kuechler, Assistant Metallurgist and Junior Ceramic Engineer, respectively, of the State Mining Experiment Station, for their assistance in conducting the investigation; to Mr. H. M. Lawrence, Acting Superintendent of the Mississippi Valley Experiment Station of the U. S. Bureau of Mines, who supervised the work; to Mr. O. W. Holmes, Cooperative Chemist of the State Mining Experiment Station, for his accurate analytical work; to Mr. C. R. Forbes, Professor of Mining of the Missouri School of Mines, who furnished the flint clay used in the investigation.
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Books.


Periodicals.


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