SYLLABUS OF CLAY TESTING
PART 1

BY

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SIDNEY GOTTLIEB, and GLENN C. TRUESDELL

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SYLLABUS OF CLAY TESTING. PART 1

By Theron A. Klinefelter, Robert G. O’Meara, Sidney Gottlieb, and Glenn C. Truesdell

INTRODUCTION

Much of the literature dealing with clays and clay products is filled with many and various methods of testing. Each of the ceramic and nonceramic industries has its own assortment of test methods. A general survey of them shows that a surprising number are merely variations or different angles of approach to evaluation of some one property. Their very multiplicity and lack of agreement are evidence of their inadequacy and empirical nature. Virtually all of the current tests are an empirical development by the user, who, of course, is interested only in a clay to fit his individual product.

Notwithstanding much theorizing in the past, it has been only recently that fundamental research has furnished data of real practical value. No doubt testing will be greatly simplified. Nevertheless, much empirical testing must continue, for, after all, clays are what has been aptly termed “geological trash.” No matter what the essential structure of the predominant component of the mixture, the ever-present impurities and exchangeable bases are always certain to modify properties.

In studying the present tests, one of the greatest annoyances is the varied, overlapping, and confusing nomenclature and terminology that have grown into current usage. The name of one type of clay is based on usage, another on its geologic or geographic origin, and another on its mineral composition. Again, a term means one thing in one test and another in another test, or different things when used by different industries.

Ceramic technologists have been attempting for years to agree on a more orderly nomenclature and have begun to reach some sort of stabilization. Meanwhile the nonceramic users have grown apace, and since their products are not considered clay products the tendency

1 Work on manuscript completed June 30, 1942.
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4 Junior mineral technologist, Southern Experiment Station, Bureau of Mines, Tuscaloosa, Ala.
5 Junior chemical engineer, Southern Experiment Station, Bureau of Mines, Tuscaloosa, Ala.
6 An unusually confusing terminology of this type is the practice of soil chemists in dubbing as "clay" any material having a certain degree of fineness. Foundrymen also have followed this practice. In the following outline, when we say “clay” we mean clay in the usual acceptance of the term by the ceramic industry and not a material of a certain degree of fineness.
has been to overlook their problems. However, with the annual consumption of huge tonnages of clay by nonceramic industries, the need for interest is apparent. In fact, the clay laboratories have been under increasing pressure in recent years from producers to evaluate clays for both ceramic and nonceramic uses. Availability of such information from a single source avoids the necessity of making the rounds of the various industries.

The Bureau of Mines has surveyed the more important tests recognized by the various industries with a view to suggesting a series whereby any given clay may be classified according to its possible uses, both ceramic and nonceramic. The work has been done under the supervision of Oliver C. Ralston. The authors are much indebted to W. H. Coghill, supervising engineer, Southern Experiment Station, Dr. T. N. McVay, Dr. Hewitt Wilson, J. J. Hazel, Dale Bogard, Ellis Creitz, Louis Berkelhamer, Ellis Hertzog, and Homer MacDonald. To make the outline or syllabus as widely serviceable as possible, only those tests are included that are possible in a laboratory moderately well equipped for making physical and chemical tests. For complete and accurate testing, more elaborate equipment or methods are needed. The final testing, of course, must involve actual incorporation of the clay into the product, which must then be tested. Obviously, this last testing is beyond the scope of this syllabus and must be done by the user himself.

It will be noted in the series of tests presented that the main and final classification is on the basis of use. For some purposes, such as prospecting for more deposits of the same kind, geologic and other types of classification are more useful. Attempts to mix such classifications have never proved very satisfactory from any standpoint and hence are avoided as much as possible in the accompanying flow sheet (fig. 1).

Naturally, some reference must be made in the outline to current terms for the various clays, but it will be noted that the final classification by usage is not dependent on such terms, excepting, of course, the broad clay-mineral types.

This presentation (pt. 1) is what might be termed a qualitative syllabus, in that uses only are indicated, no attempt being made to evaluate a clay quantitatively, that is, to grade for particular uses.

In part 2, which is to follow, the work will be amplified both qualitatively and quantitatively.

The procedure of part 1, shown in figure 1, falls into two general steps. In the first, preliminary tests are made to eliminate from consideration all nonclay minerals or clay minerals containing such large amounts of impurities as to render them unfit for normal uses and also to make a broad general classification of the clay minerals into the kaolinite (shales and clays) and montmorillonite groups. In the second step, the main tests, divided into the two general headings, ceramic and nonceramic, are detailed.

By referring to the flow sheet and making the outlined tests, each of which is described in the text, even an inexperienced operator should be able to classify clays according to possible uses. An explanation of the flow sheet follows:
SAMPLE

PRELIMINARY TESTS

OBSERVATION (color, hardness, texture, screen size, plasticity)
REHYDRATION (special samples only)
CHEMICAL (soluble salts, alumina, carbonates, sulfates, phosphates, iron)

CLAYS
Low in carbonates, iron, etc.

MONTMORILLONITES
CALCAREOUS CLAYS
ROAD MATERIALS
SOILS
BAUXITES
OCHERS, UMBER, PIGMENTS
MARLS (limestone and dolomite)
PHOSPHATES
HALloysite-Allophane

No further tests

CLAYS

NONCERAMIC TESTS

ODY TEXTURE

Filter test
Satisfactory
Slopes completely
and swells
USE
Drill muds

Satisfactory
Siakes with granules
Cracks and chips

Satisfactory
Nonswelling
bentonites

Bleaching test
Satisfactory
Oil bleacher
Activation test

Oil bleacher

CLASSIFY BY RAW COLOR

clays or...?

All colors
Reds
Grays
Creams
Very light
USES
Fillers
Paints
Molding sands
Modeling
Drill muds

<0.5%
USE
Ultramarine

<1.5%
US€
Alumina

<3.0%
USE
Alums

USES
Rubber filler

USES

Fine grain (<325)
Settling test
Satisfactory
USE
Medical

USES

Cu, Mn tests
OK

USES

Rub-out test
Not

USES

High gloss
Low gloss

USES

Color OK
Bleach test
Not

USES

Paper coat and filler
Textile filler

1. Classification of clays by uses.
PRELIMINARY TESTS

INSPECTION

In making evaluations it must be remembered that properties such as color, hardness, and texture are interrelated, and each may affect the other. Hence, it will be noted that several properties may be discussed together in the text.

COLOR

VERY LIGHT, SUCH AS WHITE TO CREAM TINTS

If the texture is soft and there seems to be little sandy grit, there are possible uses in the paper, rubber, and chemical trades and for fine ceramic wares. If, however, the sample seems coarse, hard, and sandy, it is more likely to find application in ceramic refractories or chemicals only.

DEEP REDS, BROWNS, YELLOWS

If the iron content is found to be sufficiently high in later tests, possible use as a pigment is indicated; otherwise, the clay is likely to be utilized in the ceramic heavy-clay products industry only.

REDS, LIGHT TO DARK

Usually good for heavy-clay products only.

BUFFS AND GRAYS, ALL SHADES

Most refractory clays are in this class. If the P. C. E. tests show them to be too low, they are nearly always good for heavy-clay products and stonewares.

GREENS

Usually indicative of bleaching clays.

HARDNESS AND TEXTURE

SOFT FEELING, WITH LITTLE APPARENT GRIT

Very light colors.—Paper, rubber, and chemical trades and fine ceramic whitewares.

Dark colors.—Drilling muds and certain types of filler. All the various color grades may find ceramic uses.

PISOLITIC OR OOLITIC STRUCTURE

Indicates bauxites and bauxitic clays.

HARSH, SANDY, GRITTY-FEELING

In light colors may be good for refractories, and in all various colors will probably be good for heavy-clay products.

HARD

Requires considerable pressure to break. In white to cream, it would be employed in high-grade ceramic ware and refractories.
Possible use in chemical industry for $\text{Al}_2\text{O}_3$ and alums. In dark colors, it would include the shales (good for heavy-clay products) and flint fire clays (good for refractories as well as heavy-clay products).

SCREEN SIZE

Screen size is determined by putting the material through several sieves and examining the various fractions with a magnifying glass. The screen sizes depend somewhat on the type of clay. Where the material is obviously quite fine-grained and soft, 200- and 325-mesh screens should be used. With the coarser, sandy clays and shales, 6-, 20-, 65- and 100-mesh screens are preferable.

Classifications based on sieve analyses are very broad, and the personal factor is rather large whatever procedure is followed. Hence, size and type of the sieves are left to the discretion of the technician. However, once a procedure is adopted, it should be followed consistently, or comparison will be relatively worthless.

The methods and equipment specified by the American Society for Testing Materials (Philadelphia, Pa.), for particle-size determination of ground refractory material (designated C92-36) are excellent and may be followed if preferred. If the sample in question happens to be a fine, white clay with possible uses in the paper and rubber trades, a somewhat more refined test is made with 200- and 325-mesh sieves only. The details of this sieving will be found in the discussion of nonceramic testing. The following procedure will give excellent results:

SIZING-ANALYSIS PROCEDURE

A sample of the clay is dried at 105° to 110° C. for 2 to 4 hours (2 hours is long enough for air-dried samples, but 4 hours may be necessary for wetter samples). A 100-gram sample of the dried clay is slaked with 250 ml. of water in a 500-ml. beaker for 1 hour. The mixture is then stirred until a slip of homogeneous consistency is obtained. Enough water is added to wash the sample onto a 200-mesh screen; then it is hand-jigged and finally washed until water passing through the screen comes clear. If the particles remaining on the screen are slaked, so that gentle movement causes them to “ball up,” the clay is rubbed gently and washed until only firm particles remain. If the plus-200-mesh material amounts to more than a trace, it is dried at 105° to 110° C. for 2 hours and screened on 6-, 20-, 60-, and 100-mesh, if warranted. If the minus-200-mesh is more than a trace it may be screened further on 325-mesh, provided the characteristics of the clay warrant this. In these instances, either the minus-200-mesh or the minus-325-mesh portions are discarded, and the amount is determined by difference. The minus-200, plus-325-mesh products are dried in the same manner as are the other sizes. The sizes are reported as percentage to the nearest tenth.

If the sample swells, a swelling bentonite or a fuller’s earth may be indicated. In such instances more water should be added or the amount of sample should be reduced to lower the pulp density for complete slaking and screening.
INTERPRETATION

Large amount of plus-6-mesh.—Not likely to be usable except as road material.

Large amount of minus-6- and plus-20-mesh, that appears to be mostly a silica sand. Possibility of use as a foundry sand or clay.

Small residue on 20-mesh; fair residues on 60-, 100-, and 200-mesh.—Light colors, grays, and buffs, probably satisfactory for refractories and heavy-clay products. All reds should be good for heavy-clay products.

Very fine grained, that is, not over 3 percent residue on 200-mesh and one-half percent on 325-mesh.—With light colors, fillers for paper and rubber and use for fine ceramic wares are indicated. With the darker colors fillers for other applications are possible, and uses may be indicated in ceramics, depending upon how the fired color shows in the firing tests. With very deep colors, possible utilization as pigments may be indicated.

PLASTICITY

A good idea of plasticity may be obtained by wetting a small amount of the sample with water and working the mass with the fingers, but it is best to use enough equipment to obtain somewhat more accurate evaluations. There are a number of devices for measuring plasticity or properties corollary or akin to it, and if a laboratory happens to have such apparatus it may be utilized. However, for the purpose of this outline it will be found that the old Atterberg test will do very nicely. The test is subject to a considerable personal factor, so that there is no necessity for extreme accuracy of measurement. There is much overlapping of ranges for kaolinite-type clays. The best thing to do is to say that a clay has good plasticity if the amount of water absorbed is large and the working range is relatively long. However, when montmorillonite-type clays are tested, it will be found that the amounts of water absorbed and the ranges of workability are far greater than those of the kaolinite type. Thus, a very simple method of differentiating the two types is available. With an experienced operator, finer distinctions are possible. Furthermore, the swelling type of bentonite becomes readily distinguishable.

PROCEDURE

Equipment consists of a graduated 100-cc. burette, evaporating dish, and a spatula.

A 100-gram sample (minus-20-mesh) is dried at 105° C. The sample is placed in the evaporating dish, and small amounts of water are added from the burette until the whole mass, which is worked with the spatula, is uniformly wet. At a certain point the spatula, used knife fashion, will make a clean, sharp cut with slight sticking, and the separated portions of the mass will not tend to flow together. A reading is taken on the burette of the amount of water used. This gives the low figure for the plastic range. Water is now added in small amounts, the mass being worked constantly with the spatula; frequent

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knives are made until the point is reached when the mass tends to stick and drag on the spatula. Again, a reading is taken of the amount of water added. This gives the upper limit of the workability range.

**INTERPRETATION**

Figure 2 shows some ranges of representative clays.

![Diagram of clay classification by plasticity test](image)

**Figure 2.** Classification of clays by plasticity test.

*Range under 20.*—Indicates a clay of little plasticity or a nonclay mineral.

*Range between 20 and 40.*—Indicates a clay of moderate plasticity. Shales, flint clays, and the majority of surface clays fall within this range. Some of the less plastic kaolins fall here also. Individually, the range will be short for a clay of low plasticity, 4 or 5 for a diasporite...
clay, for instance, and longer for the more plastic type—30 to 42 for a plastic surface clay, as another illustration. It is out of the question to differentiate among the above-mentioned types in this test. However, data as to amounts of water needed to obtain plasticity will be useful during the test for ceramic uses. Most of the clays in this group are kaolinitic types, but some halloysites will be found here also. The range, however, is quite narrow, seldom exceeding 2 or 3 percent. To separate out the halloysite-type clay mineral requires an additional test, which will be described later.

Range between 35 and 60.—Indicates a very plastic clay. The most important clays in this group are the kaolins and ball clays, although some of the fine-grained, highly plastic surface and glacial clays will be found here also. The various types have approximately the same range, but the more plastic ones always require the most water. In general, the kaolins have a somewhat shorter range than the balls, but it is difficult to make any real separation.

Range above 65; montmorillonites.—Includes swelling-type bentonite, nonswelling bentonites (sometimes called bentonites, subbentonites, or metabentonites), and fuller's earth. Upper limits as high as 235 to 250 have been noted. The swelling type is readily distinguishable from the others by the way it gelatinizes and begins to overflow the dish.

**OPTIONAL TEST FOR CLASSIFICATION**

When there is doubt as to the identity of a mineral type, such as halloysites and some of the metabentonites, and where the initial tests have not given clear evidence, the following test will be found reasonably accurate:

**PROCEDURE**

1. Use low-form (wide-mouth) pyrex weighing bottles with covers.
2. Obtain tare on bottles after prior ignition to 600° C.
3. Weigh approximately 1 gram of each raw sample, in duplicate (minus-100-mesh).
4. Place samples in oven at 300°±5° C. for 2 hours with covers removed (covers may be placed in oven also).
5. Remove bottles and covers; cover bottles and place in regular desiccator until cool; then weigh with covers on.
6. Place one of the duplicate samples, uncovered (see step 12 for other sample), in a preheated desiccator (see below), the desiccant of which has been replaced with water. Take proper precaution to prevent condensate from dropping into bottles, and do not allow bottles to have any contact points other than base on wire screen that replaces regular porcelain plate (see apparatus).
7. Place desiccator (water-containing) in oven at 65°±1° C. (desiccator preheated at this temperature for approximately 2 hours before use) for 2 hours. Also, place covers in oven to keep warm.
8. Remove covers from hot box first, then desiccator.
9. Remove bottle from desiccator, place on towel, wipe apparent condensate from inside (if obvious) of bottle, and stopper immediately (if sample shows wet spot, dark from dropping condensate, unreliable results can be expected).
10. Wipe outside of bottle, and place in regular desiccator to cool and weigh.
11. Record gain in weight (dehydration) as percent weight of sample dried at 300° and 5° C.

12. A duplicate sample is replaced in oven, now set at 600°±5° C., for 2 hours and, using the same precautions as for 300°±5° C. dehydration, the loss in weight that occurs between the 300° and 600° C. periods is ascertained. Record as percentage of weight of sample dried at 300°±5° C.

*Test devised by Louis Berkelhammer, associate petrographer, Bureau of Mines.*
INTERPRETATION

The classification resulting from the test may be noted by consulting figure 3.

CHEMICAL TESTS

The tests made at this point have as their general purpose the elimination of materials that are not clays or shales in the ordinary meaning, although they look like and often are mistaken for clays. For this reason they frequently turn up for examination in clay laboratories. The tests are also designed to point out clays or shales containing so many impurities as to render them useless for many purposes. Except for the water-soluble salt test, the average operator probably will need to use the tests only occasionally.

TESTS FOR CARBONATES AND IRON

For marls, ochers, and calcareous and dolomitic clays.

PROCEDURE

A small lump of the sample, or equivalent in powder, is placed in a test tube containing about 15 ml. cold HCl (1 acid : 1 water). If calcite is present it will effervesce in the cold solution. The solution is then warmed, and if dolomite is present, effervescence will occur. If solution of the sample is more or less complete, calcite (marl) is indicated. Very slight effervescence indicates a calcareous or dolomitic clay.

About 1 gram of the powdered sample is next treated with 5 ml. of concentrated HCl plus an oxidizing agent (KMnO₄, K₂Cr₂O₇) and
heated to boiling, diluted with 25 ml. H₂O. Ammonium hydroxide is then added to the solution to precipitate iron and alumina. The amount of iron is estimated from the volume of the precipitate, and the proportion of iron and alumina is indicated by the color.

This method does not give the full iron content, but the amount so found is sufficient at this point. Clays found by this method to contain more than 15 percent Fe₂O₃ are considered as being in the umber, ocher, or pigment group.

**CAUSTIC-SOLUBLE ALUMINA**

For bauxites and bauxitic clays.

**PROCEDURE**

A 1-gram sample of clay dried at 105° C. is digested in a 30-ml beaker with 1 gram of NaOH and 10 ml of water on a steam bath for 30 minutes. The sample is stirred at 5-minute intervals while heating. The sample is then cooled, diluted to 25 ml., and settled.

* Dilution A.—A 5 ml. portion of this liquid is transferred to a 25-ml volumetric flask, to which is added 2.5 ml. of acetic acid (1 acid: 3 water), and the solution is diluted to 25 ml. with water.

* Dilution B.—If a precipitate forms in dilution A, warm gently, transfer to a 250-ml volumetric flask, and redisolve with warm water, cool, and dilute to 250 ml.

Transfer 0.5 ml. of dilution A plus 405 ml. of H₂O or 5 ml. of B to a comparator tube (15 mm. in diameter, 75 mm. in height) or a 10-ml graduated cylinder; add 1.5 ml. of ammonium acetate (300 grams per liter) and 0.2 ml. of 8-hydroxyquinoline solution (2.5 grams of 8-hydroxyquinoline in 5.0 ml. of glacial acetic acid and diluted to 100 ml.), and shake. Let stand 2 minutes. If the alumina (plus titanium) exceeds 2 percent, a precipitate will form. At 10 percent the precipitate will be so heavy that a bright metallic surface placed against the bottom of the tube will be just visible when held so that reflected light is cut out. At 20 percent the precipitate is heavy enough so that the metallic surface is not visible, in which case gravimetric determination of alumina may be desirable.

In the gravimetric determination 5 ml. of solution A or 50 ml. of solution B is placed in a 250 ml. beaker, to which is added 5 ml. of HCl and 100 ml. (of 50 ml.) water, and the solution is heated about to boiling. Two drops of methyl-red solution (0.2 gram of methyl red in 100 ml. of ethyl alcohol) is added with ammonium hydroxide (1 NH₄OH : 1 water) until the solution is yellow-green. The solution is then boiled and filtered while hot, and all the precipitate is transferred to the filter paper. The precipitate is washed six times with a hot 2-percent solution of ammonium chloride and finally ignited in a tared platinum crucible and weighed. The weight-percent precipitate multiplied by 500 equals the percentage of Al₂O₃.

**ACID-SOLUBLE ALUMINA**

**PROCEDURE FOR HALLOYSITE AND ALLOPHANE**

A sample of minus-65-mesh clay is dried to constant weight at 110° C., and 1-gram sample of the dry clay is mixed with 40 ml. of 35 percent H₂SO₄ and heated to 100° C. The acid is allowed to react at this temperature for 1 hour while the mixture is being stirred constantly. The residue is then separated by filtration and washed free of soluble salts. The alumina in the filtrate may be determined by any standard procedure. A suitable method is to dilute the solution to 250 ml. and use aliquot parts for R₂O₃ and Fe₂O₃ determinations. The R₂O₃ is determined by boiling the slightly acid solution, just enough ammonia being added to turn brom cresol purple (this indicator seems to be more satisfactory than methyl red), filtering the solution, and washing and igniting the precipitate at 1,200° C. for 1 hour. (More accurate results may be obtained by redissolving and precipitating the precipitate.) The iron is determined on an aliquot part by titration with K₂Cr₂O₇. The solution is evaporated to 50 ml., and 25 ml. of concentrate hydrochloric acid is added. The mixture is heated to boiling, the iron is reduced with stannous chloride, and one drop in excess is added after the color disap-
pears. The liquid is cooled, and 8 ml. of saturated HgCl₂ solution is quickly added. After 3 minutes, dilute to 200 ml., and titrate with K₂Cr₂O₇ solution, using diphenylamine as an indicator.

DISCUSSION

If the percentage of Al₂O₃ is 30 to 40, halloysite or allophane is indicated. Many clays contain as much as 5 percent acid-solution Al₂O₃. Bauxite clays may contain more acid-soluble Al₂O₃, but the presence of bauxite can be confirmed by the alkali-soluble Al₂O₃.

PHOSPHATES

Accurately weigh 1 or 2 grams of finely ground sample rock into a beaker of suitable size. Add 20 cc. of aqua regia solution, and digest on a hot plate until free from red fumes. Dilute, and cool to room temperature. Transfer to a 250-cc. volumetric flask, and dilute to mark. Draw off an aliquot portion by means of a pipette equivalent to 0.1 gram of sample, and proceed as follows:

Transfer this aliquot portion to a beaker or jar of about 250-cc. capacity, add 65 cc. of ammonium nitrate solution, place solution in water bath, and precipitate phosphates at 65° C. with 40 cc. of molybdate solution. Keep at 65° C. for 15 minutes, stirring occasionally. Filter on asbestos pad by aid of suction, or on good, close filter paper. Wash with distilled water until free from acid. Transfer to the original beaker with as little water as possible, and dissolve the yellow precipitate with a slight excess of standard sodium or potassium hydroxide solution (0.3238 N). When all of the yellow precipitate is in solution, titrate the excess standard caustic with standard nitric acid solution, using phenolphthalein indicator.

Calculation.—Number of cubic centimeters of caustic used, minus acid equivalent, equals percentage of phosphoric acid, natural basis.

To correct to dry basis, divide percentage of phosphoric acid, natural basis, by 100, minus percentage of moisture in ground sample. Phosphoric acid, dry basis, multiplied by factor 2.184 equals tricalcium phosphate, dry basis.

WATER-SOLUBLE SALTS

All clays contain appreciable amounts of soluble salts, which, unless corrected for, may render the clays troublesome to process or even unfit for use. Hence, determination of the total amounts present is a worthwhile routine test for virtually all samples. The following procedure is accurate enough for the present work.

PROCEDURE

Grind a 500-gram sample through a 20-mesh sieve, dry at 105° C., and blunge for 30 minutes in 3,000 cc. of distilled water, using a good mechanical mixer. Then allow to settle. The liquid is then filtered, and 500 cc. of the filtrate is evaporated in a tared beaker, dried at 105° C., and weighed. The weight of the salts, in grams, multiplied by 1.2 gives percentage of soluble salts. If the filtrate is not clear, a smaller aliquot part of the 3,000 cc. may be centrifuged and the salts determined in like manner.
CLASSIFICATION RESULTING FROM PRELIMINARY TESTS

ROAD MATERIALS

Where the sample is a mixture of gravel, coarse, colored, sandy grit, and obviously quite impure clay, about the only use likely would be as a road material. Further tests of a different type would be necessary to be certain of this use, and for the purpose of this outline such a sample is of no further value. For further tests see following references:

3. Various publications of United States Bureau of Public Roads; also various State Highway Departments.

SOILS

When subjected to the Atterberg test, most soils show low plasticity with very short range. Alone they seldom make good ceramic bodies, although they may be used with other clays at times. They usually contain too much organic matter, fine sand, and too many other impurities to be very useful for either ceramics or nonceramics.

BAUXITES AND BAXITIC CLAYS

As the uses of bauxite in both ceramics and nonceramics are definite and well known, no further tests according to the present outline are indicated. Bauxitic clays, however, are likely to be used in making refractories, and ceramic tests are indicated for them.

OCHERS, UMBERS, PIGMENTS

Where the iron content is 17 to 20 percent, the clay is likely to have little use except as an ocher, umber, or pigment, and no further tests are indicated in this outline. To be used as such, the material must, of course, meet the necessary specifications of color, uniformity, grain size, etc., desired by pigment manufacturers. For further tests, see following references:

4. American Institute of Mining and Metallurgical Engineers. Industrial Minerals and Rocks. (Chapter on Mineral Pigments.)

DOLOMITIC AND CALCAREOUS CLAYS

(CONTAINS MORE THAN 15 PERCENT LIME, MAGNESIA)

Lime in some quantity is present in nearly all clays and shales. As a rule, clays containing 1 to 1½ percent lime are not very satisfactory for ceramic uses. Occasionally, a clay containing slightly more lime
than this is used in making a heavy-clay product such as brick, but this is exceptional. Hence, nonceramic tests only are indicated for the calcareous-type clays.

There is also the possibility of using such clays in making puzzolanic-type cements; however, no simple test involving the clay alone is known by which evaluation for this purpose can be made. According to a communication from John Tucker, Jr., chief, Cement Section, National Bureau of Standards, it is necessary to test the clays, "using them calcined or uncalcined, mixed with lime, and made into mortar or concrete test specimens, from which the strength is determined." Incorporation in a product in this manner takes such testing out of the scope of the present outline.

LIMESTONE AND DOLOMITIC MARLS

Marls are often mistaken for clays, as they may be quite plastic when mixed with water. The chemical tests for carbonates indicate them readily. No further testing is necessary. Marls may be of value in the cement industry, and reference to cement literature is suggested if the deposit appears to be large enough for exploitation.

PHOSPHATES

No further testing is necessary for the purposes of this outline.

HALLOYSITE; ALLOPHANE

In general, the trade does not use these claylike minerals, although some years ago the halloysites were employed to a limited extent in making alum. Most of the deposits are small, pocketed, and contaminated, making large-scale operation costly. Hence, their use has been extremely limited, and for the purpose of this outline no further tests are indicated.

CLAYS AND SHALES

The clays and shales used generally by the trade have relatively low iron and carbonate contents. When there is a deposit of either close to a portland cement plant there is a possibility that it will be used by the cement plant, provided chemical analysis of the clay or shale shows it to be suitable for combination with the particular limestone being utilized by the plant. If the analysis is favorable, further testing according to the outline is unnecessary for that particular purpose.

SHALES

These range from the soft, friable grades to a hardness merging into that of shale. The most general use of shales is for making ceramic heavy-clay products, such as building brick, structural tile, drain tile, and sewer pipe. Some deposits have a very high bituminous content, from which oil could be extracted. Such shales may be detected readily by holding a match under a sliver. If the shale contains much oil, it will catch fire. A high oil content does not, however, preclude its use in ceramics, and the shale should also be tested in the usual way.
CLAY

This term is meant to cover all clays used industrially, except the montmorillonites, and includes the ordinary surface, glacial, and aluvial clays, loesses, coal-measure or flint clays, fire clays, ball clays, kaolins, and china clays.

SANDY CLAYS

Any clay, whether of the common variety or of high grade, may contain considerable sand. If the sand content is around 50 percent or better, there are several choices.

If the clay is of the common type, reddish and coarse, and the sand is fine-grained, it may be used "as is" in making common brick and should be tested accordingly. Depending on the size and shape of the sand grain and certain properties of the clay substance, these sandy clays may also make good molding sands and clays. The tests for this purpose will be found in Testing and Grading Foundry Sands and Clays issued by the American Foundryman's Association, 222 West Adams Street, Chicago, Ill.

Where the clay is of light color or seems to have other desirable properties, such as excellent plasticity or fine grain, a sample should be washed well by dispersing, blunging, and allowing the sand to settle out. Then the clay yield is filtered out, dried, and put in line for further testing of the usual type. Some of the best clays on the market, such as Florida kaolins, are washed products from a material often containing 90 to 95 percent sand in the deposit. Furthermore, many clays of the commoner grades develop surprisingly good qualities if their sand load is dropped.

MAIN TESTS FOR CERAMIC AND NONCERAMIC USES

CERAMIC TESTS

Whatever may be the characteristics of a clay in the raw, plastic, and dried states, the final and determining test is the heat treatment. This must always be kept in mind. Undesirable impurities, such as sand, may be eliminated and corrections made for soluble salts, lack of plasticity, or undue shrinkage, but in the end the fired product must meet specifications. If this cannot be accomplished without undue cost, the clay is unfit for ceramic use. It must also be remembered that it is regular and standard practice throughout the ceramic industry to resort to mixing of clays to obtain desired results when necessary; hence, a clay should not be rejected necessarily because it makes a poor product of itself. If there is a possibility that the clay could be employed in a mixture, the fact should be noted. Judgment must be exercised under these circumstances. For instance, when a clay is obviously of such low grade that the only possible use for it would be for a cheap common brick and no other clay suitable for mixing with it is in the near vicinity, it would be out of the question to consider such clay as of any great value. For more complete tests for ceramic purposes see the June 1928 issue of the Journal of the American Ceramic Society.
OBSERVATION ON PLASTICITY, DRYING AND FIRING CHARACTERISTICS

PLASTICITY

The preliminary tests give an excellent idea as to relative plasticity. The report should be made in such terms as poor, good, very plastic.

DRYING AND FIRING CHARACTERISTICS

Lumps of the raw clay or hand-made shapes (cubes, bars, or cylinders) may be used. For quick work, to obtain a rough idea of what may be expected, lumps of clay are sufficient. However, it takes little time to make up a few bars by hand or by a simple extrusion apparatus. Such specimens will enable much more accurate observations to be made of shrinkage, warping, cracking, and strength. The reports may utilize such terms as “low,” “moderate,” “high,” and “excessive” rather than numerical phraseology.

It is rather important to note the drying behavior, particularly as to cracking, warping, and shrinkage. If this is excessive in the drying, trouble is likely to occur in the firing stage. If bars are made, linear shrinkage measurements to within one-sixteenth inch should be noted. Although a fair idea of relative strength may be obtained by breaking the dry bars by hand and the fired bars by a hammer, it is, or course, far better to break them in a machine for that purpose. A simple home-made apparatus is described in the Journal of the American Ceramic Society, June 1928, figure 514. For more elaborate equipment see Journal of the American Ceramic Society, volume 8, No. 11, November 1935, page 774.

FIRING TESTS AT CONE 4

Cone 4 is not a critical temperature, and a cone or two above or below usually makes little difference, but it is indicative of the action under heat treatment. It is slightly above the usual brick-kiln temperatures, hence gives a fair idea as to what to expect with reference to shrinkage, hardness, and color to a certain extent. Where the iron and other impurities are excessive, this temperature will expose the trouble to be expected. It must be remembered, however, that the laboratory kiln, with its quick schedule and lack of long soak, as well as difference in kiln atmospheres, does not duplicate closely what will happen under commercial conditions. Proper allowances must be made accordingly. It is suggested that the temperature be held at approximately cone 4 for 1 hour.

CLASSIFICATION BY FIRED COLOR

Only clays that show satisfactory characteristics in general during the cone 4 fire—that is, no excessive warping, bloating, or fusion—are included in this classification.

Very light color.—Here are to be classed the clays ranging from cream tints to whites. All the high-grade kaolins and ball clays fall into this group. It will be noted that sizing on the 325-mesh sieve is considered here. This is to separate out clays that may have possibilities in the enamel trade. In some instances, where the material is coarser, it may be beneficiated by processing to the required fineness. To be certain that a clay will meet the requirements for enamels, it is
best to incorporate it into an enamel and test as such. It is not within
the scope of this outline to include such tests, but they may be found
in Standard Specifications, Porcelain Enameling Supplies, by the
Porcelain Enamel & Manufacturing Co., Baltimore, Md.

In the ceramic whiteware trade (dinnerware and electrical porce-
lain are representative) all of these clays are used. No particular
attention need be paid to grain size here as a criterion, because the clays
may be processed to meet any particular demand of this sort. The
main factor is color, so far as the present outline is concerned.

Further evaluation of these clays for whiteware purposes requires
accurate observation of several different properties and the firing char-
acteristics at the usual whiteware heats of cones 10 to 14. Furthermore,
testing should be done by incorporation in whiteware bodies. For such
testing reference should be made to the publications of the
American Ceramic Society, Columbus, Ohio.

Most light clays are very refractory and often are used for such
purposes; they should be given P. C. E. tests (softening points).

Grays.—Some of the grays are light enough to be used in whiteware
bodies and should be tested accordingly. The darker grays and buffs,
however, are used mainly in stoneware, refractories, and heavy-clay
products.

Buffs.—Most clays used in the refractory industry are buffs. Any
of these buffs, whether refractory or not, usually are good for making
heavy-clay products. The refractoriness must be checked by P. C. E.
tests. (See P. C. E. Test.) They should also be given a salt-glaze
test. (See Salt-Glaze Test.)

Red and darker.—These clays all contain so much iron and other
impurities as to preclude their use as refractories. They are our main
source of supply for heavy-ware products.

Certain types of heavy ware, such as sewer pipe and electrical con-
duct, are salt-glazed. This ware is made mostly of clays of the red-
burning type, but buffs also are used. Hence, buffs, reds, and darker
colors as well should be given the test. The best test is to place a
sample within a regular commercial kiln where salt-glazing is em-
ployed. If this is not available, a small furnace may be constructed
from a few firebrick and an oxyacetylene torch. (See appendix A.)

CLASSIFICATION BY BODY STRUCTURE AFTER FIRING TO CONE 4

Includes all the samples that look unsatisfactory owing to the fol-
lowing defects:

Excessive warping, cracking, shrinkage, or scums.—If the color is good, and
especially if it is light or buff, it may pay to mix with other clays or correct
the defects by beneficiation. With the average red clay, this usually is not
worth while.

Bloats.—If the bloating is generally even and regular, with bubbles or blebs
of about the same size structure, there may be possibilities for use as a light-
weight aggregate. However, this industry is more or less localized and has not
developed on a widespread scale, so that, in general, bloating of a sample is
cause for rejection for ceramic purposes.

Fuses to a glass or slag.—The so-called slip clays, such as Albany and Michi-
gan slip clays, fuse to a glass or slag at cone 4. These clays develop character-
istic colors at various temperatures, and experience with them is necessary to
be sure of them. Slip clays are not widespread, hence most samples that fuse
down at cone 4 are to be viewed with suspicion. They are probably worthless.
If in doubt, another test should be made, using an Albany slip-clay sample as a
check for comparison.
SYLLABUS OF CLAY TESTING

P. C. E. TEST

The American Society for Testing Materials procedure (C24–35), using a standard furnace, may be followed if desired. However, an oxyacetylene furnace described in appendix A will be found adequate in most instances.

PROCEDURE

A small sample of air-dried clay is ground through 65-mesh. About 10 grams of the clay is mixed with enough 5-percent solution of dextrin, or water alone if the clay is plastic, to make a plastic mass. Cones similar to small pyrometric cones are made in the usual manner in a mold (shown in appendix A). The cones are air-dried and then dried at 110° C. before setting. The cone plaque may be made from fire clay or by mixing 50 percent of minus-120-mesh alumina and 50 percent kaolin with enough water to form a plastic mass. A 1-by 2-inch pat ¾ inch thick is a convenient size for the furnace. Six cones, in two rows of three, are mounted in a pat. Standard cones are placed on the corners, and the unknown cones are placed in the middle of each row. The cones are placed with one face parallel to the outside edge of the plaque and set at an angle of 82°, pointing toward the side. The pat and cones are air-dried and then dried at 110° C., after which they are ready for the P. C. E. test.

INTERPRETATION

Between cones 4 and 19.—The uses here indicated are for stoneware and heavy-clay products.

The refractories, which lie between cones 19 and 31 and above.—For the various specifications and requirements see the manual of American Society for Testing Materials, Philadelphia, Pa.

SALT-GLAZE TEST

A sample of air-dried clay is ground through 20-mesh. About 1 kilogram of the clay is mixed with water, wedged, and made into standard test bars. The bars are air-dried, then dried at 110° C. for 12 to 24 hours, and finally fired to cone 4. These bars are then ready for the salt-glaze test.

The furnace used is described in appendix A.

The bar of clay calcined to cone 4 is inserted into the furnace, and heat is applied. A pinch of salt is introduced from time to time to determine when the temperature is high enough for glazing to be begun. When salt fumes are visible coming through the flue, salt is added at short intervals for about 5 minutes. The bar is then withdrawn, and another may be inserted. The glazing is readily evident by the appearance of the bar.

NONCERAMIC TESTS

MONTMORILLONITES

The montmorillonite types are identified in the preliminary tests. There are three types of interest industrially—the swelling-type bentonite, the nonswelling or so-called metabentonite or subbentonite, and the fuller’s earths. The swelling-type bentonite will be indicated
in the preliminary testing, but the other two types are not so easy, and Nutting's \(^9\) method, by means of slaking, is used. The slaking test has been used in ceramic testing, also, as it is an indirect measure of bonding power and grain size; but it is not used in the present syllabus for anything but the montmorillonites, as it has been found that many kaolinite-type clays act in the same way as the metabentonites and fuller's earths. Some slake with granules, and some chip and crack. Hence, to avoid confusion, the test is used for identifying the three montmorillonites only.

**SLAKING TEST**

A 25-gram lump of clay is dried at 105° C, and placed in a beaker containing 250 ml of hot water. It is allowed to stand 1 hour. The clay is then screened on a 100-mesh sieve and washed to remove the undersize. The oversize is dried and weighed, and the undersize is determined by difference. Inspection of the oversize and the percentage of minus-100-mesh material are indicative of the type of montmorillonite. No slaking or slaking of the sample to coarse flakes with less than 25 percent minus-100-mesh suggests a fuller's earth. Slaking to small grains not much coarser than 100-mesh and 25 percent or more minus-100-mesh indicates a metabentonite. The swelling montmorillonites and hectorite are identified by complete slaking and formation of a gel. The hectorite may be distinguished readily from the swelling montmorillonite by a qualitative test for magnesium.

*Test for magnesium.*—A mixture of 1 part of finely pulverized clay and 3 parts of sodium carbonate is fused in beads over a bunsen burner in a double loop of platinum wire. Several beads are prepared so as to have an adequate sample for the magnesium test. The beads are dissolved in dilute nitric acid (1 HNO\(_3\) : 1 water), and the solution is evaporated to dryness. The residue is redissolved in a small amount of nitric acid, and the iron and aluminum and the calcium are removed in successive precipitations with ammonium hydroxide and ammonium oxalate, respectively, and filtrations each time a precipitate occurs. To the clear filtrate sodium phosphate, Na\(_3\)HPO\(_4\), is added. If magnesium is present, a crystalline precipitate of magnesium ammonium phosphate, NH\(_4\)MgPO\(_4\).6H\(_2\)O, forms.

The slaking test should not be regarded as infallible in classifying the fuller's earths or naturally active bleaching clays and the metabentonites or activable bleaching clays. In either case, a bleaching test should be made for confirmation.

Numerous types of oils are bleached industrially, and both contact and the percolation methods are used. The choice of natural bleachers or activated clay and the choice of the two methods of bleaching depend on the trade requirements for the finished product and the suitability of the clay in plant operation. As an illustration, some clays impart an undesirable flavor to edible oils, some are difficult to filter, and some crush readily, so that the losses in fines during reactivation are high. Aside from these objections, clays that are good bleachers may be used interchangeably. For simplicity in evaluating clays, the contact process, using an unbleached, refined cottonseed oil, is used.

**BLEACHING TEST**

A sample of air-dried clay is ground through 150-mesh and dried for 1 hour at 150° C. A refined, unbleached cottonseed oil is used.

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for bleaching. The amount of oil used is equivalent to 100 cc. for each 5 grams of clay. (As little as 3 grams of clay may be used.)

In the test, 100 cc. of oil is stirred by a mechanical mixer and heated to 120° C. A 5-gram sample of clay is added to the oil, and the mixture is stirred 5 minutes. During stirring the temperature is not allowed to fall below 105° C. or to rise above 120° C. At the end of the 5-minute treatment period the sample is filtered, and the clear filtrate, after cooling, is ready for color comparison. If the first filtrate is turbid, the oil is returned to the filter until a clear filtrate is obtained. For comparison of color two standard samples of oil are prepared in a similar manner. One standard is prepared with an official fuller's earth and the other with a clay, such as one of the Georgia kaolins, that is not naturally active and is not activable. For classifying the bleaching power of the clay, 4-inch columns of the oil in flat-bottomed test tubes 5 inches in height and 5/8 inch in diameter are mounted in a Nessler stand with a white, opalescent glass reflector at the bottom. The colors of the bleached-oil samples are compared by observation through the depth of the column. Thus the bleaching power of the clay may be classified readily. If the color of the oil sample is as light or lighter than that bleached with the official fuller's earth, the clay is a natural bleacher. If the color of the oil is darker than that bleached with the kaolin, the clay is not satisfactory for bleaching. On the other hand, if the color of the oil is between that bleached by the official fuller's earth and that by the kaolin the clay has bleaching properties that may be enhanced by acid activation.

However, after activation, clays vary in their ability to bleach. A measure of this cannot be predicted without a activation test.

**ACTIVATION TEST**

A sample of air-dried clay is ground through 150-mesh. A solution of 20 percent by weight of sulfuric acid in amounts equivalent to 50 cc. per gram of clay is used to activate the clay.

In the test a 12-gram sample of clay is digested with 600 cc. of sulfuric acid at 95° to 100° C. for 2 hours. The mixture is then filtered on a Büchner filter and washed twice with water. The filter cake is dried and ground through 150-mesh. The treated clay is then tested as described in the bleaching test. The color of the bleached oil is compared with that bleached by the clay before acid treatment and with that bleached by the official fuller's earth.

If the color of the oil bleached by the clay after activation is as good as or better than that bleached by the official fuller's earth, the clay is an activated bentonite. If the color of the oil bleached by a clay before activation closely resembled that achieved with official fuller's earth, and if after activation it is about the same or only slightly lighter in color, the clay is a fuller's earth. If the bleaching power of a clay only slightly better than that of kaolin is not increased so as to equal an official fuller's earth, the clay is probably contaminated with impurities. Such impurities as lime would consume acid and prevent acid activation under the same conditions as for the other clays; likewise, a large proportion of an inert diluent

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10 Supplied by American Oil Chemists Association, 500 Poydras Street, New Orleans, La.
would lower the effective bentonite per unit of weight. Thus, even after activation the impure activable clays are still low-grade bleachers.

From the information derived from the slaking tests the uses of clays are apparent. The swelling bentonites are utilized in drilling muds for their thixotropic properties, by which the cuttings are suspended when the mud sets after drilling ceases. The swelling bentonites also are used for foundry sand bond and plasticizers. The natural bleachers or fuller’s earth and the activable bleachers or metabentonites are used principally for bleaching oils. Low-grade or impure montmorillonites may possibly be beneficiated and used for the uses mentioned above; otherwise, their principal use is in drilling muds or as foundry sand bonds. The use of hectorite, an unusual type of clay in the developmental stage, at present is limited to drilling muds as a substitute for swelling bentonite and as a bond in molding sands.

CLAYS AND SHALES

The evaluation of clays and shales for nonceramic uses depends to some extent on their slakability and particle size. The sizing test (see Preliminary Testing; Particle Size) classifies clays that contain largely minus-325- and minus-200-mesh material. Clays chiefly finer than these sizes have one of the properties essential to coating and filler clays of various grades. Rubber clays are minus-325-mesh, and paper-coating and filler clays are finer than 200-mesh, but size alone is not sufficient indication of the satisfactoriness of the clay for these uses. The clays must have certain water-settling characteristics as well.

WATER-SETTLING TEST

A slip of 25 grams of clay and 225 cc. distilled water is agitated in a high-speed mixer for 7 minutes. At the end of the stirring operation the slip is transferred to 100-cc. Nessler tubes or graduates. The tubes are up-ended 10 times, then the suspensions are allowed to settle for 48 hours.

The quantity of clear liquid on the top, the quantity of sediment, and the quantity of suspended material are noted every 15 minutes for the first 2 hours and again at 6 hours. After 48 hours the quantity of suspended material is noted.

If the clay is minus-325-mesh, the amount of suspension after 48 hours’ sedimentation 12 is indicative of the different types of rubber clay. From 70 to 80 percent sediment suggests hard reinforcing clays; 58 to 68 percent, slightly lower-tensile reinforcing clays; 40 to 45 percent, high-grade soft clays; and 20 to 35 percent, low-tensile soft clays. Even traces of soluble manganese and copper, however, are objectionable in rubber clays, and their absence must be confirmed.

Test for manganese and soluble copper.—A 10-gram sample of air-dried clay is crushed through 10-mesh and extracted with 50 cc. of 0.25-percent normal ammonium acetate solution. (This solution is prepared by dissolving 10 grams of ammonium acetate, adding 19 cc of glacial acetic acid, and diluting the volume to 1 liter. When the solution is cool, the pH is adjusted to 5 by the addition of either acid or alkali, as required.) The suspension of clay and ammonium acetate solution is stirred to break up granules and is allowed to

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stand 10 minutes from the time of addition of the ammonium acetate solution; the mixture is filtered, and the filtrate, which is usually clear, is used for the qualitative tests.

The manganese is determined by the benzidine test, as follows:
A 5-cc. portion of clay extract is placed in a test tube and first mixed with 5 drops of 5-percent sodium hydroxide solution, then with 5 drops of benzidine solution (2 grams of benzidine in 100 cc. methyl alcohol), and finally with 5 drops of acetic acid (1 acid: 3 water). The test tube is agitated, and if a blue color forms, manganese is present.

Copper is determined in the following manner:
10 cc. of the clay extract is tested with excess ammonia to determine the presence of iron. If ferric hydroxide forms, the precipitate is removed by filtration. The filtrate is then acidified with acetic acid and treated with potassium ferrocyanide solution. A red precipitate, either in suspension or settled, denotes the presence of copper.

Clays that are suitable rubber fillers may also be suitable for other purposes. Likewise, clays that are unsatisfactory for use in rubber on the basis of the tests for copper and manganese may still be satisfactory coating and filler clays. In the settling tests, if the clays are dispersed, the amount of sediment at the end of the 15-minute intervals and the 2- and 6-hour intervals is a gauge of the nonclay- or clay-aggregate content. If the amount of sediment at the end of 6 hours is little more than at the end of 15 minutes, and both occupy only a small part of the clay column, the clay should be tested for coating and filler uses. On the other hand, if the clays are flocculated in the settling tests and settle at the end of 2 hours to large volume with a clear supernatant liquid, fine particle size may be indicated, and they still may be satisfactory coating or filler clays. To determine this, a dispersion test is necessary.

**DISPERSION TEST**

A slip of 10 grams of clay, 0.005 gr. sodium hexa-metaphosphate, and enough water to make a volume of 100 cc. is prepared in a sedimentation graduate. The suspension is agitated at intervals over a period of 4 hours until the clay is slaked and dispersed. After the suspension is dispersed, the graduate is up-ended 10 times and allowed to settle. The sediment is noted after 1 hour and after 6 hours.

If the same amount of sediment is present at the end of 1 hour as after 6 hours, and if at both time intervals it is only a small part of the clay column, the clay should be tested further as a coater or filler.

In the selection of clays as coater or filler, the coarsest limiting size is not as important as the mean size of the clay. A minus-200-mesh clay may be either granular or colloidal in size. The former type may be a filler, whereas the latter has a desirable property as a coating clay. A rapid means of evaluating clays in this respect is the rub-out test.

**RUB-OUT TEST**

A sample of 25 grams of clay is mixed with 25 cc. of warm, distilled water in a small evaporating dish. The samples are allowed to slake for about 4 hours. While the clay is slaking, the mixture is stirred with a spatula from time to time, and finally it is stirred to as smooth a paste as possible. The paste is applied to white blotting paper with a spatula and quickly spread to form a smooth, even coating.

Rub-outs of a typical filler and coating clay are spread on the blotter in juxtaposition to the clay being evaluated. The similarity of the
sample to either the filler or the coating clay can be seen readily. The presence of specking material, grit, or hard particles of clay may be detected either, with the eye alone or with a magnifying glass. Even slight differences in the color or cast of the wet and dry clays are apparent in this comparison. Likewise, the particle size of the samples is apparent from the gloss. Even though two samples may be off color, the gloss indicates fine particle size, which in turn would indicate a coating clay if the color were satisfactory.

If the sample has low gloss and is off color, it may be of use as a textile filler; if it is white, it may be a paper filler; if the sample has high gloss and is white, it may be a coating clay; if it has high gloss but is off color, a bleaching test is advisable to distinguish it from a fine, textile, filler clay.

**BLEACH TEST**

A slip of 200 grams of clay and enough water to make a slip of 32 to 35 percent solids is prepared by slaking the clay in a beaker and vigorously agitating the mixture. To the slip are added 0.75 gram of H$_2$SO$_4$, 0.25 gram of powdered zinc (95 to 97 percent minus-300-mesh), and 0.6 gram of NaHSO$_3$, in the order named. The slip to which the solution is added is agitated vigorously and allowed to stand 3 hours. The clay is then filtered on a Büchner filter. A rub-out on the bleached clay and with a standard coating clay readily shows the amenability to bleaching and indicates that the clay may be suitable as a coater. If the clay is not bleached, it is probably suitable for use as a textile filler or for other nonceramic uses.

**CLAY MINERALS OF ALL SIZES (FINE AND COARSE)**

In the classification of the montmorillonites by the slaking test, the swelling bentonites were classified, and their use in drilling muds was indicated. Other montmorillonites, as well as the other types of clay, may likewise be used in drilling muds. Roughly, drilling muds may be classified for three different purposes, named in the order in which the clays are most valuable and bring the best prices. They are swelling bentonites, fine-grained clays, and “back-yard” clay. The thixotropic clays give viscosity to the fluid to suspend the cutting and cause setting when drilling has ceased, which prevents the cuttings from settling out. In addition, these clays are useful for wall building; that is, a thin water-impermeable coating is formed around the hole to prevent loss of drilling fluid and thinning of the drilling mud by seepage water from the formation. The fine-particle clays have the wall-building and suspending properties to a smaller extent but are not thixotropic in their action. The back-yard clays are clays of various types that merely give consistency to the mud. The fine-particle-size clays are enough more valuable than the back-yard clays to justify a simple test such as the filtration test.

**FILTRATION TEST**

The air-dried clay is blunged with enough water to make a slip of thick consistency but readily pourable. A slip of 30 to 40 grams of clay and 70 to 60 cc. of water is usually satisfactory. A gob of this slip is placed on a filter paper, and the wetting of this paper is a
measure of the rate of filtering of the clay. Marks one-fourth inch apart are first made on the paper, and the mud is then placed three-fourths inch away from them to allow a long enough interval to time wetting between the marks. Measure of the time required for the water to creep in a certain direction is timed. If one-half minute is required to travel one-fourth inch, the filtration rate is too rapid; if 1 minute is required, the rate is fair; and 2 minutes is indicative of a good mud.\textsuperscript{12} Clays that do not have a satisfactory rate of filtration may still be usable in drilling muds as inert fillers.

The clays, other than the montmorillonites, may be usable for other purposes than is shown by the syllabus, and any one may be suitable for more than one use. Classification of the clays by raw color at this point is of value; very light colored, white, cream, or gray clays may be suitable for chemical uses. The iron content determined by the usual chemical method will give a clue to this. If the iron is less than 0.5 percent $\text{Fe}_2\text{O}_3$, the clay may be used in making ultramarine; if less than 1.5 percent $\text{Fe}_2\text{O}_3$, in alumina; and if less than 2 percent $\text{Fe}_2\text{O}_3$, for the manufacture of alum.

In conclusion, clays classified by the syllabus for certain uses may be satisfactory for other purposes as well. The syllabus covers the classifications representing the principal tonnages of clay consumed in the industry. Consideration of other uses would only be justified in a more extensive syllabus.

\textsuperscript{12} Stern, A. G., Role of Clay and Other Minerals in Oil-Well Drilling Fluids. \textit{Bureau of Mines Rept. of Investigations} 3556, 1941, 88 pp. (See p. 42.)
APPENDIX A

EQUIPMENT AND SUPPLIES

Most of the testing can be carried out with the apparatus and reagents to be found in the ordinary chemical laboratory. Virtually all of the equipment may be bought from laboratory supply houses. In some instances home-made apparatus serves quite well and may be put together at much lower cost. The following items are certain to be needed for most of the work:

GOOD MECHANICAL HIGH-SPEED MIXER

The handiest type is that consisting of a motor and propeller unit, which can be fastened to a jar or other container and can maintain vigorous agitation of 2 or 3 gallons of clay suspension. For small samples a malted-milk mixer is very handy.

SET OF SIEVES

Screens built according to different standards are available. For the type of testing involved it does not matter which standard is used. However, it will be found that sieves of 20-, 60-, 100-, 200-, and 325-mesh size are in quite common use for evaluating clays. The screen scale and size of opening are matters of individual preference. In making tests, however, one should not mix standards or sizes or switch from one to another.

DRYING OVENS

A drying oven operating at 105° to 110° C. will be used constantly. Occasionally there is need of one capable of maintaining a temperature of about 600° C.

EQUIPMENT FOR MAKING TEST SPECIMENS FOR CERAMIC TESTS

Although lumps of the crude clay often are used, it is best to have specimens of standard size and shape. For years the standard specimen has been a bar 1 by 1 inch in cross section and 7 or 8 inches in length. However, many, including the authors, prefer a short, round specimen about 1/2 inch in diameter by 2 1/2 inches in length.

The square bars may be made by hand-pressing the clay \(^{23}\) in a steel or brass bar mold. It is possible to purchase such molds, but one can be made quite easily. (See fig. 4.) The hand-made bar is slow to make and not as uniform as it might be. It is frequently used when only a small sample is available.

For more rapid production, especially if a round specimen is wanted, it is best to use an extrusion-type machine, either hand- or power-operated. Most of those obtainable by purchase depend on an auger screw to push the clay through the die. It is possible to obtain such,

\(^{23}\) The clay is worked into a soft, plastic mass with the hands by adding water slowly while the clay is pressed and kneaded by hand. When the correct workability is reached, the mold is worked full of the clay and the excess clay clay is struck off, the specimen being smoothed over with a spatula. The mold is now inverted over a drying board, and the specimen is carefully placed on the board by pushing down on the movable bottom with the thumbs, at the same time pulling up the mold frame with the fingers.
as well as a de-airing device, which insures more uniformity and greater green strength. Sausage stuffers could be used, the piston type being especially good, as an auger tends to produce laminations. A hand-operated piston-type machine is shown in figures 5, 6, and 7. This is easily built in a machine shop and makes excellent specimens. The authors prefer a 1/2-inch round shape about 21/2 inches long. A square bar die may be used if preferred. Being of the piston type, there is a minimum of lamination. A tube inserted into the head and connected to a vacuum pump permits de-airing of the clay.14

![Figure 4.—Bar mold.](image)

EQUIPMENT FOR FIRING TEST SPECIMENS

Quite a variety of small laboratory kilns and furnaces is available in different sizes and for use with different fuels. For the testing involved in the outline, a small muffle (4 by 3 by 10 inches) will be found satisfactory, unless the volume of testing is large. It is quite possible, of course, for a laboratory to construct its own furnaces, and this is often done.15 Any such furnace should be capable of reaching 2,400°F.

14 To de-air, the pump is turned on, and a greased microscope glass slide is slipped over the orifice of the die. After an interval of about 10 to 15 minutes of de-airing, the pump is cut off and a column about 2 feet long is extruded onto a receiving board, and the column cut into the 21/2 inch lengths. When the column is being extruded on the board, if a strip of newspaper the length of the board is fed onto the board from below just under the clay column, the column will travel the board with so little friction that it will not be distorted. (See fig. 7.)

15 A number of descriptions of such kilns are to be found in American Ceramic Society literature. The following are typical:

Figure 6.—Hand-operated piston-type bar-molding machine.

Figure 7.—Hand-operated piston-type bar-molding machine.
Figure 8.—Oxyacetylene PCE furnace.

Figure 9.—Close-up of furnace shown in figures 8 and 10.
Figure 5.—Bar-extrusion machine, de-airing type. (Sketch not drawn to scale; dimensions indicated.)
Several firms make furnaces for this test. However, an oxyacetylene welder’s outfit and a few refractory brick make an excellent substitute for such testing as is involved in the outline. (See figs. 8, 9, and 10). For details of cone mold, see figure 11.

To carry out this test it is necessary to have a supply of standard cones for reference. For any given test it is customary to put in three of these standard cones—the one expected to be reached, the one below that temperature, and the one above. In making tests on unknowns, a range of four or five cones will save repeat runs. Cones may be obtained from The Edward Orton, Jr., Ceramic Foundation, Columbus, Ohio; H. B. Henderson & Co., East Liverpool, Ohio; and Accurate Pyrometric Cone Co., Pataskala, Ohio.

**Furnace for Salt-Glaze Test**

Furnaces of this type are not on the market. The one shown in figure 12 is easily put together and will roughly indicate the possibilities of the clay for taking a salt glaze. It may be made of any refractory brick. The port brick and the top brick may be made conveniently from insulating brick. An ordinary oxyacetylene burner is used. A bar of clay known to salt glaze well should be tested and kept for a reference. Only a spot on the bar will glaze.

NOTE: Make 1, shellac finish

WOOD BASE

NOTE: 2 pieces, 1 right and 1 left. Finish all faces

FIGURE 11.—Standard two-piece cone mold.
Figure 12.—Salt glaze testing furnace.
APPENDIX B

To clay technologists familiar with the various types of clays used commercially, little difficulty is likely to be experienced in following the syllabus; in fact they are likely to short-cut a good deal. Inexperienced operators, however, will find some additional information in the way of common trade names for commercial clays very helpful. It is suggested that the syllabus will become a much more useful tool for them, in a much shorter time, if reference samples of typical commercial clays are obtained. Then the syllabus should be followed, using such samples, before any attempt is made to evaluate any unknowns. Some of each reference sample should be kept to refer to later, until an operator is thoroughly familiar with the various clay types.37

When we talk about “clay types,” we find more than one classification for the same clays, and an operator should be familiar with all to obtain the best results. What might be termed the fundamental classifications are those laid down by geologists and mineralogists.

The geological classes refer to the manner of formation, such as surface, alluvial, glacial, residual, sedimentary, and equivalent terms. The mineralogical classifications refer to the internal crystalline structures and include such terms as kaolinite, montmorillonite, halloysite, allophane, and others.

In clay literature, as well as in commercial parlance, a mixture of all these names will be found, as was noted in the introduction of the outline. To make matters still more confusing, a single clay is often referred to in terms embracing two or more classifications at the same time. For instance, such a clay as one used by the paper and fine-ceramics trades may be referred to as a “white-burning, plastic, sedimentary kaolin.” Here four different classifications are involved. First, “white-burning” refers to a classification based on heat treatment, “plastic” refers to a property of the clay in its raw state, “sedimentary” dips into geologic history, and “kaolin” gives a clue to the mineral structure. And, again, this clay may be called a “china clay” by the potter using it for making chinaware, or a “paper clay” by a paper maker. It is hoped that the following notes will help inexperienced operators in this confusing matter of mixed terms and classifications.

SHALES

The term “shale” is geological and signifies a type of rock. At the same time it is a type of clay that, in the process of geological formation, has been subjected to heat and pressure and as a result is quite hard and rocklike as compared with a soft clay. If, however, it is not

too hard (slaty), it may be crushed and ground and, on mixing with water, may be used like any ordinary clay.

Nearly all shales burn red, and the main uses are the same as those of the red-burning surface, glacial, and alluvial clays—that is, to make what the outline terms “heavy-clay products.” This term includes such commodities as building brick of all types, sewer pipe, drain tile, roofing tile, structural hollow tile, and electrical conduit. Shale samples may be obtained from some nearby firm making heavy-clay products from shale.

**MONTMORILLONITES**

The classification here is based on the mineral found predominantly in these types. The three principal types of materials included under this term are the swelling bentonites, nonswelling bentonites (sometimes called subbentonites or metabentonites), and fuller’s earths. There is no trade term covering all three materials. Some of the trade is inclined to use the term “fuller’s earth” to designate both the ordinary type of fuller’s earth and the nonswelling bentonites, as both are used in bleaching oils. Others dub all clays of this type “bentonite.”

Although there is some disagreement among technical men over the origin, the majority seem to think that most of these deposits are altered or weathered volcanic ashes. The degree and type of weathering seem the main factors in determining swelling, nonswelling, and natural-leaching characteristics. However, all three seem to have one thing in common—the mineral or lattice structure shown by X-ray analysis and thermal studies. All seem to have crystallized mostly in a montmorillonite mineral structure or similar type. As most of the uses of these materials have developed fairly recently, use of the term “montmorillonite” is appearing in patents and current trade literature. Hence, it is thought both convenient and in keeping with the trend to classify the three types under one heading.

Although some attempts have been made to incorporate montmorillonites in ceramic bodies, this procedure has not been adopted to any great extent. The principal uses are in molding sands and oil bleaching. It will be noted that the tests in the outline for the montmorillonites are all of the nonceramic variety. The slaking test differentiates the three main classes.

**SWELLING BENTONITES**

Principal uses are in molding sands for castings (principally steel) and in drill muds. At present the main commercial deposits are in Wyoming and South Dakota. Samples may be obtained from the American Colloid Co., Chicago, Ill.

**NONSWELLING BENTONITES**

The two principal uses for this type are in molding sands and oil bleaching. The main difference between this type and the regular fuller’s earth is that the fuller’s earth will bleach oils without receiving any previous treatment, whereas the bentonite must be activated with acid. The main commercial supplies are from Mississippi, and

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amples may be obtained from the Filtrol Corporation, Jackson, Miss., and the Eastern Clay Products Co., Eifort, Ohio.

**FULLER'S EARTH**

Clays of this type were used originally in textile work as absorbents. The main use now is in oil bleaching. The main sources of supply in this country at present are in Georgia and Florida. Samples may be obtained from the Attapulgus Clay Co., Attapulgus, Ga., and the General Reduction Co., Macon, Ga.

**CLAYS**

This heading includes all clays of the usual type employed in manufacturing ceramics and for such nonceramic uses as paper and rubber rollers. The predominant type mineral is kaolinite as contradistinguished from montmorillonites, illites, halloysites, and other clay minerals. The terms designating these clays have been in use for many years, and, as would be expected, reflect properties or uses rather than mineral structures.

**COMMON CLAYS**

This term will not be found in the outline but is one frequently heard in the trade. In general, it refers to the clays used in making the so-called heavy-clay products, such as brick and the various structural units, sewer pipe, etc. It includes virtually all clays burning to "off" colors, principally reds, but sometimes includes buff-burning, which under other classifications are often referred to as refractory or fire clays. Samples of common clays are usually obtainable from the nearest brickyard.

In origin, most of the red-burning clays are surface or alluvial deposits or, in many sections, glacial deposits. The buff-burning clays usually occur under coal measures and are frequently referred to as "coal-measure clay."

While the principal uses of these common clays are in the manufacture of heavy-clay products, there are many minor applications. A great deal of art and garden pottery, terra cotta, and old-type sanitary ware is made from such clays.

**REFRACTORY CLAYS**

Here, again, the above term, while not in the outline, is common in the trade. Although certain white-burning clays employed for fine ceramics are refractory and are sometimes used in refractories, the trade usually means the buff-burning clays. Here are to be found various trade names. The term most generally used is fire clay, which gain may be a plastic fire clay or flint fire clay. There are also other designations, such as "pot" clays, when used for making pots for glass melting, "retort" clays, "crucible" clays, etc. The term "bond" clay is commonly employed in the refractory trade to mean a plastic, sticky clay, which is good for forming and maintaining the strength of the ware, particularly in the green state. Since the "ball" clays used in whiteware are often utilized by the refractory trade as well, the term "bond" not only applies to the strong, plastic, buff-burning clays
but to the whiter-burning “balls.” The “balls” will be discussed under the heading Whiteware Clays.

It should be remembered, however, that, by and large, the majority of refractory clays are those associated with coal measures. These are usually called fire clays. When the clay is fine-grained, flinty, and hard it is called a “flint” fire clay. The trade classifies the fire clays further by the amount of heat and degree of temperature required to soften them.

In the literature of some years back, fire clays were referred to as No. 1, No. 2, and No. 3. In reverse order, the No. 3 clay was good to temperatures between cones 19 and 26 (2,750° F. to 2,900° F.), the No. 2 was good between cones 26 and 31 (2,900° F. to 3,050° F.), and No. 1 above cone 31 (3,050° F. and higher). Recent trade talk refers to about the same ranges, but in terms of “low heat duty,” “intermediate heat duty,” “high heat duty,” and “super duty,” the latter term meaning products above cone 34 (3,200° F.). These terms refer specifically to the refractory products, but the clays making them may also be referred to in the same manner.

The main producing districts are New Jersey, Pennsylvania, Ohio, and Missouri. In obtaining samples, it would be well to have a plastic fire clay and a flint fire clay. Samples may be obtained from the United Clay Mines Co., Trenton, N. J.; Harbison-Walker Refractories Co., Pittsburgh, Pa.; Laclede Christy Clay Products Co., St. Louis, Mo.; A. P. Green Fire Brick Co., Mexico, Mo.; and Ironton Fire Brick Co., Ironton, Ohio.

**STONEWARE CLAYS**

This term is not used in the outline but is seen in literature frequently. By stoneware the trade usually means such products as crocks, churns, jugs, jars, steins, and some types of artware. A special, limited product is called chemical stoneware.

The clays used are frequently the so-called common clays that happen to be of the correct degree of refractoriness for the stoneware fire, which is about cone 8 (2,230° F.). Although an increasing number of buff-burning clays are used, the real stoneware clay should burn to a fairly dense body with a gray tone. The term is more or less dying out, as that type ware is not in great demand now. The chemical stoneware is a body mix using red- or buff-burning clays with feldspar added to give the required density.

It is not necessary to obtain samples of this type of clay, as it will be covered by samples of the usual buff-burning fire-clay varieties.

**WHITeware ClAYS**

For making dinnerware or electrical porcelain, two types of clay are used in the body mix and one or two types for the kiln furniture. For the body, the clays are, of course, the white-burning varieties and in the outline show up in the “Firing Test at Cone 4,” under the heading “Satisfactory Color and Body Texture,” as light and fine-grained. For kiln furniture (that is, the containers for the ware, called saggers) the regular, refractory, buff-burning fire clays and sandy ball clays are used.
BALL CLAYS

These clays are very fine-grained and highly plastic. At white-ware heats they produce a dense, vitreous structure, which helps in the vitrification of the body. Their main function in the body mix is to give the plasticity needed in forming the ware and strength in the dry state, and, finally, to assist the feldspar or other fluxes in vitrification during the firing period. Owing to organic matter and other impurities, they tend to burn to a cream tint rather than white, and they shrink and warp more than the china clays.

Large amounts of English ball clays are sold in this country. They are roughly divided into two classes, the light and the dark, the latter containing more organic matter than the light. Samples may be obtained from Moore & Munger, Plainfield, N. J., and the United Clay Mines Co., Trenton, N. J.

The main domestic producing fields are in western Kentucky and Tennessee. The “light and dark” division does not apply to domestic grades. Most would tend to be classified as “light” on the English basis, and in general the domestic balls are somewhat more refractory than the English. Samples may be obtained from H. C. Spinks Clay Co., Newport, Ky., and the Kentucky-Tennessee Clay Co., Mayfield, Ky.

CHINA CLAYS OR KAOLINS

These terms are really synonymous, in that the type of clay, properties, and mineralization are the same. In England it is customary to refer to them as china clays. Continental Europe calls the deposits kaolins, and this country tends to follow the same practice. The mineral kaolinite is found best developed in these clays.

They are not only white-burning at porcelain heats but are almost white (light creams and yellows) in the raw state. Generally, although not always, they are coarser-grained and less plastic than the ball clays and contain little or no organic matter.

In the raw state these clays supply the main tonnage for nonceramic uses, such as paper fillers and coaters, rubber fillers, and chemical and various other uses. They are, generally, readily processed to a dry powder, the most desirable form for the nonceramics trade.

From a geological standpoint, we have two classes—residuals and sedimentaries. The English clays and some of our domestic clays, notably the North Carolina kaolin, are residuals. The other main domestic supplies, all sedimentaries, are from Georgia, South Carolina, and Florida. In general, the domestic sedimentary kaolins tend to be a little more plastic than the residuals, but the color is not quite so good. They also contain a little more rutile, which gives a cream tint.

Samples of the English china clays may be obtained from The Paper Makers Importing Co., Easton, Pa., and Moore & Munger, Plainfield, N. J.

For domestic samples, some from each producing district should be obtained, as they vary somewhat.

Florida:

United Clay Mines Co., Trenton, N. J.
Edgar Plastic Kaolin Co., Metuchen, N. J.
SYLLABUS OF CLAY TESTING

Georgia:
Moore & Munger, Plainfield, N. J.
Edgar Plastic Kaolin Co., Metuchen, N. J.
R. T. Vanderbilt, New York City.
J. M. Huber, Inc., New York City.


South Carolina:
United Clay Mines Co., Trenton, N. J.
R. T. Vanderbilt, New York City.
J. M. Huber, Inc., New York City.

SAGGER CLAYS

Most of the so-called kiln furniture, such as saggers, slabs, etc.,
is made from ordinary fire clays or sandy ball clay, the only exceptions
being the pins and stilts used by the dinnerware trades. Also, in
recent years nonclay refractories have been used increasingly for heavy
kiln furniture.

The usual “sagger” body is generally a mixture of fire clays and
sandy ball clays, so far as the clay part is concerned. The ball-clay
deposits all have strata containing a good deal of sand, and these are
shipped as “sagger” clays or “wad” clays. The latter term applies to
a very plastic type of sagger clay having high tensile strength. Wad
clays are used as a jointing material between the individual pieces of
kiln furniture.

Samples of all the various types of sagger and wad clays may be
obtained from any of the ball-clay dealers.

NONCERAMIC CLAYS

PAPER AND RUBBER CLAYS

As noted in the description of the china clays or kaolins, these clays
are used almost exclusively in the two trades. Virtually all of the
paper clays are from Georgia, and samples may be obtained from the
Georgia producers of the ceramic grades. Some of the rubber clays
also are from Georgia and are sold by the same firms. However, most
of the rubber clays are from South Carolina, and samples may be
obtained from the firms listed under that heading for ceramic clays.

CLAYS USED FOR CHEMICAL PURPOSES

China clays are used in making ultramarines and alums.

DRILLING MUDS

The swelling bentonites are much used for this purpose, but all sorts
of other clays—of the commonest varieties, in fact—may be used to a
certain extent. The only test for such that can be given in the outline
is the filter test. That is, whatever the clay used, it must not lose its
mechanically held water too rapidly.

MOLDING-SAND CLAYS

In the past most casting molds for metals have been made of what
is termed natural molding sands. These are really very sandy clays.
However, in recent years use of so-called synthetic molding sand has
increased. This is a mixture of clean sand with certain clays. Al-
though various clays of the kaolinitic-mineral type (not china clays or kaolins, but rather the fire-clay type) have been used, a great demand for the swelling and nonswelling bentonite has developed.

Since all kinds of sandy clays are brought in for inspection and not many are really suitable as molding sands, it is well to have some reference samples of natural molding sands. About three grades should be obtained—one for very light castings, one for intermediate castings, and one for very heavy castings. Such samples may be obtained from Hougland & Hardy, Evansville, Ind.

Samples of the clays used in the synthetic mixtures may be obtained from Hougland & Hardy, Evansville, Ind.; Illinois Clay Products Co., Joliet, Ill.; F. E. Schundler, Joliet, Ill.; American Colloid Co., Chicago, Ill.; and Eastern Clay Products Co., Eifort, Ohio.