
CHEMISTRY OF CLAYS

BY

J. B. WEEMS.

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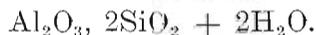
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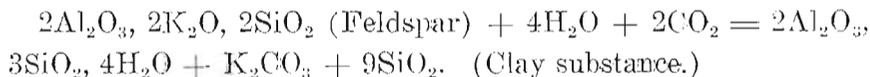
CHAPTER V.

Chemistry of Rock Composition.

Clay is a substance which we know as a deposit which is extensively distributed over the surface of the earth. It is found almost everywhere but differs greatly in its purity. This deposit has been produced by the decomposition of various rocks. As these decomposed products have been acted upon by water, the various original materials have been separated, leaving deposits of various kinds. Some of these we know, as sand, clay and various mixtures. Clay in its pure condition is comparatively rarely found, the larger portion of the deposit being composed of impure clay. Pure clay is a silicate of aluminum and when found in large quantities it is called kaolin. The various changes which take place upon the minerals present in the earth's crust, in order that clay may be produced is according to the results of Forchhammer shown in the formula



The changes which take place in the original rock to produce the clay substance may be shown by the following equation, as the result of the action of water and carbon dioxide upon feldspar.



As an illustration of the various changes which may take place during the decomposition of the original clay substance both in the mechanical as well as chemical changes, the investigation of Reichart⁽¹⁾ is of interest: The results under column (a) represent the original material. The second column (b) gives the results

(1) *Hanlwörterbuch der Chemie* Ladenburg 12, p. 17.

of the product produced from the weathering processes and the third column (c) the resulting kaolin.

| | (a) | | (b) | | (c) |
|----------------------|-------|---------|-------|---------|-------|
| Coarse sand..... | 33.95 | } 78.05 | 22.56 | } 72.11 | 2.48 |
| Fine sand..... | 36.20 | | 37.40 | | 28.52 |
| Finest sand..... | 7.90 | | 12.15 | | 18.42 |
| Clay..... | 9.27 | } 21.95 | 12.26 | } 27.89 | 20.51 |
| Fine earth..... | 7.46 | | 8.55 | | 17.69 |
| Silt..... | 5.22 | | 7.08 | | 12.38 |
| Silica..... | 77.48 | | 75.73 | | 76.48 |
| Alumina..... | 17.10 | | 21.92 | | 21.58 |
| Iron oxide..... | 2.83 | | 0.98 | | 0.97 |
| Magnesium oxide..... | 0.84 | | 0.18 | | 0.17 |
| Lime..... | 0.38 | | 0.27 | | 0.25 |
| Magnesia..... | 0.10 | | 0.10 | | 0.07 |
| Potash..... | 1.03 | | 0.55 | | 0.16 |
| Soda..... | 0.13 | | 0.08 | | 0.01 |
| Phosphoric acid..... | Trace | | | | |

The resulting product from this decomposition is naturally a complicated mixture and it is necessary to study it not only from a chemical point of view but also its physical conditions. Such physical properties, as plasticity, naturally belong to the physical consideration of the subject.

It is of interest to note that as the result of the chemical changes taking place in the production of kaolin from the original rock that the amount of sand decreases while the clay substance increases. In the chemical results the amount of silica and alumina undergo only a slight change while ferric oxide, manganese oxide, calcium oxide (lime), magnesium oxide (magnesia), and the alkalis decrease as the results of the changes taking place in the products.

Pure clay substance is insoluble in dilute hydrochloric acid or nitric acid. Concentrated sulfuric acid decomposes it at 250° to 300° C. with the separation of sand and silica. Hydrofluoric acid in the presence of sulfuric acid decomposes the sand which may be present producing silicon fluoride which is volatile.

The fact that the clay substance with many of its impurities is acted upon by sulfuric acid enables the separation of the

clay substance from the sand or quartz. The action upon impure quartz of a dilute solution of sodium hydrate or sodium carbonate, furnishes means for separating the quartz from the amorphous silica. The action of sulfuric acid and the alkali has given rise to a method of analysis, which is known as rational analysis. Work of this nature has been carried on extensively by Aron and Seger. In some cases rational analysis is all that is necessary to enable one to determine, within certain limits, the relative proportions of quartz, clay and feldspar which may be present in the clay. For the determination of other substances, however, a complete chemical analysis is necessary.

The typical clay such as kaolin has the formula $2\text{SiO}_2, \text{Al}_2\text{O}_3, 2\text{H}_2\text{O}$. Its percentage of composition would be accordingly $\text{SiO}_2, 47.1; \text{Al}_2\text{O}_3, 39.2; \text{H}_2\text{O}, 13.7$. The impure clays contain feldspar such as orthoclase, $\text{K}_2\text{O}, \text{Al}_2\text{O}_3, 6\text{SiO}_2$, or albite, $\text{Na}_2\text{O}, \text{Al}_2\text{O}_3, 6\text{SiO}_2$ and there will be also present mica having the formula $\text{K}_2\text{O}, 3\text{Al}_2\text{O}_3, 4\text{SiO}_2$ and quartz. As a natural result of the weathering processes the alkalis disappear and aluminum and silica may be washed away after it is hydrated. Some of the typical clays may be shown by the following analyses: ⁽²⁾

| | PURE KAOLIN. (Zettlitz). Per cent. | KAOLIN WITH QUARTZ. Per cent. | KAOLIN WITH FELDSPAR. Per cent. |
|-----------------------------|--|-------------------------------------|---------------------------------------|
| Kaolin clay substance | 96.6 | 63.8 | 55.9 |
| Feldspar..... | 1.1 | 0.7 | 38.2 |
| Quartz..... | 2.3 | 35.5 | 5.9 |

Kaolin clay substance containing:

| | PURE KAOLIN. (Zettlitz). Per cent. | KAOLIN WITH QUARTZ. Per cent. | KAOLIN WITH FELDSPAR. Per cent. |
|-------------------|--|-------------------------------------|---------------------------------------|
| Silica | 45.6 | 45.3 | 47.1 |
| Alumina | 38.1 | 37.1 | 36.0 |
| Ferric oxide..... | 0.9 | 1.3 | 0.6 |
| Magnesia | 0.7 | 0.8 | 3.3 |
| Alkalis..... | 1.8 | 2.0 | |
| Water..... | 13.3 | 12.1 | 12.9 |

(2) Ref. Chemistry for Engineers and Manufacturers, Vol. II, p. 143.

The clays of all classes are prepared for use in manufacturing and depend upon two physical properties: (1) The ease with which they may be molded and shaped when wet, and (2) their change of state to a hard, unalterable product when subjected to heat. The chemical composition has little to do with the first physical property. The second physical property of course is affected more or less by the chemical composition of the clay and as there is dehydration and agglomeration it is accompanied by shrinkage. The effect of impurities present in pure kaolin is to lower the fusing point.

CLASSES OF CLAYS.

The clays are generally divided into three classes:

(1) Porcelain clay, which is approximately pure kaolin. This burns to white or light-cream color.

(2) Plastic clay, which contains more impurities than the porcelain clay. It burns to a yellow-red color and is used for ordinary earthenware.

(3) Fire Clays.—These clays approach very close to the porcelain clays in composition, however they contain a larger quantity of iron, also more silica as quartz. Some of the highest fire clays are very rich in quartz. The typical fire clay and brick clay as found in England, contains the following substances:

| BRICK CLAY. | PER CENT. | FIRE CLAY. | PER CENT. |
|---------------------------|-----------|-----------------------|-----------|
| Quartz | 16.42 | Quartz | 84.59 |
| Feldspar | 3.27 | Combined silica | 5.11 |
| Calcium carbonate | 4.45 | Alumina | 5.40 |
| Calcium sulfate | .95 | Ferric oxide | 0.21 |
| True clay substance | 74.91 | Lime | 0.20 |
| | | Magnesia | 0.09 |
| | | Potash | 0.61 |
| | | Water | 3.74 |

Clay substance consisting of:

| BRICK CLAY. | PER CENT. |
|--------------------|-----------|
| Silica | 46.96 |
| Alumina | 36.42 |
| Ferric oxide | 2.80 |
| Magnesia | 0.87 |
| Alkalis. | 0.91 |
| Water | 12.04 |

OBJECT OF RATIONAL ANALYSIS.

The rational analysis of clay has for its object the separation of the clay into clay substance, quartz and feldspar. The methods used depend upon the action of sulfuric acid upon the clay. The remaining residue contains amorphous silica and quartz. The amorphous silica is then separated from it by the action of sodium carbonate or sodium hydroxide. As to the value of the rational analysis of clay, there seems to be a difference of opinion upon the subject. For example, Ries considers that it is necessary and customary for the manufacturers of porcelain or earthenware or fire brick or any product made from high grade clay to use the rational analysis as a guide for their mixtures in order to obtain a constant product. It is claimed that the advantage of this method is that it separates the clay into middle components and enables one to obtain an idea of the physical character of the material. This is claimed to be, frequently, a matter of far greater importance than is the chemical composition of the clay. It is claimed that the ordinary ultimate analysis only considers the clay as a mixture of oxides and that these substances may be present as silicates, carbonates, hydrates, sulfates, etc. Silica, it is claimed, will decrease the shrinkage and up to certain limits of temperature increase the refractoriness. If, however, it is present in combination as feldspar, it acts as a flux and somewhat increases the plasticity.

In contrast with the value of the rational and ultimate analyses of clay it may be said that each has its value and cannot replace the other.

The value of the ultimate and rational analyses and what may be indicated by them, is stated by Ries, as follows:

OBJECT OF ULTIMATE ANALYSIS.

“The ultimate analysis may be used to supply information on the following points:

(1) The purity of the clay, showing the proportions of silica, alumina, combined water, and fluxing impurities.

(2) From the ultimate analysis we can form an estimate of the refractoriness of the clay, for, other things being equal, the greater the total sum of fluxing impurities the more fusible the clay.

(3) The color to which a clay will burn may also be judged approximately, for the greater the amount of iron present the deeper red will the clay burn, provided the iron is evenly and finely distributed, and an excess of lime is not contained in the clay. If the proportion of iron to lime is as 1 to 3, then a buff product results, provided the clay is heated to incipient fusion or vitrification, the above condition will be affected by a reducing atmosphere in burning, or the presence of sulfur in the fire gases.

(4) Clays with a large amount of combined water sometimes exhibit a tendency to crack in burning. This combined water would be shown in the chemical analysis.

(5) A large excess of silica would indicate a sandy clay."

These are practically all the points which the ultimate analysis explains, and they are mostly of a chemical nature.

METHODS USED IN RATIONAL ANALYSES.

As regards the rational analysis it may be carried out in a simple way or in an elaborate one.

Most kaolins and other high grade clays consist only of kaolinite, quartz and feldspar, the kaolinite forming the finest particles of the mass while the balance is quartz, feldspar and perhaps some mica. The finest particles are known as the clay substance, which may be looked upon as having the properties of kaolinite, for the latter is present in it in such a large excess. Now as each of these three components of the kaolin—clay substance, quartz and feldspar—have characteristic properties, the kaolin will vary in its behavior as one or the other of these constituents predominates or tends to increase.

As to the characters of these three: Quartz is nearly infusible, non-plastic, has very little shrinkage, and is of low tensile strength; feldspar is easily fusible, and of low plasticity by itself; kaolinite is plastic and quite refractory, but shrinks considerably in burning.

Mica may practically be considered as kaolinite, for the reason that there is usually so little of it, and also because the experiments of G. Vogt have indicated that it acts very similarly to kaolin, being, when fine, fairly plastic and unaffected by a temperature of 1,300° C. In chemical composition it is of course different from kaolinite, but from a physical point of view it is similar.

The investigations of Vogt⁽³⁾ have called attention to the fact that in the cases where the analyses of clays were made with determinations of kaolin, feldspar, and quartz the results are not satisfactory. In the attempt, from the results obtained in the analyses, to imitate the porcelain of China and Japan the conclusion was reached that 50 parts of kaolin, 30 of feldspar and 20 of quartz would meet the requirements and upon this data the manufacturing of porcelain in Europe was founded. Vogt calls attention to the fact that in the rational analyses as carried out under these conditions, no attention was paid to the mica which may have been present. The experiment proved that a clay composed of kaolin 20, mica 25 and quartz 40 yielded merely stoneware of fine quality. In order to obtain the beautiful and transparent porcelain a quantity of feldspar was found necessary. The following mixture was prepared with this object in view: kaolin 25, mica 25, orthoclase 25 and quartz 25. In some cases, however, in the ordinary manufacture of cheaper ware and cheap clay products it is not deemed necessary to make the analysis complete as carried out by Vogt.

(3) Bull de La. Soc. Chim. d. Paris, Abst. Chem. News 62 (1890) p. 315.

From a scientific point of view, it is generally conceded that the results of the rational analysis is as stated by Doctor Bischof, "Ein Kuntschlich Theoretisches Bild."

This is due to the assumption that, in the calculation from the analysis, the mineral sand is regarded as feldspar. The separation of the mineral, quartz sand and clay substance cannot be regarded in any way as an accurate analysis.

Langenbeck⁽⁴⁾ in speaking of the analytical results obtained from the treatment of the feldspar in the same manner as the clays during the analytical process, states that pure feldspar left a residue of 81.44 per cent and in the other 83.95 per cent. Attempts have been made to remedy the action of the concentrated sulfuric acid upon feldspar, by treating the clay with dilute sulfuric acid under pressure. Yet it was found that the clay was only partially decomposed while the feldspar was acted upon to the extent of leaving a residue of 82.67 per cent which is practically the same as when treated with concentrated acid. Langenbeck states that under the existing methods used in the rational analysis it is impossible to dissolve the clay substance out of the clay without a loss of feldspar that may reach 20 per cent of the material present. The action of sulfuric acid has an effect upon the quartz, although not so much as upon the feldspar. In the examples given it was found that the acid may dissolve from the quartz 3.88 per cent as the result of its action. However, in spite of the imperfections of the methods used in rational analysis there is no doubt but that from a practical point of view the results are of value for it has enabled the manufacturer to mix the natural clays with other substances in which the clay lacks and this enables him to deal with many of the problems connected with his work. Where the ultimate method would be, under ordinary circumstances, an unintelligible analysis the rational analysis is of service to him.

(4) Chemistry of Pottery, pp. 8-12.

In some cases the fact that the action of the sulfuric acid upon the clay will dissolve a quantity of feldspar and some of the quartz, has led some investigators to reject the rational analysis. As an example of this objection the following is of interest in connection with the rational analysis,⁽⁵⁾ "On account of the fact that the Wisconsin clays contain a large amount of feldspathic material the rational analysis would have little significance and the following method was adopted to calculate the feldspar and kaolin, from the ultimate analysis, using the following percentages of composition of feldspar:

K_2O , 16.9; Al_2O_3 , 18.4; $6SiO_2$, 64.7; Na_2O , 11.8; Al_2O_3 , 19.5; $6SiO_2$, 68.7; and kaolin: Al_2O_3 , 39.5; $2SiO_2$, 46.5; $2H_2O$, 14. Under these conditions potash and soda were regarded as part of feldspar. The alumina required for this amount of feldspar was deducted from the total alumina present and the remainder was the basis on which the kaolinite substance was calculated. The difference between the total amount of silica and that required by feldspar and kaolin gives the amount of quartz present."

In connection with the calculation of the rational analysis the following results showing the composition of various feldspars will be of interest.⁽⁶⁾

| | POTASSIUM FELDSPAR ORTHOCLASE. ($K_2Al_2Si_2O_{10}$) Per cent | SODIUM FELDSPAR ALBITE. ($Na_2Al_2Si_2O_{10}$) Per cent | CALCIUM FELDSPAR ANORTHITE. ($CaAl_2Si_2O_8$) Per cent |
|---------------------------------|---|---|--|
| Potassium oxide, K_2O | 16.89 | 11.82 | 20.10 |
| Aluminum oxide, Al_2O_3 | 18.43 | 19.56 | 36.82 |
| Silica, SiO_2 | 64.68 | 68.62 | 43.08 |

There are in use two methods for the determination of silica, feldspar, and quartz in clay. The following is one of the methods which is used in Germany:⁽⁷⁾

"About five grams of clay are weighed and placed in a 200 c. c. Erlenmeyer flask. 100-150 c. c. of water and two c. c. of sodium

(5) Clays and Clay Industries of Wisconsin, p. 267.

(6) König Untersuchung Land. Gewerb. Stoffe.

(7) Ladenburg-Handwoerterbuch der Chemie, 12, p. 15.

hydrate are added and the contents boiled, covering the flask with a small glass funnel. The contents of the flask are allowed to cool and 25 c. c. of sulfuric acid is added. Continue the boiling until the fumes of the acid begin to be driven off the flask.

As a result of the reactions which have taken place the calcium carbonate has been changed to calcium sulfate, the aluminum silicate has been converted into aluminum sulfate and silicic acid, while the quartz and feldspar remain. Water is added to the flask and most of the sulfuric acid and aluminum sulfate washed out of the residue by decantation.

In washing by decantation the water which is decanted should be placed upon a filter paper for the reason that should any of the residue be removed from the flask it can be returned by making a hole in the filter and washing back into the flask.

After washing by decantation the contents of the flask are treated with hydrochloric acid (100 c. c.) and boiled. Decant off the liquid and add sodium hydroxide (100 c. c.) boil and decant. Repeat the above process with hydrochloric acid and sodium hydrate. The residue is then transferred to a filter and washed with dilute hydrochloric acid (1 to 20). The filter with contents is transferred to a platinum crucible and weight determined.

The contents of the crucible are treated with a few drops of sulfuric acid and small quantities of hydrofluoric acid, evaporated to dryness in the water bath, ignited, weighed, and from the loss calculate and determine aluminum, iron, etc. From the aluminum found in the residue the feldspar is calculated, 1 part of alumina (aluminum oxide) = 5.41 of feldspar.

Another method which has been used in much of the work of this country is as follows:

The material (two grams) is treated with dilute sulfuric acid (20 c. c.) for six or eight hours on a sand bath, and the excess of acid is finally driven off. One cubic centimeter of concentrated hydrochloric acid and a small quantity of water is added. The

residue is separated by filtration and thoroughly washed with hot water. The residue is then treated with a small quantity (15 c. c.) of a boiling ten per cent solution of sodium hydroxide, 25 cubic centimeters of hot water are added and the solution filtered through same filter paper. The residue is washed six or eight times with hot water by decantation. The residue is then treated with dilute hydrochloric acid and washed, and transferred to the filter paper. The filtrate should be free from hydrochloric acid before the washing is finished. The residue on filter paper is burned and weighed, the result is the residue, insoluble in sulfuric acid and sodium hydrate. A part of this residue is used for determination of silica, aluminum oxide, and iron oxide and the other part for determining alkalis present in the insoluble residue.

The results furnish the means for calculating the rational analysis. The aluminum oxide present in the insoluble residue is multiplied by 3.51. This factor is regarded as the ratio between the silica and aluminum oxide in orthoclase feldspar. The product found represents the amount of silica present in feldspar. The sum of the amounts of silica, aluminum and ferric oxides, and alkalis represents the "feldspathic detritus." The difference between the silica combined and the total silica in residue gives the "quartz" or "free sand." The sum of the "quartz" and "feldspar" gives the basis for calculating the clay substance.

In the rational analysis it has been seen that there is a choice in the use of sodium hydroxide or sodium carbonate to dissolve the amorphous silica after the clay has been acted upon by sulfuric acid. The question sometimes presents itself as to the strength of the alkali to be used.

Lunge and Milberg⁽⁸⁾ in their investigations, concluded that the results of Michaelis, that a 10 per cent solution of NaOH does not

(8) *Zeits. Angew. Chem.*, 1897, pp. 395-425, *Abstr. Jr. Soc. Chem. Ind.* 16 (1897) p. 762.

act upon the quartz even during several hours boiling and that the soluble silica cannot even be dissolved by sodium carbonate, do not stand the test of investigation. In their experiments, Lunge and Milberg submitted a large number of different silicates with the results that caustic alkalis can not be safely employed to separate the quartz from the soluble silica, as quartz is dissolved during the process. Hot solutions of alkaline carbonates can only be used after finely powdered material has been washed away. It is stated that some quartz is dissolved even by a 5 per cent solution of sodium carbonate at the temperature of the water bath. It is stated, however, that the error will probably not exceed .1 per cent to .2 per cent, the soluble silica being returned high. Hardened filter papers should be used in the analysis. The modification of the process as suggested by Lunge and Milburg is in the analysis of the silicates after the sample has been reduced to the finest kind of powder, treated with hydrochloric acid and dried at 110 C. The amorphous silica can be readily determined by digestion for fifteen minutes in a 5 per cent solution of sodium carbonate and the error need not exceed .2 per cent. In the determination of aluminum and iron, Meinke states that there is a tendency for the iron and aluminum to contain a certain amount of silica, this amount may vary from .3 to 1 per cent.

The Chemical Analysis of Clays.

Besides the rational analysis, many times the complete ultimate analysis of clay is of value. In order that the chemical analysis may be of value it is necessary that the analytical work have the best possible care. Langenbeck calls attention to this subject in the following words: "While it is true that the chemical analysis alone, however accurate, is insufficient without accompanying physical tests to give one the perfect characteriza-

tion of clay, its value is sufficiently great to warrant the most careful work."

Clay, according to Blair, is a mixture of silica and the silicates of aluminum, calcium, magnesium, potassium and sodium. The silicates are hydrated and as a result they may contain from 6 to 12 per cent of water chemically combined. In addition to the substances named, clay may contain ferric oxide, titanio acid, ferric sulfate, organic matter and phosphoric acid. Occasionally, rare elements are found.

In the analysis of clay the following determinations are made:

(1) *Moisture*.—Two grams of clay are heated in a platinum crucible at 100° to 105° C., until a constant weight is reached. The loss in weight is regarded as moisture.

(2) *Loss on Ignition*.—This result includes combined water, organic matter and carbon dioxide in the form of carbonates.

The clay from the determination of moisture can be used for this determination. The crucible is heated with a blast lamp until it becomes constant in weight.

(3) *Silica*.—Clay being practically unacted upon by hydrochloric acid, it is impossible to use the substance for preparing a solution and it is necessary to fuse the product in order to decompose the silicate. One gram of finely divided clay is mixed with ten grams of sodium carbonate and a little potassium nitrate in a platinum crucible. The mass is brought to fusion in a platinum crucible, using the blast lamp if necessary. After the mass is fused it is run well up on the sides of the crucible and allowed to cool. Add hot water and when the mass is thoroughly disintegrated, treat the undissolved portion with hydrochloric acid. Transfer the mass to a casserole and evaporate to dryness on water bath, heat in air bath at 110° until all the hydrochloric acid is driven off. Dilute hydrochloric acid (60 c. c.) is added to the contents of the casserole and the contents brought to boiling. Transfer the material to an ashless filter, wash it

thoroughly with hot water, dry and transfer to a platinum crucible and weigh. The filtrate is used for the determination of the other constituents. The silica present in the crucible is treated with sufficient sulfuric acid to moisten it. Hydrofluoric acid is added cautiously in very small quantities successively until the silica has disappeared. Evaporate the contents of the crucible to dryness in a sand bath or on an asbestos board, ignite and weigh. The difference in weight represents the silica.

(4) *Determination of Aluminum and Iron.*—The filtrate from the silica is treated with an excess of ammonia and boiled until the ammonia in excess is driven from the solution. Filter and wash several times with hot water. The precipitate is dissolved with a mixture of 15 c. c. hydrochloric acid and 15 c. c. of water, and allow the solution to run into a small clean beaker. If any part of the precipitate remains in the larger beaker, in which the first precipitate was made, a part of the contents of the small beaker may be used for dissolving it. Wash the beaker and filter paper in order that the small beaker may contain all of the precipitate. Re-precipitate with ammonia as usual. Filter, transfer the precipitate to the filter paper and wash thoroughly with hot water. Dry, ignite the precipitate and weigh. It may be necessary to treat the residue before igniting with a few drops of nitric acid to convert any iron or ferrous oxide which may be present into ferric nitrate. Evaporate off the acid and weigh the contents of the crucible. The results will be the quantity of aluminum oxide, Al_2O_3 , and ferric oxide, Fe_2O_3 .

(5) *Determination of Iron.*—Fuse the precipitate of aluminum and iron oxide with Na_2CO_3 , and treat the fused mass with water. Wash into a small beaker and decant off the clear liquid into the flask used for the determination of iron volumetrically and determine in the usual manner.

If desired, citric acid can be added to the solution with an excess of ammonia and the iron precipitated as sulfide. Filter,

wash the precipitate, dry and determine iron in the usual manner. The amount of iron, subtracted from the total amount of aluminum and ferric oxides, gives the amount of aluminum.

(6) *Determination of Calcium.*—The filtrate from the determination of ferric and aluminum hydroxides is concentrated to about 200 c. c. and ammonium oxalate added to precipitate the calcium. The precipitation of the calcium should be from a hot solution. Allow the precipitate to settle twelve hours. Ammonium oxalate should be in excess in order to prevent the precipitation of any magnesium. Some prefer to add one grain of ammonium oxalate to insure an excess. The precipitate is filtered and washed with hot water, ignited and weighed in the usual manner. The resulting product is calcium oxide.

(7) *Determination of Magnesia.*—The filtrate from the determination of calcium precipitation is concentrated until about 150 c. c. Allow the solution to cool and precipitate the magnesia from an alkaline solution by means of hydrogen disodium phosphate. Fifteen minutes after the precipitation add 15 c. c. of concentrated ammonium hydroxide, sp. gr. .90. The magnesia ammonium phosphate is allowed to stand for at least two hours and is filtered through an ashless filter. Wash with water containing 5 per cent of ammonium hydroxide. Place the filter, after drying, in a crucible, ignite, and weigh in the usual manner. The resulting residue is magnesium pyrophosphate, and from the result the amount of magnesia is calculated.

(8) *Determination of Alkalis.*—The determination of alkalis in clay is made by treating two grams of finely ground material in a platinum dish with four c. c. of concentrated sulfuric acid and 50 c. c. of pure hydrofluoric acid. The contents of the dish should be stirred with a platinum wire in order that the acid may come in contact with the clay. The contents of the platinum dish is heated carefully until the clay is entirely decomposed and the gritty portion has disappeared. Evaporate to dryness, and

heat until the sulfuric acid is driven off and the fumes cease to be evolved. The use of hydrofluoric acid should be carried on under a hood where there is a strong draught. It is dangerous to handle the hydrofluoric acid in a place that is not well ventilated with a strong draught. After evaporating to dryness in the hood, 50 c. c. of water and a little hydrochloric acid is added. The mass should dissolve; if it does not, heat until it does. If there is any insoluble matter, it shows that the clay has not been thoroughly acted upon by the hydrofluoric acid. The soluble material can be transferred to another dish, and the operation repeated by means of hydrofluoric acid upon the insoluble residue in the dish. After thoroughly decomposing, the solution is diluted to about 350 c. c. by adding water. After placing in a beaker, heat to boiling, add an excess of ammonia and allow the precipitate to settle, and filter. The precipitate is transferred to the beaker in which the precipitation was made, by piercing the filter and washing with hot water. Add about 200 c. c. of water and a little ammonia. Transfer to the filter and wash thoroughly. The two filtrates are united, evaporated to dryness in a platinum dish. Heat in order to volatilize the ammonium salts. Add a few drops of hydrochloric acid and evaporate to dryness and heat to low red heat. The resulting residue represents the alkalis in the form of chlorides.

If it is desired to separate and determine the amount of potassium present, this can be done by precipitating the potassium chloride and determining it as K_2PtCl_6 in the usual manner.

(9) *Smith's Method of Determination of Alkalis.*—Instead of determining the alkalis by means of hydrofluoric acid and sulfuric acid, the method given by Smith may be used. The method is as follows: one gram of finely ground clay is mixed thoroughly with an equal weight of granular chloride of ammonia in a porcelain or agate mortar. To this mixture eight grams of calcium carbonate are added. Grind the mass together thoroughly so that

it is intimately mixed. Transfer the material to a large platinum crucible covered with a close fitting cover and heat slowly in order to decompose the ammonium chloride. This is accomplished in a few minutes, then heat, gradually increasing until the crucible is red. The bottom of the crucible is kept at a bright red heat for about one hour. The crucible is allowed to cool and, if the mass can be detached easily, transfer it to a platinum dish and add 80 to 100 c. c. of water. Remove any residue which may remain in the crucible, cover with water and wash the contents of the crucible into a dish. The contents of the dish are heated to boiling and the mass is allowed to slake completely. After slaking, filter and wash the residue thoroughly with hot water.

In case the contents of the crucible, after fusion, can not be removed easily, the crucible is placed on its side in a dish, the cover washed off and 100 c. c. of water added. The contents of the dish are heated until the mass can be removed. Remove the crucible from the dish, wash thoroughly and filter the liquid from the dish and wash the residue. The filtrate is treated with about one and one-half grams of pure carbonate of ammonia and filtered. Evaporate the solution in a platinum dish until it is reduced to 50 c. c., add a small quantity of ammonium carbonate and ten or fifteen drops of ammonia. Filter off the precipitate, if any is produced, and evaporate. Heat carefully to drive off the ammonium salts. The alkalis are present in the form of potassium and sodium chlorides. The addition of a few drops of hydrochloric acid will prevent any loss of material during the process of heating in driving off the ammonium salts.

(10) *Determination of Titanic Oxide.*—The determination of titanic oxide is made by placing one-half gram of clay in a platinum crucible and thoroughly mixing with five grams of potassium bisulfate and one gram of sodium fluoride. The mass is fused, allowed to cool and then dissolved with a five per cent solution of sulfuric acid. An aliquot part of the resulting solution is

placed in a Nessler tube, hydroxide is added and the tint developed is compared with the standards made from solution of titanium sulfate.

(11) *Determination of Sulfur*.—The total sulfur present in clay is determined by fusing one-half gram with a mixture of sodium carbonate, five parts, and potassium nitrate, one part, in a platinum crucible. The fused product is dissolved by dilute hydrochloric acid. The sulfur separated in the usual manner by evaporation and heating in order to render the silica insoluble, and filtered. Hydrochloric acid is added in order to have an excess of the acid present. The sulfate present in solution is precipitated by means of barium chloride in excess. Both solutions should be hot. The barium sulfate is filtered, washed with hot water, dried, placed in a crucible and weighed, and the sulfur or trioxide calculated from the results.

(12) *Determination of Ferrous Oxide*.—Ferrous oxide can be determined by fusing one-half gram of clay with five grams of sodium carbonate. The clay in a crucible should be well covered with carbonate. The contents are heated, the crucible being covered. The fused mass is dissolved and mixed with hydrochloric acid and sulfuric acid in an atmosphere of carbon dioxide. Ferrous iron is determined by titration with standard potassium permanganate.

THE VALUE OF CHEMICAL ANALYSIS.

As previously stated, the value of the rational analysis lies in the fact that from a theoretical point of view it is designed to separate quartz, feldspar and clay substance in the clay. The chemical analysis, on the other hand, can be used to draw certain inferences which are also valuable.

According to Ries, the following facts can be drawn from the chemical analysis:

(1) In the chemical analysis of clay the depth of the red color depends upon the amount of ferric oxide. A large percentage of ferric oxide produces a deep red color, while a small amount gives the yellow colors.

(2) The analysis will determine whether lime present is sufficient to counteract the action of the ferric oxide.

(3) That an approximate idea of the fusibility of clay can be obtained from the total amount of fluxes present and also the nature of the fluxes, whether they are weak or strong.

(4) The clay containing a large percentage of silica indicates generally a sandy clay.

(5) Clays having a high percentage of alumina and combined water, as a rule, shrink considerably on burning.

It is also stated that besides the tendency to shrink as indicated by chemical analysis, other physical tendencies are largely dependent on the mineralogical composition.

THE COLORS OF CLAYS.

The iron oxide present in the clay will produce certain definite colors dependent on the amount of oxide present. According to Professor Winchell, the cream colored bricks in Minnesota are produced as a result of the iron which is present in the clay entering into combination with silica and alumina instead of remaining as an oxide. The various manifested tints which are exhibited in many clays are practically due to a mixture of carbonaceous matter and iron. It is well known that the oxides of iron will cause white aluminum silicate to change its color to a grayish-blue and even to a black color. In clays where the color is produced by carbonaceous or other organic matter it will entirely disappear in burning if an excess of air is possible. Blue clay, it is said, can not be decolorised by digestion in hydrochloric acid, and on heating it becomes more intense, owing to the separation of particles of carbon, and these finally disappear.

Colored waters, if mixed with clays, will color them slowly, while kaolin, if mixed with brown, peaty water and allowed to stand for eight or ten days, will become brownish-blue. This color becomes more distinct when it is dried. If treated the second time this residue becomes brown, and by constantly repeating the process a dark-blue clay with a brown tint has been obtained similar in color to many of the natural clays.

INCRUSTATION ON BRICK WALLS.

Under certain conditions brick walls become colored by the production of white incrustations which appear after the wall is built. This incrustation sometimes is produced even before the building is complete, and unsightly white patches disfigure the building. In some cases it has been noticed that bricks which have never come in contact with the mortar have had this incrustation upon them. On examination the incrustation was found to be sulfate of magnesium. As a rule, this sulfate is discolored by other compounds, such as oxide of iron. Under other conditions the incrustation has proved to be a mixture of calcium and magnesium sulfates, with traces of iron and aluminium oxides. Carbonates were not found to be present. The material which produces this incrustation is therefore evidently of the nature of soluble sulfate, and the question arises whether these substances are present in the original clay or not. Investigations made upon these incrustations prove that calcium or magnesium sulfates are not present, usually, in the original clays. There is a small proportion of lime and magnesia, and no doubt the burning of brick accounts for the conversion of the magnesia and lime, which is present in the clay, to the corresponding sulfates, by the sulfur oxide produced from the sulfur in the coal as sulfide of iron or iron pyrites. When the coal is burned, sulfur dioxide in the presence of air and moisture produces sulfuric acid, which combines with magnesia and lime. The acid probably decomposes

the calcium and magnesium silicates, producing sulfate of calcium and magnesium, while silica is set free. The sulfate of magnesium possesses the peculiar property of efflorescence in the presence of dry air. A small amount readily becomes conspicuous. It has been suggested that the only way to prevent the incrustation is naturally to prevent the formation of magnesium sulfate in brick. In other words, to burn the brick intended for exposure to the atmosphere with material which will not produce sulfuric acid in a large enough quantity to produce sulfate of magnesia during the process.

Another source of trouble may be said to lie in the fact that mortar is produced from magnesium limestone and is really a mixture of lime and magnesia in various proportions. Of course, in the presence of fumes of sulfur dioxide, magnesium sulfate is readily produced from the magnesia, and the salt makes itself manifest in the efflorescence. Of course it can be readily seen that houses built with mortar from lime containing a large amount of magnesia can be acted upon by smoke containing a quantity of sulfur oxide, readily producing magnesium sulfate.

If lime should be the source of the trouble, then it is necessary to use lime in building which is free from magnesia, for that portion of the brick work which is exposed. In some houses these white incrustations of magnesium and calcium sulfates are rendered still more unsightly by black streaks. This is caused from repeated washing of the black dust from the window cases and frames. Of course the problem here is for the builder to use for that part of the building which is exposed, bricks containing a minimum amount of sulfuric acid produced from coal containing a small amount of sulfur, and mortar which is free from magnesia. It is a problem for prevention instead of endeavoring to discover a remedy for the mischief.

According to the investigation of Gunther, however, the iron pyrites present in the clay is one of the causes of efflorescence and this is not the result of the material used in burning the brick. Gunther stated that almost all clays contain sulfide of iron or pyrites, and this in the presence of magnesia gives rise to the efflorescence, after the decomposition of alkalis in the presence of water.

He regards the sulfur present as the cause of the trouble. The remedy which, as suggested by Gunther, is to mix barium carbonate or chloride in clay; this substance would combine with sulfuric acid, producing barium sulfate. The sandstone of many new buildings suffer with this trouble of efflorescence. From experience it is found that it has been impossible to prevent the formation of soluble sulfate entirely, but it has been suggested that an excess of baryta would be present at all times, combining with any sulfuric acid to produce insoluble barium sulfate. The addition of baryta to the mortar has been suggested as a remedy to check the efflorescence. Of course this check would be limited if the mortar is exposed to the action of the fumes of sulfur from the chimneys where coal is used containing a large amount of sulfur.

As a matter of interest I would note the investigation of Director Parize of the Agricultural Station at Morlaix, France, whose work has led to the conclusion that the crumbling of brick is due largely to the action of microbes. The result of the action of the microbes is a red dust which on examination with the microscope was found to contain a number of microscopic organisms. This opens a wide field for the investigation of many problems connected with the decay of walls of brick buildings.

ANALYSES OF CLAYS.

| Number. | SAMPLE FROM. | Moisture. | Potassium oxide. | Sodium oxide. | Calcium oxide. | Magnesium oxide. | Ferric oxide. | Alumina. | Sulfur tri-oxide. | Carbon dioxide. | Loss in ignition. | Total. |
|---------|--|-----------|------------------|---------------|----------------|------------------|---------------|----------|-------------------|-----------------|-------------------|--------|
| 1 | Flint Brick Co., Des Moines, bottom..... | .20 | .95 | 1.50 | 2.57 | 1.57 | 4.80 | 15.75 | .97 | 1.71 | 3.22 | 100.80 |
| 2 | Flint Brick Co., Des Moines, middle..... | .76 | .63 | .89 | 1.80 | 1.44 | 5.28 | 21.12 | .95 | 2.07 | 3.75 | 99.91 |
| 3 | Flint Brick Co., Des Moines, top..... | 1.09 | .24 | .58 | 3.17 | 2.72 | 2.88 | 15.47 | .95 | 1.25 | 4.37 | 99.87 |
| 4 | Iowa Brick Co., Des Moines, No. 7, bottom. | .42 | .52 | 1.15 | 3.53 | 2.16 | 4.79 | 21.01 | .95 | | 4.51 | 100.86 |
| 5 | Iowa Brick Co., Des Moines, No. 6, top..... | .25 | .39 | 1.35 | 3.17 | 1.42 | 4.51 | 16.68 | 1.00 | 2.99 | 1.44 | 100.40 |
| 6 | Iowa Brick Co., Des Moines, No. 5, top..... | .84 | .21 | 1.90 | 6.59 | 1.87 | 1.92 | 20.43 | .45 | | 6.82 | 100.50 |
| 7 | Iowa Brick Co., Des Moines, No. 4, top..... | .83 | .80 | 1.48 | 3.17 | 1.54 | 3.63 | 21.97 | 1.15 | 1.58 | 3.59 | 100.63 |
| 8 | Iowa Brick Co., Des Moines, No. 3, top..... | .89 | .59 | 1.69 | 2.90 | 1.44 | 5.35 | 19.21 | 1.80 | 2.58 | 2.66 | 100.83 |
| 9 | Iowa Brick Co., Des Moines, No. 2, top..... | .63 | .65 | 1.95 | 1.00 | .86 | 3.83 | 11.94 | 1.65 | .90 | 4.33 | 99.57 |
| 10 | Flint Brick Co., Des Moines..... | .56 | .54 | 1.20 | 1.59 | 1.22 | 5.75 | 19.78 | 1.17 | 2.15 | 2.92 | 100.50 |
| 11 | M. E. Ward, Lake City, Iowa..... | .58 | .31 | 1.08 | 2.33 | 1.08 | 1.24 | 12.20 | 2.01 | | 5.15 | 100.70 |
| 12 | Capital City Brick & Pipe Co., Des Moines. | 1.13 | .29 | 1.08 | 3.60 | 1.44 | 2.87 | 22.33 | 1.85 | 1.78 | 5.33 | 100.26 |
| 13 | Holman, Mark X..... | .59 | .50 | 1.15 | 2.15 | .86 | 2.80 | 14.17 | .95 | .76 | 5.16 | 100.31 |
| 14 | Holman, Sergeants Bluffs..... | .79 | .24 | .70 | 1.00 | .49 | 1.43 | 10.73 | .75 | 1.84 | 6.38 | 100.30 |
| 15 | Corey Pressed Brick Co., Lehigh, red burning | .96 | .90 | 2.04 | .30 | 2.04 | 3.83 | 23.05 | .99 | | 8.10 | 100.10 |
| 16 | Corey Pressed Brick Co., Lehigh..... | .79 | .84 | 2.19 | .96 | 1.70 | 3.83 | 23.89 | 1.16 | .26 | 5.87 | 100.17 |
| 17 | Granite Brick Co., Burlington, top..... | .13 | 1.44 | 2.79 | 3.65 | 3.13 | 2.40 | 5.16 | 1.30 | 1.44 | 1.49 | 100.29 |
| 18 | Granite Brick Co., Burlington, bottom..... | .42 | .86 | .78 | 3.18 | 3.80 | 3.35 | 11.41 | 1.25 | 3.02 | .87 | 100.52 |
| 19 | L. M. Harris, Rockford, bottom..... | .38 | 1.19 | 1.76 | 9.42 | 3.03 | 3.84 | 15.54 | 1.16 | 2.02 | 3.47 | 100.06 |
| 20 | Capital City Brick & Pipe Co., Des Moines. | 1.35 | 2.92 | .87 | 1.29 | 1.94 | 11.20 | 22.26 | .16 | | 7.43 | 100.30 |
| 21 | Capital City Brick & Pipe Co., Des Moines | .49 | 2.17 | 2.41 | 1.78 | 1.58 | 10.32 | 16.22 | | | 7.43 | 100.04 |
| 22 | Capital City Brick & Pipe Co., Des Moines. | .88 | 1.04 | .54 | .81 | .42 | 3.36 | 16.39 | 1.42 | | 5.75 | 100.01 |
| 23 | Capital City Brick & Pipe Co., Des Moines. | .51 | 2.81 | 2.76 | 1.58 | 1.55 | 5.52 | 17.90 | | | 5.02 | 100.00 |
| 24 | Capital City Brick & Pipe Co., Des Moines. | .40 | 1.09 | 1.87 | .75 | 1.72 | 5.12 | 17.89 | | | 4.67 | 100.37 |
| 25 | | 6.77 | 1.25 | 3.70 | 4.05 | .94 | 8.64 | 17.71 | | 2.58 | | |
| 26 | Galesburg..... | 3.85 | 4.17 | 3.19 | 3.39 | .18 | 2.00 | 24.40 | | | 12.41 | 100.21 |
| 27 | | | | | | | | | | | | |
| 28 | Boone Brick Tile & Paving Co., Boone..... | 2.10 | 3.10 | .50 | .70 | 1.10 | 12.40 | 22.20 | 2.40 | | 7.90 | 99.80 |
| 29 | Buff Clay from Boone..... | .70 | .70 | .30 | 1.00 | 1.00 | 2.00 | 16.60 | 1.70 | | 4.30 | 99.90 |
| 30 | Red Clay from Ft. Dodge..... | 2.10 | 1.22 | 1.80 | 1.23 | 1.34 | 4.00 | 23.09 | | | 6.28 | 100.22 |
| 31 | Buff Clay from Ft. Dodge..... | 2.08 | 1.62 | .59 | 1.12 | 1.74 | 4.83 | 11.43 | .94 | | 11.50 | 100.06 |
| 32 | Buff Clay from Ft. Dodge..... | 1.15 | .81 | .49 | .92 | .62 | 2.72 | 17.26 | | | 5.57 | 99.96 |
| 33 | Clermont Brick & Tile Co..... | .43 | 5.38 | 7.41 | 19.14 | 5.40 | 3.78 | 10.37 | 3.01 | | 16.24 | 99.96 |
| 34 | Storm Lake Brick & Tile Co..... | 2.33 | 1.14 | 1.90 | 4.02 | 1.80 | 4.00 | 12.64 | | | 5.82 | 100.10 |
| 35 | Dale Brick Co., Des Moines..... | .32 | 1.84 | 1.77 | .65 | 1.36 | 5.12 | 17.73 | | | 4.21 | 100.17 |
| 36 | B. B. Jester, Danville..... | .22 | .32 | .38 | .73 | .45 | 2.72 | 15.54 | | | 4.97 | 100.15 |
| 37 | American Brick & Tile Co., Mason City..... | .42 | 1.43 | 2.59 | 4.14 | 3.36 | 7.50 | 18.34 | 2.76 | | 7.39 | 100.04 |
| 38 | Myron Mellen, Edgewood, Iowa..... | .89 | 1.56 | 5.29 | 7.88 | 6.05 | 7.80 | 13.72 | | | 12.18 | 100.00 |
| 39 | | | | | | | | | | | | |
| 40 | | | | | | | | | | | | |
| 41 | L. C. Besley, top..... | 1.05 | .55 | 1.89 | 7.20 | 3.15 | 3.83 | 6.53 | .90 | 5.53 | 2.08 | 99.99 |
| 42 | L. C. Besley, middle..... | .92 | .58 | 1.68 | 3.90 | 3.16 | 2.87 | 10.21 | 1.45 | 5.86 | 1.52 | 100.07 |
| 43 | L. C. Besley, bottom..... | 1.52 | .47 | 1.59 | 2.52 | 3.29 | 2.40 | 0.12 | 1.76 | 1.87 | 2.94 | 100.24 |
| 44 | Dale Brick Co., Des Moines..... | .71 | 1.27 | 2.72 | 1.53 | 1.01 | 5.56 | 9.68 | | | 3.88 | 99.85 |
| 45 | Gettmann Bros., Gladbrook, Iowa..... | 1.49 | 1.87 | 1.92 | 1.63 | 1.18 | 6.72 | 11.76 | | | 5.36 | 99.85 |
| 46 | Myron Mellen, Edgewood..... | 1.91 | 1.76 | .72 | 1.00 | 1.12 | 6.16 | 10.83 | | | 3.25 | 100.00 |
| 47 | Fort Dodge Stoneware Co..... | 1.00 | 1.06 | 1.92 | | 4.21 | 2.25 | 17.00 | | | 7.62 | 100.58 |
| 48 | Fort Dodge Stoneware Co..... | .37 | 1.35 | 2.74 | 3.00 | 4.09 | 1.12 | 15.25 | | | 9.31 | 100.82 |
| 49 | Marshalltown..... | .84 | 1.23 | .81 | .51 | .44 | 4.32 | 16.24 | | | 3.33 | 100.21 |
| 50 | Marshalltown..... | .98 | .64 | .55 | .99 | .42 | 4.32 | 20.21 | | | 8.50 | 100.00 |
| 51 | Clayton county..... | .22 | 1.25 | 2.77 | 19.86 | 4.81 | 5.04 | 14.59 | .68 | | 31.91 | 94.92 |
| 52 | Clayton county..... | 1.35 | 2.27 | 8.27 | 1.89 | 1.12 | 5.16 | 20.64 | .18 | 1.20 | 5.17 | 100.07 |
| 53 | A. W. Turner, Tabor, Iowa..... | .74 | 1.92 | 1.44 | 31.68 | 1.57 | 2.60 | 10.09 | .60 | | 30.16 | 100.25 |
| 54 | Geological Department..... | 3.02 | 2.52 | .43 | .12 | .45 | 4.32 | 23.28 | 1.22 | | 8.06 | 100.26 |
| 55 | Geological Department..... | 3.25 | 1.22 | .67 | .57 | .24 | 4.36 | 29.31 | .94 | | 10.38 | 100.14 |
| 56 | Geological Department..... | 2.49 | 1.82 | .98 | .82 | .71 | 7.92 | 24.63 | .16 | | 5.36 | 99.89 |
| 57 | Geological Department..... | .81 | 1.90 | 2.45 | .91 | .67 | 5.16 | 21.70 | .48 | | 6.95 | 99.87 |
| 58 | Geological Department..... | 1.85 | 1.52 | 1.06 | .46 | .86 | 9.38 | 23.79 | .35 | | 4.87 | 100.09 |
| 59 | | 3.48 | 3.44 | 3.54 | 1.74 | .90 | 7.92 | 24.43 | .09 | | 5.49 | 100.00 |
| 60 | O. J. Peterson..... | | | | | | | | | | | |
| 61 | Trenton Shale, Fayette county..... | 1.15 | 3.25 | 3.25 | 14.97 | 3.94 | 4.80 | 15.38 | 1.62 | | 18.01 | 99.97 |
| 62 | Fred Hanson, Eldora..... | 2.46 | 1.08 | .77 | .48 | .48 | 1.08 | 16.24 | .14 | | 5.18 | 100.00 |
| 63 | West Union, Iowa..... | | | | 3.87 | 1.81 | | | | 2.44 | | |
| 64 | West Union, Iowa..... | 2.18 | 1.79 | 1.33 | 1.20 | 1.87 | 3.48 | 15.29 | .61 | | 4.83 | 99.80 |
| 65 | Volunteer Coal Co., What Cheer..... | .84 | 1.93 | 1.49 | .65 | .39 | 2.2 | 22.06 | .79 | | 6.92 | 99.94 |

RATIONAL ANALYSES OF CLAYS.

| Number. | SAMPLE FROM— | | | | |
|---------|--|-------|---------|-----------|---|
| | | Clay. | Quartz. | Feldspar. | Carbonates and sul- fates of calcium and magnesium. |
| 1 | Flint Brick Company, Des Moines, bottom | 52.85 | 25.99 | 15.80 | 5.36 |
| 2 | Flint Brick Company, Des Moines, middle | 59.12 | 9.59 | 25.32 | 5.97 |
| 3 | Flint Brick Company, Des Moines, top | 53.79 | 33.31 | 8.78 | 4.14 |
| 4 | Iowa Brick Company, Des Moines, No. 7, bottom | 56.79 | 19.63 | 21.96 | 1.62 |
| 5 | Iowa Brick Company, Des Moines, No. 6, top | 31.00 | 30.56 | 32.77 | 5.67 |
| 6 | Iowa Brick Company, Des Moines, No. 5, top | 60.70 | 36.97 | 2.33 | |
| 7 | Iowa Brick Company, Des Moines, No. 4, top | 70.66 | 21.79 | 3.95 | 3.60 |
| 8 | Iowa Brick Company, Des Moines, No. 3, top | 49.30 | 34.01 | 13.09 | 3.60 |
| 9 | Iowa Brick Company, Des Moines, No. 2, top | 41.47 | 55.29 | 3.24 | |
| 10 | Flint Brick Company, Des Moines, brick | 61.57 | 20.53 | 13.47 | |
| 11 | M. E. Ward, Lake City | 42.00 | 46.16 | 8.44 | 3.40 |
| 12 | Capital City Brick and Pipe Company, Des Moines | 57.40 | 31.17 | 4.38 | 7.05 |
| 13 | Holman, Mark X | 46.44 | 46.51 | 3.80 | 3.25 |
| 14 | Holman, Sargeants Bluff | 38.20 | 51.10 | 7.62 | 3.08 |
| 15 | Corey Pressed Brick Company, Lehigh, red burning | 68.20 | 25.81 | 5.99 | |
| 16 | Corey Pressed Brick Company, Lehigh | 54.62 | 32.23 | 11.13 | 1.97 |
| 17 | Granite Brick Company, Burlington, top | 24.92 | 51.39 | 19.64 | 4.05 |
| 18 | Granite Brick Company, Burlington, bottom | 35.82 | 42.14 | 14.18 | 7.76 |
| 19 | L. M. Harris, Rockford, bottom | 64.47 | 18.67 | 11.13 | 5.73 |
| 20 | Capital City Brick and Pipe Company, Des Moines | 51.53 | 44.05 | 4.42 | |
| 21 | Capital City Brick and Pipe Company, Des Moines | 44.49 | 45.97 | 9.54 | |
| 22 | Capital City Brick and Pipe Company, Des Moines | 44.12 | 47.31 | 8.57 | |
| 23 | Capital City Brick and Pipe Company, Des Moines | 39.79 | 50.49 | 9.72 | |
| 24 | Capital City Brick and Pipe Company, Des Moines | 38.00 | 50.81 | 11.19 | |
| 25 | | | | | |
| 26 | Galesburg | 83.57 | 8.83 | 7.60 | |
| 28 | Boone Brick, Tile and Paving Company | 74.90 | 12.70 | 8.81 | 3.59 |
| 29 | Buff clay from Boone | 37.13 | 47.54 | 15.33 | |
| 30 | Red clay from Fort Dodge | 59.23 | 38.47 | 2.50 | |
| 31 | Buff clay from Fort Dodge | 55.86 | 34.73 | 8.31 | 1.60 |
| 32 | Buff clay from Fort Dodge | 47.47 | 47.50 | 5.03 | |
| 33 | Clermont Brick and Tile Company | 68.20 | 21.75 | 4.98 | 5.12 |
| 34 | Storm Lake Brick and Tile Company | 38.80 | 36.36 | 24.84 | |
| 35 | Dale Brick Company, Des Moines | 40.31 | 47.09 | 12.60 | |
| 36 | B. B. Jester, Danville, Iowa | 21.77 | 68.94 | 9.29 | |
| 37 | American Brick and Tile Company, Mason City | 47.08 | 41.45 | 6.98 | 4.49 |
| 38 | Myron Mellen, Edgewood | 40.61 | 28.00 | 4.62 | 23.77 |
| 39 | | | | | |
| 40 | | | | | |
| 41 | L. C. Besley, top of pit | 26.51 | 32.77 | 28.34 | 12.34 |
| 42 | L. C. Besley, middle | 19.72 | 40.29 | 25.74 | 14.25 |
| 43 | L. C. Besley, bottom | 27.37 | 46.09 | 19.32 | 7.22 |
| 44 | Dale Brick Company, Des Moines | 31.30 | 56.23 | 12.47 | |
| 45 | Gethmann Brothers, Gladbrook | 39.91 | 40.28 | 19.82 | |
| 46 | Myron Mellen, Edgewood | 33.55 | 50.39 | 11.06 | |
| 47 | Fort Dodge Stoneware Company | 29.00 | 59.94 | 11.06 | |
| 48 | Fort Dodge Stoneware Company | 35.18 | 44.41 | 20.41 | |
| 56 | Marshalltown | 14.81 | 38.98 | 48.21 | |
| 57 | Marshalltown | 41.10 | 46.68 | 12.22 | |
| 58 | Clayton County | 84.74 | 10.23 | 5.03 | |
| 59 | Clayton County | 70.65 | 26.73 | 2.57 | |
| 60 | A. W. Turner, Tabor, Iowa | | | | |
| 61 | Geological Department | 58.64 | 35.09 | 6.27 | |
| 62 | Geological Department | 51.46 | 42.22 | 6.32 | |
| 63 | Geological Department | 41.20 | 44.61 | 14.19 | |
| 64 | Geological Department | 50.20 | 26.22 | 23.58 | |
| 65 | Geological Department | 45.36 | 41.91 | 12.73 | |
| 66 | | 67.92 | 24.34 | 7.74 | |
| 67 | O. J. Peterson | 43.97 | 25.03 | 31.00 | |
| 68 | Trenton shale, Fayette County | 66.59 | 18.95 | 14.55 | |
| 69 | Fred Hanson, Eldora | | | | |
| 70 | West Union, Iowa | | | | |
| 71 | West Union, Iowa | | | | |
| 72 | Volunteer Coal Company, What Cheer | 40.11 | 54.37 | 5.52 | |

