

SOME CLAYS OF IDAHO

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A THESIS

PRESENTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE

DEGREE OF BACHELOR OF SCIENCE IN CHEMICAL ENGINEERING

IN THE

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The purpose of this investigation is to begin the work of classifying the clays from the various parts of the state of Idaho. As there has been no Geological Survey work done on the nature, extent, and geology of the clay beds of the state, the undertaking is difficult and the available information is necessarily fragmentary and incomplete. Since we were not able to travel to the various clay-beds of the state, take the geological and other necessary data, and collect our own samples, but had to rely upon the scanty and unscientific information furnished by the owners of the various beds, we have not made any attempt to offer any geological information as to the clays, but have merely attempted, by means of chemical and physical analyses of the few representative samples which we could secure, to so correlate chemical analyses and physical properties that the uses and approximate values of the different clays could be determined, even in untried and undeveloped deposits, by means of their chemico-physical analyses.

The clay industry of the state of Idaho is in its infancy, and indeed, the interest in the subject seems to very slight, as a general thing. As an indication of the amount of interest shown, even by the owners of clay deposits, in this kind of investigation, we may as well state that during three semesters of practically continuous correspondence we were able to secure only fifteen samples of clays. As a rule, large clay-manufacturing companies, owning and operating extensive plants, treated our requests with the greatest courtesy and offered us every aid in their power. In this connection, we

wish to especially express our indebtedness to John T. Huetter and Sons, of Huetter, Idaho, and Spokane, Wash., who made it possible for us to investigate their clay-beds in person, collect samples, and take geological notes; and who extended to us every courtesy:- to The Idaho Lime Company, of Albany Falls, Idaho, and Spokane, Wash., and to The Washington Brick, Lime, & Sewer Pipe Company, of Freeman, Wash. and Spokane, Wash., for their kindness in furnishing us with samples and information. Also, several individuals who own undeveloped clay beds kindly sent us samples, but the available information as to the extent and geology of such beds is both meager and unscientific.

The Coeur d'Alene Commercial Club sent us a sample of a very fine kaolin clay recently discovered near that city, but in general, commercial clubs and "booster" clubs, while they usually answered our letters and gave glowing accounts of the prosperity and attractiveness of their respective cities, were very unproductive of actual results.

Besides these very real difficulties, we were also hampered by lack of suitable apparatus with which to do our work, and much time was consumed in the construction of such apparatus. Thus, the construction of the small electric muffle furnace used in the ignition of our precipitates and in the burning tests of the clays; the installation of the electric power-circuit for our electrical apparatus; and other work of the same nature; while they furnished us with a great deal of very valuable experience, necessarily greatly shortened the time which we could devote to the actual investigations.

However, we hope that we have made a beginning, have, in a measure, laid a foundation upon which subsequent investigators may build a complete and accurate classification of the clays of Idaho.

In the following pages we shall first give an outline of the methods used in the chemical and physical analyses of all the clays (except as shall be noted in the descriptions of the individual clays); then we shall take up each clay separately, giving its physical properties and its chemical analysis in detail, as well as a description of the bed from which it was taken, whenever possible. And from the above information, we shall attempt to show to what other uses the clay may be put, than those to which it is being put at present.

We shall also give a brief discussion of the most common semi-refractory clays, such as the paving-brick, the sewer-pipe, the roofing-tile, and the stoneware clays; and also of the non-refractory ones, such as the flower-pot, the ornamental brick, the terra-cotta, and the common brick and tile clays; and in the appendix, we shall give a brief description of the electric furnace which we constructed for our burning tests and ignitions.

METHODS OF CHEMICAL ANALYSIS.

SAMPLING. The clay, which has been previously air-dried in the warm basement of the Engineering Building, is broken up on the clean-swept concrete floor into pieces about the size of a walnut. The sample is then thoroughly mixed and quartered, and the quarter is broken up still finer and again quartered. This operation is continued until the sample is reduced to about one kilogram, when it is spread out in a thin layer upon a rubber sheet and a 50-gram sample is taken from it by means of a small spatula, by taking portions from the various parts of the layer. This sample is then ground to a fineness of 100-mesh in a Braun Sample-Grinder and put into a tightly-stoppered bottle. The remainder of the kilogram is put away in a bottle of suitable size, for possible future reference.

MOISTURE. Loss at 110 deg. C. (Am. Jour. Sci., 3rd. Ser., Vol. XLVIII, 1894, p. 31.) One gram of the sample is weighed into a weighed platinum crucible and heated in an air-oven for one and one-half hours at a temperature of 110 deg.C. After cooling, it is again weighed and the loss reported as moisture.

COMBINED WATER AND VOLATILE MATTER. Loss on ignition. The sample from the moisture determination is covered and placed in the muffle furnace, the muffle being below red heat. The current is then turned on and the clay ignited to constant weight. The loss in weight consists of combined water, organic matter, carbon dioxide, etc.

ALKALIES. This same portion of clay, which has been used for determining moisture and loss, is treated with concentrated sulphuric and hydrofluoric acids until it is completely decomposed. The acids are evaporated off by heating upon the sand-bath. The cooled crucible ~~crucible~~ is washed out with boiling water to which several drops of hydrochloric acid have been added. The solution, after being made up to about five hundred cubic centimeters, is boiled, one-half gram of ammonium oxalate is added, and the solution is then made alkaline with ammonium hydroxide; the boiling is then continued until only a faint odor of ammonia remains. The precipitate is allowed to settle and is separated from the liquid by filtering, and is washed three times with boiling water. The filtrate is evaporated to dryness and ignited to drive off ammonium salts. The residue is treated with five cubic centimeters of boiling water, two or three cubic centimeters of saturated ammonium carbonate solution are added, and the whole is filtered into a weighed crucible or dish. The precipitate is washed three or four times with boiling water and the filtrate evaporated to dryness. Five drops of sulphuric acid are added to the residue, and then the crucible or dish is brought to a red heat, cooled in a desiccator, and the alkalies weighed as sulphates.

This method is the method of Ries, given in his book, "CLAYS? THEIR OCCURRENCE, PROPERTIES, AND USES", p.64.

SILICA. Two grams of clay are mixed with ten grams of sodium carbonate and one-half gram of potassium nitrate and brought to a calm fusion in a large platinum crucible.

This is accomplished by putting the crucible with its charge into the cold muffle, turning on the current, and allowing the furnace to come up to its full heat. This takes about forty five minutes, at the end of which time the contents of the crucible are in a state of quiet fusion and the clay is completely decomposed. The melt is softened by immersing the cooled crucible in cold water contained in a Jena glass evaporating dish. An excess of concentrated hydrochloric acid is added to the water in the evaporating dish, and the crucible and cover are thoroughly rinsed with dilute hydrochloric acid. After the melt is entirely decomposed, which may be hastened by breaking up the lumps with a blunt glass rod, the solution is evaporated to dryness upon a water-bath and the residue is dehydrated in an air-oven at 120 deg. C. for one hour. The evaporating dish is then cooled and the contents thoroughly moistened with concentrated hydrochloric acid. Hot water is now added, and the dish is heated until all soluble substances have gone into solution. The solution is now filtered, the silica washed with hot water until free from chlorides. The filtrate and washings are evaporated to dryness and the residue is again dehydrated for one hour at 120 deg. C. The residue is brought into solution as before, and the slight amount of silica secured by this second treatment is filtered off upon a second filter and thoroughly washed with hot water. Both filters, with their contained silica precipitates, are now carefully transferred to a weighed platinum crucible and ignited to constant weight in

the muffle furnace. To secure the percentage of silica in the clay, the weight of silica thus obtained is divided by two.

IRON SESQUIOXIDE. The filtrate from the silica is divided into two equal portions. To one portion is added 10 cc of 1:1 sulphuric acid and the solution is run through a reducer, slowly enough so that the iron is completely reduced. The iron is then determined by titration with a standard solution of potassium permanganate.

ALUMINIUM OXIDE. To the second portion, which must be brought to boiling, ammonium hydroxide is added in slight excess, the boiling is continued from two to five minutes, the precipitate is allowed to settle and caught upon a filter, and all the chlorides are washed out with hot water. The precipitate is ignited to constant weight in the muffle furnace and weighed as a mixture of aluminium oxide and iron sesquioxide. The amount of iron sesquioxide already determined by the permanganate titration is subtracted from this and the remainder reported as aluminium oxide.

TITANIUM OXIDE. The solution which has been titrated for iron is poured into a 300 cc measuring flask and made up to the mark with pure water. 50 cc of the solution is run into a Nessler tube, 5 cc of concentrated sulphuric acid and 3 cc of hydrogen peroxide solution are added, and the whole mixed by inverting two or three times. Another Nessler tube is prepared by filling it up to somewhat below the level of the first with water, 5 cc of

concentrated sulphuric acid, and 3 cc of hydrogen peroxide solution, as before. Into this Nessler tube the standard solution of titanium (containing ~~XXXX~~ one centigram of titanium oxide as sulphate in 10 cc) is run from a small burette or a graduated pipette, drop by drop with constant mixing, until the depth of coloration in the two tubes is the same. The percentage of titanium oxide is readily calculated from the amount of standard solution required to cause the equality of coloration.

CALCIUM OXIDE. The filtrate from the precipitate of iron and aluminium hydroxides is concentrated to about 200 cc, and the calcium is precipitated from the hot solution by adding one gram of solid ammonium oxalate. The precipitate is allowed to settle for twelve hours, filtered, washed with hot water, ignited, and weighed as calcium oxide.

MAGNESIUM OXIDE. The filtrate from the calcium oxalate precipitate is concentrated to about 100 cc, cooled, and the magnesium precipitated by means of hydrogen disodium phosphate solution in a strongly alkaline solution. The magnesium ammonium phosphate, after standing overnight, is caught upon a filter, washed with water containing at least five per cent of ammonium hydroxide, ignited to constant weight and weighed as magnesium pyrophosphate.

MANGANESE OXIDE was determined in only one of these clays, as only one gave a deep enough coloration in the fusion to indicate the presence of enough manganese to make its determination worth while. In this connection, it may be

said that very few clays contain manganese in more than traces. Out of ninety five analyses of Washington clays listed in Shedd's "Clays and Clay Industries of Washington", (pp 318-323), eighty nine are free from manganese, and in only two does the content of manganese oxide exceed one per cent. Furthermore, as to the determination of manganese oxide in igneous rocks, (and the same statement applies to clays, since clays are derived from igneous rocks), H.S. Washington, in the "U.S.G.S. Professional Paper No. 14", pp 27-28, says:-

"The question as to whether manganese oxide should be determined or not is a rather perplexing one. Of course, for the best work, and if the analyst is sufficiently experienced not to fall into the error possible in its determination by the sodium acetate method, it should be done. But on the other hand, its determination adds very materially to the time necessary for the analysis, and involves as well a method very liable to a grave error which will affect seriously a much more important constituent, aluminium oxide. Furthermore, the long list of analyses made by the chemists of the United States Geological Survey, as well as those made elsewhere, show that, while nearly always present, its amount is very small, in general little more than a trace. Thus, in all the analyses published by the United States Geological Survey it only exceeds five-tenths of one per cent twice, and falls between four-tenths and five-tenths of one per cent five times. Indeed, it is of interest to note in this connection that ----- the amount of manganese in them is only about one-sixth that of titanium, and is less even

than that of phosphorus. In view of the great variety represented by these analyses and of the very high character of the analytical work, the high figures so often found for this oxide are to be regarded with suspicion, the probability being that in them the error already spoken of has been made.

"That the non-determination of manganese oxide will affect the figures of other constituents is certain, but to what extent is not very clear. If the separation of alumina, etc., has been made with ammonium hydroxide, a small portion will be thrown down and weighed as aluminium oxide. Part of that which passes through in the filtrate will be thrown down with the calcium oxalate and weighed as calcium oxide. But as manganese oxalate is somewhat soluble in water, some of the manganese will be thrown down as phosphate with the magnesia and weighed as magnesium pyrophosphate. Little is known of the various proportions of the manganese which will be thus distributed, and the matter is one which calls for further investigation.

"At any rate, in view of the small amount of this substance present, its probable distribution among several constituents, thereby affecting each of them only to a negligible extent, and the loss of time and the possibility of error affecting the determination of aluminium oxide, involved in its determination, it may well be held that the determination of manganese oxide is not essential or called for, even in what is otherwise very complete and accurate work".

A few experiments were made using the

Bismuthate-Arsenite method of determining manganese described in Lord and Demorest's "Metallurgical Analysis" (1913) p.p. 82-84, as applied to iron and steel analysis, to test its simplicity and accuracy as a method for estimating manganese in silicate rocks and clays. The investigation was not extensive enough to thoroughly test the method and to ascertain whether the difficulties that were encountered were amenable to solution. The results that were obtained were not of a sufficiently high degree of accuracy to recommend the method for general laboratory use.

To prepare the standard arsenite solution, sodium arsenite, J.T. Baker's c.p., formula assumed to be NaAs_2O_5 (Abegg- Auerbach, Vol. 5, p. 524), was dissolved in distilled water in the proportion of 2.95 grams to the liter. A more dilute solution, e.g., one gram per liter, would have been more suitable.

The method of standardization was as follows:-A sufficient volume of the KMnO_4 solution used in the estimation of iron was measured into a flask from a burette to give 0.005 grams of Mn. The solution was diluted to about 50 cc., and 10 cc. of concentrated HNO_3 were added. The solution was then shaken with 0.2 gram of sodium bismuthate, J.T. Baker's, free from Mn and Cl, and boiled. If the permanganate color had disappeared, more bismuthate was added. A small crystal of KNO_2 was now added to dissolve any MnO_2 that may have formed, and boiling was continued until all nitrous fumes were expelled. The solution was then cooled with tap water. When cold, the solution was shaken thoroughly with 0.3-0.4 g. of bismuthate. 30 cc. of water were then added, and the solution again shaken. After settling, the excess of bismuthate was filtered thru a filter prepared by mixing thoroughly

together equal quantities of glass wool and asbestos fibre by shaking with water in a flask. A sufficient amount of this mixture was poured into a funnel to fill it about one-third full, and was washed free from HCl. The material was found to filter well without clogging readily, and could be used for a number of filtrations without changing. The method of filtering thru a gooch crucible was found to be unsatisfactory, as two or three filtrations effectually clogged the filter. The filter was washed with water, and the filtrate and washings titrated with the ~~the~~ standard solution. The MnO equivalent of the arsenite solution per cc. was then calculated.

The end-point of these titrations was not clear and definite; the solution changing from pink to golden-brown and then to greenish yellow. It was also found extremely difficult to obtain concordant results. It was evident that the conditions of the titration should be very carefully regulated. The effects of temperature, dilution, amount of free acid, the time period elapsing between final filtration and titration, and the rate of titration should be studied to determine their relative influence and the most favorable conditions.

The same process was followed in making the actual determinations. For accurate results, it is necessary to make a manganese determination in the iron-alumina precipitate, the calcium precipitate, and the magnesium pyrophosphate precipitate, as some magnesium is thrown down whenever an excess of ammonia is used in the precipitation. The tests seem to show however, that the bulk of the manganese tends to precipitate with the magnesium. In testing the iron-alumina precipitate for Mn, the solution of the fusion with KHSO₄ was divided into two aliquot parts, one of which was used for iron and titanium deter-

-minations and the other for manganese determination. The end-point in the mineral titrations was clear and distinct; the solution changing from pink to colorless.

The sodium arsenite solution proved to be fairly stable, but should be checked up every few days. The method seems to show possibilities for very good work, and if the exact conditions for titration were ascertained would evidently be capable of giving very reliable results. The labor required to test the three precipitates would, in our experience, be less ~~than~~ than that involved in a basic-acetate precipitation.

ALKALIES by the J. Lawrence Smith method. (See p. 5) One gram of the finely ground clay is intimately mixed in a mortar with an equal weight of sublimed NH_4Cl , then all but a small portion of eight grams of pure CaCO_3 is added and the whole thoroughly mixed together. The contents of the mortar are transferred as completely as possible to a long platinum crucible of the J. Lawrence Smith type. The remainder of the carbonate is used to rinse the mortar and pestle, and as a cover for the contents of the crucible. The crucible is capped and placed in an inclined position thru a hole in a piece of stout asbestos board clamped in a vertical position. Heat is applied to the bottom of the crucible from a low flame ~~until~~ ^{is} the odor of ammonia ^{no} longer perceptible, taking care that no ammonium chloride is driven off instead of being broken up to form CaCl_2 and ammonia. The full heat of two Bunsen burners is then applied from 40 to 50 minutes. The cake usually detaches readily from the crucible but if not it may easily be softened up with hot water. The cake is transferred to a casserole and digested with water (using only ^{until it is completely disintegrated.} 2 or 3 cc. at first) Any lumps are broken up and the residue filtered and washed well with hot water.

To the hot filtrate are added excess of NH_4OH and $(\text{NH}_4)_2\text{CO}_3$. The CaCO_3 is filtered off and washed with water containing a very little NH_4OH .

The filtrate is concentrated to a small volume in a casserole and transferred to a small porcelain evaporating dish, then evaporated to dryness. It is then ignited carefully at a low red heat until all NH_4Cl is expelled and no more fumes form. After the dish has been allowed to cool, there are added one cc. of 10 per cent $(\text{NH}_4)_2\text{CO}_3$, and a little water, a drop of BaCl_2 , one cc. of $(\text{NH}_4)_2\text{C}_2\text{O}_4$. The solution is heated and filtered from any precipitate into a weighed porcelain dish. Two or three drops of HCl are added to the filtrate. It is evaporated to dryness, ignited as before, and weighed as the combined chlorides, NaCl and KCl . The chlorides must be white, and if there is any insoluble residue in water solution, it must be weighed.

The amount of PtCl_4 (Kahlbaum's 10% solution) required to convert the two chlorides into chloroplatinate is calculated (Treadwell-Hall, "Analytical Chemistry", (1911) Vol. 2.

Quantitative Analysis, p. 44) and a few tenths of a cubic centimeter in excess of the calculated amount are added to the water solution of the chlorides. It is then carefully evaporated until only a drop of liquid is left. Twenty cc. of alcohol (80%) are added, and allowed to stand until the platinum salts dissolve. It is then filtered on to a weighed gooch filter, and the K_2PtCl_6 washed with 80% alcohol. The gooch filter is then dried at 110°C . and weighed. The K_2O from the weight of the chloroplatinate thus obtained, and the Na_2O is then calculated from the remainder obtained after deducting the weight of the KCl from that of the combined chlorides.

This is the method described by W.F. Hillebrand in "Analysis of Silicate and Carbonate Rocks", U.S.G.S. Bulletin 422, p. 172.

PHYSICAL TESTS.

One kilogram of the air-dried sample was weighed out on the laboratory balance and ground in a Braun sample-grinder to a fineness of about 25-50 mesh. Water was then added from a 500-cc graduate, with thorough working and kneading of the mass, until the clay had attained its maximum plasticity, when it had come to the best possible condition for molding. The reading of the graduate was called grams of water, and this, divided by ten, gave directly the percentage of water necessary to work the clay.

In molding the clay into briquettes, brass gang molds were used, holding three molds each. The molds were shaped according to the specifications laid down by the American Society of Civil Engineers, as described on page 188 of Waterbury's "Laboratory Manual for Testing Materials of Construction".

In breaking the briquettes, a Fairbanks machine was used, the property of the Civil Engineering department of the University of Idaho. From eight to twelve briquettes of each sample were broken, and the tensile strength of the clay was figured from the average tensile strength of all the perfect briquettes broken. Results of briquettes which showed faults after fracture, such as cracks at or near the mid-section, were discarded.

As the briquettes shrank considerably in drying, the average cross-section of all the briquettes of each

sample, measured by means of a vernier caliper, was divided into the average tensile strength of them, the quotient giving the tensile strength of the clay in pounds per square inch.

The air shrinkage of each clay was computed from the linear measurement of the briquettes just before breaking. Thus, if the average length of all the briquettes of a sample was 2.8 inches, (the original length of all was three inches) the percentage of air shrinkage would be 0.20×100 divided by 3, or 6.67%.

Plasticity was determined only by noting the way the clay worked up, as there seems to be no standard method of determining and expressing precisely, the plasticity of clays.

Burning test. The color of the sample after ignition was noted, as this is the color which the burned product will have, provided the ware is not vitrified.

Fusion tests were not carried out, on account of lack of a suitable furnace. Our small muffle furnace, as well as the large muffle furnaces of the Assay department, proved inadequate for these tests.

BURNING TEST.

Fifty grams of each sample were made into a plastic paste, and four ~~sides~~^{disks} one inch in diameter and one-fourth inch thick were molded in an improvised wooden mold. These were ~~dried~~^{dried} in the air for twelve hours, then at 110 C. for two hours. The diameter of three disks of each set was then measured by means of a vernier caliper; the fourth disk being preserved ~~as~~^{as} an unbaked sample. The disks were then introduced into the electric furnace at a low heat, and then submitted to the full heat of the furnace for about two hours. At the end of this time, the disks were again measured with the verniers along the same diameters. In this way an idea of the relative fire shrinkage was obtained, altho maximum shrinkage does not take place until the point of vitrification is reached, which was not attained at the temperature of the furnace. The fire shrinkage thus obtained expressed in per cent was as follows:-

CLAY NO.	I	2	3	4	5	6	7	8	9	10	11
Fire Shrinkage											
Per Cent	2.52	5.46	7.25	2.26	0.82	2.48	2.6	1.75	.96	nil	1.83

Summary of the Origin, Composition, and Uses of Clays.

"Clay is the term applied to those earthy materials occurring in nature whose most prominent property is that of plasticity when wet. Physically, clay is made up of a number of small particles, mostly of mineral character ranging from grains of coarse sand to those that are of microscopic size. Mineralogically, it consists (1) of many different mineral fragments in all stages of decay, and representing chemically many different compounds; (2) of colloidal material which might be either organic or mineral".

Clays are always of secondary origin, the product of the decomposition of other rocks. A residual clay is one which remains in the same location as the parent rock, and may occur as a mantle over a considerable area, but is more usually found in veins.

I. This summary is based on H. Ries' "Clays, their Occurrence, Properties, and Uses" (1906).

Residual Clays rarely remain on the steep slopes where they are formed, but are carried away by rain or wind, forming the more common beds of sedimentary clay.

Sedimentary clay is to be distinguished in that it usually occurs in stratified layers, and usually bears no direct relation to the underlying rock on which ^{it} ~~they~~ may rest. In the process of transportation the finer and the coarser particles are like to become separated as the fine particles can only be deposited in still water. Also it may become mixed with impurities, and layers of sand are often found in sedimentary clay deposits.

Oftentimes the sedimentary layers ^{be come} ~~are~~ covered with enormous amounts of other deposited material, and the great

pressure to which they are subjected causes them to harden ~~it~~ into a firm rock-like mass called "shale". Shale has the same composition as the clay from it is formed, and may be used for many of the same purposes as clay.

Clays have a very complex mineralogical composition and therefore no definite chemical formula. Kaolinite has been supposed to be very widely distributed in clays, but little evidence seems to be ^{available} ~~reliable~~ in proof of this supposition. It is a hydrated silicate of alumina, represented by the formula $Al_2O_3, 2SiO_2, 2H_2O$ corresponding to the composition:—

Silica 46.3%

Alumina 39.8%

Water 13.9%

Other minerals closely related to kaolinite are halloysite, indianite and pholerite.

Quartz, mica, the iron ores including limonite, hematite, magnetite, siderite, and pyrite, calcite, gypsum, rutile, ^{zircon} ~~line~~ ~~ite~~, glauconite, dolomite, magnesite, manganese oxides, as well as many other minerals are of common occurrence in clays.

An ultimate analysis does not afford a knowledge of the mineralogical constituents of the clay, but indicates many important facts. It gives an idea of the purity of the clay, ~~silica, alumina, and water~~. The refractoriness of the clay may be judged to some extent from the amount of the fluxing impurities, ferric-oxide, lime, magnesia and alkalies. The quantity of water is of economic importance, as well as excess of silica which in large amounts indicates a sandy clay. Iron has a great influence on the fluxing properties and the color of the clay.

The importance of clay in the industrial world may be judged from the many applications to which it may be put as shown by the following table. (Ries, p. 217)

DOMESTIC--Porcelain, white ware, stone-ware, yellow ware, and Rockingham ware for table service and for cooking; majolica stives; polishing brick, fire brick and fire kindlers.

STRUCTURAL--Brick; common, front, pressed, ornamental, hollow, glazed, adobe; terra cotta; roofing-tile; glazed and encaustic tile; drain-tile; paving-brick; chimney-flues; chimney pots; door-knobs; fire-proofing; terra-cotta lumber; fence-posts

REFRACTORIES--Crucibles and other assaying apparatus; gas retorts; fire-bricks; glass-pots and blocks for tank furnaces; saggars; stove and furnace bricks; blocks for fire boxes; cupola bricks; mold linings for steel castings.

ENGINEERING--Puddle, Portland Cement, railroad ballast, water conduits, turbine-wheels; electrical conduits, road metal.

HYGIENIC--Urinals, closet bowls, sinks, wash-tubs, bath-tubs, pitchers, sewer-pipe, ventilating-flues, foundation-blocks, vitrified bricks.

Minor USES--Food adulterant; paint fillers; paper filling; electric insulators; pumps; fulling cloth; scouring soap; packing for horses' feet; chemical apparatus; ink-bottles; ultramarine manufacture; emery-wheels; marbles; battery-cups; pins; stilts, and spurs for potters' use; shuttle eyes and ^{thread} ~~thread~~ guides; smoking-pipes; umbrella stands; pedestals; filter-tubes; castor-wheels; pump-wheels; electrical porcelain; foot-rules; plaster; alum.

A BRIEF DISCUSSION OF CLAYS FROM ECONOMIC STANDPOINT.

The following statistics concerning the clay-working industries are taken from U.S.G.S. Bulletin "Mineral Resources of the United States, Part 2-Non-Metals, 1912," (1913) pp. 525-620. The total value of all clay products marketed in 1912 was \$172,811,275 compared with ~~\$36,504,164~~^{\$162,236,181} in 1911. Of ~~this~~^{to} former sum, the brick and tile products amounted ~~\$36,307~~^{valued at \$36,504,164 or} 21.12% of the total; and the clay products were 78.88% of the total. The brick and tile division showed the greatest increase.

The clay-working industries are very little developed in Idaho as no pottery clays are being worked and only a few of the more available deposits are being made into brick and tile. The total value of the industry in Idaho and Nevada combined as given as \$176,108 in 1912, showing a decrease from \$198,479 in 1911.

The total amount of clay mined and sold is given as 2,530,265 tons with a value of \$3946,020. The clay as mined had therefore an average value of \$1.56 per ton which represents largely the cost of mining. The average value per short ton of various kinds of clay as mined is Kaolin, \$8.53; paper clay, \$4.37; slip clay, \$1.69; ball clay, \$3.50; fire-clay, ~~\$0.89~~^{1.40}; miscellaneous clay, \$1.03. It will be seen then that a clay ~~or~~^{stone}ware clay, \$0.93; brick clay, \$0.89; ~~bed~~^{has} has very little economic value in itself, but it is of value

to its owner only so far as he is able to produce a valuable finished product at a reasonable cost of production

It is ~~not~~^{not} probable that there will be a great development of clay-working industries in this state for several years to come. At the present time clay products are limited almost exclusively to common brick, pressed brick, and tile to satisfy the local market for structural purposes.

Even if valuable kaolin, ball clay or the higher grades of clay should be discovered in extensive and commercial deposits the problems of competing with established wares, if fuel, transportation, and operating costs would serve to retard and check clay-working on a large scale.

The continuation of such investigations as the present one should be of value in indicating those clays most suitable for commercial development, and preventing useless expenditure on clays of no value.

DESCRIPTION OF SAMPLES.

Samples Nos. 1, 2, 3, and 4 were secured from the brickyard of John T. Huetter and Sons, at Huetter, Idaho, a small town about three miles from Coeur d'Alene, Idaho. Mr. Huetter and his son Theodore accompanied one of us, E.E. Smith, out to the brickyard from Spokane and aided him in securing the samples.

The clay at Huetter is in three strata, the upper one beginning immediately below the surface and the lower one merging into coarse sand at an average depth of seven to eight feet. Geologically, it is evidently of sedimentary origin, having been deposited in the bed of Lake Coeur d'Alene at a time when it was vastly larger than it is at the present time.

Huetter and Sons had been making brick from the whole of the deposit, mixing the clays from the three strata, and the product had been of an inferior quality. Knowing that Mr. Smith was collecting clay samples, they employed him to investigate their clay deposit, in the hope of discovering the cause of the inferiority and finding a remedy for it.

Mr. Smith secured samples from each of the three strata, numbering them 1, 2, and 3 from the upper layer down, and also secured sample No. 4 from another bed, of uniform composition, belonging to the same company but lying on a high table-land across the Spokane River from Huetter.

Geologically, this clay differs from the other bed in that it is a residual clay, formed from the decomposition of gneiss and schistose rocks in the place where it is found.

Sample No. 1 is from the first stratum of the bed. It is from two to three feet in thickness, beginning immediately beneath the grass roots, and is very dark brown in color, evidently containing considerable organic matter. In structure it is rather coarse-grained, and as a result, it is not very plastic. It slakes easily and quickly, and requires 41% of water to bring it to its most plastic condition. Owing to the large amount of water necessary in the working of this clay, it cannot be dried rapidly, but cracks unless the drying is slow.

After drying, the briquettes measured two and five eighths inches in length and seven eighths of an inch square at the breaking section. Thus, the air shrinkage of this clay is 12.5%.

Eight perfect briquettes were broken, the breaking loads being the following:- 85, 80, 80, 75, 115, 90, 85, and 80 pounds; average, 86 pounds, or an average tensile strength of 112 pounds per square inch.

The chemical analysis of this clay is as follows:-

Moisture and Ignition Loss	18.22%
Silica (SiO_2)	44.46
Alumina (Al_2O_3)	26.97
Iron (Fe_2O_3)	8.03
Calcium (CaO)	2.02

Magnesia	(MgO)	.69
Alkalies	(Na ₂ O-K ₂ O)	.98
	TOTAL	101.37
	TOTAL FLUXES	11.72

Sample No. 2 is from the second stratum of the bed. It forms a sharply-defined white seam, varying from one to two feet in thickness. This clay is very light and porous, and has very little body. It slakes quickly, and requires 40% of water to bring it to its maximum plasticity. It is a very fine-grained clay, but is almost entirely lacking in plasticity, being of a chalky, powdery nature.

Its air shrinkage, as figured from linear measurements before breaking the briquettes, is 3.3%.

Six perfect briquettes of this sample were broken, the breaking loads being as follows:- 15, 11, 13, 15, 13, and 11 pounds; average, 13 pounds, corresponding to a tensile strength of 15 pounds per square inch.

The chemical analysis of this clay is as follows:-

Moisture	(110deg C)	6.21
Loss on Ignition		5.30
Silica	(SiO ₂)	52.02
Alumina	(Al ₂ O ₃)	18.95
Iron	(Fe ₂ O ₃)	4.07
Calcium	(CaO)	3.20
Magnesia	(MgO)	.48

Alkalies ($\text{Na}_2\text{O}-\text{K}_2\text{O}$)-	9.60
TOTAL	99.83
TOTAL FLUXES	17.35

It was this seam of white clay that caused the inferiority of the product. Its extremely low tensile strength made the brick weak, and its extraordinarily high proportion of total fluxes (17.35%) made the brick fuse together in the kiln, at temperatures below that which was necessary to give good color and strength to the finished product. Huetter and Sons had thought that it was this white clay which was spoiling their brick, but had hesitated in going to the expense of stripping it off until they were certain. After these tests and analyses, however, there was no doubt that it was the white clay which was doing the damage, and in the next run, they threw as much of the white clay as possible back into the pit. The brick from this run were of high quality, well above the standard, ~~and~~ did not fuse together in the kiln, and had a good red color.

Sample No. 3 is from the third strata of the bed, the main layer of the bed. It varies from three to four feet in thickness, and is light brown in color. It slakes easily and quickly, and requires 29% of water to bring it to its most plastic condition.

In structure it is rather fine-grained, but contains considerable sand, especially in the lower portion. As its plasticity is fair, and its tensile strength is very good,

however, the sand does no harm, but on the other hand, is a distinct advantage, in brick making, in that it enables the clay to stand rapid drying without cracking or warping.

This clay has an air shrinkage of 18.4%, and burns to a good red color. Eight perfect briquettes of this clay were broken, the breaking loads being as follows:- 100, 105, 145, 120, 115, 97, 110, and 95 pounds, corresponding to an average tensile strength of 145 pounds per square inch.

The chemical analysis of this clay is as follows:-

Moisture		6.48%
Ignition Loss		5.98
Silica	(SiO ₂)	54.84
Alumina	(Al ₂ O ₃)	20.15
Iron	(Fe ₂ O ₃)	7.41
Calcium	(CaO)	1.54
Magnesia	(MgO)	1.13
Alkalies	(Na ₂ O-K ₂ O)	.47 (By difference)

TOTAL FLUXES 10.55%.

Sample No. 4 was secured, as has already been stated, from a deposit lying on the South side of the Spokane River, opposite Huetter, Idaho. The geology of this bed has already been mentioned, but its extent and depth are unknown, as the deposit has been opened up in only one place.

This is a very good clay, yellow in color, of a dense, hard body, eminently plastic, and, with the exception

of some pebbles and small fragments of undecomposed rock scattered through it, is very fine grained. This clay slakes slowly, is hard to work, and requires 35% of water to bring it to its most plastic condition.

Owing to the plastic nature of this clay, it will not stand rapid drying, but if dried slowly, very good results may be obtained. The air shrinkage of this clay is 6.7%, and it burns to a good red color.

Seven perfect briquettes of this clay were broken, the breaking loads being as follows:- 90, 100, 85, 85, 110, 95, and 100 pounds, the average being 95 pounds, corresponding to a tensile strength of 108 pounds per square inch.

The chemical analysis of this clay is as follows:-

Moisture,		3.20
Ignition Loss		9.56
Silica	(SiO ₂)	53.03
Alumina	(Al ₂ O ₃)	24.82
Iron	(Fe ₂ O ₃)	5.58
Calcium	(CaO)	None
Magnesia	(MgO)	.53
Alkalies	(Na ₂ O-K ₂ O)	3.28 (By difference)

TOTAL FLUXES 9.93%.

From the analysis and physical tests, it is apparent that this clay could be used for many other purposes than common brick making. It should make good paving brick, sewer pipe, and roofing tile, and, if the pebbles could be

removed economically, it should make good stoneware, pottery, and other kindred products.

Sample No. 5 was secured from the Idaho Lime Co., of Spokane, Wash. and Albany Falls, Idaho. As the company sent us this sample, we could learn nothing of the geology, extent, or occurrence of this clay, beyond the fact that it was deposited from the bed of the Pend d'Oreille River. This clay is very uniform in composition throughout the entire deposit, and is a white, eminently plastic clay, extremely fine grained and free from sand or other impurity,

It slakes rather slowly, but is easily worked, though it requires 35.5% of water to bring it to its most plastic condition. The air shrinkage of this clay is 8.33%, and it burns to a good red color.

Eight perfect briquettes of this sample were broken, the breaking loads being as follows:- 70, 75, 80, 85, 80, 70, 70, and 85 pounds, the average being 77 pounds, corresponding to a tensile strength of 98 pounds per square inch.

At present, this clay is being used only in the manufacture of brick, but since this is a large deposit of high grade clay, it is certain that it will be used for many other purposes, such as stoneware and pottery, for which it is eminently suitable, as soon as the company becomes able to enlarge its scope of operations.

The chemical analysis of this clay is as follows:-

Moisture		1.34 %
Loss on Ignition		10.10
Silica	(SiO ₂)	59.21
Alumina	(Al ₂ O ₃)	17.05
Iron	(Fe ₂ O ₃)	6.05
Calcium	(CaO)	2.66
Magnesia	(MgO)	1.32
Alkalies	(Na ₂ O-K ₂ O)	3.01
		<hr/>
TOTAL		100.74 %
TOTAL FLUXES		13.04 %

Sample No. 6 was sent us by the Coeur d'Alene Commercial Club. It is a very high grade white kaolin clay, very fine grained and eminently plastic, with a dense, hard body. This clay slakes very slowly and is hard to work, requiring 46% of water to bring it to its most plastic condition. Owing to its very plastic nature, and the excessive amount of water necessary in its working, this clay must be dried very slowly in order to avoid cracking. Its air shrinkage is 16.67%. On burning, it assumes a light buff color.

Eleven perfect briquettes of this sample were broken, the breaking loads being 110, 115, 140, 150, 120, 165, 125, 155, 160, 155, 125, pounds, the average being 138 pounds. Thus, the tensile strength of this clay is 180

pounds per square inch.

The chemical analysis of this clay is as follows:-

Moisture		5.40	70
Ignition Loss		9.52	70
Silica	(SiO ₂)	52.10	
Alumina	(Al ₂ O ₃)	27.55	
Iron	(Fe ₂ O ₃)	3.62	
Calcium	(CaO)	.65	
Magnesia	(MgO)	.49	
Alkalies	(Na ₂ O-K ₂ O)	.67	(BY DIFFERENCE)
TOTAL FLUXES		5.43	70

We have no information as to the extent, depth, location, or geology of this deposit, but since it is being exploited by the Coeur d'Alene Commercial Club as one of the assets of the country, it must be of considerable extent. Judging by the tests and analysis, it is a very high grade clay, and should be of considerable industrial importance when developed. It is apparently suitable for high grade stoneware and pottery, and might be suitable for chinaware.

Sample No. 7 was secured from Mr. Neill, of Hope, Idaho. We have no information as to the extent, depth, location, or geology of this clay, as the deposit is entirely undeveloped. Geologically, however, it is of sedimentary origin, and was deposited in the bed of Lake Pend d'Oreille.

This clay is gray in color, fine grained and eminently plastic, free from sand and other impurities. It is of medium dense body, slakes easily and quickly, and requires 29% of water to bring it to its most plastic condition. It dries well, without cracking or warping; has an air shrinkage of 8.33%, and burns to a light brownish red color.

Seven perfect briquettes of this clay were broken the breaking loads being 120, 105, 105, 135, 100, 100 pounds, the average being 111 pounds, corresponding to a tensile strength of 145 pounds per square inch.

This is a rather high grade of clay, and should be suitable for the manufacture of all the ordinary clay products that require a red color. It should make excellent stoneware as it possesses all the qualities of a stoneware clay.

The chemical analysis of this clay is as follows:-

Moisture	3.24
Ignition Loss	4.83
Alumina (Al_2O_3)	11.94
Iron (Fe_2O_3)	12.83
Titanium (TiO_2)	.22
Calcium (CaO)	.79
Magnesia (MgO)	2.42
Alkalies (Na_2O-K_2O)	5.76
by difference.	
Total Fluxes	21.80

The high proportion of fluxes in this clay make it unfit for all purposes requiring a refractory or semi-refractory clay; but its plasticity, tensile strength and easy working properties would apparently make it eminently desirable for the manufacture of sewer-pipe. It would probably be suitable for pressed brick, if mixed with suitable sandy material.

Sample No. 3 was sent to us by Mr. Philip Heinrich of Midvale, Idaho. This clay is of a dark earthy color, has a dense hard body; is fine-grained and free from large amounts of sand and is plastic. It slakes slowly, and 34% of the water was used to bring it to its most plastic condition. Its air shrinkage is 13%, showing the need of careful drying to prevent cracking and warping. On burning it assumed a deep red color, and caked together in the crucible, indicating a very low fusing point.

Six perfect briquettes of this sample were broken, the breaking loads being 220, 190, 165, 190, 185, and 185 pounds, averaging 189 pounds, corresponding to a tensile strength of 248 pounds per square inch.

The chemical analysis of this clay was as follows:

Moisture	4.37
Ignition Loss	4.75
Silica (SiO_2)	54.34
Alumina (Al_2O_3)	7.77
Ferric Oxide (Fe_2O_3)	19.51
Lime (CaO)	3.26
Magnesia (MgO)	1.62
Titanic Oxide (TiO_2)	.93
Manganous Oxide (MnO)	.20
Alkalies (by difference)	3.26
Total Fluxes	27.65

The unusually high amount of fluxes make this rather a low grade of clay. It is, however, very strong and tough and should be very suitable for the manufacture of brick-sewer pipe and the like, where a dense hard body is essential and a deep red color is desirable.

SAMPLE NO. 9 was secured from the campus of the University of Idaho, from a pit dug to contain the gasoline reservoir for the gasoline generator of the Engineering Building. This clay is gray in color, of medium grain, and fairly plastic. It slakes easily and quickly and requires 25% of water to bring it to its most plastic condition, and dries without cracking or warping. The air shrinkage is 12.5%.

Eight perfect briquettes of this sample were broken, the breaking loads being 100, 90, 85, 100, 105, 105, 110, and 95, the average being 99 pounds, corresponding to a tensile strength of 129 pounds per square inch. This clay is similar to No. 10, being from a different part of the same bed, and no chemical analysis was made of it.

SAMPLE NO. 10 was also secured from the campus of the University. It was obtained from the excavations on the athletic field which was ^{being} constructed at the time this investigation was being made. This clay comes from an older and deeper stratum than No. 9, though of the same formation. It is light brown in color, fine-grained and plastic, and is of moderately dense body. It slakes easily and quickly, and works up well, requiring 30.5% of water to bring it to its maximum plasticity and dries without cracking. Its air shrinkage is 14%, and it burns to a good red color. This is the clay that was used in the manufacture of the brick for the first Administration Building of the University of Idaho. The brick were of very high grade.

Ten perfect briquettes of this clay were broken, the breaking loads being 130, 145, 125, 135, 120, 110, 155, 130, 155 and 120 pounds, the average being 135 pounds, corresponding to a tensile strength of 176 pounds per square inch.

The chemical analysis of this clay was as follows:

Moisture		4.00
Ignition Loss		4.49
Silica	(SiO ₂)	63.35
Alumina	(Al ₂ O ₃)	10.27
Ferric Oxide	(Fe ₂ O ₃)	11.67
Lime	(CaO)	1.46
Magnesia	(MgO)	1.37
Titanic Oxide	(TiO ₂)	.49
Manganous Oxide	(MnO)	.08
Alkalies (by difference)		2.82
TOTAL FLUXES		17.32

SAMPLE NO. II was sent to Prof. D.C. Livingston of the Department of Mining from Deary, Idaho, and he kindly turned it over to us, for investigation and analysis. No information of a definite sort regarding its formation and extent is available, but it is said to exist in quantities of possible commercial importance.

The air-dried clay is almost pure white and, on burning assumes a very light buff or cream-colored hue. It is very fat and plastic, slakes readily, and, while for the most part is very fine-grained, contains a small amount of fine black and white sand, and white pebbles, *ranging in size up to 1/8 inch in diameter*. About 35% of water should be used to bring it to its most plastic condition, when it shows an air shrinkage of 8 1/3%.

The briquettes used to determine tensile strength were imperfect, being laminated on account of incorrect methods

of molding them, and shrunk so that they fitted the testing machine very poorly, giving low results. The breaking loads on 7 briquettes were 65, 105, 70, 75, 70, 70, and 90 pounds, averaging 78 pounds, and corresponding to a tensile strength of 115 pounds per square inch?

The chemical analysis of the clay was as follows:

Moisture	4.65
Ignition Loss	9.78
Silica (SiO_2)	49.54
Alumina (Al_2O_3)	27.23
Ferric Oxide (Fe_2O_3)	5.63
Titanic Oxide (TiO_2)	.51
Lime (CaO)	.35
Magnesia (MgO)	.53
Potash (K_2O)	1.444
Soda (Na_2O)	<u>.31</u>
TOTAL	99.97
TOTAL FLUXES	8.26

The analysis of this clay indicates a high-grade semi-refractory clay, probably of feldspathic origin, containing a very large proportion of Kaolin. It should be suitable for the manufacture of terra cotta, and with the proper admixture of suitable material could probably be used for making paving brick, fire-proofing and hollow-brick.

SAMPLE NO. 12 while not an Idaho product, is included here as an example of clay used in Portland cement manufacture. It was obtained from the clay quarry of the Superior Portland Cement Co. of Concrete, in north-western Washington.

The deposits from which this sample was obtained are of recent sedimentary origin. The ^{deposits} ~~quarries~~ that are being

worked at the lower end of Skagit River canyon, are up to fifty feet in ~~height~~, from one-half to one mile in ~~length~~ ^{width} and several miles ~~wide~~. ^{in length}.

The following analysis was made by C.F. Smith in the laboratory of the S.P.C.Co. during the summer of 1913.

Moisture		2.65
Ignition Loss		2.69
Silica	(SiO_2)	58.10
Alumina	(Al_2O_3)	15.29
Ferrie Oxide	(Fe_2O_3)	8.86
Lime	(CaO)	5.47
Magnesia	(MgO)	4.06
Sulphur	(S)	0.13
Alkalies	(by difference)	2.75
TOTAL FLUXES		21.14

This clay is representative of the clays used in actual cement manufacture. Its magnesia and alkali contents come just within the limits that make it suitable for cement material, while iron is added as ore or slag to increase the iron-alumina content. The silica in the slag is not as finely divided is most desirable, necessitating extra expense in grinding.

DESCRIPTION OF THE VARIOUS KINDS OF CLAYS

MENTIONED IN THIS THESIS, "IDAHO CLAYS".

1. PAVING BRICK CLAYS.

Paving brick clay must be plastic enough so that it may be worked in an auger machine, and worked as cheaply as possible; though it should not be plastic enough to produce lamination. Air and fire shrinkage should be as small as possible, to avoid cracking and warping; and the brick should be able to stand comparatively rapid drying. The clay should be of such a character as to produce dense brick, and should burn to a very strong, tough body. It should also have a very high tensile strength.

The difference in temperature between the point of incipient fusion and vitrification should be from 300 to 400 degrees F., in order to minimize waste in the kiln.

2. SEWER PIPE CLAYS.

The clays from which sewer pipe may be made are very similar to those used in the manufacture of paving brick. The sewer pipe have to be vitrified in burning, hence a clay high in fluxes is needed. The clay must be plastic enough so that it can be easily molded, and should burn to a dense, smooth body, and the shrinkage should be as low as possible. Dark colored sewer pipe seems to meet with the most ready sale, hence the clay should contain enough iron to give this color to the finished ware.

3. ROOFING TILE CLAYS.

Clays for the manufacture of roofing tile in general should fuse at low temperatures and produce a vitrified product. The tile when burned should have a dense, tough body, so that it will not absorb much water and will not break in handling. Shrinkage should be as low as possible so as to avoid loss by cracking. The clay must have good plasticity and bonding power, and should contain enough iron to give the red color usually desired.

4. STONEWARE CLAYS.

Wheeler (Mo. Geol. Surv., Vol. XI, pp299-300) gives the following as the properties required in a stoneware clay:

- 1) Eminent plasticity,
- 2) Freedom from coarse sand or other coarse matter,
- 3) Smallest possible iron content,
- 4) Capability of burning to a close, incipiently vitrified body at a temperature less than 2200 deg. F,
- 5) A range of at least 200 deg. F. between the point of incipient and complete vitrification,
- 6) Capability of drying and heating at moderate speed,
- 7) Tough and strong body when burned,
- 8) Freedom from carbonates, sulphates, or other salts that are liable to cause blisters in burning.

5. FLOWER POT CLAYS.

Clays used for the manufacture of flower pots are usually low grade clays, similar to common brick clays. They are burned at low temperatures, and the ware has an open and porous structure. These clays should have good plasticity and fair tensile strength.

6. ORNAMENTAL BRICK CLAYS.

The manufacture of ornamental brick, which is usually by the dry-press or the semi-dry process, calls for a higher grade of clay than is used in ordinary brick. The product may be red, white, or buff, but in general, the clay should fuse at medium temperatures. The most important physical requirements of a clay of this type, according to Ries, are:-

- 1) Uniformity of color in burning,
- 2) Freedom from warping and splitting,
- 3)---Absence of soluble matter,
- 4) Sufficient hardness and low absorption.

7. TERRA COTTA CLAYS.

Terra cotta clays should give uniform colors, strong body, and burn without checking, cracking, or warping. Formerly, the color which was secured after burning was very important, but a uniform color is now usually obtained by

means of slips applied to the terra cotta before it is burned. Shrinkage should be as small as possible, as the blocks must be exactly standard size after burning, so that they will fit into the building, each into its place

They should contain no soluble salts, should have good plasticity, and should burn to a dense, hard body at medium temperature.

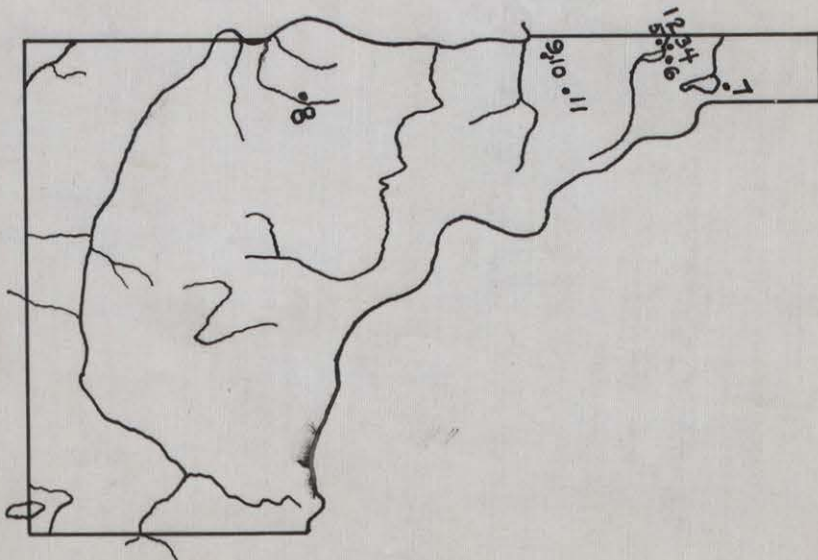
8. PORTLAND CEMENT CLAYS.

The physical properties of clays are of small importance for cement making purposes as compared with their chemical composition, and the fineness, or state of subdivision. In general terms, the clay should ^{contain} ~~be~~ at ~~least~~ ^{or} ~~2~~ ³ times as much silica as alumina. Alumina may be replaced by iron to a great extent. Magnesia should be low, not over 4%. Alkalies should not be present in amounts over 3%.

The uncombined silica in a clay is in the form of sand or pebbles. Pebbles may be removed by mechanical means, but the sand must be in a finely divided condition. If over 5% is present in form of grains not passing a 100-mesh sieve, the clay is not suited to cement manufacture.

Summary of Physical Tests of Clays.

Clay No.	Color (Dry)	Water for making plastic paste (%)	Tensile Strength lbs per in ²	Air shrinkage (%)	Color (Burned)	Fire Shrinkage (%)
1	Dark Brown	41	112	12.5	Brownish Brick Red	2.52
2	Light Yellowish Brown	40	15	3.3	Light Brick Red	5.46
3	Light Brown	29	145	18.4	Brick Red	7.25
4	Brownish Yellow	35	108	6.7	Light Brick Red	2.26
5	Dark gray	35.5	98	8.33	Light Brick Red	.82
6	White	46	180	16.67	Buff	2.48
7	Earthy gray	29	145	8.33	Light Brick Red	2.6
8	Earthy Brown	34	248	13	Dark Brick Red	1.75
9	Light Brown	25	129	12.5	Brick Red	.96
10	Buff	30.5	176	14	Brick Red	nil
11	White	35	115	8.33	Buff	1.83



Sketch showing approximate location of various clay investigations

Summary of Chemical Analyses

Clay No.	1	2	3	4	5	6	7	8	10	11	12
Silica (SiO_2)	44.46	52.02	54.84	53.03	59.21	52.10	57.97	54.34	63.35	49.54	58.10
Alumina (Al_2O_3)	26.97	18.95	20.15	24.82	17.05	27.55	11.94	7.77	10.27	27.23	15.29
Ferric oxide (Fe_2O_3)	8.03	4.07	7.47	5.58	6.05	3.62	12.83	19.51	11.67	5.63	8.86
Lime (CaO)	2.02	3.20	1.54	.00	2.66	.65	.79	3.26	1.46	.35	5.47
Magnesia (MgO)	.69	.48	1.13	.53	1.32	.49	2.42	1.62	1.37	.53	4.06
Potash (K_2O)	{ 9.8	9.60	.47 ^a	3.28 ^a	3.01	.67 ^a	5.76 ^a	3.26 ^a	2.82 ^a	1.44 ^b	{ 2.75
Soda (Na_2O)										.31 ^b	
Titanic oxide (TiO_2)							.22	.93	.49	.51	
Manganous oxide (MnO)								.20	.08		
Sulphur (S)											.13
Moisture (110°C)	{ 18.22	6.21	6.48	3.20	1.34	5.40	3.24	4.37	4.00	4.65	2.65
Loss on Ignition		5.30	5.98	9.56	10.10	9.52	4.83	4.75	4.49	9.78	2.69
Total Fluxes	11.72	17.35	10.55	9.93	13.04	5.43	21.80	27.65	17.32	8.26	21.14

Analyses 1-6 by E.E. Smith; 7-12 by C.F. Smith

^a by difference ^b by J. Lawrence Smith method

APPENDIX.

CONSTRUCTION OF THE ELECTRIC MUFFLE FURNACE.

Since a muffle furnace is much better than a blast lamp for making ignitions, in that it yields a much evenner heat and does not tend to blow away any particles of the finely divided precipitate, and also in that it gives a destructive distillation of the filter, which is much better than burning it off, we decided to construct such a furnace, to be heated by a "Nichrome" resistance wire. Nichrome was chosen as the resistance material because it will stand the highest heat of any resistance material of its kind except platinum wire, which was out of the question on account of its cost.

From the pamphlet entitled "Resistance Materials", published by the Driver-Harris Wire Company, manufacturers of "Nichrome" and other resistance materials, we found that the resistance of No. 17 Nichrome wire at 1100 deg. C. (the maximum temperature at which the wire may safely be used) is 0.564 ohms per foot, and its carrying capacity at the same temperature is 14.5 amperes when coiled in air with free radiation. If insulated with asbestos, to prevent heat radiation, about half the current is all that may be safely be carried. Then, assuming that the formula $I = \frac{E}{R}$, which gives the relation between current, voltage, and resistance in direct current, would apply to the calculation for this coil, since its inductance, impedance, and capacity must be so very small as to be negligible, and calculating for 7.5

amperes of current at 1100 deg.C, we have:-

$$\text{XXXX} \quad 7.5 = 110/R, \quad R = 110/7.5, \quad = 14.7 \text{ ohms.}$$

Since the resistance of No. 17 Nichrome at the desired temperature is 0.564 ohms per foot, we required $14.7/0.564$, or 26 (nearly) feet of wire.

Taking an 18 cm battery-cup, we wrapped the wire tightly and evenly around it, leaving about three quarters of an inch of each end of the cup bare. Around these bare ends we clamped metal bands, to which the wire was connected with solder and to which the power wires were connected by means of brass binding posts. The bands were bolted with small stove bolts to strap iron legs, which in turn were fastened by means of screws to a heavily braced board, 1x12x16 inches in size.

The cup was then covered with asbestos cement, which was intended to serve both to keep the coils of wire from springing together and short-circuiting, and to retain all the heat possible. The whole was then surrounded with asbestos heat-insulating material and a small door made of a piece of asbestos card. After the cement had dried, the current was turned on, and in about twenty minutes a very satisfactory temperature was attained. After this trial, the furnace was allowed to cool:- and next morning, the cup was found checked into nineteen pieces. The battery cup was of too poor quality to stand the extreme temperature.

As we had found, even in this one short trial, that the circular type of furnace was not satisfactory on account

of its small size, inefficiency, and instability, another type of furnace was next considered. Securing a small furnace-muffle, of the "D" type, three inches by four inches by six inches in size, we wrapped the resistance wire from the previous furnace tightly and evenly around it, throughout its entire length, coated it thickly with asbestos cement, and set it aside to dry. While it was drying, we made a base of fire brick, laid in asbestos cement, upon the base-board of the previous furnace; covered it with a liberal layer of the cement; placed the partially-dried muffle in position and cemented it solidly in place.

The next problem was to hold the heat-insulating material in place around the furnace, and at the same time permit free access to the mouth of the muffle. After ~~the~~ several unsuccessful attempts to insulate it sufficiently by draping sheets of asbestos over it and by wrapping layers of asbestos pipe-covering around it, we finally made a hood from a five-gallon kerosene can by cutting off the top and bottom and cutting an opening in the side, the same shape as, and a trifle larger than, the mouth of the muffle. This hood, or more properly, jacket, fitted snugly over the base, was flush with the front of the muffle, and gave about three inches of space on the two sides and the end of it for asbestos packing, which we made by shredding the insulating material from around a piece of eight-inch steam pipe.

Copper conductors (No. 17 wire) were led from the ends of the resistance wire through the metal jacket and down

to the wood base in porcelain tubes, and were firmly fastened to binding posts screwed into the board. As soon as it was dry, the furnace was tried out, and current ceased to flow after about an hour's run. Upon dismantling it, we found that the wire had burned out in two places and was very brittle throughout its entire length. As this was a sure sign of overheating, we revised our figures, going over them very carefully, with the following result:-

Checking our figures of the resistance and carrying capacity of No. 17 Nichrome wire with the handbook, we proved them correct. Looking up the formula for current, electromotive force, and resistance in alternating current circuits, in Sheldon-Mason-Hausmann's "Alternating Current Machinery", (p 71), we found the relation to be

$$I = \frac{E}{\sqrt{R^2 + (2\pi fL - \frac{1}{2\pi fC})^2}}$$

in which I is the current in amperes,

E is the E.M.F. in volts,

R is the resistance in ohms,

f is the frequency,

L is the inductive reactance in henrys, and

C is the capacitance in farads.

The coil in this case is so small that the effects of inductive reactance and capacitance are practically the same as for a straight wire, and therefore the quantity

$$(2\pi fL - \frac{1}{2\pi fC})^2$$

is extremely small and may be neglected. (Authority, Prof. Corbett of the Electrical Dept.) Therefore, the relation

$$I = \frac{E}{R}$$

should hold in this case, and the burning out of the furnace must have been due to the fact that our heat-insulation was better than that provided for in the handbook, so that one-half the open-air current could not be safely carried. This ~~XXXXX~~ supposition is rendered highly probable by the fact that the same wire which gave good results in the small round furnace, which had relatively poor heat-insulation, burned out in the muffle furnace, in which the heat insulation was excellent.

Acting upon this assumption, we wrapped the muffle again, using twenty nine feet of the No. 17 Nichrome wire instead of twenty six feet, as before, to lessen the current flowing through it; covered it loosely with asbestos packing, and connected it through an ammeter and a voltmeter to determine its resistance at its maximum working temperature. Before the wire had become sufficiently heated to render observations possible, however, the expansion due to the rise in temperature and the stiffness of the wire with its consequent tendency to "spring" caused two loops to come together, short-circuit the current, and burn the furnace out.

We removed the burned-out wire, cleaned the muffle, and again took twenty nine feet of the resistance wire. This time, however, we stretched it upon a ladder and allowed the current to flow through it for ten minutes, in order to take some of the spring out of it. Then, with the wire still upon the ladder, we wound it upon the muffle, holding the muffle in our hands and slowly moving along the ladder as the

wire was wound up, using as much tension as seemed safe. In this way, we secured a very even, tight winding. We then cemented the muffle solidly in place, as in the first case, and allowed it to dry. Then, after packing the space inside the jacket with loose asbestos packing, as before, we turned the current on and awaited results. It heated up evenly and smoothly to a bright red, and after cooling, it showed no evil effects. Since it appeared satisfactory, we took the furnace to the laboratory of the Physics Department, and calibrated it by means of their thermoelectric pyrometer.

DATA OF CALIBRATION.

On the indicator of this particular pyrometer,

523.0	indicates	600	deg. C.
733.0	"	800	" .
957.0	"	1000	" .
1070.0	"	1100	" .

Voltage during test, 100 volts.

TIME	READING	
3:00 PM	700	
3:10	790	
3:15	860	Furnace taking
3:20	900	8.5- 9 amperes.
3:25	926	
3:30	952	
3:35	992	
3:40	1000	Connected in a
		sliding resistance
3:45	952	8.25 amperes.

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DATA OF CALIBRATION.

On the indicator of this particular pyrometer,

523.0	indicates	600 deg. C.
733.0	"	800
957.0	"	1000
1070.0	"	1100

Voltage during test, 100 volts.

TIME	READING	
2:00 PM	700	
2:10	720	
2:15	820	Furnace taking
2:20	900	8.5-9 amperes.
2:25	925	
2:30	932	
2:35	932	
2:40	1000	Connected in a
2:45	952	sliding resistance
		8.25 amperes.

TIME	READING
3:50	962
3:55	988
4:00	1000
4:05	1010 At this point we
4:10	1010 slid in more res-
4:15	1010 istance, till only
4:20	1016 8 amps flowed.
4:25	1020
4:30	1022 Regulating resist-
4:35	1022 ance so that temp.
4:40	1022 stays close around
4:45	1022 1022 (1075 deg C)
4:50	1022 CONSTANT.

Upon measuring the amount of external resistance necessary to maintain the temperature below 1100 degrees, it was found to be 1.42 ohms, which amount was measured off on another variable resistance and permanently connected in series with the furnace.

The furnace was used without change for about two months, both for our thesis work and for the Sophomore class in Quantitative analysis. At the end of this time, one of the copper leads burned out, due to the oxidation caused by the prolonged high temperature. In replacing these leads with much heavier ones (No. 8 copper wire) it was discovered that the intense heat of the muffle, even through the thick

base of fire-brick, had burned through the wooden base of the furnace and through the top of the bench beneath. Therefore it was deemed advisable to remove the wooden base and place the furnace upon loose fire-brick in such a way as to leave an air space between it and the bench, which was done.

This arrangement (shown in the accompanying photograph) proved perfectly satisfactory, and was the way in which the furnace was worked the remainder of the year. The furnace was in perfect condition at the close of the school year, which is a very favorable comment upon the endurance of "Nichrome" resistance wire.

