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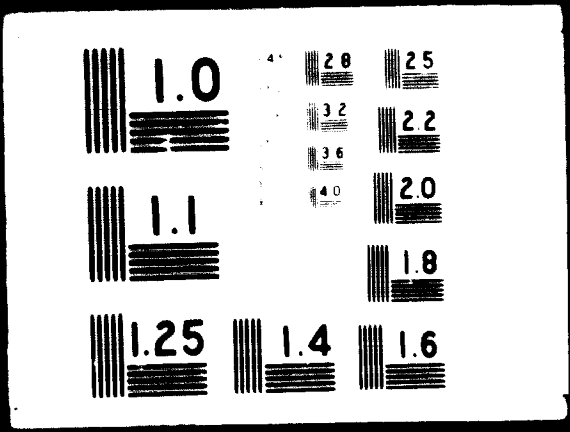

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The Interregional Seminar on the
Development of Clay Building Materials
Industries in Developing Countries

Copenhagen, 12-25 August 1968

TESTING AND EVALUATION OF BRICK CLAYS[1]

by

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(revised and expanded by T. Chvatal)

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[1] The views and opinions expressed in this paper are those of the authors and do not necessarily reflect the views of the secretariat of UNIDO. The document is presented as submitted by the authors, without re-editing.

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Introduction

1. This paper on "Testing and Evaluation of Brick Clays" has been prepared for the International Seminar on the Development of Clay-Building Materials Industries in Developing Countries.

2. Clay has been used for the production of brick for a very long time. Today's architecture is seeking for light constructing material and a rational way of building. The brick industry is making every effort to fulfill the demands of this development, by producing large bricks, of accurate dimensions, and bricks with hollow spaces. Therefore the raw material has to be of high quality. On the other hand the development in ceramic technology has lead to economically possible methods of benefication of poor quality clays. By means of these the range for using available clays has been widely enlarged.

3. Relevant for the estimation of clays are the minimum requirements of the desired products. These are stipulated in the standards. As regards the production of bricks the manufacturing depends on local conditions. Because of the need for economy, expensive additives are unreasonable. An intensive testing of the clay has to answer the question, whether a clay as such, or a clay processed by certain technologic methods will fulfill the required standards. Thus a correct estimation of the clay is not only of importance to determine whether a production is possible, but also to set up the process and equipment.

4. With regard to the conditions prevailing in various countries, the main emphasis was laid on the technological tests, which can be carried out by means of simple laboratory equipment. At the same time the more complicated tests were not neglected, as a further step of development, in order to stimulate a rise in technical standard of the tests and evaluation.

5. As clays are employed not only in the ceramic industry but also in the chemical industry, in the food industry, in the pharmaceutical industry, in the paper industry, in metallurgy, in the building industry, in drilling techniques etc. and are important for agriculture, detailed research on clays is carried out all over the world, making use of physico-chemical tests, such as X-ray analysis, thermography, electron and neutron diffraction, nuclear magnetic resonance, electron and high temperature microscopy etc. in order to clear up their structure and texture and to explain their behaviour and properties under certain conditions. The broad ranging uses of clay bricks indicates why information from other branches can contribute to a better understanding of clays, employed in the ceramic industry.

I. GENERAL DISCUSSION OF THE PROPERTIES OF CLAYS

6. The ceramic raw materials employed in the manufacture of various ceramic products, especially the structural clay products, are of mineral origin and are composed of silica, alumina, lime, magnesia, iron oxide, alkalis and of compounds made of these oxides. The first place among the ceramic raw materials is occupied by various clays, known long ago to be capable of being shaped into different vessels or products, employed for use or building.

7. The definition of clay is not simple. For example, the conception of clay is different for a ceramist than it is for an agricultural chemist.

8. According to the American Ceramic Society the definition of clay is: "A fine-grained rock, which when suitably crushed and pulverised, becomes plastic; when wet, leather-hard; when dried and on firing is converted to a permanent rock-like mass."

9. To this definition it is possible to add that in ceramics the term "clay" means not only the natural material but also the same material, after it has been beneficated, purified or treated in some other way. If various accompanying minerals are removed and the substance of clay is formed by clay minerals only, that substance is sometimes referred to as "true clay" or "clay substance".

10. As the difference in chemical, mineralogical and dispersion composition of clays influences their technological properties and their fitness for a particular sort of ceramic product, the clays are often differentiated in their application. Some of the clays are called brick clays, others refractory clays, pottery clays, sintering clays etc. The clays for building materials, especially the brick clays are, as a rule, not pure clays but mixtures with other non-clay minerals, so that the quantity of the clay substance is somewhere between 30 and 40 $^{w}/o$.

11. The term "brick clay" is more industrial than geological and is applied to all clays, loams and earths that can be used for the manufacture of bricks.

Varieties of clays

12. Taking into account the geological origin, clays are divided into two groups:

a) Primary or residual clays that remained <u>in situ</u>. These are characterised by large grains and by the great number of primary minerals that were not weathered enough;

b) Secondary or sedimentary clays, the nature of which is determined by the kind of transport and deposition.

We distinguish:

i) The fluvial and alluvial clays, originated by sedimentation in rivers;

ii) The glacial clays, originated in transportation of great masses of glaciers;

iii) The deltaic clays, sedimented in mouths of rivers;

iv) The lacustrine clays, originated by sedimentation in freshwater lakes;

v) The marine clays, originated by deposition in seas and oceans;

vi) The aeolian clays, that were wind-blown in origin.

13. All these sorts of clays can be employed, sometimes directly, sometimes after some sort of benefication, for the manufacture of brick products. Some clays, even of the same origin, have their specific features. For example:

a) Marl (or malm) is a clay, containing finely dispersed calcium carbonate;

b) Red-burning clays are clays whose colour, after being fired, is red;

c) Strong clays are very plastic and relatively pure clays;

d) Mild clays are mild plastic clays, weakly consolidated, containing sand;

e) Loams are clays containing gravel and sand;

f) Loess are wind-blown, silty clays;

g) Rock clays are the consolidated clays, known as shales, slates, and fire-clays.

14. The brick clays can contain non-clay minerals. When necessary and according to the properties of the clay, these minerals can be added in order to optimize the technological properties of the mixture. For such additions we can employ: sand, crushed rocks, waste from burned bricks, limestone, fly ash, coal slag,

clinker etc.; the additives can be of natural or artificial origin. The result of such additions is a mixture that has suitable properties for manufacture, when the raw material, as found in the deposit, was not suitable for this purpose.

Basic properties of clays

15. Clays in general, and the brick clays as well, consist of a great number of various mineral particles that are of colloidal to macroscopical size.

16. The term 'earths" is applied to a product of natural rock decomposition only if it contains more than 50 $^{w}/o$ of particles smaller than 2 mm. Ceramic technology divides them according to the contents of the size fractions as follows:

a) Clay substance: particles of a diameter under 2 microns;

b) Silt: particles of a diameter between 2 and 50 microns;

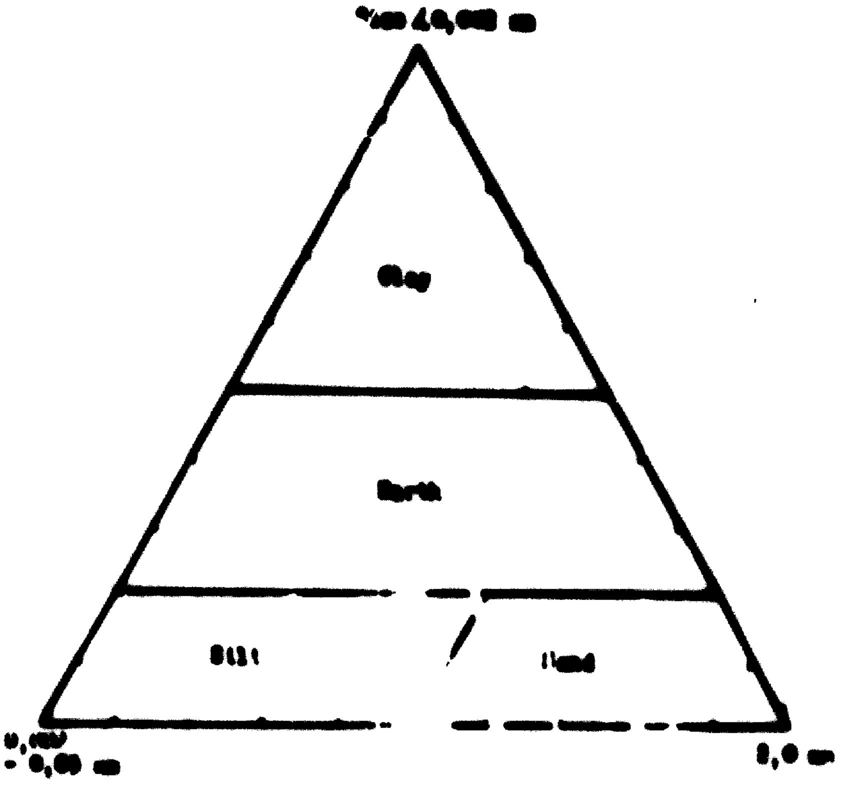
c) Sand: particles of a diameter above 50 microns (to 2 mm).

According to the amount of these fractions it is possible to distinguish various sorts of clays.

17. The ceramic clays contain more than 50 $^{w}/o$ of fine-size fractions; the brick clays can have contents of small-size particles up to 20 $^{w}/o$. The "earths" containing less than 20 $^{w}/o$ of fine-size fractions are practically non-plastic earths that can be used in ceramics only as grog (Fig.1 below).

Figure 1

Classification of sediments



Chemical composition of clays

18. The average composition of the clay earths is usually in these ranges:

	%
SiO_2	50 - 70
Al_2O_3	10 - 35
TiO_2	0,1 - 2
Fe_2O_3	2 - 8
CaO	0,5 - 15
MgO	0,2 - 5
SO_3	0,0 -0,5
Alkalis	0,5 - 4
Loss on ignition	3 - 12

19. The particular elements are, of course, not presented in the form of oxides, with the possible exceptions of silica (SiO_2), hematite (Fe_2O_3), rutile (TiO_2), but are bonded in the form of silicates, aluminosilicates or other compounds.

20. On the basis of the amount of oxide, we can judge the probable technological properties e.g., with respect to processing, drying and firing.

Mineralogical composition of clays

21. As a function of the disintegrated rocks and of the conditions of weathering and sedimentation, the compositions of clays and earth is very different. It is not possible to find two clays which are equal. Table I gives a survey of almost all minerals met with clays. This table is also used for the nomenclature of the clays and earths. The minerals occurring in brick clays are underscored.

22. The clay minerals are essentially the hydrated aluminosilicates. They are crystalline (with the exception of amorphous alophan) and have a layer or chain structure.

23. The most important of clay minerals are kaolinite, montmorillonite, illite and chlorite.

Table 1

Minerals in clays

Kaolin-group (kandites)

Nakrite
Dickite
Kaolinite
Fireclay
Halloysite

Montmorine-group (smectites)

Montmorillonite
Nontronite
Beidellite
Volkonskoite
Saponite
Hectorite
Sauconite
Stevensite

Micaceous minerals

Hydromuscovite
Illite
Ledikite
Brammalite

Muscovite, Sericite
Paragonite
Biotite
Phlogopite
Lepidolite
Zinnwaldite
Glauconite
Celadonite

Other silicatic clay minerals

Allophane
Pyrophyllite
Talcum
Attapulgite
Corrensite
Hydrobiotite
Rectorite

Vermiculite

Chlorites

Serntinite

Aluminum oxides and hydroxides

Gibbsite, hydrargillite
Diaspore
Boehmite

SiO_2 - minerals

Quartz, Chalcedone
Tridymite
Cristobalite
Opal

Feldspar-group

Orthoclase
Plagioclase
Foides

Carbonates

Calcite
Dolomite
Ankerite
Siderite
Brennerite

Other accompanying minerals

Limonite
Hematite
Magnetite
Pyrite, marcasite
Ilmenite
Rutile
Anatase
Zircon
Tourmaline
Garnet
Apatite
Phosphorite

Salts (soluble)

Rock salt

Gypsum
Alunite

Carbonaceous material

Coarse aggregates, fragments of rock
soils.

Figure 2

Structure of kaolinite

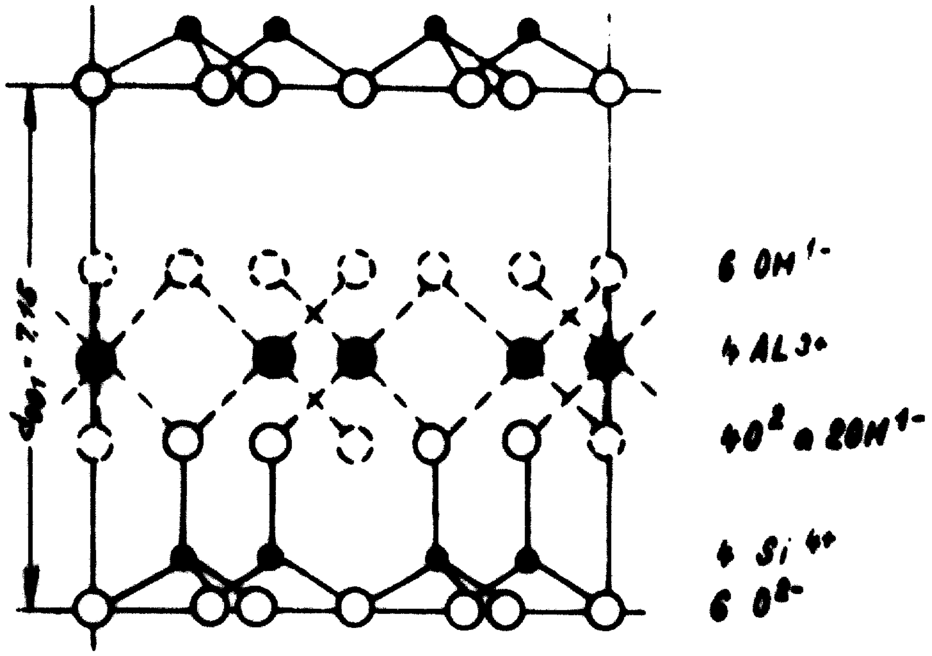


Figure 3

Structure of montmorillonite

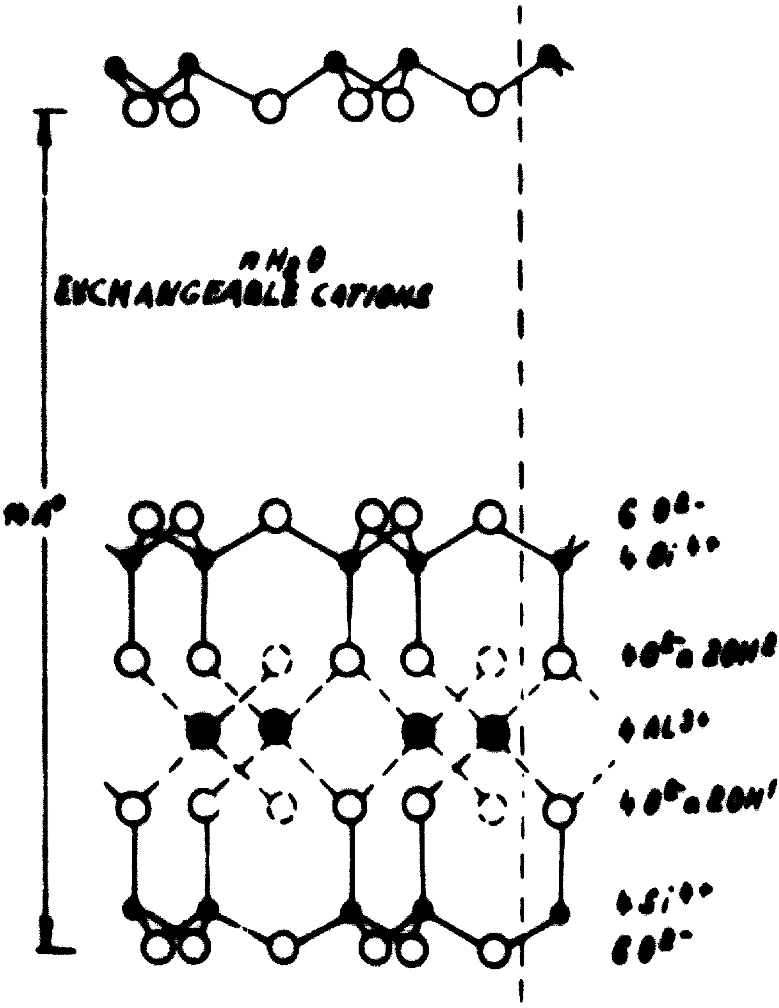


Figure 4

Structure of illite

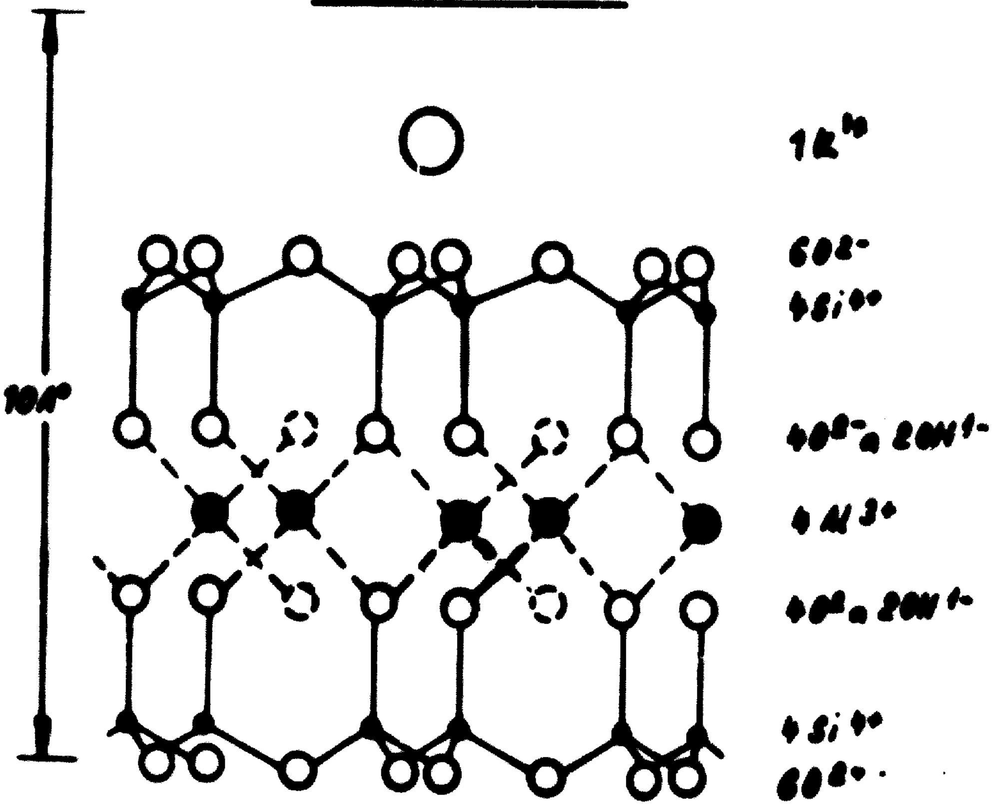
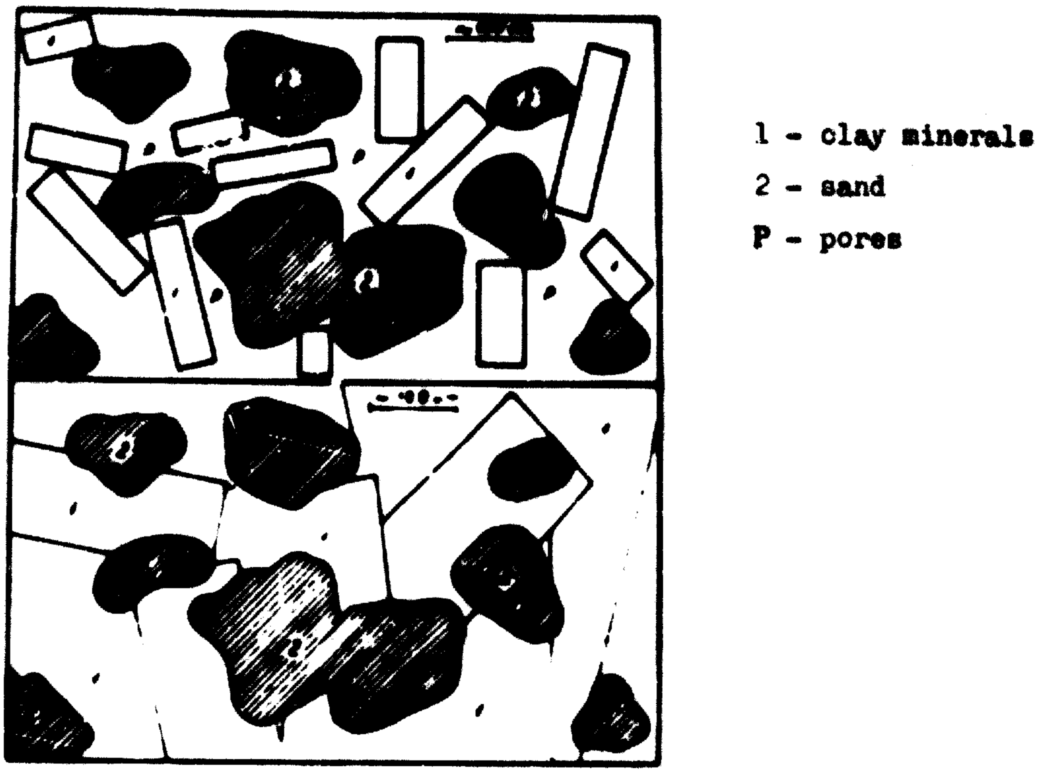


Figure 5

Swelling



24. Kaolinite (Fig. 2) is a double-layer mineral. The chemical compositions correspond to the formula $Al_2O_3.2\ SiO_2.2\ H_2O$.

25. Montmorillonite (Fig. 3) has the chemical composition approximately $Al_2O_3.4\ SiO_2.H_2O$. The montmorillonites cause the high plasticity.

26. Illite (Sericite), (Fig 4) in its structure resembles the montmorillonite. Illite, compared with the montmorillonite, has no inner swelling.

27. The group of the chlorites includes Fe-Mg-Al-silicates with water of constitution.

28. Some of the properties of the clays are exceptional. These properties are based on the crystalline structure and on the morphology of the clay particles. The plasticity is conditioned by the form and the size of the particles. The ionic exchange on the surface of the particles is caused by negative charge because of the substitution of Si by Al in the crystal latices. The deflocculation and the swelling is a result of exchangeable cations in the hydratic layers in the clay particles or among them.

Hygroscopicity and water-bonding

29. The particles form a very complicated system of larger to very fine capillaries that can very intensively suck the water. The arising capillary forces are proportionate to the inner surface of the clay and to the surface tension of water, and can reach very considerable values (to 100 kg/cm^2).

30. The clay, placed in moist medium, bonds certain amounts of water. A capillary phenomenon takes place, called absorption, that in this case appears as a concentration of the liquid phase on the furface of the particles, where they form thin or monomolecular layers of the liquid.

Swelling

31. On contact of clay mineral and water, the ions OH from water concentrated on the surface of the clay, give to it a negative character. The water dipoles become oriented and form a water film round the particle or lyosphere. The particle is hydrated, which means that it has not only the free water, which is movable between the particle, but also the bonded water.

32. The relatively great dimensions of the lyosphere as compared with the particles of the clay have the effect that the earth on contact with water becomes voluminous or swells. The hydrated particles of the clay develop sometimes a considerable pressure on the surroundings, the so-called swelling pressure, and change the physico-chemical properties of the dispersed system. The scheme of swelling is shown inFig. 5.

33. With monmorillonite the water can enter into the lattice and expand it. Therefore the swelling ability of monmorillonite is markedly higher than that of other clay minerals.

34. According to the degree of swelling, brick clays can be divided into mildly swelling (under 20 $^{w}/o$), medium swelling (20-30 $^{w}/o$) and strongly swelling (above 30 $^{w}/o$).

35. The swelling has no upper limit; a material can pass from a suspension to a solution. In the case of clays this appears as a disintegration of coarser aggregated particles and as a destruction of the texture and the outer structure of clay. This process is called the slaking of the earths and its mechanism can be explained as a surface dispersion. The slaking depends not only on the nature of clay but also on its density, moisture, particle size etc. The consolidated and strongly plastic earths slake with difficulty, while the sandy ones slake very easily.

Absorption capacity of clays

36. If the clay particle is placed in water, it absorbs on the surface the ions of the same charge only, while the ions of opposite charge remain in the solution.

37. The absorption of cations on clays depends on valency, ionic radius and hydration of the cation. Very hydrated cations are accompanied by relatively bulky water layer and so the alkalis (Na^{1+}, K^{1+}) are more loosely bonded. Less hydrated and therefore more strongly bonded are Ca^{2+}, Mg^{2+}, Sr^{2+} and Ba^{2+}. The cations H^{1+} are most strongly bonded and are not hydrated.

38. This ion exchange is expressed in milliequivalents for 100 g of the earth and is called "sorption capacity", which is in direct relation to plastic properties of clays.

39. The sorption capacity of some clay minerals is as follows:

Kaolinite	under 15 meqv./100 g
Illite	20 - 40 meqv./100 g
Montmorillonite	60 - 150 meqv./100 g

Flocculation and dispersion

40. If we add to the clay suspension an electrolyte, this can cause a rise in the negative charge of the micelles. The mutual repellency of micelles increases and so a decrease in viscosity occurs.

41. The decrease in viscosity we call liquefying or deflocculation. The most common of the electrolytes, employed for the liquefying of the slurries, are soda, water-glass, protective colloids and condensed phosphates.

42. If this addition of electrolytes exceeds certain limits, the potential of the double-layer decreases, as the thickness of double-layer decreases under the influence of great number of the cations presented. This can lead to the charge compensation on the surface of the double-layer so that the repulsive forces cease to act. On the contrary, under the action of the molecular forces, the mutual attraction takes place and formation of cluster occurs; in other words the flocculation takes place.

43. Electrolytes suitable for flocculation, are the divalent and trivalent cations or acids. In technical practice e.g. calcium hydrate is employed. An organic reagent, dimethylamine, is also used for these purposes.

44. An opposite effect is produced by alkaline compounds capable of precipitating or screening the polyvalent cations, especially Ca^{++}, in the clays. These salts could be neutral also. Some alkaline salts of organic acids (e.g. sodium oxalate), NaF and particularly the condensed phosphates (e.g. sodium hexametaphosphate) are good deflocculants.

Plasticity and viscosity

45. If the ceramic paste, which is a mixture of clay and water, is acted upon by a certain degree of force (pressure), this paste can be seen to be reshaping and gaining various shapes, without any failure in the microstructure. This property we call plasticity.

46. The plasticity of the paste is influenced by many factors, especially by those which are connected in some way with the nature of clay minerals. They are:

a) The particle size;
b) The shape of particles;
c) The number of layers of the liquid enveloping the particles;
d) The thickness of the liquid layers;
e) The magnitude of bond-forces;
f) The absorption of ions on the clay particles;
g) The presence of foreign matter.

47. It is possible to distinguish between the kinetic or latent plasticity and the actual plasticity. The first one is an inner property of the clay and represents the maximum plasticity. The actual plasticity, which can be better called the workability, characterizes the plastic properties in the given conditions. It can be influenced by the addition or removal of water, the addition of grog (inert substance), grinding (that is raising the proportion of small-size particles), etc.

48. As to the causes of the plasticity of clay minerals there are many theories, e.g. the morphologic theory, coming from the shape and size of the particles; the colloid-chemical theory, explaining these properties on the ground of colloid-chemical reactions; and the physical theory, according to which the most significant are the properties of the liquids.

49. The plasticity is a function of the binding power of the water films. This is determined by the specific surface, by ions in the water, and by the nature of the clay mineral. These values are decisive for the swelling and for the absorption capacity also. Therefore it is possible to estimate the plasticity from the mentioned properties.

50. The content of the clay minerals in the earth and its nature decide the plasticity. The illitic clays are more plastic than the kaolinitic ones, but less plastic than the montmorillonites (bentonites).

51. The explanation of the plasticity is very complicated and its objective determination is difficult. Many methods have been worked out, but the values obtained can often be compared only with difficulty. For the production of bricks the plasticity can be estimated to a certain extent by means of simple empiric tests.

Thixotropy

52. Thixotropy is a phenomenon, which can be found with some (especially very plastic) earths. After mixing with water, they give suspensions, which when left at rest become solid and gain shear strength, the characteristic of a solid substance. It can be explained that the flake-shaped lyospheres form together a sort of inner structure, like to a house of cards, in the suspension and it appears as a gel. By mixing or by shaking, this structure is disturbed and the suspension becomes liquid again. If we stop these mechanical actions the suspension becomes solid again. The thixotropic behaviour of the earth depends on:

a) Size and shape of the particles;
b) The porportion of the particle surface to their volume;
c) The concentration of the electrolytes;
d) The polarization of the liquid;
e) The temperature.

53. Deflocculated clay suspensions show often thixotropy. It can occur at brick clay also, especially in silts.

Particle size and specific surface

54. In brick clays the dispersion size distribution can very within these limits:

Particle size	above	1,0	0,2	0,09	0,06	0,02	0,002	under
in mm:	2 mm	2,0	1,0	0,2	0,09	0,06	0,02	0,002
%	0-2	0-3	0-20	8-25	10-30	15-45	20-65	15-50

As the particle size very much influences the properties of the dispersed system, especially colloid systems, there are relations between the particles size of a clay and its physic-chemical and technological properties. From the particle size it is possible to estimate the specific surface of the system.

Technological properties

55. From the employment point of view the most important properties of the brick clays are the technological properties, which influence mostly the manufacture process and the treatment of the earth, and can be taken as a manifestation of physical and chemical properties of the earth.

Forming water

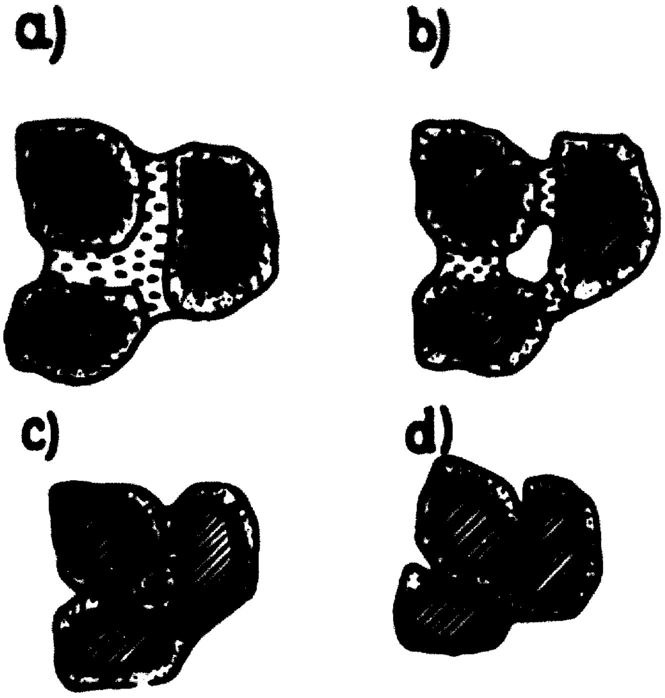
56. The contents of water in the mixture, necessary for obtaining the workable paste, is one of very important technological properties of raw material. According to the amount of this water the forming process differs. We distinguish:

a) Soft-mud process, where the content of water is about 25 $^{w}/o$;
b) Stiff-mud process, where the amount of water is about 12 $^{w}/o$ - 15 $^{w}/o$;
c) Dry-press process, where the content of water is 5 - 7 $^{w}/o$.

Drying

57. Drying we call the process during which the pieces, shaped from the plastic paste, are relieved of water in order to get a body with only minimum amount of water ($w < 2\%$). During drying various changes in the formed piece (or in the paste) takes place (Fig.6).

Figure 6
Mechanism of drying



58. In the first place the removal of water enveloping the particles and which is here also present as pore water, shrinkage takes place. It originates stresses which can have various values and character on the surface and inside of the piece. According to the conditions of the drying this tension can rise and it can lead to the cracks and deformation of the piece. Of great importance is the fact that according to the plasticity the removal of water can be quick or slow and the volume changes can be small or very big.

Firing

59. The chemical and mineralogic composition of the clays is decisive for their behaviour on firing. At higher temperatures, beginning 300°C, the clays lose their water of constitution and become amorphous. The carbonates decompose at higher temperatures (800-900°C). Since these effects go together with a remarkable shrinkage of the material these temperature ranges are critical. The analogous is true of the transformation of quartz at 573°C, particularly on cooling of the burned bricks. Thus it is obvious, that a thorough investigation of the firing behaviour is of great importance if a firing schedule is to be established.

60. The amorphous and dehydrated materials form new compounds by solid state reactions. Kaolinite forms mullite and cristobalite at higher temperatures, after going through a spinelic phase at medium temperatures. Illite forms a glass, CaO from $CaCO_3$ and SiO_2 from quartz combined to form wollastonite at higher temperatures. Finally a glassy phase is formed, which effects sintering and leads to mechanical strength after cooling.

Bloating (expanding)

61. With rising temperature the amount of the glassy phase increases, while the material becomes viscous and finally melts. If the material evolves gases (either by thermal decomposition or by chemical reactions) in a certain range of viscosity (10^6 - 10^8 poise) bloating or expanding occurs. Brick clays frequently show this effect, which is not desirable for the production of bricks. Based on this very effect, however, light expanded clay aggregates can be produced, where the expansion is enhanced by sudden heating. These spherically shaped aggregates are cheap and are excellently suited as insulating construction materials.

II. METHODS OF EXAMINATION AND TESTS

Survey and general discussion

62. What has been said so far about clays already clearly shows how difficult and complex their properties are. A complete description of a clay is only possible in a very well equipped laboratory, where chemical, physico-chemical and technological methods of investigation can be combined. It is interesting to note that there is a marked shift from chemical to physico-chemical methods. For the practical purposes of production, technological tests will be preferred.

63. The examinations for the evaluation of brick clay are not so extensive as is necessary for other ceramic clays. The laboratory tests have mainly two objects:

a) To determine the nature and/or the properties of the raw material or of the raw material mixture,

b) To determine the character and/or the effect of the equipment or of the process.

In the appendix an example of a complete examination and evaluation of a brick clay is given. This test-report is made by a factory for extrusion machines. The costs of this analysis are considerable, but are refunded if a machine is bought. Own examinations and tests should be carried out according to the standards. These are not only explicit, but enable us to compare our results with other analyses. At present not all of the tests for the brick clays are standardized. If possible, corresponding standards for ceramic materials are to be detailed for these cases.

Physico-chemical methods

Microscopic examination

64. Microscopical examination of the clays or of these fractions after dry or wet sieving is a simple method for the determination of the minerals and of the inclusions in the clays. The binocular microscope with medium magnifications is very practical. In the polarization microscope the quartz can be easily identified. The high temperature microscope is useful for studying the firing behaviour of the clays, especially the bloating. Further details on this method are given in paras. 73-75.

(DTA) Differential thermal analysis

65. This method is used more and more frequently in the ceramic industry. It not only enables the determination of the minerals occurring in the clay but renders information on the processes taking place during the heating and firing of clays.

66. The principle of the thermal analysis is the simultaneous measuring of temperatures in the sample and in an inert standard (mostly calcined alumina) during heating or cooling. In Fig. 7 an apparatus is schematically given. The rise of temperature is controlled, and is usually 10°C/min. If at certain temperatures physico-chemical reactions take place, a temperature difference appears, recording an exothermic or endothermic effect. This difference of temperature is plotted against the temperature of the system and we obtain the thermogram which is typical for clay and for single clay minerals (Fig. 8 and Fig. 9 of the test-record).

Thermogravimetric analysis

67. The thermal gravimetric analysis is similar to the DTA. In this case we determine the loss of weight of the sample in relation to the temperature. This can either be done periodically or continually. The loss of weight is expressed as w/o and plotted in a diagram in relation to the temperature. The heating is carried out at temperatures of 200, 300, 400°C etc. or in steps of 50°C for 30 min. Characteristic curves are given in Fig. 9.

Dilatometry

68. Dilatometry is used for the determination of reversible and irreversible changes of the sample. Reactions which occur at firing or cooling are reflected in the dilatometric curve. The sample in the form of a rod is heated up in an electrical furnace and we measure the rod length at different temperatures. The recording of the changes in the length and the temperature can be effected by various methods, automatically or by hand. In most cases the difference of the dilatation between the sample and the rods of the dilatometer is recorded.
Fig. 10 shows a cheap apparatus, which can be self-made. Plotting these length changes against the temperature we obtain the typical curve. On this curve we also notice the regions of sintering accompanied by shrinkage. From this it is possible to estimate the temperature at which the material should be fired and the schedule of burning and cooling.

Figure 7

DTA-Apparatus (Saladin)

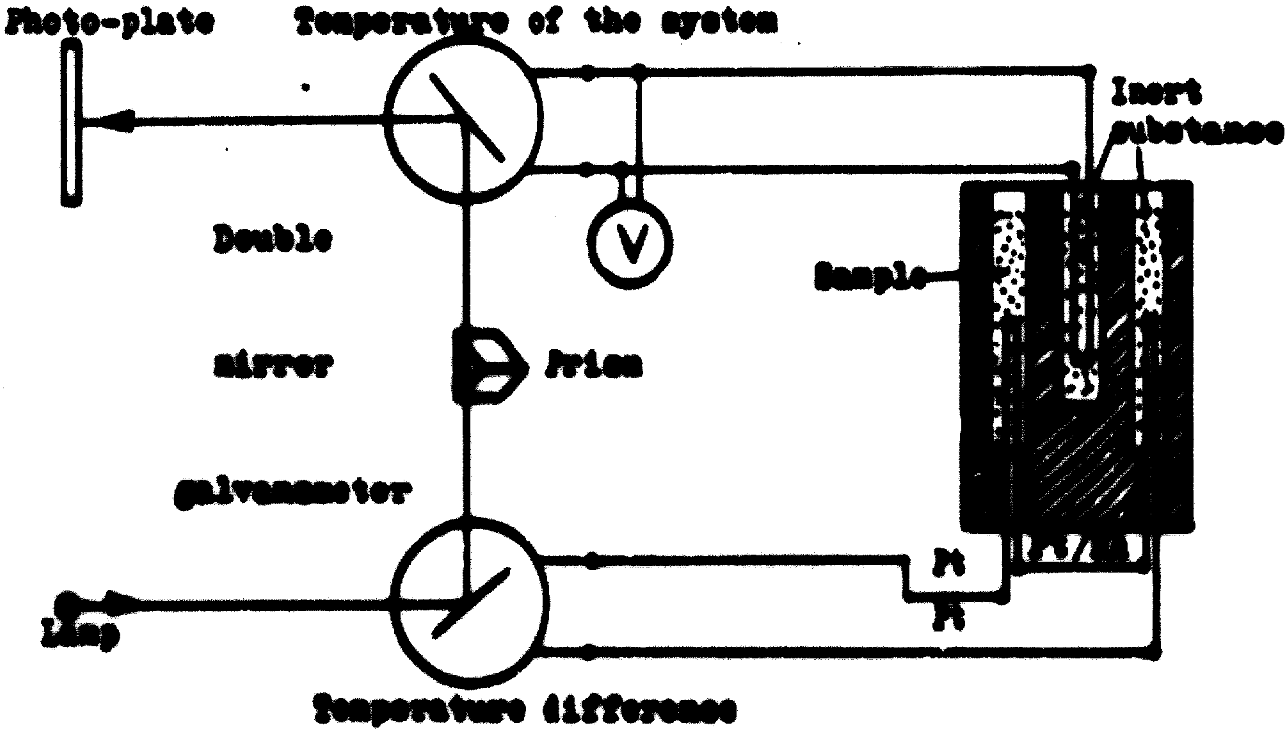


Figure 8

DTA-curves of clay minerals

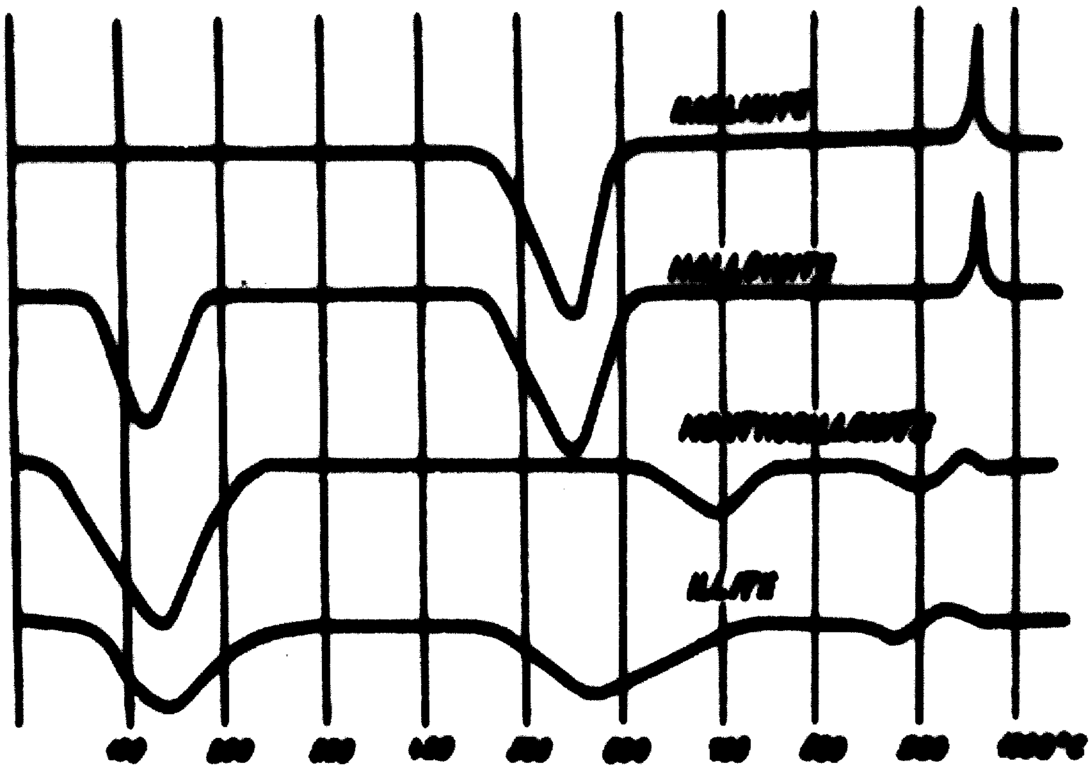


Figure 9

Thermogravimetric curves

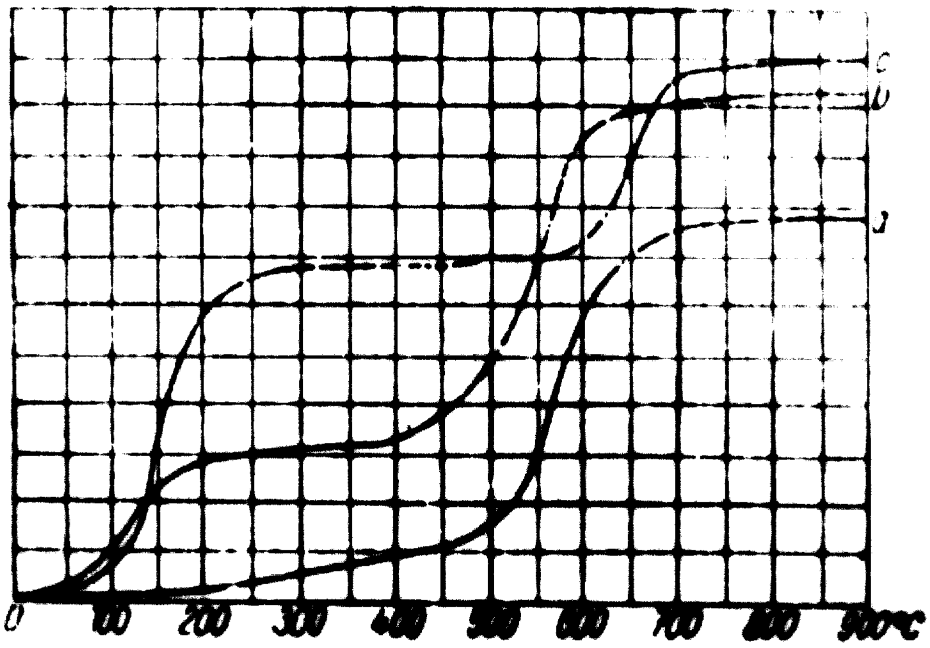
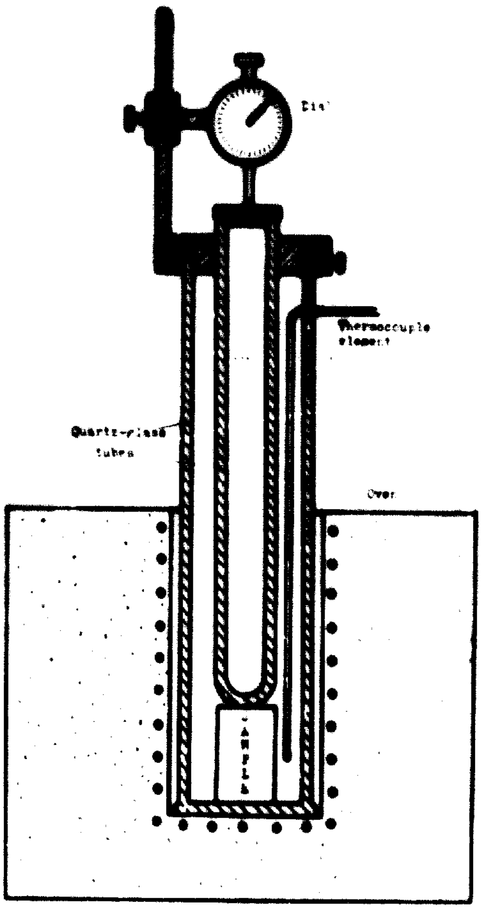


Figure 10

Dilatometer



Chemical analysis

69. The chemical analysis is of great importance for the complete characterization of the clay. If it is desired, the analysis should be carried out according to the ASTM standard C 323-56, "Chemical analysis of ceramic whiteware clays".

70. In general the determination of the content of carbonates ($CaCO_3$) suffices. This can be carried out by means of the calcimeter (Fig. 11). The lower vessel contains HCl (D = 1.17). The weighed sample is put in the boat, the apparatus is closed and the surplus of a saturated solution of NaCl is captured in a graduated cylinder. This volume is reduced to 0°C and 760 mm Hg (a) and used for the calculation of CO_2 content in w/o according:

$$CO_2 = \frac{a . 0.001978 . 100}{\text{weight of sample}} \quad (w/o)$$

The amount of carbonates in the sample is w/o CO_2 .2.27. The dolomite or magnesite is calculated as $CaCO_3$.

Rational analysis

71. The rational analysis is carried out only in exceptional cases with brick clay. It discloses the mineralogic constitution of the clay. There are several methods, which are all based on the fact that if clay is heated to 600-700°C it becomes amorphous by dehydration and thus becomes soluble in strong mineral acids. Thus it is possible to separate the clay minerals from the quartz and feldspar, which are insoluble in mineral acids. The separation of feldspar from quartz is achieved by the determination of the alkaline oxides, or Al_2O_3, which only occur in feldspar. The calcite is determined in the manner as was mentioned above.

72. We should like to point out that the mineralogic composition can be calculated approximately from the complete chemical analysis.

Mineralogical examination of clays (according to P. S. Keeling)

73. Simple tests and techniques have been used to investigate the mineral constitution. The coarse constituents are amenable to low-power microscope examination, but it is first necessary to separate them from the fine material and this is done by standard methods of dispersion and sedimentation. The procedure is designed to remove all material smaller than 20 µ equivalent spherical diameter. The residue is split into two size fractions (coarse and

Figure 11

Calcimeter

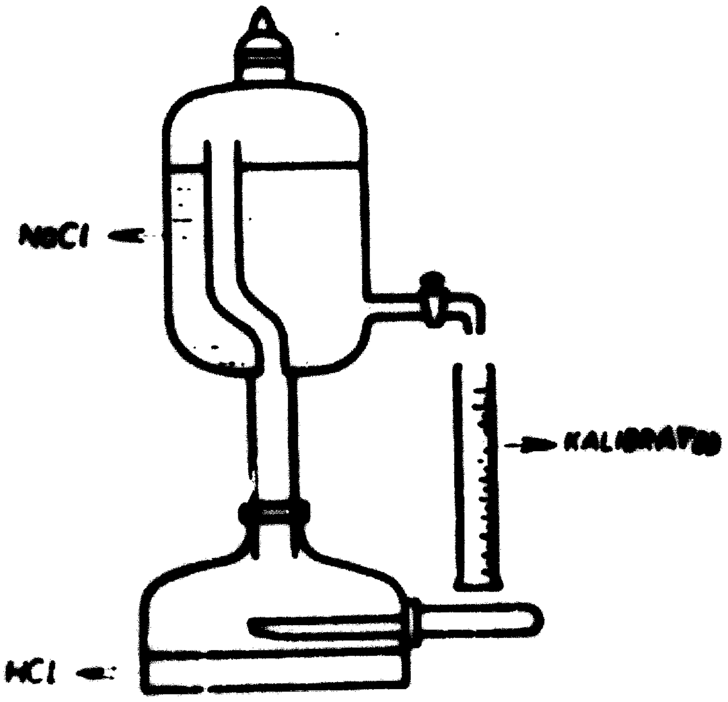
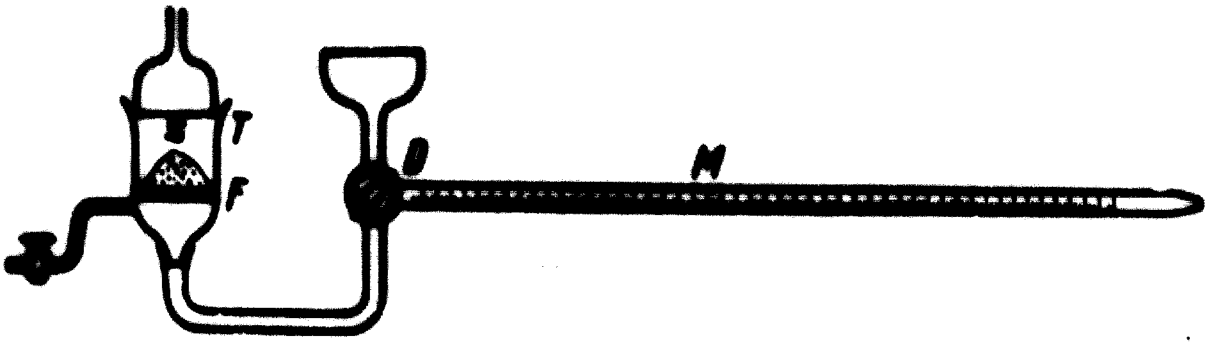


Figure 12

Emlin-Apparatus



S - sample; F - glass frit; T - container for the sample;
M - micropipette; D - three-way cock.

medium) by wet sieving through a 200 mesh (76 μ) BS sieve. The two fractions are weighed and their combined weight is subtracted from the dry weight of the sample. The weights of the three fractions are expressed as percentages of the dry weight and, in the order fine, medium, coarse, give a grading figure that, to some extent, is characteristic of the formation from which the sample was derived.

74. The coarse and medium fractions are examined by means of a binocular microscope with a magnification of X 20. Apart from clay aggregates that occur in the more consolidated clays, the coarse fractions usually contain one or more of the following minerals: quartz, calcite, dolomite, siderite, pyrites, marcasite, gypsum, mica, carbonaceous material, iron oxides and glauconite. Carbonates can be identified by treating with some drops of HCl. The medium fractions generally contain a much larger proportion of quartz grains. Minerals that occur in nodular form in the coarse fractions are not usually well represented. In consequence the medium fractions generally contain a smaller variety of common minerals. Apart from quartz, the following materials commonly occur: calcite, dolomite, mica, carbonaceous material and iron oxides.

75. Information about the clay minerals is obtained from standard ignition loss and moisture adsorption tests. To obtain the ignition loss of the clay minerals, it is necessary to determine or estimate the ignition loss due to non-clay minerals and deduct this from the total ignition loss. The value obtained is dependent on the nature and proportion of the clay mineral. The moisture adsorption values appear to depend solely on the nature and proportion of the clay mineral. For any particular clay, the ratio of clay mineral ignition loss to moisture adsorption (IL/MA) gives a figure that is characteristi of the clay mineral.

Physical tests

Sampling

76. Sampling is an important part of testing and evaluation. The tests have to comprise a representative average of the mineral. The sampling is to be carried out according to ASTM standard C 322-56, "Sampling ceramic whiteware clays".

Appearance of the earth

77. This gives important clues for the experienced man. According to the appearance of the earth it is possible to estimate some of its technological properties, e.g. the presence of coarse impurities rules out the use of such earth for the manufacture of thin-walled products.

78. The colour of the earth varies and mainly depends on the contents of organic matters and on some compounds, especially on iron, calcium and magnesium. The colour shade depends on the moisture. When dried, the earths have a brighter colour. Some of the brick earths are of typical colour, e.g. yellow granites, grey clays, green shales, etc.

79. The facture of the surface of the earth was tested by breaking greater fragments. It varied according to the nature of the earth and its mineralogical composition, and was mostly earthy to granular. The cut of the earth is determined by means of a sharp instrument, the best being a knife. The cut can be smooth and bright, which gives evidence of a finely-grained composition and of the purity of the raw material. In the opposite case, the presence of sand and other impurities will become evident. It is possible, too, to dribble water on the cut and to observe the rate of absorption. The more finely grained the earth is the slower the rate of absorption will be.

80. Calcite can be identified by a simple test. The wet earth is treated with dilute hydrochloric acid. An effervescence indicates carbonates.

81. The homogeneity of the earth can be estimated according to the appearance of the sample. Thus we distinguish between different or identical fragments or grains or perhaps of different layers. The heterogeneous earth needs much more thorough treatment.

Moisture content

82. Moisture content is determined according to the ASTM standard C 324-56, "Free moisture in ceramic whiteware clays".

83. Apart from the standard method, there exists a quick method for raw material and batches. The moisture content is determined by measuring the volume or pressure of acetylene evolved by the reaction between CaC_2 and water. The necessary equipment, consisting of a pressure pot and a manometer, is on the market. Values obtained in this way are somewhat lower than values obtained by drying.

Absorptive capacity (water absorption)

84. The determination of the absorptive capacity expressed as the so-called "Enslin-value" is indicative for the plasticity of the clays and for their mineralogical composition. The apparatus is simple (see Fig. 12), and is based on a glass frit in connexion with a micro-pipette. This micro-pipette records the amount of water absorbed by the weighed sample (0.5-1 g of pulverize and dried clay) on the bottom of the glass frit crucible.

85. The Enslin-value expresses the w/o of water absorption and is calculated according to the equation:

$$EV = \frac{\text{water absorption x 100}}{\text{weight of sample}}$$

The EVs are: for quartz powder 40
for kaolinite or illite 80-100
for bentonite up to 800.

Plasticity (PL) and Workability (WOR)

86. Plasticity is the property of mass to be shaped without any failure in its cohesion. As regards plasticity, we already mentioned in paras. 45-51 that it constitutes a rather complex quantity. Therefore, a great number of different tests is known by means of which it should be determined. For our purposes it is useful to apply the simple method of Rieke-Atterberg.

87. We determine the number of plasticity according to the formula equation

$$PL = w - a$$

where PL = plasticity number.
w = amount of water in w/o in the plastic paste in that moment when a notch, cut into the paste by a knife, does not disappear (flow limit)
a = amount of water in w/o of the paste at the cohesion limit, at which the grains cease to stick to one another.

The cohesion limit is determined as follows:
Rods having a diameter of 3 mm are rolled out. If this rod remains plastic and is coherent, we repeat the rolling until the pieces cease to stick to one another. The water content of this paste is determined by drying and given in whole w/o (a). The results are given in whole w/o of dry earth.

88. According to the plasticity number we distinguish:

Plasticity number (PL)	clay
to 5	low plasticity
5 - 3	middle plasticity
8 - 10	good plasticity
10 - 12	high plasticity
above 12	very high plasticity

89. The workability is directly connected with the plasticity, that is, the ability of the clay to be easily shaped during the technological process. For brick clays workability tests are not standardized. An analogous test for refractory masses is given in the ASTM standard C 181 - 47. A formula for workability is given in para. 110.

Particle size determination

90. The size of mineral particles and their contents in the earth sometimes directly determine or at least influence the decisive properties of the earth such as, for example, plasticity, slaking, shrinkage, green strength, etc. According to the particle size, it is possible to determine the suitability of the raw material for the manufacture of a particular product, for example by means of Winkler's triangle (see chapter III). The particle size determination can be effected in several ways:

a) Sieve analysis:
   i) Wet
   ii) Dry
b) Sedimentation methods (for the determination of the particles under 0.063 mm):
   i) Static
   ii) Floating

Dry sieve analysis

91. This method can be used for dry earth. Especially the coarse impurities in the clays are separated and determined by dry sieving. For the application, see ASTM standard No. C 92-46, "Sieve analysis and water content of refractory materials".

Slaking

92. This test indicates the ease with which the pieces of earth break when placed in water. According to the velocity of breaking, we distinguish:

easily slaking - 0-10 minutes
well slaking - 10-30 minutes
poor slaking - 30-60 minutes
very poor slaking - more than 60 minutes

Wet sieve analysis

93. This analysis is important for the particles the size of which is above 0.06 mm (230 mesh). The wet sieve analysis should be carried out according to the ASTM standard C 325-56, "Wet sieve analysis of ceramic whiteware clays".

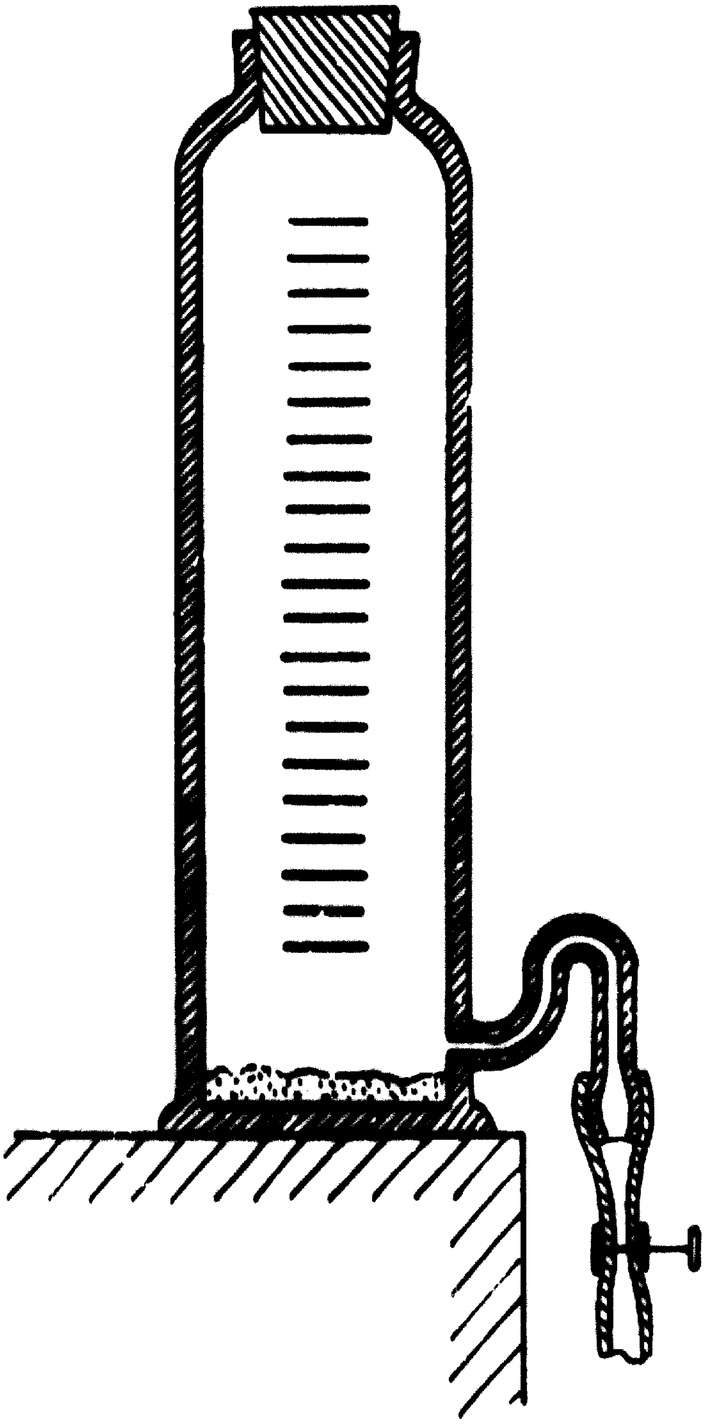
Sedimentation analysis

94. The finest particles below 10 µ which are very important for the properties of brick clays can be determined using the sedimentation method. The sedimentation analysis is based on Stoke's Law, which describes the relationship between the velocity of the particle sinking in a liquid and the diameter of the particle For particles below 10 µ (up to 1 µ), the method of the fixed position pipette (according to Andreassen) is useful. By this method the sum of those finer particles is recorded the falling time of which is longer than the applied time and which therefore are suspended in water. This method is fully described in the British Standard 3406: Part 2: 1963. For this analysis an automatic scale is also available.

95. For particles of 10-60 µ the falling time is short, so that errors during the pipetting easily occur. Therefore the method of separation of the fractions by sedimentation should be applied for these particle sizes. According to the Atterberg method, the fractions in the sediment are weighed after a calculated falling time (according to Stoke's equation) and after which the finer fractions in the suspension are poured out (see Fig. 13). This operation is repeated until the water above the sediment remains clear after the calculated falling time. The sediment is dried and weighed. It gives the sum of the larger particles.

Figure 13

Sedimentation Apparatus (Atterberg)



Representation of the results

96. The results of the particle size determination should be represented graphically. Two variants are possible (Fig. 14). In the upper diagram we find the integral (summation) curve, in the lower diagram there is the distribution curve. The first curve is used for coarser powders, the second curve is very suitable for clays or earths. An example for the comparison of real distribution curves with the optimum curve is given in the test-report.

Technological tests

Preparation of plastic paste

97. The plastic paste is a mixture of a clay and water (w_a = 20-30%), which can be shaped. For its preparation we add water to a clay, pre-treated according t para. 75, until a paste of optimum consistency is formed. As a paste of optimu consistency we take the paste which, while kneaded, does not stick to the hand and at the same time is workable without forming any sort of cracks.

98. After being formed, the paste (about 3-5 kg) is wrapped in a wet cloth or a polythene bag in order to prevent any water being lost, put into a moisture chamber and left there to mature. After one day of maturing the paste is kneaded and homogenized and used for the preparation of test specimens.

99. From the plastic paste prepared as given above the test specimens are prepared either by hand or by a machine. From the hand preparation we use smal size moulds, the sizes of which are:

70 x 35 x 12 mm
100 x 50 x 20 mm

or some other size as the need may be. Before starting to press the paste, th walls of the mould are wetted and a wetted paper of the same size is placed at the bottom to prevent sticking of the paste. The plastic paste is pressed int the mould by hand (Fig. 15). This pressing in is done in parts, and care must be taken that the edges and corners of the mould are also filled. After the mould is filled, the excess paste is cut off by means of a steel wire and the surface is smoothed with a suitable scraper. Then the test specimen is remove from the mould by pushing the bottom upwards.

Figure 14

Grain-size distribution curves

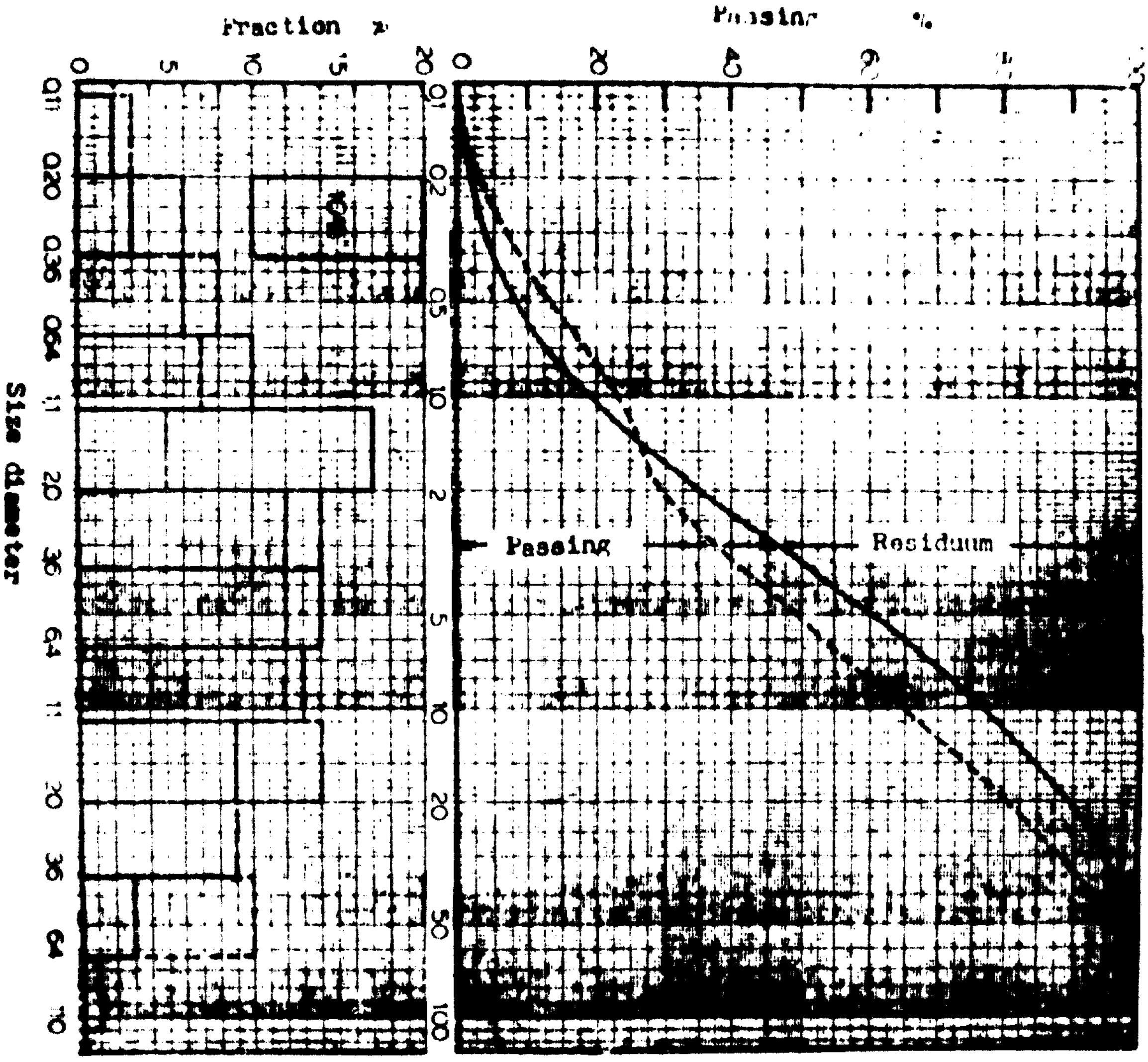


Figure 15

Brass mould (2)

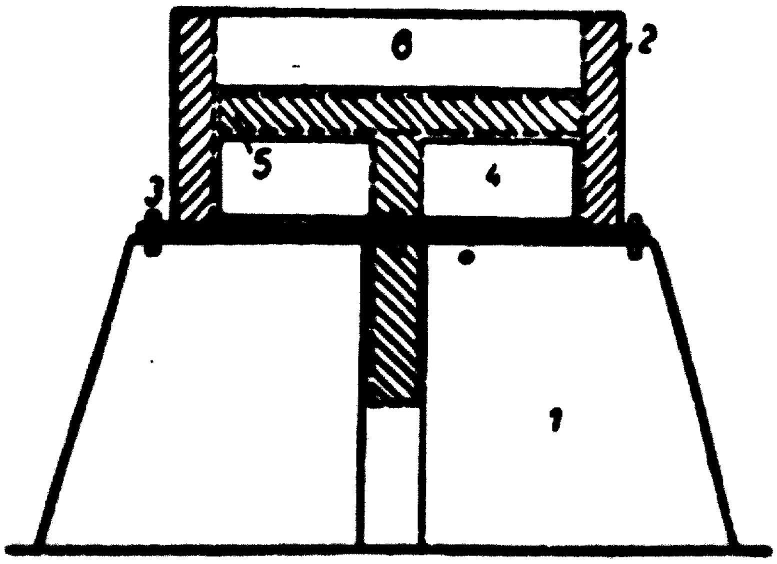
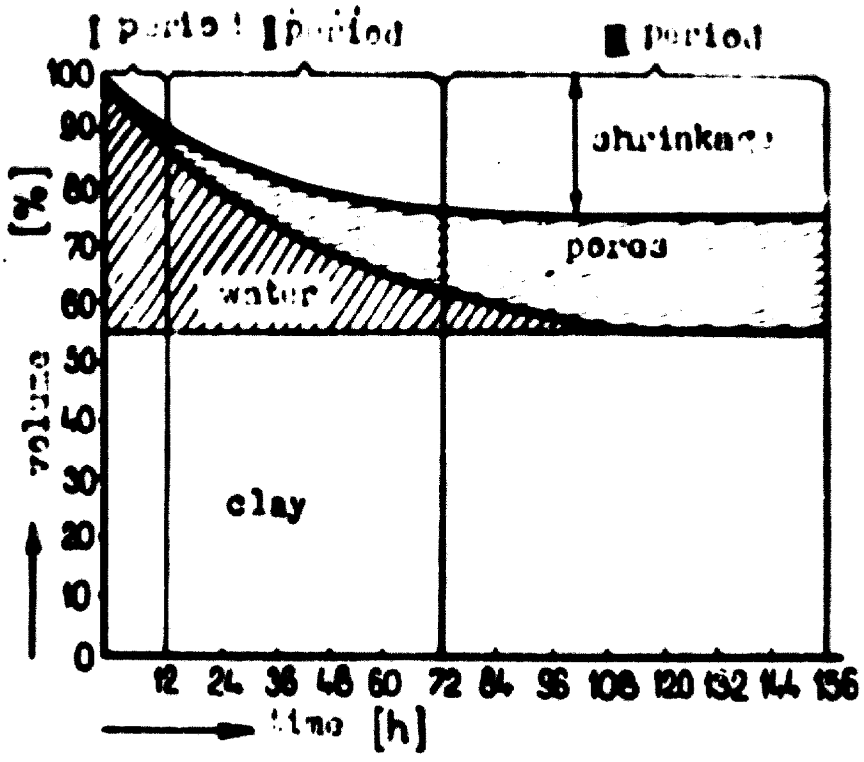


Figure 17

Bourry's diagram



100. For machine preparation we use a suitable auger, from which the paste is extruded in the form of a ribbon 30-50 x 15 mm. This ribbon is cut into strips of 100 to 120 mm length.

101. For the determination of forming water according to ASTM standard C 324-56, all of the test specimens must be weighed immediately after being prepared. The test specimens are then placed on a perforated wooden or steel plate and dried at room temperature.

Drying tests

102. The water migrates through the capillaries to the surface and evaporates there. The way in which the particles come closer is schematically shown in Fig. 6, and the consequences to which this mechanism leads are given in Bourry's diagram in Fig. 17. During the drying process the particles of the paste can approach more closely to one another, since the forming water is gradually removed.

103. The drying shrinkage is finished before all the water which is present in the paste is lost. We distinguish: .

a) Colloidal water (w_o). Water which is removed from the beginning of the drying to that moment when the shrinkage is finished.

b) Pore or critical water (w_k), which is being removed without any shrinkage taking place. This water is located in the pores between the particles.

104. During the drying some shrinkage takes place. According to the rate of removal of the water, stresses in the bodies develop and can cause cracks or deformations. The tendency of the clay to deform and to crack is called the sensitivity of drying. Very often this is estimated by means of Bigot's curve in the form of the coefficient of sensitivity at drying.

Drying shrinkage (DS)

105. The drying shrinkage shows the percentage of linear shrinkage of the test specimens after drying. For the measurement we apply the ASTM standard C 326-56.

106. From the value of the drying shrinkage we can estimate the sensitivity of the clay at drying. If the drying shrinkage is 6-8 per cent or more the earth is considered to be sensitive at drying. This is the case with clays or earth with high amounts of fine particles.

Determination of Bigot curve

107. Bigot's curve gives the relationship between the contents of water (w_a) and the drying shrinkage. The value for Bigot's curve is obtained in the following way: the test specimens are weighed and on the surface two marks are made at a distance of 80 mm. Test specimens are then allowed to dry at room temperature, care being taken that this drying takes place with the same rate on all surfaces. During this process the samples are to be measured and weighed repeatedly (e.g. daily).

108. After reaching the critical moisture, we dry out the test specimens in the oven at 110°C, and after cooling in the desiccator the final values of weight and length are determined. From these values the Bigot curve is constructed (Fig. 18). We determine the critical point. The parallel with axis through the critical point will divide the area of the Bigot curve into two regions. The first region in which the shrinkage occurred is called the danger area; the second region is the security area. The larger this area, the higher is the amount of critical water and the less sensitive is the earth at drying.

Sensitivity at drying (DS)

109. The drying sensitivity can be expressed as the so-called coefficient of drying sensitivity, which can be calculated from the values obtained by Bigot's curve according to the formula:

$$DS_e = \frac{W_a - W_k}{W_k}$$

Where W_a = amount of the water present in plastic paste.
W_k = amount of the water present as critical water.

According to the value of this coefficient we distinguish:

DSe 1 not sensitive at drying.
DSe = 1-2 sensitive at drying.
DSe 2 very sensitive at drying.

Figure 18

Bigot's curve

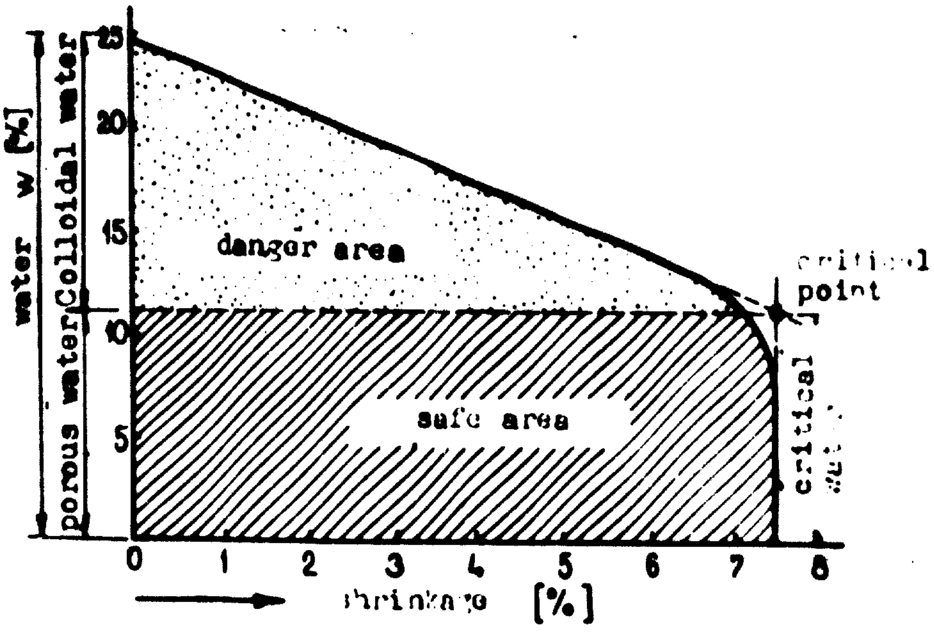
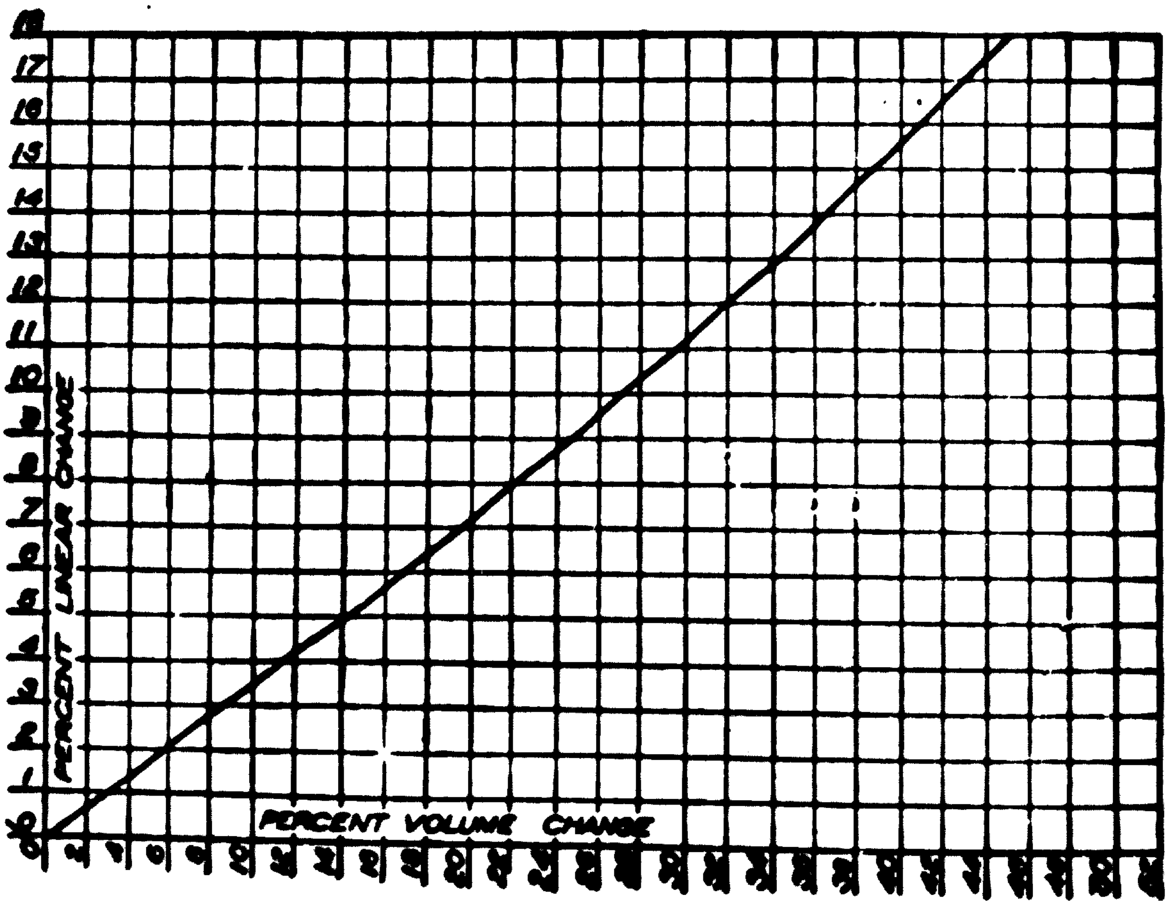


Figure 19

Curve for the conversion of volume to linear shrinkage based on initial dimensions



110. From the mentioned values Bigot has established an equation for the workability index:

$$\text{Workability} = \frac{W_k \times \%\ \text{drying chrinkage}}{W_a}$$

Green strength

111. The green strength is a function of the finest particles. For the determination of green strength we use test specimens 120 x 20 x 20 mm, prepared from the plastic paste usually by extending. After being dried in the usual way they are tested in the apparatus for bending strength according to ASTM standard C 67-57, paras. 7 and 8.

Firing of the test pieces

112. For this test we use the test specimens of the same size as those used for the determination of green strength.

Firing curves (schedule)

113. To examine the relationship between the temperature and the properties the test specimens are fired in an electric furnace at several temperatures. For brick products we choose, for example:

800°, 900°, 1000°, 1100°C or
850°, 950°, 1050°C.

The test pieces, dried at 110°C, must be placed in the furnace in such a way to enable the firing to be applied to all sides of the test specimen. The rate of firing is usually between 100 and 300°C per hour. When the required temperature is reached, it is maintained for two hours. The test specimens are left to cool in the furnace overnight.

Sintering

114. By this we mean all physico-chemical reactions occurring during firing in the bodies and resulting in the strengthening and sintering of the body. This compacting of the body can result in total sintering or even in vitrification. The body reaches the minimum of its water absorption. The sintered bodies have AW 2%, the vitrified bodies have AW = 0.

115. The sintering properties depend on the chemical and mineralogical composition. Very important is the amount of the so-called fluxing oxides, that is, the sum of the oxides Fe, Ca, Mg, K, Na, which cause the formation of easily-melting eutectic mixtures.

116. The amount of the melt depends not only on the amount of these oxides but also on the temperature. The increasing amount of melt during the firing results in filling up of pores and in compacting difficult melting grains, so that the sintered body is of negligible porosity and of a great strength. We can estimate the sintering properties of the body according to the ratio between the melting oxides and the amount of silica/alumina (see diagram, Fig. 20).

117. From the water absorption we can estimate:

The compacting temperature: This is the temperature at which the water absorption reaches AW = 6%.

The sintering temperature: This is the temperature at which the water absorption reaches AW = 2%.

Sometimes the degree of compacting is the ratio between the bulk density (VW) and the specific gravity (SG):

$$\text{degree of compacting} = \frac{\text{Bulk density}}{\text{Spec. gravity}}$$

The nearer this value is to 1, the more sintered the body is.

Bloating

118. The tendency of clays to expand at higher temperatures can limit the maximum burning temperature and therefore debase the quality of bricks. The bloating can be estimated by progressive firing at higher temperatures and by controlling the corresponding shrinkage. Very useful for these studies is the high temperature microscope. For the fabrication of light expanded aggregates, the clays are tested by forming small cylinders, drying them and inserting them in an already heated furnace for a few minutes.

Refractoriness

119. The refractoriness is a property of earth to resist high temperatures without deformations and melting. It is a function of the chemical composition of the clays. This test is unusual for brick clays.

Sound (ring) test

120. For the determination of the degree of firing we can use a simple empiric test by which we estimate the sound of the fired sample. We strike the sample with a hammer and find out the sort of sound (like a metallic "clink"). The more the sample is sintered the more strident the sound will be. When the sound is heavy or duller, this may be due to fine cracks or to a low firing temperature.

Appearance of the test pieces after firing

121. When describing the fired test specimens we particularly notice the colour of the bodies. This colour is to be described precisely, e.g. very bright red, bright red, dark red, etc. After firing at higher temperatures the colours become darker. The colour of the body depends on the furnace atmosphere. With a reducing atmosphere the shade of colour becomes darker. Besides the information about the colour, we describe the formation of cracks, deformations, efflorescence, etc. (see ASTM standard C 62-58 also).

Firing shrinkage

122. Firing shrinkage is a linear contraction, expressed as a percentage of length of the original, non-fired test specimen. The firing shrinkage can be determined according to the ASTM standard C 326-56.

123. Cubic shrinkage can be determined directly according to the volume of the test specimen before and after firing. If the test specimen is regular, its volume can be calculated.

124. Cubic shrinkage can be calculated from the linear shrinkage also. The equation is given in the ASTM standard C 326-56; Fig. 19 shows a conversion nomogram for this purpose.

Water absorption

125. Water absorption gives us the amount of water absorbed by the test piece when completely saturated. It is determined according to the ASTM standard C 67-66, "Sampling and testing brick", paras. 17-19.

Suction

126. The initial rate of the water absorption, i.e. the velocity of absorption, is determined by the suction test according to ASTM standard C 67-66, paras. 25-28.

Saturation coefficient

127. This coefficient can give us some information for the evaluation of the frost resistance of the body. A good frost resistance is expected at a saturation coefficient < 0.85. It is estimated from the difference between the absorption after boiling and absorption at ordinary temperature (see ASTM standard C 67-66, para. 20).

Freezing and thawing

128. The frost resistance is determined by the freezing and thawing test. This test is described in the ASTM standard C 67-66, paras. 21-24.

Apparent and true porosities

129. In ceramics we distinguish the apparent or open porosity and the true or total porosity. The apparent porosity (AP) is the proportion of the open pores of the body to its volume, expressed as a percentage. It can be determined from the water absorption and bulk density. Apparent porosity = Bulk density x water absorption (v/o).

130. True or total porosity TP is the proportion of all pores (open and closed) of the body to its volume, the volume of the pores included. The total porosity can be calculated from the bulk density and specific gravity as follows:

$$\text{Total porosity} = \frac{\text{spec. gravity} - \text{bulk density}}{\text{spec. gravity}} \cdot 100$$

Bulk density

131. The bulk density is the proportion of weight of the body to its volume, including the pores. For the determination see ASTM standard C 20-46.

Specific gravity

132. The specific gravity gives the proportion between the weight and the volume of the mass without the pores. (According to the ASTM standard C 20-46).

Mechanical strength

133. Strength of the fired test specimen can be determined in several ways. We can determine the compression strength, modulus of rupture or the tensile strength - usually the first two. Modulus of rupture (flexure strength) and compressive (crushing) strength should be determined according to ASTM standard C 67-66, paras. 6-12.

Modulus of elasticity

134. The elasticity modulus or Young's modulus (E) is (in the limits of reversible deformations) the relation between the tensile load and the relative elongation of the test specimen. This test is applied only very occasionally to bricks. The elasticity modulus varies between 1 and 2.5 X 10^5 kg/cm^2 and is in direct relation to the strength. An acoustical determination of E is also possible.

Efflorescence and scumming

135. On drying soluble salts, especially Na_2SO_4, K_2SO_4, $MgSO_4$ and $CaSO_4$, in the fired bricks can promote efflorescence by migration with water. These salts can already be found in the raw materials, but they can also be formed on firing in an SO_2-containing atmosphere. The soluble salts in the raw clays can be transformed into insoluble compounds by adding certain chemicals or by suitable conditions at firing (either reducing atmosphere and/or a higher temperature).

136. The tendency to efflorescence should be tested according to the ASTM standard C 67-66, paras. 29-33. The analysis of the soluble salts is described in the BS 3921: 1965, "Bricks and blocks of fired brick earth, clay or shale", para. 45.

137. Scum is a fire indurated insoluble coating, usually white, developed during the process of manufacture on the surface of the bricks from salts within the bodies. The sources of scum are calcium sulfate directly and pyrite indirectly. The calcium sulfate has a high dissociation temperature and remains unreacted after firing. Pyrite is insoluble but at higher temperature, when water is present, forms sulfuric acid which reacts with lime, forming gypsum. For preventing scumming, similar steps as in para. 135 should be taken. For the determination of scumming tendency, an analysis of the soluble salts in the clay should be made. (BS 3921: 1965, para. 45.)

Lime blowing

138. If the raw materials contain limestone nodules (concretions of calcium carbonate), it is necessary to find out their influence on the products after firing.

139. The samples fired at various temperatures are saturated with water. After four hours we observe to what extent the samples are damaged. If there are no apparent cracks or some other deterioration we take the limestone nodules as not dangerous. Even if the limestone nodules are not dangerous they can cause an efflorescence.

Tests on benefication of clays

Freezing through

140. A sample of 500 g clay from the mine is treated at -5°C for eight hours. The mass is thawed at room temperature. After adding two litres of water it is allowed to stand for two hours and then the mass is worked through with the hands and sieved out with a sieve of 170 mesh. The residue is washed with 500 ml of water, dried and weighed. The moisture content of the original sample is also determined. The residue of the frozen sample is compared with the residue of the equally treated reference sample which has not been frozen.

Treatment of dry earth

141. The clay from the mine is preliminarily crushed and spread out to dry. The dry clay is disintegrated to pass through a sieve of 5 mm. Thereafter 500 g of the sample is wet sieved and compared with the reference sample.

Hot water or steam

142. 500 g of clay smaller than 5 mm is heated in a dish on the water bath up to 70°C, mixed with two litres of water of 60-70° and treated on the water bath for two hours. After the wet sieving according to No. 140, the residue is compared with the reference sample.

Vacuum

143. 500 g of clay which is dry and smaller than 5 mm is charged in a vacuum desiccator and treated with steam until the clay is saturated with condensed water. The clay is then evacuated up to a vacuum of 80 per cent. Thereafter the sample is treated as under No. 140.

Grinding and maturing

144. This test is to be carried out mainly for shale clays. These clays are ground up to a size of 3 mm. Several samples of 500 g each are mixed with two litres of water and stored for one, ten and 100 days. The respective residues according to No. 140 are compared with the reference sample. The same test series can also be performed by adding deflocculants.

Leaning

145. The leaning of the too plastic clays for regulation of the drying and firing properties is primarily a technological and an economical problem. Sho tests for leaning are not developed. The mixtures should be examined like the pure clays.

Deflocculation

146. The test is simple. A thick paste is prepared. A part of the paste is separated and approximately 0.3 w/o of sodium metaphosphate (dry) is added and mixed thoroughly. After ten minutes this part of the paste should be markedly softer. If this test is positive, systematical experiments with sodium carbonate can be carried out to determine the optimal amount of the addition.

Flocculation

147. This effect can be achieved with caustic lime or calcium hydroxide. For testing five samples with 2-3 kg containing 0, 0.1, 0.2, 0.3 and 0.5 w/o CaO are prepared by mixing dry clays with lime and then with water until a strong paste is obtained. After fifteen minutes' storage, truncated cones with 10 cm height, 6 cm upper diameter and 10 cm lower diameter are formed. The cones are subjected to 100 strokes on a jarring table to determine the stiffest sample.

III. EVALUATION OF BRICK CLAYS

148. On the ground of testing and evaluation of brick clays we can determine their suitability or, if we make the evaluation in the course of manufacture, the stability of properties for which it was decided to use them.

Optimisation of properties

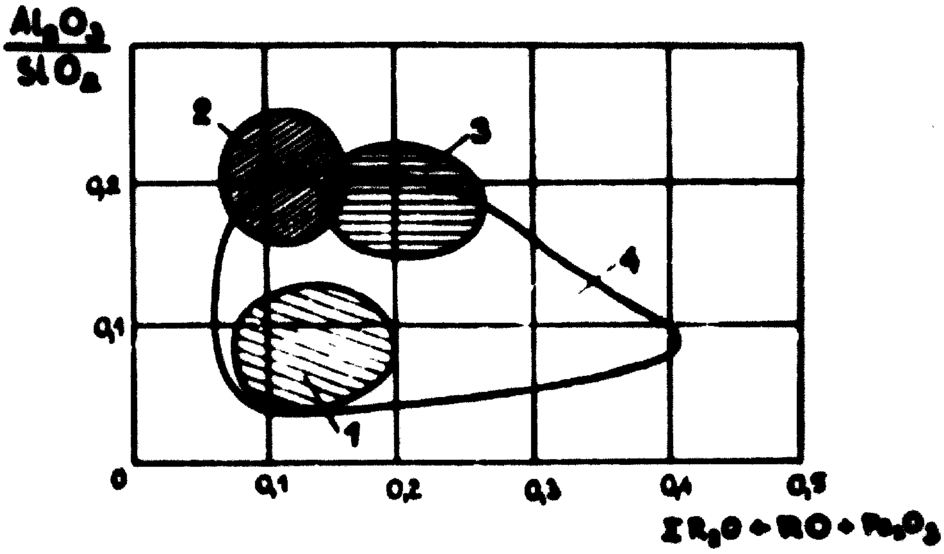
149. Table 2 summarizes the properties, which are required from the bricks, corresponding to the average of various countries.

Various types of brick clays

150. The most important types of brick clays, their properties and the suitab processing methods are summarized in Table 3.

Figure 20

Avgustinik diagram



1 - stoneware; 2 - terracota and pottery;
3 - roofing tiles; 4 - bricks.

Table 2

Criteria of brick clays

Properties	Quality of clays: lean	medium fat	fat	montmorillonitic and illitic
Coarser grains (1)				
max. 7 mm	5–20	2–5	< 2	0
2 mm	35–70	15–35	1–15	1
Forming water (%)	< 20	20–25	25–30	> 30
Drying shrinkage at 110°C (%)	2–4.5	4.5–7	7–9	12–20
Drying sensitivity	< 1	1–2	2–3.5	> 3.5
Modulus of rupture dried (kp/cm²)	15–6	25–15	50–25	50
Firing shrinkage (2) (total) 850°C	2.5–4.6	4.6–7.2	7.2–9.3	13.[illegible]–22
950°C	2.0–4.6	4.6–7.1	7.1–10	14.5–23
1050°C	2.1–4.9	4.9–7.8	7.9–10	16.0–25
Water absorption after firing (2)(3) at 850°C				< 10
950°C	≥15	< 12	8–12	< 5
1050°C				< 2
Modulus of rupture fired at 950°C (kp/cm²)	20	40–20	150–40	150–90
Water elevation (mm) after 90 mm (4)	100 mm	≥70	70–20	20
Suitable for	full or perforated bricks	bricks for great strength, radial bricks	tiles, hollow bricks, drainage pipe	admixture for increasing of plasticity

Notes

(1) residue on sieve 2.0 mm must not contain a greater amount of harmful matters: limestone, pyrite etc.

(2) authoritative are the temperatures employed in the plant

(3) the values of water absorption for earth containing limestone and sulphates are not included in these criteria and must be determined separately

(4) test specimens are of the size stated above.

Table 1
Survey on the brick clays

Indication	Characteristics	Processing
Shales	Slow slaking. Dark coloured by carbonaceous materials. Tendency to bloating.	Weathering, disintegration, adapted leaning and firing schedule.
Marles	40 - 75 $^{w}/o$ calcium carbonate	Benefication by grinding, elimination of coarse limestone, admixture of plastic clays.
Loess, Loams	Containing finest quartz, calcium carbonate and plastic rocks.	Elimination of rock fragments and grinding of coarser impurities.
Silts	A mixture of mineral dust and clays with micas. Simulates plastic clay because of a large drying shrinkage. Low green strength. Expand at low firing. Tendency to crack.	Admixture of plastic clays and simultaneous leaning with coarse material.

Chemical composition and technological properties

151. We find silica (SiO_2) in all clays. Silica is present as a quartz (sand - grog) or as a component of the silicates. With high contents of SiO_2 the earths are sandy, more coarse-grained and therefore less plastic. These earths easily slake in water, are less sensitive to drying and firing but the body is more porous and has a lower mechanical strength.

152. Alumina (Al_2O_3) is part of clay materials and is also present in other aluminosilicates like feldspar, mica etc. Higher contents of alumina lead to a broader interval between the temperature of sintering and of fusion and therefore the bodies are lighter and of greater strength.

153. Iron oxide (Fe_2O_3) is found in earth as a part of various minerals having valencies of two and three. The iron compounds can be an advantage or disadvantage according to their amount and to the type of dispersion in the earth. The greater the amount of the iron oxide in the earth, the lower the fusion temperature. If the iron minerals are present in form of large crystals (pyrite, magnetite, biotite) they may cause defects in the product. Pyrite can yield SO_3, leading to scumming and efflorescence as well. The iron oxide gives red colour of bricks.

154. Lime (CaO) is usually present as in limestone, dolomite, gypsum etc. When a greater amount of finely dispersed limestone is present in the earth we call them limestone earths. At temperatures above 1.100°C, CaO acts as a flux. At lower temperatures and when present as a carbonate it increases the porosity of the product. The presence of CaO also diminishes the interval between the fusing and sintering temperature and changes the colour of the body into a light yellow.

155. Magnesia (MgO) is present in the earth as dolomite, magnesite, or silicate. It causes the compacting of the product, and does not decrease the interval between the temperature of fusion and sintering to such an extent as CaO does.

156. Oxides of potassium and of sodium (K_2O, Na_2O) are predominantly in feldspar and micas. They can yield soluble salts after firing. The alkalis cause the peptisation of the colloids. On firing they act as very effective fluxes.

157. Ignition loss (I.L.) represents that amount of components which is driven off during firing. It consists of water and of carbon dioxide. Ignition loss is greater with the carbonate earths and with earths rich in organic matters.

The organic matters can be humus, bitumen and carbon. Humus acts as a protective colloid and improves the plasticity of the earth.

158. In some cases we determine the amount of the soluble salts in the earth, that which lead to efflorescence and sometimes to the destruction of the products. They are usually sulphates of sodium, magnesium and calcium.

159. Apart from the above mentioned oxides, TiO_2, P_2O_5, MnO and V_2O_5 can be found in traces. They are of no great technological importance. V_2O_5 can cause yellow efflorescence.

160. If the sum of Al_2O_3 and SiO_2 is plotted against the sum of the other oxides in a diagram, we can estimate the applicability of the clays for various products. Figure 20 shows the diagram established for these purposes by Avgustinik.

Mineralogical composition and technological properties

161. The determination of the mineralogical composition, especially the quantitative determination, is much more useful for the determination of the technological properties than the chemical composition. According to the amount of the present minerals we can then estimate the technological properties.

162. The clay minerals are the carries of plasticity. The greater the amount of these minerals in the mixture, the more plastic the mixture will be and the better will be its workability. The amount of clay minerals is, however, subjected to a limit, because of their behaviour during drying and firing. If the limit is exceeded, the drying shrinkage increases and great stresses in the body develop, which can cause cracks and deformation of the products. Similarly the firing shrinkage increases.

163. The less plastic kaolinite can be present in greater amount than the plastic illite, without causing any deformation. Kaolinite increases the temperature of fusion while the temperature of sintering remains constant, thus enlarging the firing range.

164. Montmorillonite increases markedly the flow limit and thus the plasticity and the drying shrinkage. The deformation temperature is lowered while the melting temperature remains constant and consequently the firing range is enlarged. Clays with montmorillonite tend to bloat. The water absorption of fired bricks is lowered.

165. Mica, if present in greater amounts, is inclined to support the texture during the extrusion. If it is very fine-grained and in the form of mica clay minerals (hydromicas), it acts favourably on the strength of the body and on the plasticity of the mixture. Illite influences the firing properties like montmorillonite.

166. Chlorites have a small influence on the firing properties of brick clays.

167. Quertz is usually present in the brick clays in great amounts. Its non-plastic properties and its fire resistance influence the technological process. With increasing amounts of quartz the plasticity, especially the flow limit and the drying and firing shrinkage, decreases. On the other hand a great amount of quartz acts against the strength of the dried and fired products. Quartz increases the sintering and melting temperatures. Undesirable are large grains of quartz which, because of volume changes during firing, cause cracks and deformation in the body. If $CaCO_3$ is present in concretions it may cause a destruction of the fired body owing to the hydration of the calcium oxide, originated during firing.

168. Calcite influences the properties of brick clay more than quartz. Calcite acts on the plasticity through the Ca ions, lowering it. The flow limit is markedly reduced. Calcite decreases the sintering temperature, but the firing range is narrowed. The water absorption is considerably increased.

169. Carbonates, present as magnesite and dolomite, usually present in small amounts, influence the fusion temperature and other properties during the firing, like shrinkage, porosity, strength etc.

170. Iron minerals act unfavourably, provided that they are not finely dispersed or that they are present in certain forms e.g. as pyrite. Fine dispersed $Fe(OH)_3$ has small effect on the technological properties, with the exception of the colour.

171. Feldspars, if present in greater amounts, influence the fusion during firing by their contents of alkalis.

172. Fine-grained silica is not harmful compared to coarse grained silica. The same is true of calcium carbonate, mica or iron compounds. Obviously not only the type of mineral but also its dispersion is important.

Particle size distribution and technological properties

173. In 16-17 and in Figure 1 the size distribution in the clays was discussed. It was stated that the finest fraction consisted mainly of clay minerals and some finest quartz, the middle fraction consisted mainly of quartz and of lime, and the coarse fraction consisted of undesired inclusion like coarse quartz, calcite, gypsum, pyrite etc. The knowledge of the size distribution obviously enables us to draw conclusions about the mineralogical constitution of the clays and to predict their technological properties.

Winkler's triangle

174. To make the technological tests in full is time consuming. There were many suggestions for simplifying these tests. The most useful method of them is a method by Winkler.

175. Studies of more than 50 European clays have shown a distinct dependence of the technological properties on the grain size classification. There were some exceptions, which however were caused by the unusual mineralogical composition. It would be interesting to examine whether other clays, formed under different climatic conditions, would also fit into Winkler's triangle.

176. This method is based on the results of only one test, namely, the size determination. According to Winkler, the decisive factor is the content of the fractions under 0.002 mm, 0.020 to 0.002 mm and above 0.020 mm. The value of this fraction plotted in a diagram gives Winkler's triangle (Figure 21), which can be used not only for the estimation of the suitability of the clay for a certain product (region I - full bricks, region II - perforated bricks, region III - tiles, region IV - hollow blocks) but also for the solution (graphic) of the composition of mixtures from several raw materials or for the determination of the amount of grog.

177. The relationship between the contents of a single fraction of earth and the suitability is proven. From the Winkler's diagram following minimal amounts of grain fractions for various structural clay products can be derived:

Fraction	solid bricks			roofing tiles		large-sized bricks with thin walls		
2 μ minimal	20	24	28	25	30	25	35	45 %
20 μ maximal	50	55	60	45	50	35	32	25 %

Winkler's triangle

I =	II =	III =	IV =
Vollsteine	Gittersteine	Dachziegel u Hohlwaren	dünnwandige großformatige Deckensteine u Hohlwaren
solid bricks	lattice bricks	roofing tiles and hollow blocks	large-sized ceiling bricks with thin walls and hollow blocks
briques ordinaires	briques multicellulaires	tuiles et briques creuses	hourdis à parois minces et de grand format et briques creuses
ladrillos macizos	ladrillos de rejillas	tejas y ladrillos perforados	hourdis de paredes delgadas de gran tamaño y ladrillos perforados
tijolos maciços	tijolos multicelulares	telhas e tijolos furados	hourdis de paredes delgadas de grande tamanho e tijolos furados
mattoni ordinari	mattoni molticellulari	tegole e mattoni forati	mattoni di soffitto a pareti sottili di gran formato e mattoni forati

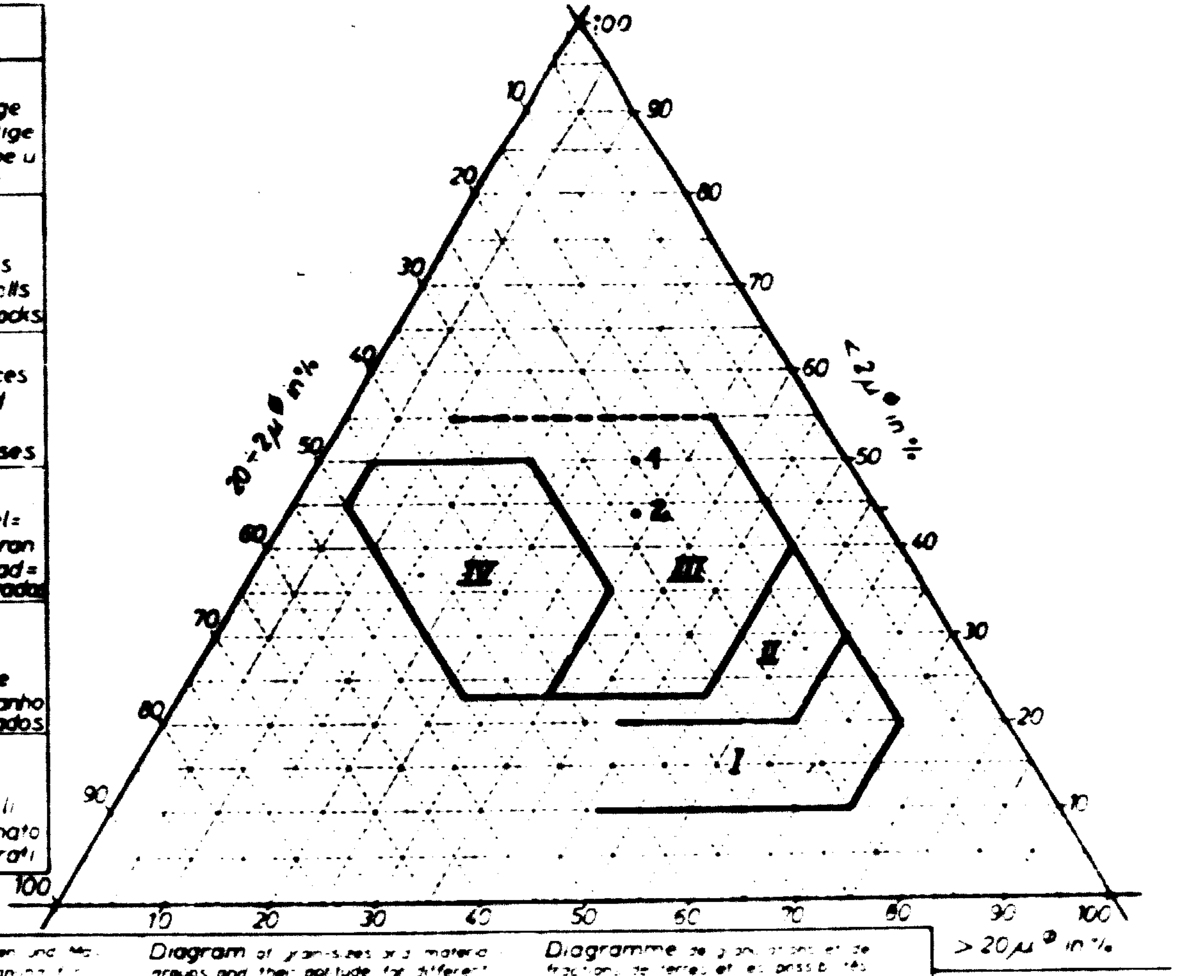


Diagramm von Korngrößen und Materialgruppen und deren Eignung für verschiedene Ziegeleierzeugnisse

Diagram of grain-sizes and material groups and their aptitude for different clay-products

Diagramme de granulations et de fractions de terres et les possibilités de fabrication qui s'offrent

Diagrama de granulaciones y grupos de materiales y sus aptitudes para los diferentes productos cerámicos

Diagrama de granulações e grupos de materiais e as suas aptitudes para os diferentes produtos cerâmicos

Diagramma di granulazione e gruppi di materiali e la sua idoneità per differenti prodotti di laterizi

178. The grain size distribution can also be used to preduct some other important technological properties. Thus the behaviour during drying is a function of the grain size distribution. The higher the portion of finest particles the higher the sensitivity at drying.

179. The frost resistance does not depend on the S value as was assumed up until now, but is a function of the grain size distribution. A high portion of the fractions between 10 and 40 μ at least is decisive for the frost resistance. There is a relatively high amount of quartz in the mentioned fraction. Thus a higher amount of pores having a diameter of 0.8 μ is formed after burning and in addition the texture is diminished on extrusion.

180. The green strength is also a function of the specific surface and thus dependent on the grain size distribution. The plasticity is also partly influenced by the grain size distribution.

Favourable properties of brick clays

181. The favourable properties of brick clays can be summarized as follows: The brick clays or their mixtures must possess such properties that after forming, drying and firing, they give the brick body of prescribed properties regular shape, strength and weather resistance.

182. The favourable properties are such, which lead to:

good slaking with water,
sufficient plasticity necessary for forming,
good workability,
low thixotropy
small drying shrinkage not over 8% and a low sensitivity of drying,
optimal course of sintering process,
small volume changes,
the required physico-mechanical properties of the body, like absorption, porosity, mechanical strength, colour, appearance, bulk density, weather resistance, and other specific properties.

Unfavourable properties of brick clays

183. These properties are opposite to the properties mentioned above. Undesirable are all minerals which influence the properties during the processing unfavourably. This influence depends on the dispersity and on the amount of the unfavourable minerals.

184. Unfavourable properties are too small or too great plasticity, too great sensitivity at drying, low strength after drying and firing, bloating limestone nodules and soluble salts.

Possibilities of benefication of brick clays

185. If the quality of brick clays is not good enough for some sorts of products, it is possible to improve its properties by benefication. The possibilities of clay benefication are various, and it is necessary to consider the character of the raw material, the technical and economic conditions. Table 3, page 40, gives some information concerning this subject.

186. Before molding, the clays are crushed and ground. Thus the coarser components are disintegrated and dispersed. The grains of the non-plastic materials are covered with a coat of clay minerals. By these steps a sufficiently homogenous, plastic and technologically suitable mixture is obtained.

187. The benefication of brick clays in other than mechanical ways, is also possible. Usually, however, considering the expenses, these other ways are but limited.

188. If the raw material contains too much plastic material, and its plasticity is therefore too high (with all other consequences) it is possible to modify its properties by an addition of low plastic clays, of finely grained sand or of grog. If the earth is not plastic enough, its plasticity can be improved by means of grinding, by addition of plastic clay or by removing the coarse grains, e.g. by sieving or by floating.

189. The air bubbles in the clay paste act as a leaning agent. By removing the air from the clay by vacuum, the plasticity and the workability are improved. Vacuum extrusion machines are used for high quality ceramic products.

190. In some cases the earth slakes with difficulty. In this case it is advisable to let the weather act on them. The grain aggregates formed by clay minerals will be dispersed. It is possible to mix the clay with water and leave it to mature.

191. Introducing steam into the extrusion machine the slaking of the clay is accelerated and thus the plasticity is improved. The drying of the already warm bodies is effected much more easily, and the quality of the dried products is improved.

192. An addition of chemicals can improve the workability also. In the case of thixotropic silts loams $^{W}/o$ of CaO (powder of caustic lime) correct the plasticity. On the other hand if a deflocculation of the clay is possible, the quality of the bricks can be improved at lower costs! The plasticity is increased, the amount of tempering water is reduced, the power consumption and the wear of the auger machine are reduced, the running column is smoother, the green strength is increased, and the fired bricks are denser and of a deeper red colour.

Exploitation requirements

193. Besides the technological requirements we must take into account some further requirements which sometimes are not directly connected with the properties of the clays. It is necessary that the nature of the deposit should correspond to the economic conditions and as regards the capacity of the factory, that it should guarantee sufficient exploitation. It is necessary that the mining of the clay should be independent of the weather conditions, as far as possible.

194. The raw material should be deposited in the mine in such a way that the mine may be exploited easily, and at low costs (by hand or by machine). The exploitation must be carried out in such a way that the clays of different layers are always mixed according to the technological evaluation and come into the production as homogeneous mixture. It must be possible to beneficate the clay and to remove the undesired admixtures.

13. 3. 72

2 OF 3

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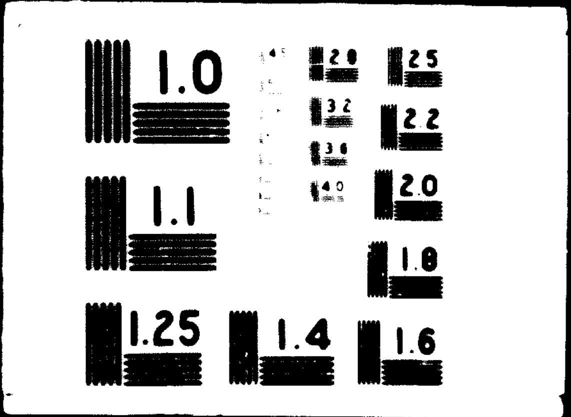


IV. LABORATORY AND EQUIPMENT

General statement

195. Purpose and the function of the laboratory is not only to examine and to control the raw material (clays or earths) and the fired products, but also to find out and to eliminate the faults which can occour from time to time. Therefore the laboratory equipment should have the minimum requirements.

Laboratory rooms

196. The laboratory should consist of two rooms; one 3 x 4 m for the preparation of samples and test specimens so that the other equipment of the laboratory should not be contaminated. In the same room there is a cupboard for the deposit of the samples. The other room is the actual laboratory (4 x 6 m) in which all other operations are performed. A third separated room (or a booth) for the analytical scale or of other corrodible instruments is very useful. This weighing room should be free of external disturbances. It would be advantageous to have a shed in the yard to keep bigger samples or for their preparation.

197. Supply of water, electricity and probably gas should be installed in the laboratory.

198. To the simple equipment belong: laboratory tables 2 x 1 and 1 x 1.5 m; writing desk; a concrete slab 100 x 30 cm, on support, for preparation of pastes or for crushing of samples by hand; cupboard for apparatus; cupboard for chemicals; stand for scales; supports for furnaces.

199. For the physical, chemical and technological tests suitable equipment and apparatus are needed. The price and complexity of apparatus rise according to the nature of tests. The most complicated is the apparatus for the physico-chemical analysis of the clay. The technological tests, especially some of them, need only simple apparatus and tools.

List of most important equipment

200.
Mortar and pestle
Vessels for mixing and boiling (1-5 l)
Equipment for heating of water
Drying oven (controllable)
Furnace for 1100°C
Scale with an accuracy of 0.1 g
Analytical balance
Box of weights 0.1-100 g

Graduate
Thermometer (150°C)
Slide gauges (200 mm)
Sieves: 0.063; 0.09; 0.2; 1.0; 2.0; 6.0 mm
Andreassen pipette
Sedimentation apparatus (Atterberg)
Porcelain evaporating dishes, beakers, bottles
Mould for test specimens
Calcimeter
Enslin apparatus
Apparatus for determination of strengths
Spatula, knife, forceps, crucible tongs
Test tubes
Test tubes rack

List for a complete ceramic laboratory

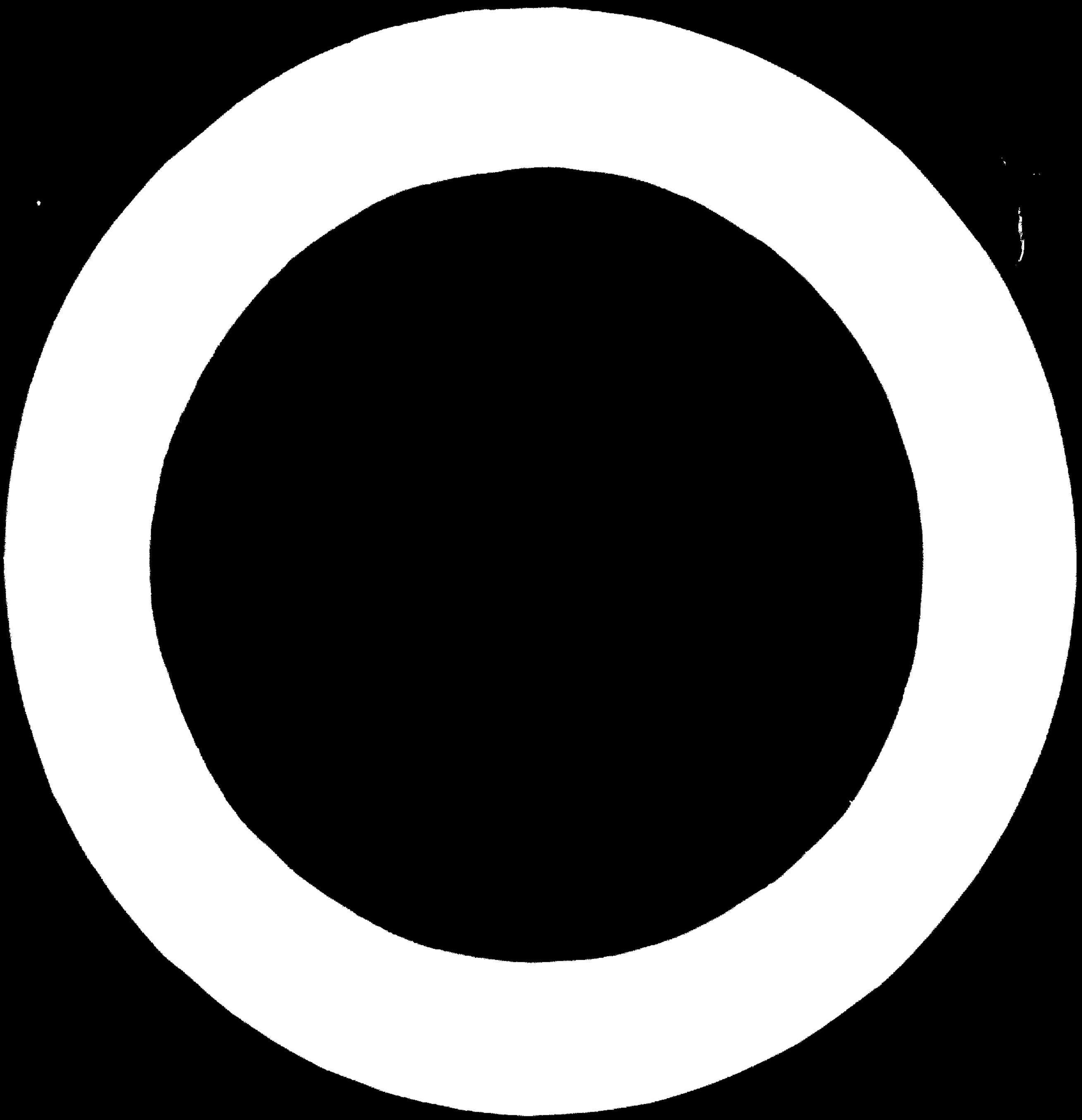
201. Microscope (binocular)
Dilatometer
Apparatus for differential thermal analysis
Laboratory crusher
Laboratory mill
Laboratory auger
Desiccator (vacuum)

Utensils for a complete chemical analysis: platinum dish; platinum crucible; beakers; wash bottle; flasks; clock glass; graduated flasks; pipettes; burettes; glass rods; filter funnels; crucibles; stands; bosser reagents; filters; indicators; burner sand bath etc.

V. CONCLUSION AND RECOMMENDATION

202. This paper was prepared in order to give a summary of the testing and evaluation of brick clays. It is clear that it was not possible to treat all methods of testing in detail and therefore it was necessary to restrict ourselves to simpler tests. If continually testing the earth of the same deposit, the worker will gain not only the necessary laboratory skill but great experience, so that according to the appearance and several tests he can at once evaluate the clay. This can be made even more easily if he prepares samples from different places of the deposit in advance. These samples can be attached to a board and described so that a kind of standard is prepared, and the test specimens can be compared with those standards.

203. Finally, we should like to point out that it is advisable to send the clays or products periodically to special laboratories or research institutes for a detailed examination to make sure of the correctness of the results.



ANNEX 1

Test report

KARL HÄNDLE & SÖHNE Keramikmaschinen — 7130 **MUHLACKER** (West-Deutschland) Transportanlagen

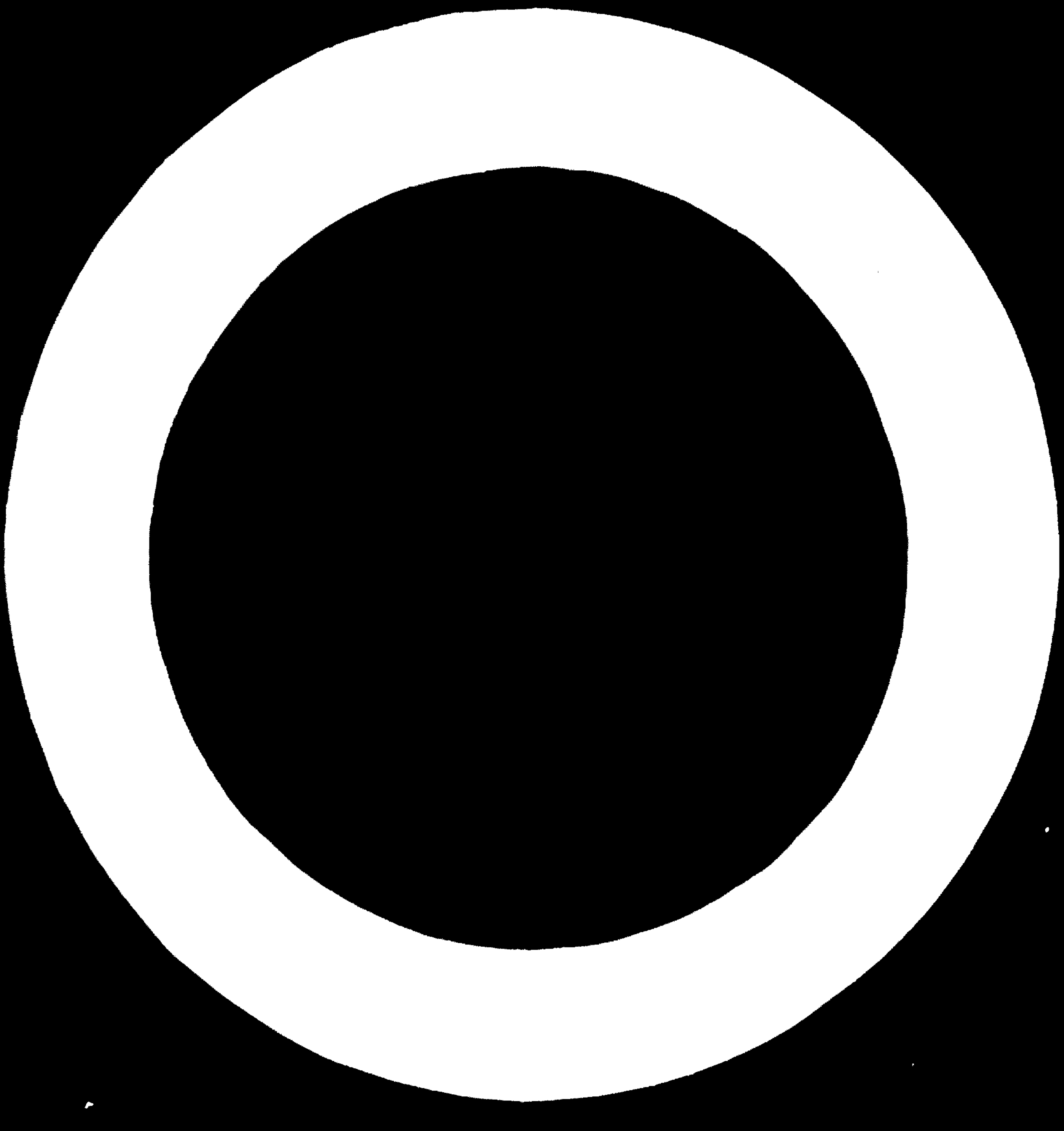
Fragebogen für grobkeramische Betriebe / Enquiry Form

Sorgfältigste Beantwortung des Fragebogens ist Vorbedingung für den Vorschlag der zweckmäßigsten Maschinen. Die Fragen mit Index „x" müssen bei Steinzeug- und Schamotteanlagen **zusätzlich** beantwortet werden.

All questions must be answered most carefully to enable us to quote the most suitable plant for the purpose required. The questions marked with „x" have to be answered **additionally** by stoneware and refractory works.

Firma/Firm in

Rohmaterial — Raw Material Wird der Rohstoff hart oder weich gewonnen?	Is the raw material obtained in a hard or a soft condition?	
x Wird Rohschamotte oder Schamottebruch verwendet und in welchem Verhältnis?	Is raw grog or refractory breakage used and in which proportion?	
Wie groß ist der Feuchtigkeitsgehalt in %, gerechnet auf 100 g trockenes Material?	What is the humidity in per cent, computed on the basis of 100 gm. (3.53 ozs) of dry material?	
Schichtung der Materialsorten in der Grube? (Skizze mit Angabe der einzelnen Materialhöhen und geologische Bezeichnung)	How are the different grades of material stratified in the pit? (Supply sketch indicating thickness of individual materials and geological designations).	
x Wie groß ist der Bindetonanteil in %? Sind es mehrere Sorten Ton?	How high is the share of binding clay in %? Have you various kinds of clay?	
Gewinnung des Materials in der Grube (Bagger, Handbetrieb, Sprengung)?	How is the clay obtained in the pit (by digging, excavating or blasting)?	
Welche Einschlüsse wurden festgestellt (Steine, welcher Art, wie groß und wie hart)?	What inclusions have been found (stones, what kind, size and hardness)?	
Aufbereitung — Preparation In welcher Stückgröße gelangt das Rohmaterial von der Grube zur Aufbereitung?	What is the lump size of the clay as it leaves the pit for preparation?	
x Welche Endkorn-Feinheit wird von den einzelnen Rohmaterialien nach der Aufbereitung verlangt?	Which final grain size of the single raw materials do you require after preparation?	
7 Wurde das Material bisher auf Halde gewintert, gesumpft oder gemaukt? Wie lange?	Has the clay in the past been weathered in the open air, sumped or matured? How long?	
8 Ist beabsichtigt auch eine Sumpfanlage zu bauen?	Is it planned to build a sump?	
9 Wurden verschied. Materialien gemischt und in welchem Verhältnis?	Were different clays mixed, and in what proportions?	
10 Wurden irgendwelche Zusätze bereits zugegeben oder sind sie beabsichtigt? Art und Menge?	Were any admixtures added, or is their addition planned? State types and quantities	
11 Welche Maschinen sind für die Aufbereitung des Materials bis jetzt vorhanden?	What machines are available for preparing the clay?	
12 Ist die Aufstellung einer Mühle zur Ziegelbruchverwertung vorgesehen?	Is a crusher to be installed to utilize broken bricks?	
13 Wie wird das Material von der Grube zur Aufbereitungsanlage gebracht?	How is the clay to be conveyed from the pit to the preparing plant?	



f) **Produktion — Production** 26 Welche Erzeugnisse wurden bis jetzt hergestellt? (Skizze mit Maßen und Abbildungen)	What articles have been produced so far? (Supply dimensioned sketch and illustrations).	
26 x In welchem Umfang der Gesamtproduktion ist Handformerei vorgesehen?	In which percentage of the total production do you intend to use hand-forming?	
27 Welche Erzeugnisse sollen hergestellt werden?	What articles are to be produced?	
27 x Welche Ø von Rohren wollen Sie herstellen und welche Stückzahlen von jedem Ø?	What diameters of pipes will you make and how many pieces of each diameter?	
27 xx Wie lang sollen die fertigen Rohre sein?	Wished length of the ready pipes?	
28 Wieviel der einzelnen Sorten sollen täglich hergestellt werden?	What are the quantities to be produced of each article daily?	
29 Wieviel Stunden werden täglich gearbeitet?	How many hours is the plant operated daily?	
30 Soll in zwei Schichten gearbeitet werden?	Is the plant to be operated in two shifts?	
31 Wie ist bis jetzt die Qualität der einzelnen Erzeugnisse?	How has the quality of each article been in the past?	
32 Zeigen sich Strukturen, S-Risse, Absprengungen, Ausblühungen und wann?	Are S-cracks, lamination, cracking-off and scum encountered, and when?	
32 x Ist eine zusätzliche horizontale Presse zur Herstellung von Rohrbogen vorgesehen?	Do you intend to install an additional horizontal Press for making pipe-bows?	
g) **Transport — Transport** 33 Sollen die Preßlinge von der Presse durch Handkarren, Etagenwagen, Elevatoren und Absetzwagen oder Ringtransporteur in die Trockenräume befördert werden?	Are the clay goods coming from the press or extrusion machine to be conveyed to the dryer on hand transfer stillages, elevators and finger cars or on a monorail conveyor?	
34 Wie liegt die Trocknerei zum Pressenraum? (Skizze mit Entfernungen und Höhenunterschieden)	Where is the dryer located relative to the machine shop? (Supply sketch showing distances and differences in height).	
35 Wie gelangen die Rohlinge zum Ofen?	How are the dried goods conveyed to the kiln?	
h) **Betriebskraft — Power** 36 Welche Betriebskraft ist vorgesehen oder vorhanden? (Dampf mit oder ohne Abdampfverwertung, Dieselmotor, Wasserkraft).	What power is to be used or is available? (steam - with or without utilization of exhaust steam; diesel engine; water power).	
37 Ist elektr. Gruppen- oder Einzelantrieb der Maschinen vorgesehen? Eigenstromerzeugung oder Fremdstrom?	Are the machines to have group or single electric drive? Is the electric power to be produced in the plant itself or purchased?	
38 Welche Stromart, Betriebsspannung und bei Drehstrommotoren welche Periodenzahl ist vorhanden?	State kind of current, main voltage and (if three-phase motors are used) cycle.	
39 Ist genügend Wasser vorhanden und woher kommt es?	Is a sufficient quantity of water available, and where does it come from?	

i) **Aufstellung einzelner Maschinen** **Installation of Individual Units**		
40. Wie ist der Einbau der neuen Maschine vorgesehen?	How is the new machine to be installed?	
41. Welche Kraft in PS steht zur Verfugung?	What is the horse power available?	
42. Wie ist die Tourenzahl u. Drehrichtung der Transmission? (Skizze)	What are the speed and direction of rotation of the transmission shafting? (Supply sketch).	
43. Wie tief liegt der normale Grundwasserspiegel?	What depth is the normal ground water level?	
Sonstiges — Remarks 44. Wieviel Meter ü. d. Meer liegt der Platz?	How many metres above sealevel ist the place situated?	
45. Wie sind die klimatischen Verhältnisse?	How are the climatic conditions?	
46. **Leistung – Capacity** **Die Leistungsangaben zu Pos. 28 sind besonders wichtig!**	**The details of capacity asked for under item 28. are especially important!**	
47.		

3. 63 / 4000

KARL HÄNDLE & SÖHNE 7130 **MÜHLACKER** (West Deutschland)
Keramikmaschinen Transportanlagen

Untersuchungsbericht Nr. 101/67 - 53939 Test-Report

(Verkürzter Untersuchungsbericht ist mit x bezeichnet) (Short Test Report x)

Firma / Firm: in

Materialeingang / Sample received Aug.2 & 14,1967 20 kg Ausgefertigt am / Date December 12,1967

Untersuchungsaufgabe / Test order: General teat of material

Blatt 1, Seite 1
Sheet 1, page 1

1 x	Materialsorte:	Class of clay:	1 white-burning	2 red-burning	
2 x	Mischungsverhältnis:	Mixture ratio:	mass mixed with different materials		
3 x	Unser Kennzeichen:	Our identification No.:	101/1	101/2	
4. x	Art und Zusammensetzung des Materials:	Kind and composition of clay:	kaolin clay poor in alkali (refractory clay)	aluminiferous loam with slight impurities by lime (face brick and clinker clay)	
5 x	Lage und Tiefe der einzelnen Schichten:	Location and depth of the single layers:			
6 x	Materialfarbe:	Colour of clay:	light grey	light brown	
7. x	Beschaffenheit des Materials:	Nature of clay:	prepared material mixes more lean	more fat (plastic)	
8. x	Zustand des Materials nach Eingang:	Condition of clay upon receipt:	semi-dry material mixes, as well as 1 air-dry brick (3-hole brick)		
9 x	Wassergehalt in % auf 100 g trock. Mat. (normal 15—30%):	Water content in %, computed on the basis of 100 gm. dry clay (normally 15—30 %):	11 - 12	8,5 - 9,5	
10. x	Wasseraufnahmefähigk.:	Ability to absorb water (solubility):	altogether well water-soluble		
11 x	Quellfähigkeit: Enslin-Wert	Swelling ability: Enslin-value	good 67	good 64	
12. x	Stückgröße in mm, maximal u. durchschnittl.:	Lump size in mm (max. and average):	not known with these material samples; as grinding stock 4-5 mm in diameter		
13. x	Einschlüsse außer Kalk in %, in welcher Größe und Form:	Impurities (not including lime) in %, giving size and shape:	fired scrap of white material as grog- or brick-dust degreasing and sand other admixtures originating in the clay materials being used, can hardly be traced or noticed	fired scrap of red material	
13.a x	Max. Härte nach Mohs:	Max. hardness as per Mohs:			
14. x	Kalkbeimengungen in %, in welcher Größe u. Form (feinstverteilt nicht mehr als 30%)	Contents of lime in % giving size and shape (not more than 30 % if finely dispersed):	none	sporadic also coarse grained	
	~~CaCO$_3$~~, in %	~~CaCO$_3$~~, in %	-	0,5 - 1%	
15.	~~SO$_3$~~-Gehalt in %	~~SO$_3$~~ contents in % : sulfur	0,034	0,011 (total sulfur)	
	Art und Größe:	Kind and size:	dispersed sulfates besides vanadium ~~with sample 1~~		
	ausblühfähig ja/nein:	Efflorescent yes/no:	le	no (see samples "A")	

3000/7.66

Blatt 2 Sorte 1
Sheet 2 page 1

No.	German	°C	English	°C	1	2	
	Materialsorte (siehe unter 1)		Class of clay: (see under 1)		1 white-burning	2 red-burning	
	Mischungsverhaltnis (siehe unter 2)		Mixture ratio: (see under 2)		according to your information, as mentioned on page 5 of this Report		
	Unser Kennzeichen (siehe unter 3)		Our identification No.: (see under 3)		101/1 1V	101/2 2V	(V = extruded under full vacuum)
16	Wassergehalt bei der Verpressung in % auf 100 g trock. Mat. bezogen (normal 20-25 %):		Water content during forming in %, computed on the basis of 100 gm (3.53 oz.) of dry material (normally 20-25 %):		24,5	19,8	
17	Wasserentzug durch Vakuum in % (normal ca. 0.5 %)		Removal of water by de-airing in % (normally abt. 0.5 %):		- 0,5	- 0,5	
18	Vakuum in % (normal 95 %)		Vacuum in % (normally 95 %):		- 95	- 95	
19	Plastizitat nach Pfefferkorn (mittel 1.35):		Plasticity according to Pfefferkorn (average 1.35):		1,21 1,16	1,21 1,16	
20	Preßdruck in kg/cm² Vollstein:		Pressure in kg/cm² solid brick:		7	7	
	Platte:		plate:		10	10	
	Dachenstein:		ceiling brick:		13	13	
21	Spezifisches Gewicht vom trock. Material in g/cm³		Specific gravity of dry material in g/cm³:		2,55	2,55	
22	Trockenschwindung in % linear (mittelfettes Mat. 6-7 %)		Drying shrinkage in % linear (medium fat clay 6-7 %):		3,7	6,2	
	trockenempfindlich ?		Sensitive in drying ?		no	yes	
23	Gesamtschwindung in % linear (mittelfettes Mat. 7-8 %):	°C	Total shrinkage in % linear (medium fat clay, 7-8 %):	950 °C	4,7	6,7	
				1000	5,5	7,8	
				1050	6,0	8,7	
				1100	7,5	9,7	
				1150	8,5	10,0	
				1200	8,8	start of melting	
				1250	9,0		
				1300	10,5		
				1400	11,0		
24	a) brennempfindlich ?		Sensitive in burning?		no	no	
	b) Garbrandtemperatur in °C:		Full-fire temperature in °C		around 1250	around 1100	
25	Sinterungspunkt in °C:		Point of sintering in °C:		around 1400	around 1100	
26	Schmelzpkt in °C: (Sinter- und Schmelzpunkt mögl. 100° auseinander):		Melting point in °C: (sintering and melting points to be 100° apart if possible):		above 1400	around 1200	
27	Brennfarbe:		Colour after firing:		yellowish-white	brick-red red-brown	
28	Wasseraufnahme in % bei Viellech- und Dachensteinen ca 17 %, bei Dachziegel ca. 13 %	°C	Water absorption in % of perforated and ceiling bricks approx. 17 %, of roofing tiles approx. 13 %	950 °C	20,2	10,2	
				1000	19,4	8,1	
				1050	17,3	5,8	
				1100	13,3	4,4	
				1150	11,4	2,3	
				1200	9,8	-	
				1250	9,0		
				1300	6,5		
				1400	2,6		
29	Frostbeständigkeit, Sättigungswert S: < 0,8 beständig, 0,8-0,9 zweifelh., > 0,9 nicht gewährleistet	°C	Frost resisting power, Saturation value S: < 0,8 frost-proof, 0,8-0,9 dubious, > 0,9 no guarantee.	950 °C	0,98	0,90	
				1000	0,98	0,81	
				1050	0,97	0,72	
				1100	0,95	0,66	
				1150	0,93	0,46	
				1200	0,86	-	
				1250	0,84		
				1300	0,77		
				1400	0,46		

2000/9.66

Blatt 3 Seite 1
Sheet 3 page 1

30	Wasserdurchlässigkeit nach DIN 2250	Permeability to water according to DIN 2250:	101/1 1V	101/2 2V	
	feuchte Flecke nach: feuchte Unterseite nach glänz. Wasserhaut nach Tropfenbildung nach: Tropfenabfall nach: (nach DIN 456 nicht vor 1½ Std)	Damp spots after: Damp under side after: Glossy film of water after: Drops formed after: Dripping after: (not before 1½ hours, according to DIN 456.	-	-	
31	Druckfestigkeit DIN 105: Vollsteine (40 150 kg/cm²) Klinker (350 kg/cm²)	Compressive strength according to DIN 105: solid bricks (600 2130 lb./sq. in) Face bricks (4980 lb./sq. in)	test values with small solid brick samples, with full standard size you will obtain about half of these values: - 860 [illegible]	1400 1700 -	at 1100°C at 1150°C at 1300°C
32	Tragfähigkeit (Mindestbruchlast in kg) Biberschwänze 50 kg Falzziegel 125 kg Flachdachpfannen 200 kg (lufttrocken)	Bearing capacity (lowest ultimate load in kg): Flat Tiles 50 kg Interlocking Tiles 125 kg Flat pan Tiles 200 kg (air dried)	-	-	
	Bruchfestigkeit des getrockneten Formlings in kg/cm²	Breaking strength of dried products in kg/cm²	low 26	high 102	
33	Chemische Analyse in % SiO₂ Al₂O₃ Fe₂O₃ CaO MgO Alkal. Glühverlust:	Chemical analysis in %: SiO₂ Al₂O₃ Fe₂O₃ CaO MgO Alkalies Burning loss:	we have only tested the mineralogical structure according to the DTA-(differential thermal analysis-)Curve, sheet Pt 116		
34	Rationelle Analyse in % Quarz Feldspat: Tonsubstanz.	Rational analysis in %: Quartz: Felspar Clay substance:	-	-	
35 a	Siebanalyse vom Rohmaterial in Gew. %	Sieve analysis of the raw material in weight %	after your crushing resp. preparation:		
	> 2 mm	> .08"	0,4	1,4	
	2-1 mm	.08-.04"	3,3	3,9	
	1-0,5 mm	.04-.02"	5,4	2,1	
	0,5-0,3 mm	.02-.012"	3,8	1,0	
	maximale Korngröße	maximum size of grains:	5 mm in dia.	5 mm in dia.	
36 a	Korngrößenanalyse des aufbereiteten Materials in Gew. %	Grain size analysis of the prepared material in weight %.			
	> 20 µ	> 20 µ	30	33	
	20-2 µ	20-2 µ	20	23	
	< 2 µ	< 2 µ	50	44	
37.	Spezifisches Gewicht vom gebrannten Material in kg/dm³:	Specific gravity of burnt material in kg/dm³	2,5 2,7	2,55 -	at 1100°C at 1300°C
38.	Raumgewicht in kg/dm³	Volume weight in kg/dm³	1,97 2,26	2,19 -	at 1100°C at 1300°C

39

Explanations to the methods of investigations applied by us.

Item 4: Kind and composition of the material is determined according to geological formation and mineral existence by dilatometrical and differential thermal analytical tests.

Item 9: The water content ist determined immediately after receipt of the material and shows us the approximate pit humidity, provided that at least one small sample has been packed air-proof.

Item 10: The ability of the dry material to absorb water and to desintegrate is found by measuring the time of desintegration under water.

0—10 minutes = easily desintegrating
10—30 minutes = good desintegrating
30—60 minutes = bad desintegrating
above 60 minutes = very bad desintegrating

Item 11: The determination of the swelling ability is done in the Enslin apparatus with already prepared material. Mostly the pit material is showing worse values. Hard shales or sands do not swell or only a little. The pure moistening of the surface found hereby can reach up to 20 units.

Approximate values:

above 80 = very high swelling ability
70—80 = high swelling ability
60—70 = good swelling ability
50—60 = less good swelling ability
40—50 = sufficient swelling ability
below 40 = low swelling ability

Item 13: The size and quantity of impurities above 0,2 mm in dia. is determined by the coarse screen analysis (see item 35). The hardness of the material or of the impurities contained therein, is indicated according to Mohs.

Mohs'hardness scale:

1 = talc
2 = plaster
3 = calcit
4 = fluorite
5 = apatite
6 = felspar
7 = quartz
8 = topas
9 = corundum
10 = diamond

Up to hardness 2: can be scratched with finger-nail.
Up to hardness 3: can be easily scratched with knife.
Up to hardness 5: can still be scratched with knife.
Up to hardness 6: window-glass can be scratched.
Above hardness 7: sparking when striking with steel.

Item 14: The determination of lime is made with a carbonic acid determination apparatus according to Baur-Cramer and is indicated as carbonic lime ($CaCO_3$).

Item 15: The determination of the total sulphur content in the raw material is done with a rapid determination apparatus according to Holthaus-Southe.

The determination of the soluble salts in the burnt products is done according to the Percolator method. The content of SO_3 is determined gravimetrically. The alkalis and alkaline earth (Na, K, Ca and Mg) are found by hydrogen flame photometer.

The tendency of the products to efflorescences is determined by lixiviation of samples in distilled water in 1 cm depth of immersion over a period of 14 days with still room air. The kind of occuring efflorescences is determined by the hydrogen flame photometer.

The most white efflorescences arise in form of sulphates of alkalis and alkaline earth as follows: sodium sulphate (Glauber's salt), potassium sulphate, calcium sulphate (plaster) and magnesium sulphate (Epsom salts). Efflorescences of chlorides and nitrates (rot of walls) cannot be contained in the freshly burnt brick, as they are destroyed during burning.

Vanadic and molybdic acid salts, which cause yellow-green efflorescences, occur only in single cases; the same applies to iron sulphate, which causes more rust-red efflorescences.

Also carbonate efflorescences are possible, if, for example, a very calciferous brick contains free lime in higher quantity which is not silicate bound.

Item 19: The ratio of the original to the pressed height ($h_0 : h_1$) of the body tested in the Pfefferkorn-apparatus shows the plasticity number or forming stiffness with the water-content found under item 16.

Item 20: Normal extrusion requires for solid bricks a pressure of 4—6 kg/cm², which may go up to 7 kg/cm² with deairing. If products with holes are made, the pressure goes up to 6—10 kg/cm², depending on number and size of the holes, resp. up to 12 kg/cm² with de-airing. Pressures above 12 kg/cm² indicate a very stiff forming, which is suitable only for single clay-materials and requires reinforced Extruding machines. As maximum pressure, 18 kg/cm² can be taken.

Item 21: The specific gravity of the dried raw material γ_0 is found by means of pycnometer and petroleum. Mostly the values are between 2,5 and 2,65 g/cm³.

Item 22: The drying reaction is tested:

1. directly at the green brick in the drying oven with air circulation, with a temperature of 80° C;
2. by means of a Barelattograph, which indicates in a curve the temporal separation of water and the drying shrinkage.

Item 23: The burning reaction is tested:

1. By drawing an expansion-shrinkage curve, which is taken up in the Netzsch-dilatometer,
2. by burning rows, which are made in the electric kiln with a heating speed of 150° C per hour on an average, and a stop period of 4½ hours at the different full-fire temperatures. The control of temperature is done by thermo-electric element and Seger cone.

Blatt 5 Seite 1
sheet 5 page 1

40	Zusammengefaßte Materialbeschreibung, Vor- und Nachteile,	40	Brief description of material, advantages and disadvantages
41	Verbesserungsmöglichkeiten durch Zumischungen	41	Possibilities of improving clay by admixtures
42	Aufbereitung des Materials und die dazu zweckmäßigen Maschinen,	42	Preparation of the clay and machines recommended therefor
43	Verformung des Materials und die dazu zweckmäßigen Maschinen Evakuierung, Heißverformung vorteilhaft?	43	Forming of the clay and machine recommended therefor Is de-airing and steam introduction considered of advantage ?
44	Trocknung der Erzeugnisse,	44	Method of drying the products,
45	Brand der Erzeugnisse,	45	Method of firing the products,
46	Ziegelei-Erzeugnisse, die aus dem Material hergestellt werden können,	46	Clay-products that can be manufactured from this material,
47	Qualität der Erzeugnisse,	47	Quality of products,
48	Vorgeschlagene Maschinen-Anordnung,	48	Recommended arrangement of machines,
49	Sonstiges	49	Further information
50		50	
51		51	

The light grey, white-burning material from was forwarded to us as a readily prepared material mix, non-extruded, and also as a dry three-hole brick. We have carried out an extrusion test with this prepared material sample followed by a series of fired samples. The samples marked 101/1V (V = extruded under vacuum) were produced in these tests. It is understood that this light-burning mass comprises 60% of a refractory clay taken from different quarries and 40% of a more cream-burning plastic clay deposit. To this clay mix approx. 12% of grog and 6% of a white-burning sand are still added so that the mass is made up of approx. 84% clay portions and approx. 16% pure degreasing portions. This composition of mass is lacking too much of fluxes to manufacture light-burning face bricks, so that abnormally high full-fire temperatures of 1250 - 1300°C will be essential in order to eliminate the yellowish-green vanadium efflorescences, which were still noticed at a temperature of 1150°C (see lixiviation samples "A" fired at 1150°C). As is well known vanadium salts occur very frequently with materials which are rich of kaolinite, and the most secure method to subdue such ugly efflorescences in the brickwork is to fire the material at a very high temperature or to obtain a premature compression of the body resp. sintering. Sometimes a small amount of fluorspar powder is added for neutralization too. The essential high firing temperature used at the moment is very much equivalent to that used for refractory bricks, for which these kaolin clays, which are poor in alkali, can be used better and more economical in the first instance. If it is desired to manufacture white-burning face bricks from this material too, we would recommend to use a light-burning flux in the form of finely ground feldspar or pegmatite in place of a higher addition of sand, so that the firing temperature could be brought down by 50 - 100°C increasing the compression of the body at the same time.

In view of the small Laboratory Extruder which we use for our tests, we were able to extrude the mass at a moisture content of 24.5% related to dry weight, whereby we achieved a moisture content in the brick of 24% after removal of the water due to the full vacuum.

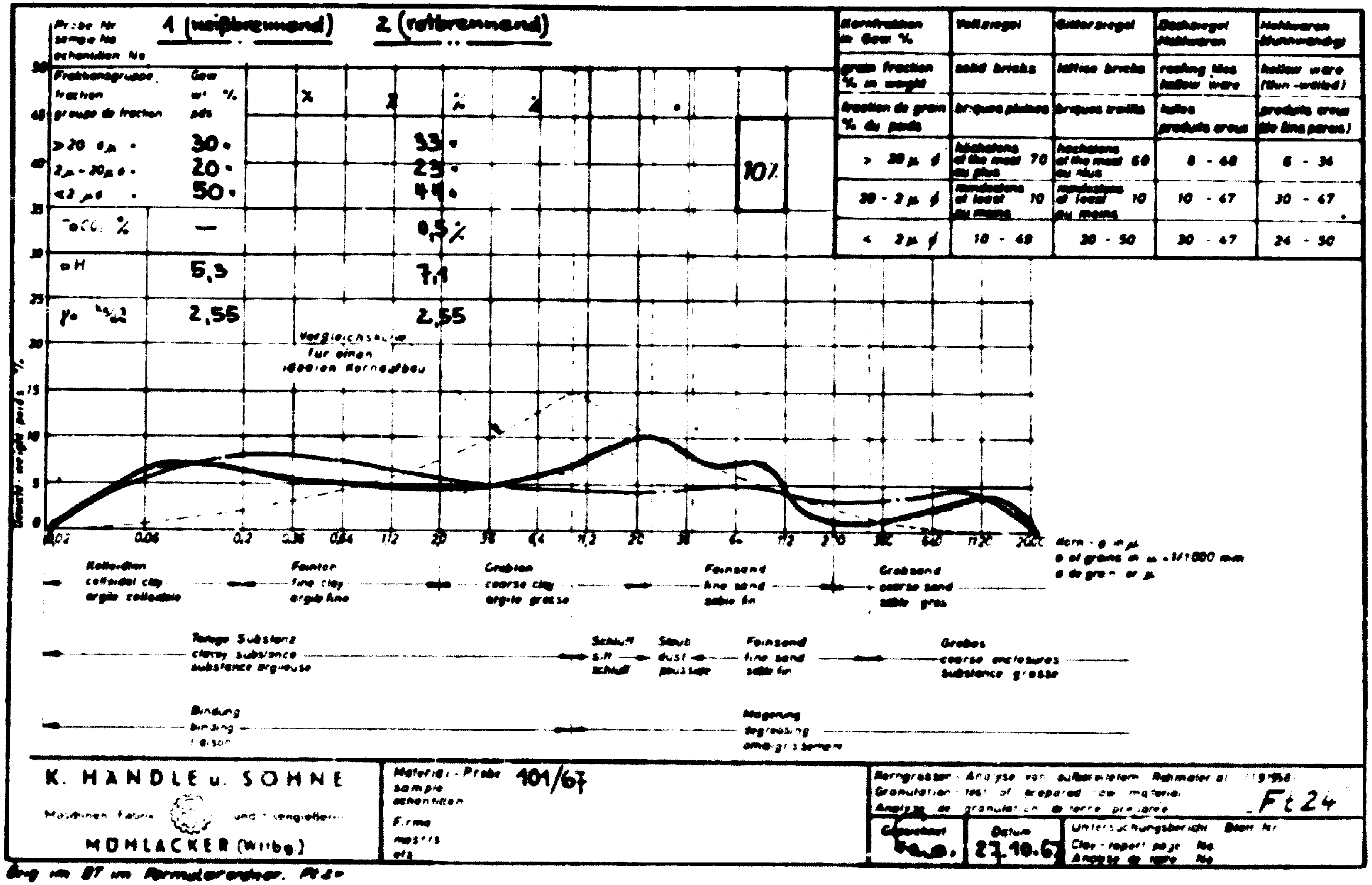
We do, however, also manufacture larger and reinforced Extruders resp. Combined De-Airing Extrusion Machines, which are quite capable of extruding material with a moisture content of only approx. 22% and even lower. For the preceding homogenisation and mixing of the material we recommend our Combined Double-Shaft Mixer with shredding device which will give a much better mixing effect in opposition to the Single-Shaft Mixer. To increase homogenization even more, we would propose a second Double-Shaft Mixer with shredder-knife arrangement to deal with such stiff material mix, instead of a Clay-Shredder, because the final moistening and mixing of the material can be accomplished in such Double-Shaft Mixer more easily and in a more accurate manner. In other respects this white-burning material mix can be extruded easily under full vacuum and there are no difficulties whatsoever in drying and burning. Only for firing face bricks, the firing temperature is much too high with a lacking compression of the body due to the missing portion of feldspar.

The light brown red-burning material from was also despatched to us in an unprepared state, namely a non-extruded sample and a green three-hole brick. On checking the moisture content of this brick we found that the humidity was 17,3% related to dry weight, which is equivalent to 14,7% related to wet weight. Therefore the moisture content mentioned by you is presumably related to the wet weight and not the dry weight as stated in your correspondence. After additional moistening we have also accomplished an extrusion test with this prepared material sample, followed by series of fired samples. These samples bear the number 101/2V (V = extruded under vacuum). We have been advised by you that this red-burning mass consists of a red-burning alluvial clay deriving from the Mississippi River Delta, and of a more buff burning clay, both material being degreased by means of grog (ground fired brick waste). We understand that the mixture ratio is 50% of alluvial clay, 35% of buff burning clay and 15% of brick dust (grog). Here again we do not know in detail the properties of the individual material samples, same as with the white-burning material mix. When drying up the air-tight packed brick already, for the purpose of determining the moisture content in the extruded brick, we noticed a sensitivity in drying with this red-burning material, in opposition to the white-burning mass. Therefore the red-burning mix does not have a larger shrinkage only, but it also shows a tendency towards the formation of cracks. As mentioned above we determined the moisture content in extrusion to be 17,3% related to dry weight, i.e. 14,7% related to wet weight. Consequently there must have been an error at your end when stating a moisture content of 14,7% to be related to dry weight. We could not check anymore if a similar mix-up happened with the white-burning material mix too, since the brick made from this mass reached us in an already dried condition. As the case may be, we tried to extrude both material mixes at the same stiffness in extrusion. In doing this we found that the plasticity and workability was very much the same with both materials, whilst there were bigger differences in the water content in the two material samples. The moisture content in extruding material No. 1 (yellow-burning) on our Laboratory Extruder was 24,8% related to dry weight, whilst it was only 19,8% related to dry weight with material No. 2 (red-burning). However, in spite of its lower moisture content material No. 2 turns out to be more sensitive and also has a larger shrinkage. It is, of course, quite possible with the red-burning material No. 2 to reduce the moisture content in extrusion even more by using stronger Extruders, to enable the bricks to be set straight on tunnel kiln cars. But still this red-burning material mix remains more sensitive, because the Bigot Drying Curves on sheet Pt 101 show a delay in the delivery of water with a longer secondary shrinkage which is demonstrated by the larger curve radius between delivery of water and shrinkage. On top of that there is the greater shrinking ability in spite of the lower moisture content. The sensitivity in drying that results from this, even when employing

stiffer extrusion, is not only defined by the modified structure of grains, but also by the entirely different mineralogical structure of the mass. It is assumed that this drying sensitivity could be overcome more easily or at least diminished by the intense heating up of the mass during stiff extrusion, resp. by steam-heating the material, but this feature must still be taken into consideration whilst it is not in existence with material No. 1.
The series of burnt samples produced from this material No. 2 reveal that this material mix can be used for the manufacture of brick-red face- and common bricks and even for clinkers (engineering bricks) and split tiles if fired carefully. The firing temperature is between 1050 and 1150°C. In view of the varying burning colour and density of the body within this small firing range, it is advisable to delay the final temperature of 1080 - 1100°C for a few more hours. In doing this, endemical (local) differences in temperature or even over-heatings could be balanced more easily, all the more so as deformation and melting must be taken into account from a temperature of 1150°C onwards already. In our screen analysis we have found single coarse grained remains of lime, amongst others, and these will not cause chippings only but also start to flow out from a temperature of 1150°C on. We consider the grinding of the materials with a final grain size of 4 - 5 mm in diameter - according to our screen analysis item 35 - to be too coarse, especially if detrimental lime portions are in existence as it is the case with material No. 2. We would therefore recommend a secondary grinding by using a large size Fine Roller Mill (Smooth Roller), as it will certainly be difficult to pass the material through an even finer screen to obtain a grain size down to 2 mm in diameter in view of the high residual moisture. For Mixing and homogenisation of the materials, also if manganese is added to get a brown colour, we would recommend the same procedure as suggested for material No. 1, namely be installing an additional Double-Shaft Mixer.

1 small box with fired samples is being forwarded to you under separate cover.

December 12, 1967
Lab/Fe/
BVEx/08/Lo.201I



Probe Nr sample No échantillon No	1 (weißbrennend)	2 (rotbrennend)
Fraktionsgruppe fraction groupe de fraction	Gew % wt % pds	
> 20 µ ø	30	33
2 µ - 20 µ ø	20	23
< 2 µ ø	50	44
$CaCO_3$ %	—	0,5 %
pH	5,3	7,1
γ_0 kg/dm³	2,55	2,55

Kornfraktion in Gew % grain fraction % in weight fraction de grain % du poids	Vollziegel solid bricks briques pleines	Gitterziegel lattice bricks briques treillis	Dachziegel Hohlwaren roofing tiles hollow ware tuiles produits creux	Hohlwaren (dünnwandig) hollow ware (thin-walled) produits creux (à fine paroi)
> 20 µ ø	höchstens at the most 70 au plus	höchstens at the most 60 au plus	8 - 48	6 - 34
20 - 2 µ ø	mindestens at least 10 au moins	mindestens at least 10 au moins	10 - 47	30 - 47
< 2 µ ø	10 - 49	30 - 50	30 - 47	24 - 50

Kolloidton / colloidal clay / argile colloidale — Feinton / fine clay / argile fine — Grobton / coarse clay / argile grasse — Feinsand / fine sand / sable fin — Grobsand / coarse sand / sable gros

Tonige Substanz / clayey substance / substance argileuse — Schluff / silt / schluff — Staub / dust / poussière — Feinsand / fine sand / sable fin — Grobes / coarse enclosures / substance grasse

Bindung / binding / liaison — Magerung / degreasing / amaigrissement

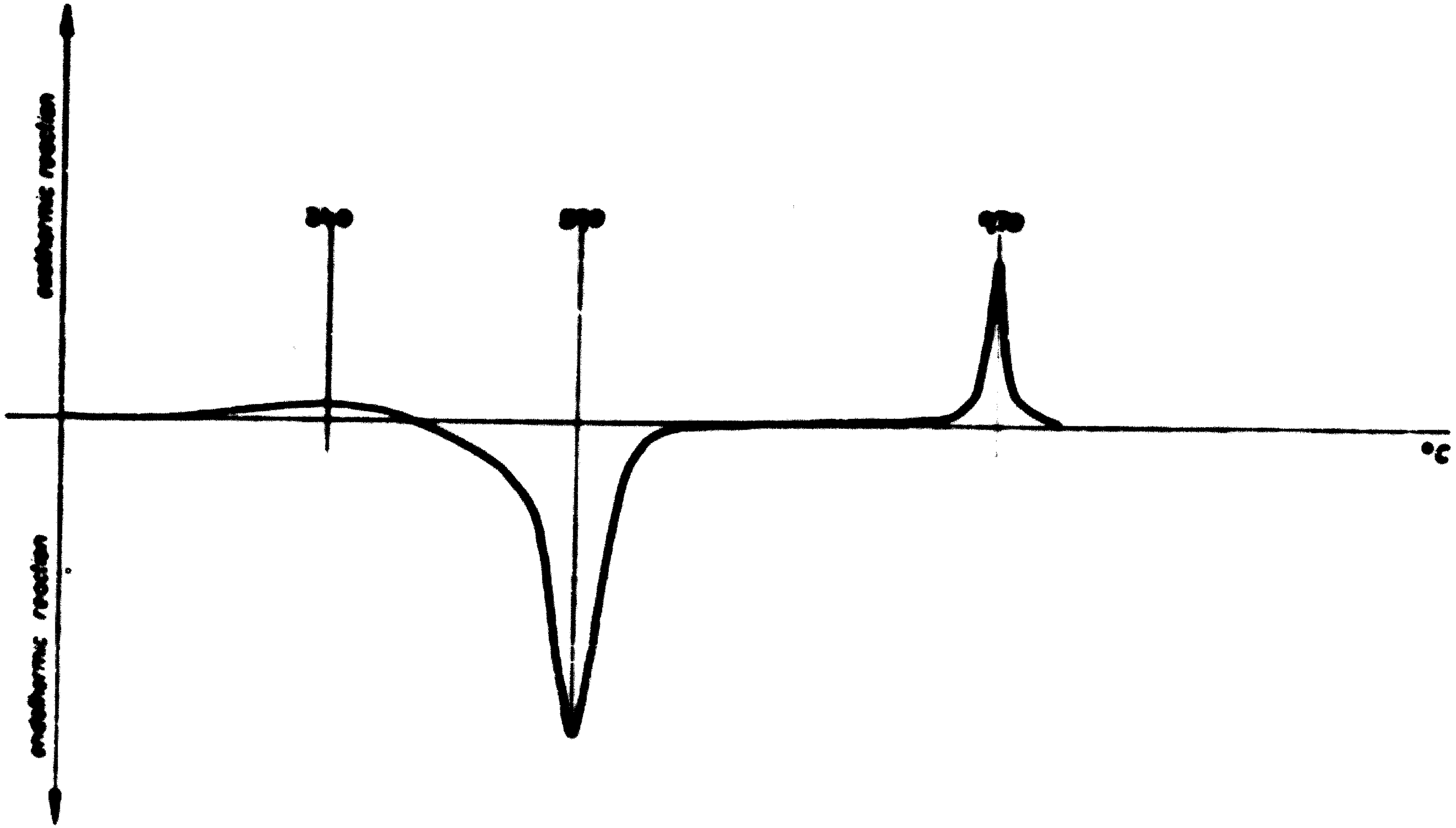
K. HANDLE u. SÖHNE
Maschinen-Fabrik und Eisengießerei
MÜHLACKER (Wttbg.)

Material-Probe / sample / échantillon 101/67
Firma / messrs / ets

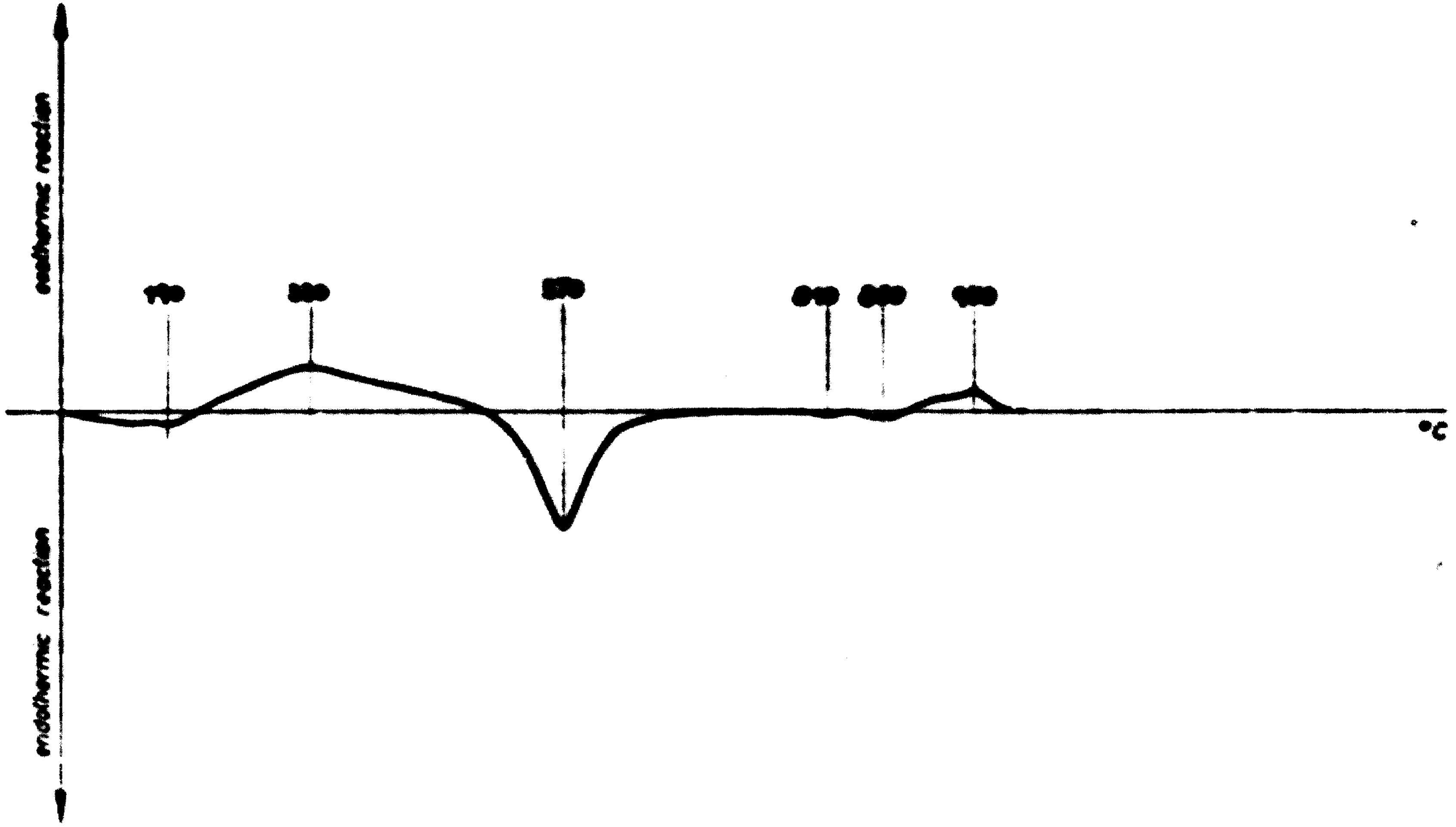
Korngrößen-Analyse von aufbereitetem Rohmaterial (1958)
Granulation test of prepared raw material
Analyse de granulation de terre préparée
Ft 24

Gezeichnet	Datum	Untersuchungsbericht Blatt Nr Clay-report page No Analyse de terre No
[illegible]	27.10.67	

Orig im BT im Formularordner. Pt 2



K. HÄNDLE u SÖHNE Maschinen-Fabrik und Eisengießerei MÜHLACKER (Wttbg.)	**DTA** (differential thermal analysis) **Curve** with a heating-up speed of 10°C/min and a sensibility of 1/10	**Sample No.: 101/1-67**			
		[illegible] [illegible]	[illegible] 10 5 65	[illegible] —	**Fl 116**



K. HÄNDLE u. SÖHNE
Maschinen Fabrik und Eisengießerei
MÜHLACKER (Wttbg.)

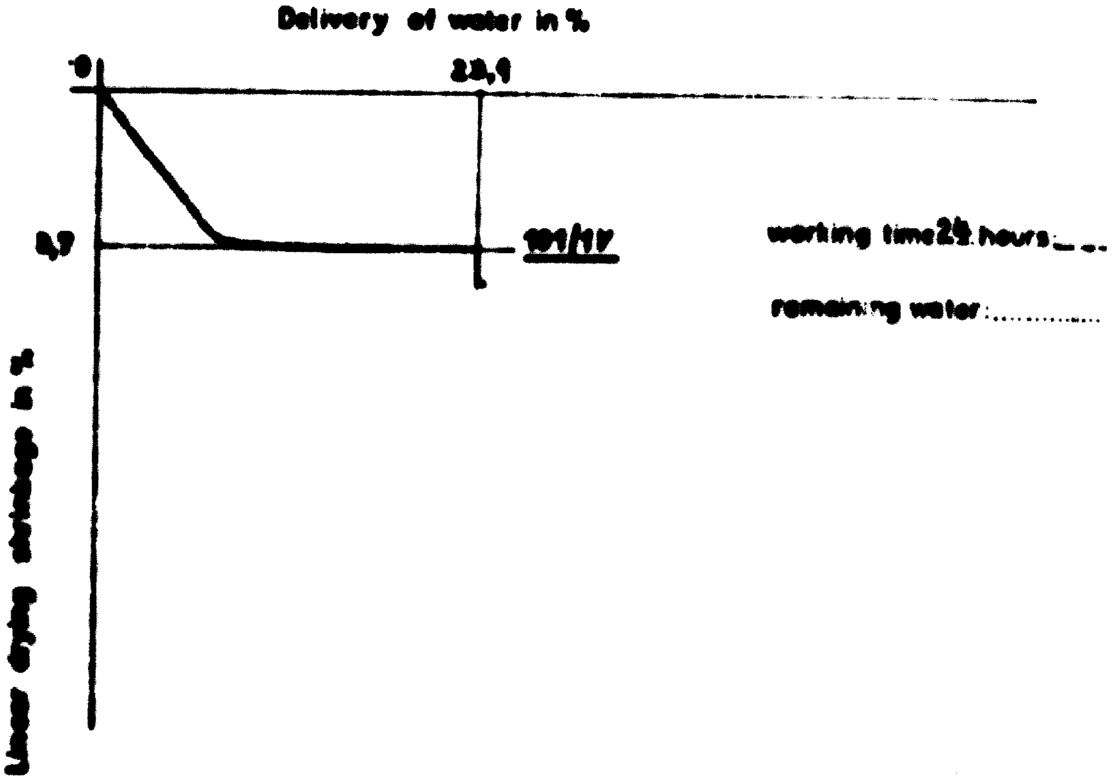
DTA (differential thermal analysis) **Curve** with a heating-up speed of 10°C/min and a sensibility of 1/40

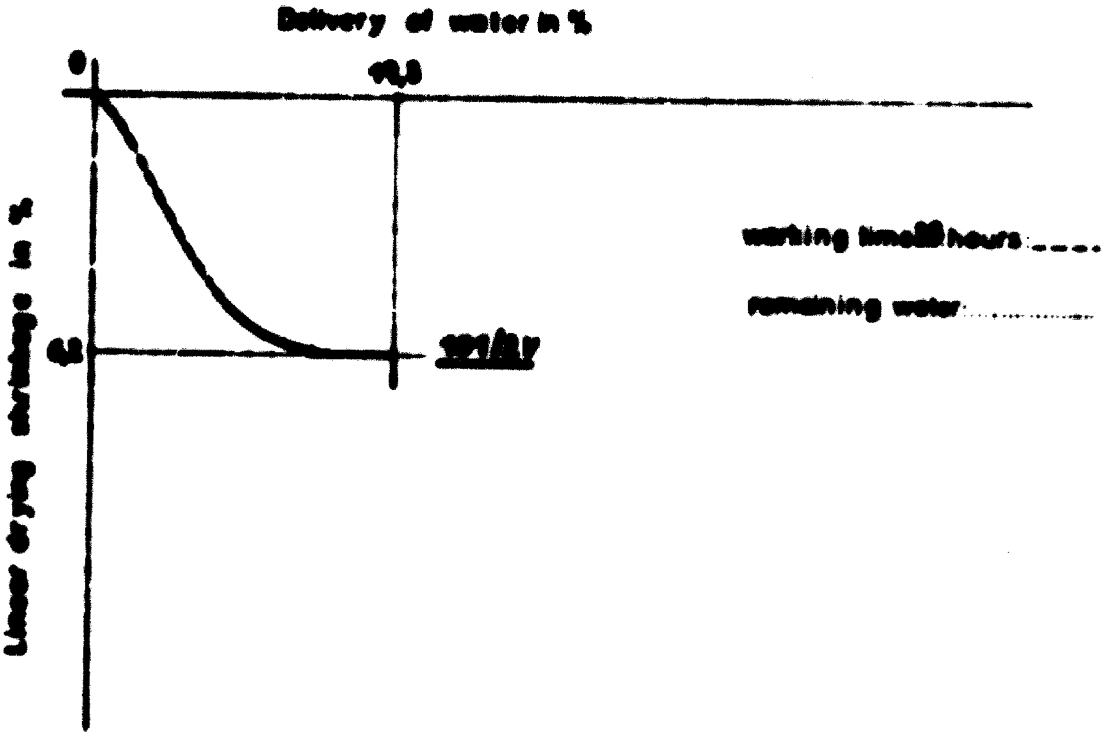
Sample No.: 101/2-67

Datum 10 5 65

—

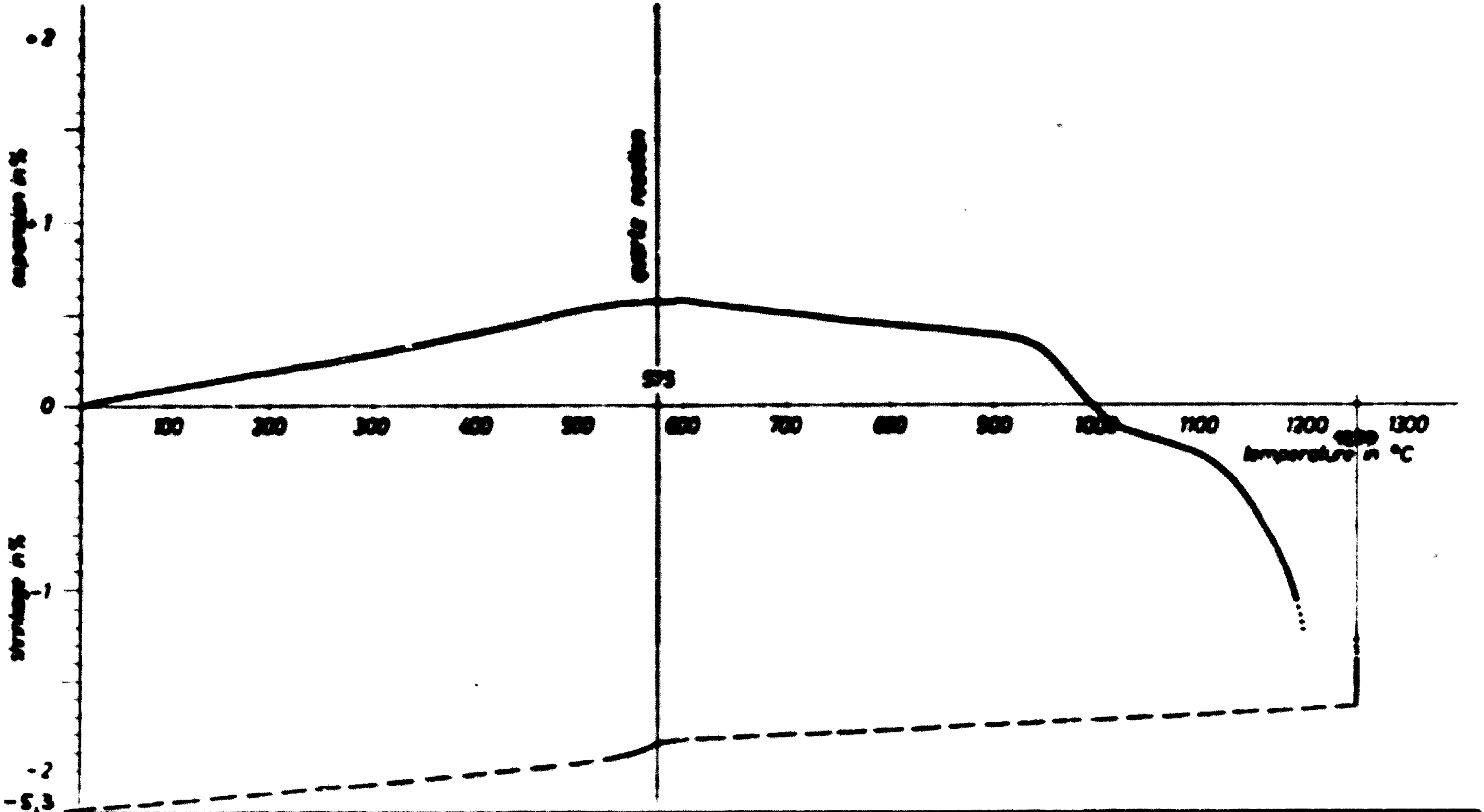
Fl 116



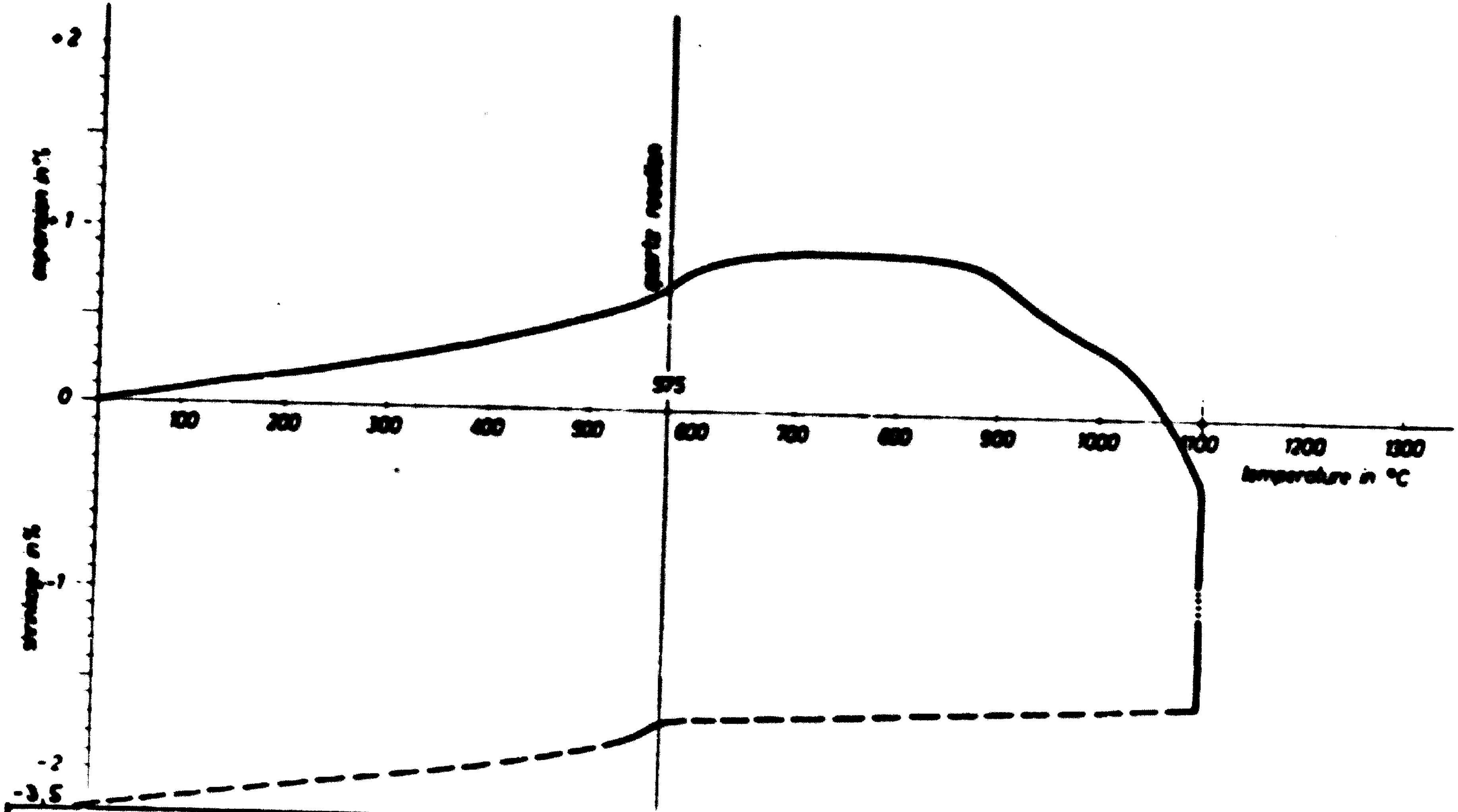


K. HÄNDLE u. SÖHNE MÜHLACKER (Wttbg)	Bigot-Curve with hourly division				
	[illegible]	8 3 65	—	FI 101	Sample Nr 101/67

18.2.67



K. HÄNDLE u. SÖHNE Maschinen Fabrik und Eisengießerei MÜHLACKER (Wttbg.)	*Dilatometer-Curve* with a heating-up speed of 5°C/min and a stop period of 4½ hours at 30 mm length of the test body	heating up ——— cooling down — — —	sample No 101/1-67	
		Gezeichnet Rda. Gosh.	Datum 8 3 65	Maßstab —
				Ft 100



K. HÄNDLE u. SÖHNE
Maschinen Fabrik und Eisengießerei
MÜHLACKER (Wttbg)

Dilatometer-Curve with a heating-up speed of 5°C/min and a stop period of 4½ hours at 30 mm length of the test body

heating up ————
cooling down — — — —

Sample No **101/2-67**

Gezeichnet	Datum	Maßstab	
Fuhr. Conrad	8.3.55	—	Ft 100

KARL **HÄNDLE** & SÖHNE

MASCHINENFABRIK EISENGIESSEREI PRESSWERK · KERAMIKMASCHINEN UND TRANSPORTANLAGEN

Karl Händle & Söhne · 7130 Mühlacker (Württ.) · [illegible]

MÜHLACKER
[illegible]
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[illegible]

Ihre Zeichen	Ihre Nachricht vom	Unsere Zeichen	7130 Mühlacker, den

Betreff: Clay-Test

Dear Sirs,

The knowledge of the raw materials to be handled is always of basic importance for eliminating any existing difficulties in production or when it is contemplated to convert the production to other new products, or for planning a new Ceramic Works.

Mode of Tests:
Our examinations cover the type and composition of the materials and their nature, the ability to absorb water as well as the swelling ability, we determine if the materials include detrimental admixtures such as lime, efflorescent salts etc., we find out the best mixture ratio and determine the shrinkage in drying and burning, the sensibility in drying, the full-fire temperature, sintering- and melting point, colour after firing, porosity (water sorption of the burnt product - with roofing tiles we determine the S-value and the water peremability), structure of grain sizes, we suggest possibilities how to improve the quality of the products by using admixtures and by processing the materials accordingly, we state how the material should be prepared best and which machinery and equipment is required for this purpose, how the material should be moulded with the appropriate machines, if it is advantageous to use de-airing, furthermore we furnish information on how the products are dried and burnt best, what kind of products can be made and what quality can be expected for the final product.

Which amount of material do we require for our examinations?
We need at least 15 kg (33 lbs.) of every type of material. In case where materials for degreasing or other additives (e.g. sand) are processed, we require approx. 15 kg (33 lbs.) of these too, so that we can also test their nature and prepare different mixtures. The material samples have to be forwarded to our address station Mühlacker, postage paid, and every type of material should be packed in an extra bag or tin and marked separately. An extra label should furthermore be provided inside the packing showing the name of the consigner and designating the materials.

p.t.o.

Test-Order:
It will facilitate our examinations, if we can issue exact instructions to our laboratory department in which respect the material samples should be tested, and if we have sufficient information on the size of deposit of the individual samples, the mixture ratio applied until now, the existing machinery for preparing and moulding, the dryers, the burning temperature etc. Any existing difficulties in production, or if it is desired to convert the production to any other products should also be reported to us. It is therefore requested to complete and return to us the attached Questionnaire (1 copy) when giving us an order to carry out laboratory tests. Furthermore 3 dried and 3 burnt bricks should be forwarded to us from the present production.

Time required for the Test:
As we accomplish our tests with every thoroughness, we require in average a period of approx. 3 months from the date of arrival of the material samples.

Charges for our Test:
Since the clay-tests are mainly carried out in connection with the purchase of new machinery, we invoice to our clients a share in costs of DM 600,-- only for one type of material and an extra of DM 200,-- for every additional type of material. There is no extra charge for additives such as sand, brick dust etc. (The costs for such extensive tests if made in laboratory institutes are about 5 times above these figures).

After receipt of the material we confirm the order for the clay-test as we usually do with any other order, and the share in costs are mentioned in the order confirmation. The Test-Report will be submitted after receipt of the amount which has been invoiced.

Any costs charged to us by shipping companies for the handling of the sample consignment, particularly in the event of clay samples expedited from overseas countries, will have to be fully invoiced as an extra.

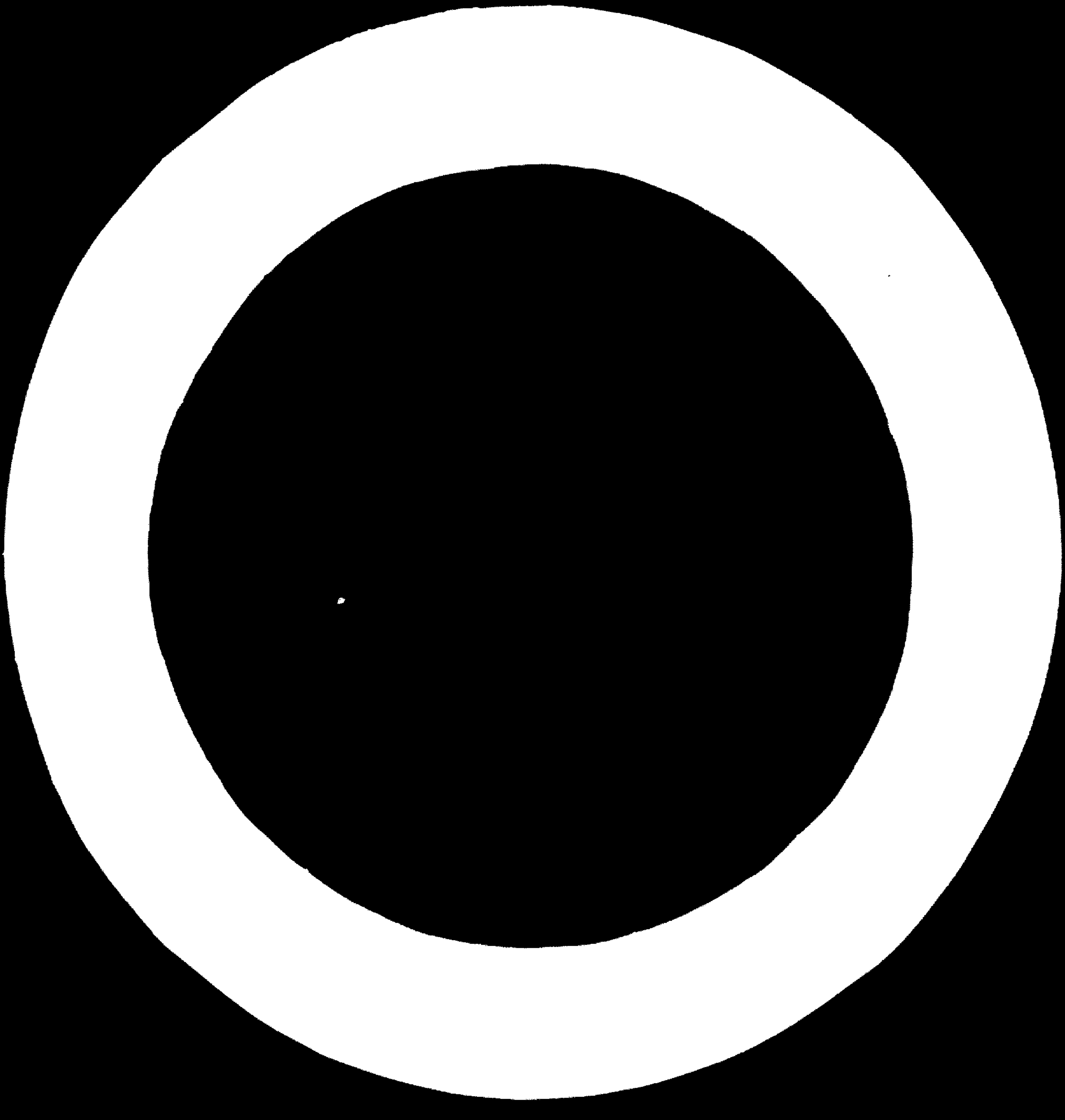
The examinations of clay materials which we have carried out have shown that our findings are reliable and that costly full-scale tests and experiments can be saved if our results are observed. Therefore the above mentioned charge in costs cannot be compared with the advantages that every Works can take from these test results.

Our findings are not of a theoretical nature; they are based on practice for practice. Therefore chemical and rational analyses are only prepared on special request, for which DM 50,-- will be charged for every type of material.

We should be pleased to assist you too by accomplishing such a clay-test and to advise you further based on the findings of our laboratory tests.

Yours faithfully,
KARL HÄNDLE & SÖHNE

Encl.:
Questionnaire (in 2 copies)



ANNEX 2

A.S.T.M. Standards

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO. A11 [illegible]
UDC 666 [illegible]

Standard Methods of Test for

APPARENT POROSITY, WATER ABSORPTION, APPARENT SPECIFIC GRAVITY, AND BULK DENSITY OF BURNED REFRACTORY BRICK[1]

ASTM Designation: C 20 – 46

ADOPTED, 1941; REVISED, 1946.[2]

Reapproved in 1958 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation C 20; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods of test cover procedures for determining the following properties of burned refractory brick:

Apparent porosity,
Water absorption,
Apparent specific gravity, and
Bulk density.

NOTE.—These methods are not applicable to refractories attacked by water.

Preparation of Sample

2. (*a*) The sample shall consist of at least five 9-in. straight brick or similar units, from each of which a single test specimen, having a volume of approximately 25 or 26 cu. in. shall be cut or broken. When testing 9-in. straight brick, the specimen shall be a quarter-brick obtained by halving the brick along a plane parallel to the 9 by $2\frac{1}{2}$-in. face and along a plane parallel to the $4\frac{1}{2}$ by $2\frac{1}{2}$ in. face. Four of the surfaces of the resultant quarter-brick specimen include part of the original molded faces. These surfaces are half of one end, half of one side and one quarter of each of the two 9 by $4\frac{1}{2}$-in. faces. When testing large shapes, the sample shall consist of several specimens cut or broken from each shape from both the center and outer portions.

(*b*) Each specimen shall be freed of all loosely adhering particles.

(*c*) Visibly defective specimens shall not be used.

Dry Weight, *D*

3. (*a*) The test specimens shall be dried to constant weight by heating to 220 to 230 F. (105 to 110 C.) and the dry weight, *D*, in grams determined to the nearest 0.1 g.

(*b*) The drying procedure may be omitted only when the test specimens

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to their present adoption as standard, these methods were published as tentative from 1918 to 1920. They were adopted in 1920, published as standard from 1920 to 1939, being revised in 1931 and 1933, but withdrawn and republished as tentative from 1939 to 1941.

are known to be dry, as may be the case with samples taken directly from kilns.

(c) The drying of the specimens to constant weight and the determination of their dry weights may be done either before or after the boiling operation (Section 4). Usually the dry weight is determined before boiling; if, however, the specimens are friable or evidence indicates that particles have broken loose during the boiling operation, the specimens shall be dried and weighed after the suspended weight, *S*, and the saturated weight, *W*, have been determined as described in Sections 5 and 6. This second dry weight shall be used in all appropriate calculations.

Boiling

4. (a) The test specimens shall be placed in water and boiled for 2 hr. During the boiling period, they shall be kept entirely covered with water, and should not be in contact with the heated bottom of the container.

(b) After the boiling period, the test specimens shall be cooled to room temperature while still completely covered with water. The cooling may be accelerated by running cold water into the container.

Suspended Weight, *S*

5. (a) The weight, *S*, of each test specimen after boiling and while suspended in water shall be determined in grams to the nearest 0.1 g.

(b) This weighing is usually accomplished by suspending the specimen in a loop or halter of 22-gage copper wire hung from one arm of the balance. The balance shall be previously counterbalanced with the wire in place and immersed in water to the same depth as is used when the refractory specimens are in place.

Saturated Weight, *W*

6. After determining the suspended weight, each specimen shall be blotted lightly with a moistened smooth linen or cotton cloth to remove all drops of water from the surface and the saturated weight, *W*, determined in grams by weighing in air to the nearest 0.1 g. The blotting operation shall be performed by rolling the specimen lightly on the wet cloth, which has previously been saturated with water and then pressed only enough to remove such water as will drip from the cloth. Excessive blotting will introduce error by withdrawing water from the pores of the specimen.

Exterior Volume, *V*

7. The volume, *V*, in cubic centimeters of the test specimens may be obtained by subtracting the suspended weight from the saturated weight, both in grams, as follows:

$$V = W - S$$

NOTE.—This assumes that 1 cu. cm. of water weighs 1 g. This is true within about three parts in 1000 for water at room temperature.

Volumes of Open Pores and Impervious Portions

8. The volume of open pores and the volume of the impervious portions of the specimen may be calculated as follows:

$$\text{Vol. of open pores in cu. cm.} = W - D$$

$$\text{Vol. of impervious portion in cu. cm.} = D - S$$

Apparent Porosity, *P*

9. The apparent porosity expresses as a percentage the relationship of the volume of the open pores of the specimen to its exterior volume and shall be calculated as follows:

$$P = \frac{W - D}{V} \times 100$$

Water Absorption, A

10. The water absorption, A, expresses as a percentage the relationship of the weight of water absorbed to the weight of the dry specimen and shall be calculated as follows:

$$A = \frac{W - D}{D} \times 100$$

Apparent Specific Gravity, T

11. The apparent specific gravity, T, of that portion of the test specimen which is impervious to boiling water shall be calculated as follows:

$$T = \frac{D}{D - S}$$

Bulk Density, B

12. (*a*) The bulk density, B, in grams per cubic centimeter of a specimen is the quotient of its dry weight divided by the exterior volume, including pores and shall be calculated as follows:

$$B = \frac{D}{V}$$

(*b*) This method of determining bulk density is useful for checking bulk density values obtained by the direct measurement method[5] (Note). While it is more accurate than the direct measurement method, and generally gives higher values (by about 0.02 to 0.04), the direct measurement method is better suited for plant and field testing, since it is a less involved technique. The present method is preferable for specimens which are branded deeply or irregular in contour.

NOTE.—The values for bulk density determined according to Section 12 will be expressed in metric units, for example, grams per cubic centimeter. The bulk density, however, determined by the direct measurement method will be expressed in English units. The conversion from metric to English units may be calculated as follows:

Bulk density in g. per cu. cm. × 62.43 = Bulk density in lb. per cu. ft.

Report

13. (*a*) For each property, the average of the values obtained with at least five specimens, and preferably also the individual values, shall be reported.[6]

(*b*) Apparent porosity and water absorption results shall be reported to one decimal place, and apparent specific gravity and bulk density results to two decimal places.

NOTE.—When values are reported for water absorption but not for porosity, it is suggested that the report shall also give the results for bulk density. This makes it possible to calculate the corresponding apparent porosity values as follows:

$$P = A \times B$$

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[5] Methods of Test for Size and Bulk Density of Refractory Brick (ASTM Designation: C 134), see p. 380.

[6] When there are pronounced differences among the individual values, another sample of five specimens shall be tested. These shall consist of the quarter brick diagonally opposite from the original specimens. The average of all ten determinations shall be reported.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A75.1-1956
UDC 691.[illegible]:691.3

Standard Specifications for
CONCRETE BUILDING BRICK[1]

ASTM Designation: C 55 - 55

Adopted, 1934; Revised, 1937, 1952, 1955.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 55; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover concrete building brick manufactured from a mixture of portland cement and suitable aggregates such as sand, gravel, crushed stone, bituminous or anthracite cinders, burned clay or shale, pumice, volcanic scoria, air-cooled or expanded blast-furnace slag and other slag, and intended for use in brick masonry. Two grades of brick are covered:

Grade A.—Brick intended for use where exposed to temperature below freezing in the presence of moisture.

Grade B.—Brick intended for use as back-up or interior masonry.

Note.—As an example, brick in exposed masonry protected by a facing of 3 in. or more of suitable masonry.

(*b*) When brick are required having strengths greater than prescribed by these specifications, the purchaser should specify the desired minimum compressive strength.

(*c*) If brick having a particular color, texture, finish, or uniformity are desired, these features should be specified separately by the purchaser.

Physical Properties

2. (*a*) The brick shall conform to the physical requirements for the grade specified, as prescribed in Table I.

TABLE I.—PHYSICAL REQUIREMENTS.

Designation	Minimum Compressive Strength (brick flatwise), psi, average gross area		Water Absorption, max, lb per cu ft	Moisture Content, max, percentage of total absorption
	Average of 5 Brick	Individual		
Grade A.....	2500	2000	15	40
Grade B.....	1500	1250	..	40

(*b*) Unless otherwise specified by the purchaser, brick of grade A shall be accepted in lieu of grade B.

Permissible Variations in Dimensions

3. No over-all dimension (width, height and length) shall differ more

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the ASTM Committee C-15 on Manufactured Masonry Units.

[2] Prior to adoption as standard, these specifications were published as tentative from 192[illegible] to 1934, being revised in 1928 and 1933.

than ⅛ in. from the specified standard dimensions.

NOTE.—Standard dimensions of units are the manufacturers' designated dimensions.

Visual Inspection

4. Brick shall pass a visual inspection for freedom from cracks and irregularity.

Sampling and Testing

5. (*a*) For purpose of tests, brick that are representative of the commercial product shall be selected by a competent person appointed by the purchaser, the place or places of selection to be designated when the purchase order is placed. The manufacturer or the seller shall furnish specimens for tests without charge.

(*b*) The brick shall be sampled and tested in accordance with the Methods of Sampling and Testing Concrete Masonry Units (ASTM Designation: C 140)[1]

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[1] See p. 110.

Standard Specifications for

BUILDING BRICK (SOLID MASONRY UNITS MADE FROM CLAY OR SHALE)[1]

ASTM Designation: C 62 - 60

ADOPTED, 1944; LAST REVISED, 1960[2]

This Standard of the American Society for Testing and Materials is issued under the fixed designation C 62; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These specifications cover brick made from clay or shale and burned, and intended for use in brick masonry. (See Explanatory Note at end of these specifications.) Three grades of brick are covered, as follows (see Section 2(*a*)):

Grade SW.—Brick intended for use where a high degree of resistance to frost action is desired and the exposure is such that water permeating the brick may be frozen.

NOTE.—As a typical example, brick used for foundation courses and retaining walls in portions of the United States subject to frost action should conform to this grade. Compliance with this grade is also recommended where a high and uniform degree of resistance to disintegration by weathering is desired.

Grade MW.—Brick intended for use where exposed to temperatures below freezing but unlikely to be permeated with water, or where a moderate and somewhat nonuniform degree of resistance to frost action is permissible.

NOTE.—As a typical example, brick used in the face of a wall above ground should conform to this grade. Such exposure is not likely to result in permeation of a brick by water if horizontal surfaces are protected. (See discussion of grade MW in Explanatory Note.)

Grade NW.—Brick intended for use as back-up or interior masonry.

(*b*) In these specifications the term brick shall be understood to mean brick or solid clay masonry unit.

Physical Properties

2.(*a*) *Appearance.*—If brick having a particular color, texture, finish, uniformity, or freedom from cracks, warpage, [illegible] stones, pebbles, or particles of

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[1] Under the standardization procedure of the Society, these specifications are under the jurisdiction of the ASTM Committee C-15 on Manufactured Masonry Units. A list of members may be found in the ASTM Year Book.

[2] Prior to adoption as standard, these specifications were published as tentative from 1937 to 1939, being revised in 1938. They were adopted in 1939, published as standard from 1939 to 1950, being revised in 1950. A revision in the form of separate tentative specifications was issued from 1950 to 1954, being revised in 1950, 1957, 1958, 1960, and 1961.

Prior to their publication as tentative in 1937, these specifications comprised a portion of the Standard Specifications for Building Brick (C 21 - 30) which were published as tentative from 1930 to 1933, being revise [illegible] 4 in 1933, but withdrawn an [illegible] 62 - 37 T in 1937.

tone are desired, such brick should be purchased under the Specifications for Facing Brick (Solid Masonry Units Made from Clay or Shale) (ASTM Designation: C 216).[5]

(b) *Durability.*—The brick shall conform to the physical requirements for the grade specified, as prescribed in Table I.

(c) Unless otherwise specified by the purchaser, brick of grades SW and MW shall be accepted in lieu of grade NW, and grade SW in lieu of grade MW. When the grade is not specified, grade MW shall govern.

(d) If the average compressive strength is greater than 8000 psi or the average water absorption is less than 8.0 per cent after 24-hr submersion in cold water, the requirement for saturation coefficient shall be waived.

(e) *Freezing and Thawing.* The requirements specified in Paragraph (b) for [illegible] boiling and saturation coefficient shall be waived provided a sample of five brick, meeting all other requirements, complies with the following requirements when subjected to 50 cycles of the freezing and thawing test:[a]

Grade SW	No breakage and not greater than 1.0 per cent loss in dry weight of any individual brick.
Grade MW	No breakage and not greater than 3.0 per cent loss in dry weight of any individual brick.

A particular lot or shipment shall be given the same grading as a previously tested lot, without repeating the freezing-and-thawing test, provided the brick are made by the same manufacturer from similar raw materials and by the same method of forming; and provided also that a sample of five brick selected from the particular lot has an average and individual minimum strength not less than a previously graded sample, and has average and individual maximum water absorption and saturation coefficient not greater than those of the previously tested sample graded according to the freezing and thawing test.

(f) *Waiving of Absorption and Saturation Coefficient Requirements.*—If brick are intended for use exposed to weather where the weathering index is less than 100 (see Fig. 1 and Explanatory Note C at the end of these specifications), unless otherwise specified the requirements given in Paragraph (b) for water absorption (5-hr boiling) and for saturation coefficient shall be waived, and a minimum average strength requirement of 2500 psi shall apply.

TABLE I.—PHYSICAL REQUIREMENTS.

Designation	Minimum Compressive Strength (brick flatwise), psi, gross area		Maximum Water Absorption by 5-hr Boiling, per cent		Maximum Saturation Coefficient[a]	
	Average of 5 Brick	Individual	Average of 5 Brick	Individual	Average of 5 Brick	Individual
Grade SW	3000	2500	17.0	20.0	0.78	0.80
Grade MW	2500	2200	22.0	25.0	0.88	0.90
Grade NW	1500	1250	no limit	no limit	no limit	no limit

[a] The saturation coefficient is the ratio of absorption by 24-hr submersion in cold water to that after 5-hr submersion in boiling water.

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[a] Brick are not required to conform to the provisions of Section 3 (e), and these do not apply unless the sample fails to conform to the requirements for absorption and saturation coefficient prescribed in Table I or the strength and absorption requirements in Section 3 (d).

[5] Appears in this publication.

(*g*) *Strength.*—When brick are required having strengths greater than prescribed in Table I, the purchaser should specify minimum strength according to the classification given in Table II.

(*h*) *Rate of Absorption.*—See Explanatory Notes.

TABLE II.—CLASSIFICATION BY COMPRESSIVE STRENGTH.

Designation	Minimum Compressive Strength (brick flatwise), psi, gross area	
	Average of 5 Brick	Individual
[illegible] lb.	[illegible]	[illegible]
[illegible] lb.	[illegible]	[illegible]
[illegible] lb.	[illegible]	[illegible]

TABLE III.—PERMISSIBLE VARIATIONS IN DIMENSIONS.

Specified dimension, in.	Maximum Permissible Variations from Specified Dimension, plus or minus, in.
Up to 3, incl	[illegible]
Over 3 to 4, incl	[illegible]
Over 4 to 6, incl	[illegible]
Over 6 to 8, incl	[illegible]
Over 8 to 12, incl	[illegible]
Over 12 to 16, incl	[illegible]

NOTE.—For a list of modular sizes, see American Standard Sizes of Clay and Concrete Modular Masonry Units, A62.3. All of the sizes listed in this standard are not produced in some parts of the United States, and purchasers should ascertain the size or sizes available.

Size and Coring

3. (*a*) *Size.*—The size of brick shall be as specified by the purchaser. The maximum permissible variation in dimensions of individual units shall not exceed those given in Table III.

(*b*) *Coring.*—Unless otherwise specified in the invitation for bids, brick shall be either solid or cored at the option of the seller. The net cross-sectional area of cored brick in any plane parallel to the bearing surface shall be at least 75 per cent of the gross cross-sectional area measured in the same plane. No part of any hole shall be less than ¾ in. from any edge of the brick.

(*c*) *Frogging.*—Unless otherwise specified in the invitation for bids, one bearing face of each brick may have a recess or panel (frog) not exceeding ⅜ in. in depth, except that in brick containing deep frogs any cross-section through the frogs parallel to the bearing surface shall conform to the requirements of Paragraph (*b*). No part of the recess shall be less than ¾ in. from any edge of the brick.

Visual Inspection

4. (*a*) The brick, as delivered to the site, shall, by visual inspection, conform to the requirements specified by the purchaser or to the sample or samples approved as the standard of comparison and to the samples passing the tests for physical requirements. Minor indentations or surface cracks incidental to the usual method of manufacture, or the chipping resulting from the customary methods of handling in shipment and delivery, should not be deemed grounds for rejection.

(*b*) Unless otherwise agreed upon by purchaser and the seller, a delivery of brick shall contain not less than 95 per cent whole brick.

Sampling and Testing

5. (*a*) For purpose of tests, brick that are representative of the commercial product shall be selected by a competent person appointed by the purchaser, the place or places of selection to be designated when the purchase order is placed. The manufacturer or the seller shall furnish specimens for tests without charge.

(*b*) The brick shall be sampled and tested in accordance with the Methods of Sampling and Testing Brick (ASTM Designation: C 67).[9]

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[9] [illegible]

Cost of Tests

6. Unless otherwise specified in the purchase order, the cost of tests shall be borne as follows:

(a) If the results of the test show that the brick do not conform to the requirements of these specifications, the costs shall be borne by the seller.

(b) If the results of the tests show that the brick do conform to the requirements of these specifications, the costs shall be borne by the purchaser.

EXPLANATORY NOTES

4. *Significance of Grades.*—Extensive tests of brick masonry as well as observations of masonry structures in the field indicate that the important properties of brick which affect the appearance and performance of masonry in buildings are size, color and texture, compressive strength, durability, and suction when laid. Data indicate that a low rate of suction (20 g per min or less) is desirable both from the standpoint of bond and watertightness, and since the suction rate of brick that normally have high rates of suction can be reduced to any predetermined value by wetting before laying, this property should not be included in specifications for brick, but may properly be made a part of specifications governing workmanship.

Other properties of brick such as density, soluble salt content, and homogeneity probably also affect the performance of the masonry. Data are not available, however, from which measures of these properties or their effects upon the masonry may be determined, and consequently there are no bases for controlling these factors through a specification. Their effects may be best judged by the record of performance of similar products.

These specifications provide a basis for specifying the following properties of brick, which, as indicated above, appear to be important:

Size.—The dimensions of the standard size brick, together with permissible variations, are given in Section 3 of these specifications. Many brick and solid units are produced in other sizes, however, and when sizes other than standard are acceptable or required, such sizes, together with permissible variations in dimensions, should be specified by the purchaser.

Color and Texture.—Brick are manufactured in a wide variety of colors and textures, neither of which has been standardized. Both color and texture are difficult to describe and a complete list of the products now produced, if obtainable, would be too voluminous to include in a specification. These properties are covered in Section 2 (h) of these specifications, which provides that color, texture, finish, and uniformity should be specified by the purchaser. The common practice is to refer to an approved sample.

Compressive Strength.—The compressive strength of brick produced in the United States ranges from 1000 psi or less (under-burned) to over 20,000 psi. Data are available from which the compressive strength of masonry walls may be predicted with reasonable accuracy if the strength of the brick and the strength of the mortar are known. In the great majority of cases, however, the compressive stresses in masonry walls are relatively low (under 100 psi), and for such structures minimum compressive strengths of brick of from 1500 to 2500 psi are ample. These minimum values are included in Section 2 (b) of these specifications; when brick having higher strengths are desired, the required strength should be specified by the purchaser as provided in Section 2 (g). Allowable working stresses for brick masonry laid with units having several compressive strengths in several types of mortars are provided in the American Standard Building Code Requirements for Masonry (ASA No.: A 41.1).

Durability.—Experience has indicated that any well-burned brick will resist the action of freezing and thawing over a long period of time and, from a structural standpoint, may be considered durable. There is a reasonably close correlation between the performance of brick in the freezing-and-thawing test and under the agents of weathering in masonry structures, and at the present time this test appears to be the best measure of the durability of brick. Freezing-and-thawing tests consist in subjecting the brick to alternate cycles (50 or more) of freezing and thawing in the presence of moisture, which requires a period of 10 weeks or more to complete. This makes it impractical as an acceptance test, and for this reason extensive research has been carried on to correlate other physical properties of brick with their resistance to the freezing-and-thawing test.

For brick produced from the same clay deposits and by the same method of manufacture, either compressive strength or total absorption may be taken as a fairly accurate measure of the resistance of such brick to the freezing-and-thawing test; however, limits on these properties that apply to one product do not apply to products produced from different raw materials or by different manufacturing processes, and consequently they alone cannot be used as measures of durability in general specifications. A third property known as "saturation coefficient," when used in conjunction with compressive strength and total absorption by 5 hr boiling, has been found to provide a means of predicting the resistance of most types of brick to freezing and thawing tests with greater accuracy than any other method developed to date.

The saturation coefficient is the ratio of the absorption by 24 hr submersion in cold water to the absorption after 5 hr submersion in boiling water and is defined generally as the ratio of easily filled to total fillable pore space. The theory of the saturation coefficient is that if only a part of the total pore space is occupied by water there is room for expansion on freezing into the remaining pore space without disruption of the material. The data indicate that if the easily fillable pore space, that is, the maximum water that might be absorbed by a brick in a wall subjected to excessive moisture, does not exceed 80 per cent of the total pore space, the remaining space will relieve the pressure due to expansion on freezing.

While this theory seems to be applicable to many types of brick, it has been found that it does not apply to certain types of de-aired products. Strength and absorption are, therefore, used as measures of durability for these products, and their acceptance should be based upon Section 2 (*d*) of these specifications.

The relationship also does not appear to hold for some brick of very high absorption (exceeding the maximum permitted in these specifications), and the acceptance of these products should be in accordance with the Section 2 (*e*) which provides for special measures based upon actual freezing and thawing tests of the particular product.

In classifying brick according to their resistance to the freezing and thawing test, they fall into three general groups as indicated in Table I of these Specifications:

Grade SW, which are not affected by the test, and whose appearance and structure remain unchanged.

Grade MW, which are for the most part well-burned brick but may include some brick which change materially in appearance when exposed to weathering.

The limits for absorption and saturation coefficient in the grade MW classification have been set to include the average production of those districts which do not grade or classify their kiln output beyond elimination of extremely underburned (salmon) brick. These "kiln run" shipments frequently include a small percentage of brick which, on exposure to weathering, will lose their surfaces by powdering, flaking, or spalling, and thus produce an unsightly appearance of the exposed masonry surface. Data indicate that brick cannot be classified into intermediate durability. Actually grade MW includes a mixture of durable and nondurable brick. It should be emphasized, however, that disintegration is not necessarily a characteristic of brick in this grade. Certain plants may supply brick under the grading, all of which remain unchanged in appearance even under severe conditions of exposure. The purchaser is advised to examine the field behavior of brick in districts where production classifies them as grade MW, and reach his own decision as to whether the appearance and condition of masonry at the age of 10 or 20 years is satisfactory.

Grade NW, which includes underburned brick that will disintegrate when subjected to freezing and thawing. Such brick should not be used in structures that will be subjected to severe weathering.

In using these specifications the purchaser is urged to consider both the requirements of the structure and the physical properties of the brick available. To a degree at least, brick are a natural product, since such properties as color, compressive strength, and absorption are more or less inherent in the raw material and frequently can be changed only within narrow limits by different methods of manufacture. While the committee believes that the specifications as they now stand provide the best means available of specifying the desirable properties of brick, it recognizes that the specifications are not perfect and that, due to the wide variation in raw materials and methods of manufacture, it is probable that some brick which do not conform to the requirements of grade SW still have satisfactory durability. It may also be true that some products which meet these requirements, particularly of grade MW, do not have satisfactory resistance to weathering. For this reason, and because of the lack of data on some properties that may have an important bearing upon the performance of masonry, the purchaser should be guided to a degree by the record of performance of any particular product.

B. Initial Rate of Absorption (Suction).—Both laboratory and field investigations have shown that strong and watertight joints between mortar and masonry units are not achieved by ordinary construction methods when the units as laid have excessive initial rates of absorption. Mortar that has stiffened somewhat because of loss of some mixing water to a unit does not make complete and intimate contact with the second unit, resulting in poor adhesion, incomplete bond, and water-permeable joints of low strength. Hence, the initial rate of absorption of the units should be determined by the method described in Section 27 of the Methods of Sampling and Testing Brick (ASTM Designation: C 67)[4] if it is not known that it is less than 20 g per min per 30 sq in. Units having initial rates of absorption exceeding 20 g per min per 30 sq in. should be well wetted prior to laying. They may be wetted immediately before they are laid, but it is preferable to wet them thoroughly 3 to 24 hr prior to their use so as to allow time for moisture to become distributed throughout the unit.

(c) *Weathering Index.* The effect of weathering on brick is related to the weathering index, which for any locality is the product of the average annual number of *freezing cycle days* and the average annual *winter rainfall* in inches, defined as follows:[5]

A Freezing Cycle Day is any day during which the air temperature passes either above or below 32 F. The average number of freezing cycle days in a year may be taken to equal the difference between the mean number of days during which the minimum temperature was 32 F or below and the mean number of days during which the maximum temperature was 32 F or below.

Winter Rainfall is the sum, in inches, of the mean monthly corrected precipitation (rainfall) occurring during the period between and including the normal date of the first killing frost in the fall and the normal date of the last killing frost in the spring. The winter rainfall for any period is equal to the total precipitation less one tenth of the total fall of snow, sleet, and hail. Rainfall for a portion of a month is prorated.

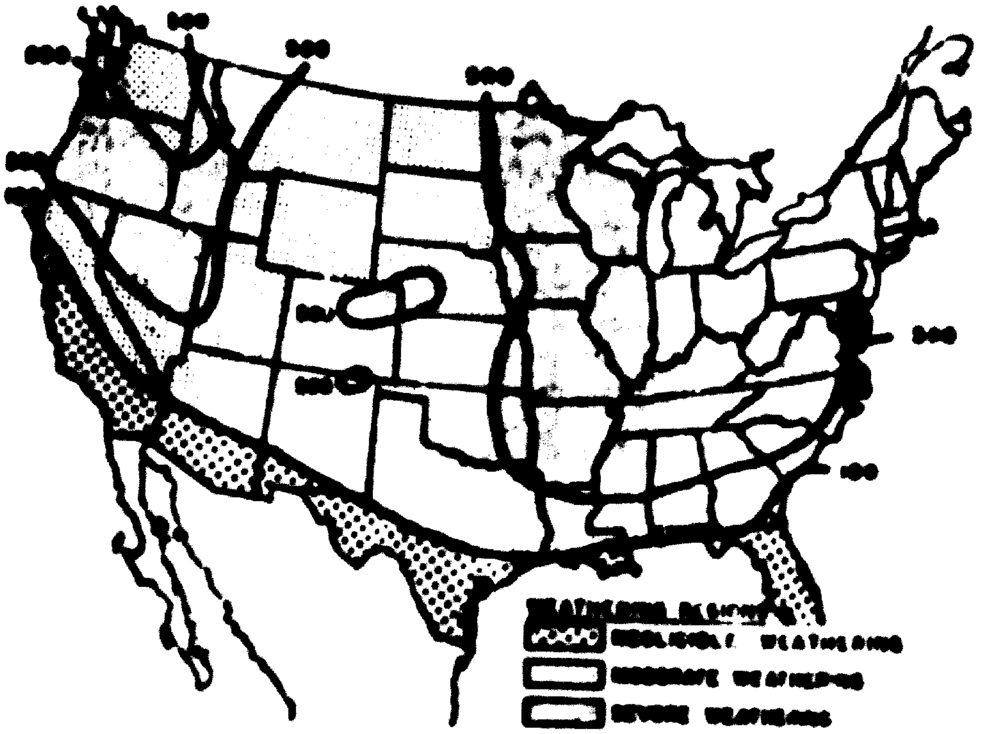

Fig. 1. Areas in Which a Weathering Index of Brick Is Less Than 500

Figure 1 indicates general areas of the United States in which brick masonry is subject to severe, moderate, and negligible weathering. The severe weathering region has a weathering index greater than 500. The moderate weathering region has a weathering index of 100 to 500. The negligible weathering region has a weathering index of less than 100.

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[5] Data needed to determine the weathering index for any locality may be found or estimated from the tables of Local Climatological Data, published by the Weather Bureau, U. S. Department of Commerce.

The use of Grade MW brick in a wall area above grade is structurally adequate in the severe weathering region, but Grade SW would provide a higher and more uniform degree of resistance to frost action. The degree of durability called for by Grade SW is not necessary for use in wall areas above grade in the moderate weathering region. Grade MW brick performs satisfactorily in wall areas above grade in the no weathering region, where the average compressive strength of the units is at least 2500 psi. Grade SW brick should be used in any region when the units are in contact with the ground, in horizontal surfaces, or in any position where they are likely to be permeated with water.

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Standard Methods of

SAMPLING AND TESTING BRICK[1]

ASTM Designation: C 67 - 66

ADOPTED, 1931; LAST REVISED, 1966

This Standard of the American Society for Testing and Materials is issued under the fixed designation C 67; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. These methods cover procedures only for the sampling and testing of brick for modulus of rupture (flexure test), compressive strength, absorption, saturation coefficient, effect of freezing and thawing, initial rate of absorption (suction), efflorescence, measurement of size, and measurement of warpage.

Definitions of Terms

2. The Definitions of Terms Relating to Methods of Mechanical Testing (ASTM Designation: E 6)[2] shall be considered as applying to the terms used in these methods.

SAMPLING

Selection of Test Specimens

3. For purposes of tests, full-size brick shall be selected by the purchaser or by his authorized representative. Specimens shall be representative of the whole lot of brick from which they are selected and shall include specimens representative of the complete range of colors and sizes of the brick in the shipment.

Number of Specimens

4. For the modulus of rupture, compressive strength, absorption, and freezing-and-thawing determinations, at least ten brick shall be selected from each lot of 50,000 brick or fraction thereof. For lots of more than 500,000 brick, five individual brick shall be selected from each 100,000 brick or fraction thereof contained in the lot. In no case shall less than ten brick be taken. Additional specimens may be taken at the discretion of the purchaser.

Identification

5. Each specimen shall be marked so that it may be identified at any time. Markings shall cover not more than 5 per cent of the superficial area of the specimen.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the ASTM Committee C-15 on Manufactured Masonry Units. A list of members may be found in the ASTM Year Book.

[2] 1966 Book of ASTM Standards, Part 30.

Modulus of Rupture (Flexure Test)

Test Specimens

6. Five dry (Note) full-size brick shall be tested.

Note. Reference should be made to Section 15 (a) for the definition of a dry specimen.

Procedure

7. (*a*) The test specimen shall be supported flatwise on a span of 7 in. and loaded at mid-span. If the specimens have recesses (panels or depressions) they shall be placed so that such recesses are on the compression side. The load shall be applied to the upper surface of the specimen through a steel bearing plate ¼ in. in thickness and 1½ in. in width and of a length at least equal to the width of the specimen. The direction of the load shall be perpendicular to the loaded surface of the specimen.

(*b*) The supports for the test specimen shall be free to rotate in the longitudinal and transverse direction of the test specimen and shall be adjusted so that they will exert no force in these directions.

(*c*) *Speed of Testing.*—The rate of loading shall not exceed 2000 lb per min, but this requirement may be considered as being met if the speed of the moving head of the testing machine during the application of the load is not more than 0.05 in. per min.

Calculations and Report

8. (*a*) Calculate the modulus of rupture of each specimen as follows:

$$\text{Modulus of rupture, psi} = \frac{3Wl}{2bd^2}$$

where.

W = maximum load indicated by the testing machine, in pounds,

= distance between the supports in inches (7 in.),

b = average over-all width, face to face, of the specimen, in inches, and

d = average over-all depth, face to face, of the specimen, in inches.

(*b*) Report the average of the modulus of rupture determinations of all the specimens tested as the modulus of rupture of the lot of brick.

Compressive Strength

Test Specimens

9. The test specimens shall consist of pieces of brick with the length equal to the width ± 1 in. A specimen may be obtained by any method that will produce, without shattering, a specimen with approximately plane and parallel ends. Five specimens shall be tested.

Capping Test Specimens

10. (*a*) If the flatwise faces of the test specimen are recessed or paneled, the depressions shall be filled with a neat portland cement paste, which shall then be aged at least 24 hr before the specimen is capped. The test specimens shall then be capped by one of the two procedures described in Paragraphs (*b*) and (*c*).

(*b*) *Gypsum Capping.*—Coat the two opposite flatwise faces of each specimen with shellac and allow to dry thoroughly. Bed one of the dry shellacked faces of the specimen in a thin coat of a neat paste of calcined gypsum (plaster of Paris) that has been spread on an oiled nonabsorbent plate such as glass or machined metal. Repeat this procedure with the other dry shellacked face. Take care that the opposite bearing surfaces so formed will be approximately parallel and the thickness of the caps will be approximately the same. Age the caps at least 16 hr before testing the specimens.

(*c*) *Sulfur-Filler Capping.*—Use a mixture containing 40 to 60 per cent sulfur (by weight), the remainder being ground fire clay or other suitable inert material passing a No. 100 (149-μ) sieve, with or without plasticizer. The casting surface plate shall be plane within 0.003 in. in 16 in. and sufficiently rigid and so supported that it will not be measurably deflected during the capping operation, and shall be lightly coated with oil. Place four 1 in. square steel bars on the surface plate to form a rectangular mold approximately ½ in. greater in either inside dimension than the specimen. Heat the sulfur mixture in a thermostatically controlled heating pot to a temperature sufficient to maintain fluidity for a reasonable period of time after contact with the capping surface. Take care to prevent overheating, and stir the liquid in the pot just before use. Fill the mold to a depth of ¼ in. with molten sulfur material. Place the surface of the unit to be capped quickly in the liquid, and hold the specimen so that its axis is at right angles to the capping surface. The thickness of the caps shall be approximately the same. Allow the unit to remain undisturbed until solidification is complete. Allow the caps to cool for a minimum of 2 hr before testing the specimens.

Procedure

11. (*a*) All specimens shall be tested flatwise (that is, the load shall be applied in the direction of the thickness of the brick) and the specimens shall be centered under the spherical upper bearing within $\frac{1}{16}$ in.

(*b*) The testing machine shall conform to the requirements of the Methods of Verification of Testing Machines (ASTM Designation: E 4).[6]

(*c*) The upper bearing shall be a spherically seated, hardened metal block firmly attached at the center of the upper head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to turn in any direction, and its perimeter shall have at least ¼ in. clearance from the head to allow for specimens whose bearing surfaces are not exactly parallel. The diameter of the bearing surface shall be at least 5 in. A hardened metal bearing block shall be used beneath the specimen to minimize wear of the lower platen of the machine. The bearing block surfaces intended for contact with the specimen should have a hardness not less than C60 Rockwell number (Brinell number 620). These surfaces shall not depart from plane surfaces by more than 0.001 in. When the bearing area of the spherical bearing block is not sufficient to cover the area of the specimen, a steel plate with surfaces machined to true planes within ±0.001 in., and with a thickness equal to at least one third of the distance from the edge of the spherical bearing to the most distant corner shall be placed between the spherical bearing block and the capped specimen.

(*d*) *Speed of Testing*.—The load, up to one half of the expected maximum load, may be applied at any convenient rate, after which the controls of the machine shall be adjusted so that the remaining load is applied at a uniform rate in not less than 1 nor more than 2 min.

Calculations and Report

12. (*a*) Calculate the compressive strength of each specimen as follows:

$$\text{Compressive strength, psi} = \frac{W}{A}$$

where:

W = maximum load indicated by the testing machine, in pounds, and
A = average of the gross areas of the upper and lower bearing surfaces of the specimen in square inches.

(*b*) Report the average compressive strength of all the specimens tested as the compressive strength of the lot of brick.

ABSORPTION

Accuracy of Weighings

13. The scale or balance used shall have a capacity of not less than 2000 g and shall be sensitive to 0.5 g.

24-hr Submersion Test

Test Specimens

14. The test specimens shall consist of half brick conforming to the requirements of Section 9. Five specimens shall be tested.

Procedure

15. (*a*) Dry the test specimens in a ventilated oven at 230 to 239 F (110 to 115 C) for not less than 24 hr and until two successive weighings at intervals of 2 hr show an increment of loss not greater than 0.2 per cent of the last previously determined weight of the specimen.

NOTE 1.—Storage of brick, unstacked, with separate placement, in a ventilated room for a period of 4 hr, with a current of air from an electric fan passing over them for a period of at least 2 hr, will cool the specimens to approximately room temperature. Specimens noticeably warm to the touch should not be used for the absorption test.

(*b*) *Saturation.*—Submerge the dry specimen, without preliminary partial immersion, in clean water (soft, distilled, or rain water) at 60 to 86 F (15.5 to 30 C) for 24 hr. Remove the specimen, wipe off the surface water with a damp cloth, and weigh the specimen. Weighing of any one specimen shall be completed within 5 min after removing the specimen from the bath.

Calculations and Report

16. (*a*) Calculate the absorption of each specimen as follows:

$$\text{Absorption, per cent} = \frac{100(W_2 - W_1)}{W_1}$$

where:

W_1 = dry weight of the specimen, and

W_2 = saturated weight of the specimen after 24-hr submersion in cold water.

(*b*) Report the average absorption of all the specimens tested as the absorption of the lot of brick.

5-hr Boiling Test

Test Specimens

17. The test specimens shall consist of half brick conforming to the requirements of Section 9. Five specimens shall be tested.

Procedure

18. (*a*) Return the specimen that has been saturated 24 hr in the cold-water submersion bath to the bath, and subject it to a 5-hr boiling test as described in Paragraph (*b*).

(*b*) Submerge the specimen in clean water (soft, distilled, or rain water) at 60 to 86 F (15.5 to 30 C) in such a manner that water can circulate freely on all sides of the specimen. Heat the water to boiling within 1 hr, boil continuously for 5 hr, and then allow to cool to 60 to 86 F (15.5 to 30 C) by natural loss of heat for not less than 16 or more than 19 hr. Remove the specimen, wipe off the surface water with a damp cloth, and weigh the specimen. Weighing of any one specimen shall be completed within 5 min after removing the specimen from the bath.

NOTE.—If the tank is equipped with a drain so that water at 60 to 86 F (15.5 to 30 C) can be passed through the tank continuously and at such a rate that a complete change of water takes place in not more than 2 min, weighings can be made at the end of 1 hr.

Calculations and Report

19. (*a*) Calculate the absorption of each specimen as follows:

$$\text{Absorption, per cent} = \frac{100(W_b - W_d)}{W_d}$$

where:

W_d = dry weight of the specimen, and
W_b = saturated weight of the specimen after 5-hr submersion in boiling water.

(*b*) Report the average absorption of all the specimens tested as the absorption of the lot of brick.

Saturation Coefficient

20. Calculate the saturation coefficient of each specimen as follows:

$$\text{Saturation coefficient} = \frac{W_s - W_d}{W_b - W_d}$$

where:

W_d = dry weight of the specimen,
W_s = saturated weight of the specimen after 24-hr submersion in cold water, and
W_b = saturated weight of the specimen after 5-hr submersion in boiling water.

FREEZING AND THAWING

Apparatus

21. (*a*) *Compressor, Freezing Chamber, and Circulator* of such design and capacity that the temperature of the air in the freezing chamber will not exceed 16 F (−9 C) 1 hr after introducing the maximum charge of brick, initially at a temperature not exceeding 90 F (32 C).

(*b*) *Trays and Containers.*—Shallow metal trays having an inside depth of 1½ ± ½ in., and of suitable strength and size so that the tray with a charge of frozen brick can be removed from the freezing chamber by one man.

(*c*) *Balance*, having a capacity of not less than 2000 g and sensitive to 0.5 g.

(*d*) *Drying Oven* that provides a free circulation of air through the oven and is capable of maintaining a temperature between 230 and 239 F (110 and 115 C).

(*e*) *Thawing Tank* of such dimensions as to permit the complete submergence of the specimens in their trays. Adequate means shall be provided so that the water in the tank may be kept at a temperature of 75 ± 10 F (24 ± 5.5 C).

(*f*) *Drying Room*, maintained at a temperature of 75 ± 15 F (24 ± 8 C), with a relative humidity between 30 and 70 per cent, and free from drafts shall be provided for the air-drying prescribed in alternate Method B.

Test Specimens

22. The test specimens shall consist of half brick with approximately plane and parallel ends. The specimens shall be free from shattering or unsoundness resulting from the flexure test or from the absorption tests (24-hr submersion test and 5-hr boiling test). Five specimens shall be tested.

Method B

Procedure

23. (*a*) Dry the test specimens as prescribed in Section 15 (*a*).

(*b*) When cool, weigh the specimens on a scale or balance to the nearest 0.5 g.

(*c*) Immediately submerge the test specimens, without preliminary partial immersion, in the water of the thawing tank for 4 hr before the start of the freezing-and-thawing test.

(*d*) Stand the test specimens on edge in the trays so that a space of at least ½ in. separates the specimens. Pour sufficient water into the trays so that each specimen stands in ½ in. depth of water, and then expose the trays and their contents to the temperature of the freezing chamber for 20 hr.

(*e*) Remove the trays from the freezing chamber after 20 hr and totally immerse them, with their contents, in the water of the thawing tank for 4 hr.

(*f*) Freeze the test specimens by the procedure prescribed in (*d*) and thaw them to the procedure prescribed in (e), five times. Following the 4-hr thawing after the fifth freezing, store the specimens for 40 hr on the floor of the drying room. They shall not be stacked or piled, and there shall be a space of at least 1 in. between any two specimens. Following this period of air-drying, inspect the specimens, submerge them in the water of the thawing tank for 4 hr, and again subject them to 5 cycles of freezing and thawing in accordance with (d) and (e).

(g) Continue the alternations of drying and submersion in water for 4 hr, followed by 5 cycles of freezing and thawing, until a total of 50 cycles of freezing and thawing has been completed, unless the test specimen has broken or appears to have lost more than 3 per cent of its original weight as judged by visual inspection.

(h) After completion of 50 cycles, or when the specimen has been withdrawn from test as a result of evident disintegration, dry the specimen in an oven as prescribed in Section 15 (a).

Calculations and Report

24. Calculate the loss in weight as a percentage of the original weight of the dried specimen, or report the number of cycles causing breakage or withdrawal of the specimen.

INITIAL RATE OF ABSORPTION (SUCTION)

Apparatus

25. (a) *Trays or Containers.*—Watertight trays or containers, having an inside depth of not less than ½ in., and of such length and width that an area of not less than 300 sq in. of water surface is provided. The bottom of the tray shall provide a plane, horizontal upper surface, when suitably supported, so that an area not less than 8 in. in length by 6 in. in width will be level when tested by a spirit level.

(b) *Supports for Brick.*—Two noncorrodible metal supports consisting of bars between 5 and 6 in. in length, having triangular, half round, or rectangular cross-sections such that the thickness (height) will be approximately ¼ in. The thickness of the two bars shall agree within 0.001 in. and, if the bars are rectangular in cross-section, their width shall not exceed $\frac{5}{16}$ in..

(c) *Means for Maintaining Constant Water Level.*—Suitable means for controlling the water level above the upper surface of the supports for the brick within ±0.01 in. (Note 1), including means for adding water to the tray at a rate corresponding to the rate of removal by the brick undergoing test (Note 2). For use in checking the adequacy of the method of controlling the rate of flow of the added water, a reference brick or half brick shall be provided whose displacement in ⅛ in. of water corresponds to the brick or half brick to be tested within ±2.5 per cent. The reference brick shall be completely submerged in water for not less than 3 hr preceding its use, as prescribed in Section 15 (b).

NOTE 1.—A suitable means for obtaining this accuracy in control of the water level is provided by attaching to the end of one of the bars, two stiff metal wires that project upward and return, terminating in points one of which is ⅛ − 0.01 in. and the other ⅛ + 0.01 in. above the upper surface or edge of the bar. Such precise adjustment is obtainable by use of depth plates or a micrometer microscope. When the water level with respect to the upper surface or edge of the bar is adjusted so that the lower point "dimples" the water surface when viewed by reflected light and the upper point is not in contact with the water, the water level is within the limits specified. Any other suitable means for fixing and maintaining a constant depth of immersion may be used if equivalent accuracy is obtained. As an example of such other suitable means, there may be mentioned the use of rigid supports movable with respect to the water level.

NOTE 2.—A rubber tube leading from a siphon or gravity feed and closed by a spring clip will provide a suitable manual control. The so-called "chicken feed" devices as a rule lack sensitivity and do not operate with the very small changes in water level permitted in the test.

(d) *Balance.*—A scale or balance having a capacity of not less than 3000 g, and sensitive to 0.5 g.

(e) *Drying Oven.*—A drying oven conforming to the requirements of Section 21 (d).

(f) *Constant-Temperature Room.*—A room maintained at a temperature of 70 ± 2.5 F (21 ± 1.4 C).

(g) *Timing Device.*—A suitable timing device, preferably a stop watch or stop clock, which shall indicate a time of 1 min to the nearest second.

Test Specimens

26. The test specimens shall consist of whole brick or of half brick conforming to the requirements of Section 9. Five specimens shall be tested.

Procedure

27. (a) *Drying.*—Dry the test specimens in accordance with Section 15 (a).

(b) *Cooling.*—After drying, cool the test specimens in the constant-temperature room (Section 25 (f)) by storage, unstacked, with separate placement, for a period of at least 4 hr.

(c) *Measurement and Weighing.*—Measure to the nearest 0.05 in. the length and width of the flatwise surface of the test specimen that will be in contact with the water. Weigh the specimen to the nearest 0.5 g.

(d) *Adjustment of Water Level.*—Set up the tray for the absorption test in the constant-temperature room. Adjust the position of the tray so that the upper surface of its bottom will be level when tested by a spirit level, and set the saturated reference brick (Section 25 (c)) in place on top of the supports. Add water until the water level is ⅛ ± 0.01 in. above the top of the supports.

(e) *Absorption and Reweighing.*—After removal of the reference brick, set the test brick in place flatwise, counting zero time as the moment of contact of the brick with the water. During the period of contact (1 min ± 1 sec) keep the water level within the prescribed limits by adding water as required. At the end of 1 min ± 1 sec, lift the brick from contact with the water, wipe off the surface water with a damp cloth, and reweigh the brick to the nearest 0.5 g. Wiping shall be completed within 10 sec of removal from contact with the water, and weighing shall be completed within 2 min.

NOTE.—Placing of the brick in contact with the water shall be done quickly but without splashing. Setting the brick in position with a rocking motion will avoid the entrapping of air on its under surface. Brick with frogs or depressions in one flatwise surface shall be tested with the frog or depression uppermost.

Calculations and Report

28. (a) The difference in weight in grams between the initial and final weighings is the weight in grams of water absorbed by the brick during 1-min contact with the water. If the test specimen is a whole brick and the area of its flatwise surface (length times width) does not differ more than ±0.75 sq in. (±2.5 per cent) from 30 sq in., report the gain in weight in grams as the initial rate of absorption in 1 min.

(b) If the test specimen is not a whole brick or the area of its flatwise surface differs more than ±0.75 sq in. (±2.5 per cent) from 30 sq in., calculate the equivalent gain in weight for 30 sq in., as follows:

$$X = \frac{30W}{LB}$$

where:

X = gain in weight corrected to basis of 30 sq in. flatwise area,
W = actual gain in weight of specimen in grams,
L = length of specimen in inches, and
B = width of specimen in inches.

Report the corrected gain in weight, X, as the initial rate of absorption in 1 min.

(c) If the test specimen is a cored brick, calculate the net area and substitute for *LB* in the formula given in Paragraph (*b*). Report the corrected gain in weight as the initial rate of absorption in 1 min

EFFLORESCENCE

Apparatus

29. (*a*) *Trays and Containers.*—Watertight shallow pans or trays made of metal or other material that will not provide soluble salts when in contact with distilled water containing leachings from brick. The pan shall be of such dimensions that it will provide not less than a 1-in. depth of water. Unless the pan provides an area such that the total volume of water is large in comparison with the amount evaporated each day, suitable apparatus shall be provided for keeping a constant level of water in the pan.

(*b*) *Drying Room.*—A drying room conforming to the requirements of Section 21 (*f*).

(*c*) *Drying Oven.*—A drying oven conforming to the requirements of Section 21 (*d*).

Test Specimens

30. (*a*) Ten dry full-size brick shall be tested.

(*b*) The ten specimens shall be sorted into five pairs, so that both specimens of each pair will have the same appearance, as nearly as possible.

Preparation of Specimens

31. The specimens shall be tested as received, except that any adhering dirt that might be mistaken for efflorescence shall be removed by brushing.

Procedure

32. (*a*) Set one specimen from each of the five pairs on end, partially immersed in distilled water to a depth of approximately 1 in., for 7 days in the drying room. When several specimens are tested in the same container, the individual specimens shall be separated by a space of at least 2 in.

NOTE 1.—Testing specimens from different sources simultaneously in the same container is not recommended, because specimens with a considerable content of soluble salts may contaminate the salt free specimens.

NOTE 2.—The pans or trays should be emptied and cleaned after each test.

(*b*) Store the second specimen from each of the five pairs in the drying room without contact with water.

(*c*) At the end of 7 days inspect the first set of specimens and then dry both sets in the drying oven for 3 days.

NOTE 3.—A drying period of 24 hr is sufficient for the purpose of an efflorescence test.

Examination and Rating

33. After drying, examine and compare each pair of specimens, observing the top and all four faces of each specimen. If there is no observable difference due to efflorescence, report the rating as "no efflorescence." If any difference due to efflorescence is noted, the specimens shall be viewed from a distance of 10 ft under an illumination of not less than 50 ft-candles, by an observer with normal vision. If under these conditions no difference is noted, report the rating as "slightly effloresced." If a perceptible difference due to efflorescence is noted under these conditions, report the rating as "effloresced." Record the appearance and distribution of the efflorescence.

MEASUREMENT OF SIZE

Apparatus

34. Either a 1-ft steel rule, graduated in $\frac{1}{32}$-in. divisions, or a brick gage or caliper having a scale ranging from 1 to 12 in., graduated in $\frac{1}{32}$-in. divisions, and having parallel jaws, shall be used for measuring the individual brick.

Test Specimens

35. Ten dry full-size brick shall be

measured. These brick shall be representative of the shipment and shall include the extremes of color range and size as determined by visual inspection of the shipment. (The same samples may be used for determining efflorescence and other properties.)

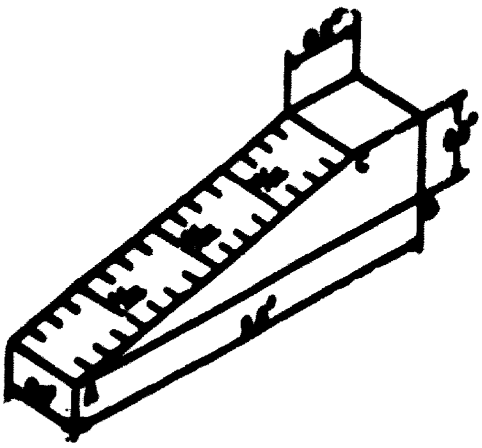

FIG. 1.—Measuring Wedge.

Individual Measurements of Length, Width, and Depth

36. The length shall be measured along both beds and along both faces from the mid-points of the edges bounding the ends. These four measurements shall be recorded to the nearest $\frac{1}{32}$ in. and the average recorded to the nearest $\frac{1}{16}$ in. as the length. The width shall be measured in like manner across both beds and both ends from the mid-points of the edges bounding the faces. These four measurements shall be recorded to the nearest $\frac{1}{32}$ in. and the average recorded to the nearest $\frac{1}{16}$ in. as the width. The depth shall be measured across both faces and both ends from the mid-points of the edges bounding the beds. These four measurements shall be recorded to the nearest $\frac{1}{32}$ in. and the average recorded to the nearest $\frac{1}{16}$ in. as the depth. Either the steel rule or the brick gage described in Section 35 may be used. Retests shall be made by the same method when required.

MEASUREMENT OF WARPAGE

Apparatus

37. (*a*) *Steel Straightedge.*

(*b*) *Rule or Measuring Wedge.*—A steel rule graduated from one end in $\frac{1}{32}$-in. divisions, or alternately a steel measuring wedge 2.5 in. in length by 0.5 in. in width by 0.5 in. in thickness at one end and tapered, starting at a line 0.5 in. from one end, to zero thickness at the other end. The wedge shall be graduated in $\frac{1}{32}$-in. divisions and numbered to show the thickness of the wedge between the base, *AB*, and the slope, *AC*, Fig. 1.

(*c*) A flat surface of steel or glass not less than 12 by 12 in. in area and plane to within 0.001 in.

Sampling

38. The sample of ten brick selected for determination of size shall be used.

Preparation of Samples

39. The specimens shall be tested as received, except that any adhering dirt shall be removed by brushing.

Procedure

40. (*a*) Where the warpage is concave, place the straightedge lengthwise or diagonally along the surface to be measured, selecting the location that gives the greatest departure from straightness. The maximum warpage as shown by the greatest distance of the brick surface from the edge of the straightedge should be selected and measured either by the steel rule or wedge. Measure this distance to the nearest $\frac{1}{32}$ in., and record as the concave warpage.

(*b*) When the warpage is convex, lay the brick with the convex surface in contact with a plane surface and with the corners approximately equidistant from the plane. Take measurements from the plane surface to the four corners of the brick. Record the average of the four measurements as the convex warpage of the brick.

APPROVED AS
AMERICAN STANDARD
BY THE AMERICAN STANDARDS ASSOCIATION
ASA NO.: A[illegible]
UDC [illegible]

Standard Methods of Test for
SIEVE ANALYSIS AND WATER CONTENT OF REFRACTORY MATERIALS[1]

ASTM Designation: C 92 - 46

Adopted, 1946.[2]

Reapproved in [illegible] Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation C 92; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These methods of test cover procedures for a wet method and a dry method for sieve analysis of refractory materials, and also procedures for determining the water content of refractory materials in the wet condition.

(*b*) *Wet Sieve Analysis.*—Water promotes the slaking of clays and helps to separate fine particles and to wash them from the larger grains. Consequently, this method will provide more reliable information than the dry method on materials to which water is added and in which slaking would occur in normal industrial use. This method is recommended, therefore, for use with such materials.

(*c*) *Dry Sieve Analysis.*—The dry method is not as effective as the wet method in determining the amount of material present in the smaller particle sizes. It is recommended (*1*) for clays only when the slaking action of water is undesirable, (*2*) when the material is in the form of coarsely ground grog and calcine, and (*3*) when the clay is to be used in such a way that the size of the ultimate particles is of secondary importance.

(*d*) *Water Content.*—There is included a method for determining the water content of air-dry samples as received, so that the sieve analysis can be calculated on the dry basis. There is also included a method for obtaining the water content of other refractory materials, such as plastic refractories and wet mixes.

Sieves

2. Sieves of appropriate mesh from either list given in Table I shall be used. The wire cloth for the sieves[3] shall be woven (not twilled) and shall be mounted without distortion or looseness in circular frames 8 in. in diameter. Pans and covers shall be provided for the sieves.

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories.

[2] Prior to their present adoption as standard, these methods were published as tentative from [illegible] to [illegible], being revised in [illegible]. They were adopted in [illegible], but were withdrawn, revised, and republished as tentative from [illegible] to [illegible].

[3] See the Specifications for Sieves for Testing Purposes (ASTM Designation: E 11), see p. [illegible]

TABLE I—A S T M SIEVES AND EQUIVALENT TYLER STANDARD SIEVES

A S T M Sieves (U. S. Standard Sieve Series Number)	Tyler Standard Sieves (Mesh Number)
No. 3	3[a]
No. 4	4
No. 6	6
No. 8	8
No. 12	10
No. 16	14
No. 20	20
No. 30	28
No. 40	35
No. 50	48
No. 70	65
No. 100	100
No. 140	150
No. 200	200

[a] This sieve was editorially added to the list in Table I January, 1960.

Sample

3. The sample submitted shall be representative of the material to be tested, and shall weigh at least four to five times the required weight of the actual test sample. Material of which the water content is to be determined shall be packed in a water-tight container.

Water Content

4. (*a*) *Wet Type Air-Setting Refractory Mortars.*—The material shall be sampled immediately after opening the original container and after carefully mixing the contents. The test samples shall weigh approximately 50 g. To facilitate handling the test sample, it is recommended that it be placed on a tared piece of waxed paper or tared aluminum weighing dish. The exact weight of the test sample shall be determined to the nearest 0.1 g. both before and after drying for 24 hr. at 220 to 230 F. (105 to 110 C.). The percentage of water shall be calculated on the as-received basis to the nearest 0.1 per cent.

(*b*) *Materials Other Than Wet Type Air-Setting Refractory Mortars* (such as ground fireclays, fireclay mortars, dry type air-setting mortars, plastic refractories, and similar materials).—If the material is shipped in the wet condition, care shall be taken that no loss of water occurs before the test sample is taken. The test sample shall weigh approximately 250 g. The exact weight of the test sample shall be determined to the nearest 0.1 g. both before and after drying for 3 hr. at 220 to 230 F. (105 to 110 C.). The percentage of water shall be calculated on the as-received basis to the nearest 0.1 per cent. The dried test sample may be required for further use (see Section 5 (*a*) and (*b*) and Section 6 (*a*) and (*b*)).

Wet Sieve Analysis

5. (*a*) *Dry Materials.*—If the material is received in the dry condition, the test sample (Note 1) shall consist of the dried and weighed test sample prepared in accordance with Section 4 (*b*).

(*b*) *Wet Materials.*—Materials prepared with water (plastic refractories, wet type high-temperature bonding mortars, etc.) shall be tested as received. Two test samples shall be taken immediately upon opening the original container, and in the case of mortars after carefully mixing the contents. One sample shall be used for obtaining the water content in accordance with Section 4 (*a*) or (*b*), depending upon the type of material to be tested. The other sample (Note 1) shall be used for the sieve analysis and shall weigh approximately 250 g. It shall be weighed to the nearest 0.1 g. and then transferred to the 1 liter container (see Paragraph (*c*)). The utensils used during weighing (to which a small part of the sample may adhere) shall be washed by means of a small jet of water from a ¼-in. hose in such a manner as to insure a quantitative transfer of the weighed sample to the container.

(*c*) *Procedure.*—The test sample shall be placed in a container of about 1-liter capacity, to which sufficient water shall be added to form a slurry. Slaking shall be allowed to proceed for 1 hr., after which a further addition of water may be necessary. The test sample shall then be transferred without loss to the finest sieve to be used in the analysis, and shall then be washed by means of a small jet of water from a ½-in. rubber hose until the water passing through the sieve contains only traces of the sample. Care shall be exercised in the washing operation to prevent loss by splashing. It may be necessary to break up lumpy material by gentle rubbing between the fingers, but never by rubbing or pressing against the sieve. The washed residue in the sieve shall then be dried to constant weight at 220 to 230 F. (105 to 110 C.). This usually requires about 2 hr. If desired, a preliminary drying period at a lower temperature may be used. The dried residue shall then be transferred to the top or coarsest sieve of the series to be used. The sieving and weighing operations shall then be completed in accordance with Section 6 (*a*) or (*b*).

(*d*) *Calculation and Report.*—The wet sieve analysis shall be calculated for the test sample on the dry weight, and the results reported to the nearest 0.1 per cent of the material retained on each sieve (Note 2). The percentage passing the finest sieve shall be reported as the difference between 100 per cent and the sum of the percentages retained on the various sieves.

Dry Sieve Analysis

6. (*a*) *Machine Sieving.*—When the sieving is to be done by machine, the sieves shall be arranged in the order of size with the coarsest sieve at the top of the nest. The sample for sieving (Note 1) shall consist of the dried and weighed material prepared in accordance with Section 4 (*b*). It shall be transferred to the top sieve of the nest, and shall then be subjected to the mechanical sieving operation until less than 0.1 g. of material passes through each sieve after 1 min. of sieving by hand, as described in Paragraph (*b*). The machine sieving operation usually requires about 15 min. The sieves shall then be carefully separated and the amount of material retained on each sieve shall be determined by weighing to the nearest 0.1 g.

(*b*) *Hand Sieving.*—The sample for sieving (Note 1) shall consist of the dried and weighed material prepared in accordance with Section 4 (*b*). The sieving operation shall be carried out using one sieve at a time beginning with the coarsest sieve and from then on using successively finer sieve sizes. The sieve, with pan and cover attached, shall be alternately tapped and rotated while held in slightly inclined position so that the test sample will be well distributed over the sieve. The operation shall be continued until less than 0.1 g. of the material passes through each sieve in 1 min. of continuous sieving. The amount of material retained on each sieve shall be determined by weighing to the nearest 0.1 g.

(*c*) *Calculation and Report.*—The dry sieve analysis shall be calculated for the test sample on the dry weight, and the results reported to the nearest 0.1 per cent of the material retained on each sieve (Note 2). Dust loss shall be reported as material passing the finest sieve.

EXPLANATORY NOTES

NOTE 1.—The size of the test sample may be changed by reason of the nature of the material. For example, some clays, when ground to exceedingly fine particle size, tend to pack or cake on the sieves, in which case a 100-g. sample may be used. In the case of plastic refractories or coarsely ground mixes, the size of the sample might well be increased to 500 g. in weight.

NOTE 2.—As an alternative, the results of sieve analysis may be reported on the cumulative basis, either as the total percentage retained on or passing each sieve.

Standard Method of

SAMPLING CERAMIC WHITEWARE CLAYS[1]

ASTM Designation: C 322 - 56

ADOPTED, 1956.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 322; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers procedures for pling shipments of ceramic white- e clays.

pling Procedure

2. (a) Where a bulk shipment con- of lumps, a number of pieces shall picked from different parts of the o that the final sample will repre- in average of all parts of the ship- from top to bottom. For a 30-ton no less than 20 samples of approxi- ely 10 lb each shall be taken from ent parts of the car. This may be by digging holes at equally spaced ls in the loaded car, or by removal loading or unloading, at spaced vals. The lumps shall then be broken es no larger than 4 in., in the t dimension, and the several es shall be made into a composite y turning with a shovel on a clean The composite shall then be quartered or riffled to provide a 10-lb laboratory sample.

(b) Where a bulk shipment consists of shredded or coarsely ground clay, sampling shall be done at 20 points for a 30-ton unit; the samples may be taken with a shovel, or with a grain sampler, if the form of the clay permits. The samples so taken shall then be mixed thoroughly, and quartered or riffled to form a 10-lb (or proportionately larger) laboratory sample.

(c) For bagged lots of ground or air-floated clay, the number of samples taken shall depend on the number of units in a shipment. A grain-sampler or similar sampling instrument shall be used to take samples which then shall be combined, mixed, and quartered or riffled to obtain a 10-lb laboratory sample. Where a shipment consists of 100 bags or less, the number of bags sampled at random shall be not less than 5 and preferably 10. When the number of bags is greater than 100 but less than 500, the number of bags sampled shall not be less than 15. For lots of from 500 to 1000 bags, 20 bags shall be sampled. For shipments of 1000 to 2000 bags, 30 bags shall be sampled at random.

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[1] r the standardization procedure of the this method is under the jurisdiction of M Committee C-21 on Ceramic White-

[2] r to adoption as standard, this method hed as tentative from 1953 to 1956.

Standard Methods for

CHEMICAL ANALYSIS OF CERAMIC WHITEWARE CLAYS

ASTM Designation: C 323 - 56

Adopted, 1956.[3]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 323; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (*a*) These methods cover procedures for the chemical analysis of clays employed in the manufacture of ceramic whitewares.

(*b*) The analytical procedures appear in the following order:

	Section
Moisture	7
Loss on Ignition	8
Silica	9
Iron, Aluminum, and Titanium Oxides	10
Iron Oxide	11
Titania	12
Alumina	13
Lime	14
Magnesia	15
Alkalies	16

NOTE.—These methods have been compiled as standard procedures for use in referee analyses. These methods, however, when the determination of iron oxide as Fe_2O_3 is involved, are not intended to preclude the use of other procedures that give results within the permissible variations. For the sake of uniformity the classical Zimmerman-Reinhardt procedure is specified for the determination of iron oxide. I recognized that numerous other procedures equally accurate and often more convenient. I other procedures commonly in use include re tion of an oxidized solution with zinc or o metal, and titration with standard KMn $K_2Cr_2O_7$, as well as titration with a sta solution of titanous chloride in an ox solution. These procedures shall be consid acceptable, provided the analyst has obta results by his special procedure that check the Zimmerman-Reinhardt procedure w the limits specified in Section 6. It is sugg that the National Bureau of Standards' sta samples be used for checking the accurac procedures.

It will be understood that the making of a complete analysis of a ceramic whiteware is a difficult procedure requiring a wide k edge of the chemistry involved in the opera and a thorough training in carrying out the A skilled analyst of good training is th required to do the work. The descriptions given cover the vital points of procedure. frequent reference in regard to the deta the various manipulations should be ma "Applied Inorganic Analysis" by Hill and Lundell[4] and to similar publications ticularly in the determination of alumina, re ence should be made to *Scientific Paper No* of the National Bureau of Standards.[5]

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the ASTM Committee C-21 on Ceramic Whiteware.

Except for certain modifications in Sections 1, 3, and 6, these methods are identical in substance with Sections 1 to 16 of the Standard Methods of Chemical Analysis of Refractory Materials (ASTM Designation: C 18), see p. 361.

[3] Prior to adoption as standard, these methods were published as tentative from 1951 to 1956.

[4] W. F. Hillebrand and G. E. F. Lundell, "A Inorganic Analysis," Wiley and Son, New Y (1929).

[5] W. Blum, "Determination of Alumina as O National Bureau of Standards *Scientific Paper* N

Reagents

2. Unless otherwise indicated, it is in- ded that all reagents shall conform to e specifications of the Committee on alytical Reagents of the American emical Society, where such specifica- s are available.[10] Other grades may e used, provided it is first ascertained t the reagent is of sufficiently high rity to permit its use without lessening e accuracy of the determination. Un- otherwise indicated, references to ter shall be understood to mean dis- ed water.

Paragraphs (a) to (n) include those gents common to two or more of the lytical procedures. Other reagents l be found listed with the particular thod in which they are prescribed.

) *Concentrated Acids and Ammo- m Hydroxide.*—Concentrated acids ammonium hydroxide of approxi- ely the following specific gravities or entrations will be required:

chloric acid, HCl	1.18 sp gr
c acid, HNO_3	1.42 sp gr
ric acid, H_2SO_4	1.84 sp gr
luoric acid, HF	40 per cent
loric acid $HClO_4$	60 to 70 per cent, cp[a]
rous acid	6 per cent solution[b]
nium hydroxide, NH_4OH	0.90 sp gr

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[a] Lower purity varieties may contain aluminum oxide, as an impurity.

[b] As supplied by reagent manufacturers.

) *Diluted Acids and Ammonium Hy- de.*—The diluted acids and ammo- hydroxide referred to are of varying entages by volume. They shall be e up by mixing proportional volumes e concentrated reagent and water. diluted sulfuric acid mixtures shall ade up by slowly stirring the acid the water. These diluted acids and ammonium hydroxide are designated in the methods as (1:4), (1:9), etc, except very diluted solutions which are referred to by the percentage of reagent added. The designation in parentheses indicates the ratio of the volume of the concentrated reagent to the volume of water; for example, H_2SO_4 (1:9) contains 10 per cent by volume of H_2SO_4 (sp gr 1.84). The following will be required:

	Percentage by Volume
Hydrochloric acid	50 20
Sulfuric acid	50 10 5
Nitric acid	30 3
Ammonium hydroxide	50 5

(c) *Ammonium Chloride (2 per cent).*—Dissolve 2 g of NH_4Cl in 100 ml of water.

(d) *Ammonium Oxalate Solution (saturated).*—Dissolve 4 g of $(NH_4)_2C_2O_4$ in 100 ml of water.

(e) *Standard Potassium Permanganate Solution (0.1 N).*—Dissolve 3.25 g of $KMnO_4$ in 1000 ml of water. Allow to stand for a week, filter through an asbestos mat, porous glass, or procelain filter, and keep in a dark place. Standardize against the National Bureau of Standards standard sample No. 40c of sodium oxalate.

(f) *Standard Potassium Permanganate (0.04 N).*—Dissolve 2.5 g of $KMnO_4$ in water and make up to 2 liters. Allow to stand for a week, filter through an asbestos mat, porous glass, or porcelain filter, and keep in a dark place. Standardize against the National Bureau of Standards standard sample No. 40c of sodium oxalate.

(g) *Standard Titania Solution.*—Weigh out 0.05 g of calcined TiO_2. Fuse with 10 g. of $K_2S_2O_7$ in a clean platinum crucible, keeping the temperature as low as possible to maintain fluidity.

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[10] Reagent Chemicals, American Chemical Specifications," Am. Chem. Soc., Wash- D. C. For suggestions on the testing of not listed by the American Chemical e "Reagent Chemicals and Standards," h Rosin, D. Van Nostrand Co., Inc., York, N. Y., and the "United States copoeia."

Cool, and dissolve in about 300 ml of H_2SO_4 (1:5). Cool, transfer to a 500-ml volumetric flask, dilute to the mark with water, and mix thoroughly. To standardize the solution, take two 50-ml portions in 400-ml beakers, dilute, boil, and precipitate with NH_4OH. Filter, and wash with hot water. Place the papers in the original beakers, add 15 ml of HCl, stir to macerate the paper, dilute, and precipitate again with NH_4OH. Filter, and wash with hot water until free of alkali salts. Ignite carefully, blast, and weigh. From the weight determined, calculate the strength of the solution.

(*h*) *Standard Sodium Arsenite Solution.*—Dissolve 0.908 g of arsenious oxide, As_2O_3, in a small amount of hot Na_2CO_3 solution, cool, filter, and dilute to 1 liter. Standardize against a steel of known manganese content.

(*i*) *Stannous Chloride Solution.*—Dissolve 50 g of $SnCl_2$ in 100 ml of HCl and dilute to 1000 ml. Keep a few pieces of metallic tin in the bottle.

(*j*) *Mercuric Chloride Solution* (saturated).—Prepare a saturated solution of $HgCl_2$.

(*k*) *Manganese Sulfate Solution.*—Dissolve 70 g of crystalline $MnSO_4$ in 500 ml of water. Add 140 ml of phosphoric acid, H_3PO_4, (sp gr 1.7) and 130 ml of H_2SO_4 (sp gr 1.84). Dilute to 1 liter.

(*l*) *Hydrogen Peroxide (30 per cent).*

(*m*) *Diammonium Phosphate Solution.*—Dissolve 10 g of $(NH_4)_2HPO_4$ in 100 ml of water.

(*n*) *Chloroplatinic Acid Solution (10 per cent).*

(*o*) *Ethyl Alcohol (80 per cent).*—Prepare a solution containing 80 per cent by volume of ethyl alcohol in water.

(*p*) *Ethyl Alcohol (absolute).*—Certain commercial brands of denatured absolute alcohol are satisfactory as well as being considerably less expensive than the reagent grade absolute alcohol.

Sampling

3. (*a*) *Selection of Sample.*—The sample shall be obtained in accordance wit the Method of Sampling Ceramic Whiteware Clays (ASTM Designation: C 322).[5]

(*b*) The sample shall be crushed in a small jaw or roll-type crusher wit hardened tool-steel faces to pass a No. (2380-micron) sieve.[6] The sample sha then be crushed to pass a No. 20 (8 micron) sieve,[6] mixed, and quartered to about 50 g. This 50-g sample shall the be ground so that it will all pass a No 100 (149-micron) sieve,[6] unless otherwise specified, mixed thoroughly, and placed in a container that will insure freedom from contamination. Fine grinding shall be done in a suitable mortar (agate, mullite, alumina, or boro carbide) to prevent the introduction of impurities. Precautions shall be taken to prevent contamination of the sample by steel particles from the samplin equipment during crushing or grindin

Statement of Analysis

4. Moisture shall be determined on the sample in its ordinary air-dried condition. All other percentage compositi shall be determined on moisture-fre samples and reported, accordingly, on a moisture-free basis. The drying te perature recommended for all moistu determinations is 105 to 110 C. Whe ever a sample is weighed out for any de termination other than moisture, it sh be moisture-free. If preferred, the sa ple may be dried in a weighing bott from which the required samples shall be weighed out.

Blank Determinations

5. Blank determinations on the re agents shall be made for each consti

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[5] See p. 421.

[6] Detailed requirements for these sieves are give the Specifications for Sieves for Testing Purposes (Designation: E 11), see p. 1043.

nt in the whiteware clay and this k deducted in each case. For the ermination of the silica blank, ap- ximately 0.25 g of Al_2O_3 should be led as aluminum chloride.

eproducibility of Results

In all cases, check determinations ll be made, and the results shall be etermined if satisfactory checks are btained. Results shall be considered factory if the differences between k determinations do not exceed the wing limits:

	Permissible Variations Between Check Determinations, max, per cent
silica or other constituent amounting to 30 per cent* or over	0.3
alumina or other constituent mounting to 10 to 30 per cent*	0.2
any other constituent ounting to under 10 per cent*	0.1

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* These figures are stated in terms of the whole sample per cent.

isture

Weigh 1.00 g of the sample and t to constant weight at a temperature under 105 nor over 110 C. Record loss in weight as moisture.

on Ignition

Weigh 1.000 g of the moisture-free to 110 C) sample and heat to con- weight over a blast lamp, or in an ric muffle furnace, at 900 to 1000 C. rd the loss in weight as the ignition

ca

Weigh 0.5000 g of the moisture- (105 to 110 C) sample into a plati- crucible containing about 5 g of powdered anhydrous Na_2CO_3 and mix well with a platinum wire. Cover the mixture with a little more Na_2CO_3. Heat gradually to the full heat of a good burner (1000 to 1100 C) maintained for about 1 hr until complete solution is obtained. Place the crucible cover on a triangle, and when the melt has partially cooled, pour it on the lid (Note). When cool, place the crucible and lid in a 150-ml beaker, placing the button on a watch glass above the beaker. Add 30 ml of HCl (1:1). When solution is complete wash off the crucible and lid with HCl (1:4), taking care to remove all SiO_2. Place the button in the solution. Transfer the contents of the beaker to an evaporating dish and evaporate to dryness on a steam bath. Bake for 1 hr at 110 C. Add 20 to 30 ml of HCl (1:1) and 50 ml of hot water. When all salts have been dissolved, allow to settle for several minutes and then filter through a No. 40 Whatman paper, or equivalent. Wash the SiO_2 three times by decantation using 20 to 30-ml portions of first hot water, then HCl (1:1), then hot water again. Transfer the precipitate to the filter paper, removing all SiO_2 from the dish with a policeman. Wash the paper and precipitate with hot water until free from salt. To recover the small amount of SiO_2 remaining in the filtrate, evaporate to dryness, using the same procedure for baking and filtering as before. Combine the two precipitates, place in a platinum crucible, and burn off the paper carefully to prevent any loss of SiO_2. Ignite the sample to constant weight at 1100 to 1200 C (15 to 20 min. is usually sufficient), cool in a desiccator, and weigh. Moisten the residue with several milliliters of water, add 10 ml of HF and 3 or 4 drops of H_2SO_4. Evaporate the solution to dryness, ignite carefully to prevent decrepitation, and blast for several minutes at 1100 C. Cool the crucible in a desiccator, weigh, and repeat blasting to constant weight. The loss in weight from the original silica residue represents the SiO_2 content, except for that part of the SiO_2 which is later recovered from alumina, etc.

NOTE. Another scheme to aid in subsequent solution of the fused melt is to rotate the crucible as it cools, spreading the mass up the side walls.

Iron, Aluminum, and Titanium Oxides

10. Fuse the residue with 1 g of fused $K_2S_2O_7$ or $Na_2S_2O_7$, dissolve in a small amount of water, and add to the filtrate from the silica determination (Section 9). Add 5 g of NH_4Cl and 3 drops of methyl red solution (0.1 per cent). Heat the solution almost to boiling, and add slowly NH_4OH (1:1) until the indicator has changed to a yellow color. Boil for several minutes to remove the excess ammonia. Allow to settle for 30 min and decant through a No. 41 Whatman filter paper or equivalent, transferring the precipitate to the paper and washing the beaker and paper several times with a warm NH_4Cl solution (2 per cent). Reserve the filtrate, *A*, for the determination of CaO and MgO (Section 14). Return the precipitate and paper to the original beaker, add 50 ml of hot water and 10 ml of HCl (sp gr 1.19). Stir until the precipitate is dissolved and the paper is well macerated. Dilute to about 200 ml with hot water, precipitate and filter as before. Combine this filtrate *B* with filtrate *A*. Wash the paper and precipitate with a warm NH_4Cl solution (2 per cent). Place the precipitate in a weighed platinum crucible and ignite. Continue the ignition at 1200 C to constant weight (15 to 20 min is usually sufficient). Cool in a desiccator, and weigh with the crucible covered with the lid. The R_2O_3 consists of the Al_2O_3, TiO_2, and Fe_2O_3 present in the sample. In addition there may be small amounts of P_2O_5, ZrO_2, V_2 and Cr_2O_3.

Iron Oxide

11. (*a*) *Method A: Fe_2O_3 Determine on R_2O_3 Sample.* Heat the R_2O_3 precipitate (Note 1) obtained in the dete mination of iron, aluminum, and ti nium oxides (Section 10) with fuse $K_2S_2O_7$ or $Na_2S_2O_7$ until solution is com plete. Dissolve the fusion in 50 ml H_2SO_4 (1:9) and evaporate to fume Cool, dilute with water, and filter off t SiO_2, washing with hot water. Reserv the filtrate for the determination Fe_2O_3 and TiO_2. Ignite the SiO_2 in platinum crucible and weigh. Treat th precipitate with 5 ml of HF and 2 3 drops of H_2SO_4. Evaporate to dry ness, ignite, and weigh. The loss weight represents extra SiO_2 whi should be added to that determined previously and also deducted from t weight of the R_2O_3 precipitate. Eva rate the filtrate obtained in correcti the R_2O_3 precipitate for SiO_2 to ab 75 ml. Cool, and dilute to 100 ml in volumetric flask. Reserve 25 ml the determination of TiO_2 (Section 1 To the remainder, add 25 ml of H (1:1) and heat to boiling. Reduce iron by adding $SnCl_2$ solution drop drop from a pipet with constant sw ing of the beaker until the solution colorless. Then add one drop in exc Cool quickly in running water, then at one stroke 15 ml of saturated H solution. Allow to stand for 3 then transfer with washing to a beaker containing 300 ml of cold tilled water and 25 ml of $MnSO_4$ s tion. Titrate with standard 0.0 $KMnO_4$, added very slowly while ring constantly, until a permanent end point is obtained.

NOTE 1.—Instead of fusing directly in platinum crucible in which the R_2O_3 was ig e precipitate may be brushed into a porcelain -ible and then fused with $K_2S_2O_7$ or $Na_2S_2O_7$. s avoids loss of platinum by the action of the -sulfate, and no platinum is present in the -tion to interfere with the iron determination.

(b) *Method B: Fe_2O_3 Determined on a Separate Sample.*—Weigh 1.00 g of e finely ground, moisture-free (105 to 0 C) sample into a platinum crucible, 1 10 drops perchloric acid ($HClO_4$) 1 20 ml of HF, and heat almost to ness on a hot plate. Add 5 to 10 ml $HClO_4$ and heat until residue has dis-ved (Note 2). Cool, place crucible in a -ed beaker, add 100 ml of water, and t to boiling. Any residue present, er than SiO_2, should be filtered off l fused with $K_2S_2O_7$ or $Na_2S_2O_7$ in a elain crucible and added to the main tion.

Note 2.—Decomposition of ceramic white- e clays and some fired whiteware materials e effected equally as well by substituting al volume of H_2SO_4 (1:1) for the $HClO_4$. is case, heat to fumes once, cool, dilute, , and fuse any residue remaining undis- ed.

(c) Determine iron, using one of the oved methods referred to in the e under Section 1.

Titania

12. Determine TiO_2 colorimetrically e of a photometer, as follows: Trans- the 25-ml portion reserved for the rmination of TiO_2 (Section 11) to a ml volumetric flask. Add 5 ml of $_4$ (1:1). Cool to room temperature ilute to the mark. Transfer exactly l the solution to another 100-ml metric flask. Dilute one of the flasks e mark with H_2SO_4 (1:19). To the r add 5 ml of 3 per cent H_2O_2, then e nearly to the mark with H_2SO_4 19), adjust temperature to 25 ± 1 C, djust volume exactly and let stand 5 min. Transfer a portion from the first flask to a cuvette and set the potentiometer scale reading at zero. Then measure the absorption of the solution in the second flask. Read the percentage of titania present from a calibration curve. Construct this curve by adding varying amounts of the standard titania solution to H_2SO_4 (1:19), develop color with 5 ml of 3 per cent H_2O_2, let stand 5 min and read the absorption, using H_2SO_4 (1:19) for the zero setting of the potentiometer scale.

Note 1.—If a spectrophotometer is used, measure the absorption at mean transmission of 420 mμ. If a filter photometer is used, use a glass with a maximum transmission in the region of 420 mμ.

Note 2.—As an alternate method, TiO_2 can be determined in the 25-ml portion reserved for this purpose (Section 11) by oxidizing both the sample and the standard TiO_2 solution with several drops of a H_2O_2 solution (30 per cent). Compare the colors either in Nessler tubes or in a suitable colorimeter. Use a H_2SO_4 solution (5 per cent) for diluting purposes in matching the colors.

Alumina

13. Subtract the calculated weight of Fe_2O_3 (Section 11 (*a*) or (*b*)), TiO_2 (Section 12), and SiO_2 (Section 9) from the weight of R_2O_3 (Section 10). The remainder is the weight of Al_2O_3 plus small amounts of the oxides which may include those previously mentioned in Section 10. These are generally considered as Al_2O_3 in reporting the analysis of ceramic whiteware clays.

Lime

14. Evaporate the combined filtrates reserved (Section 10) for the determination of CaO and MgO to about 200 ml, add 10 to 15 ml of the saturated ammonium oxalate solution and 2 to 3 ml of NH_4OH. Heat for 1 to 2 hr, by which time the volume should be about 75 to 100 ml. Allow the precipitated calcium oxalate to settle. Decant through a dense filter paper (Whatman No. 40 or,

equivalent), taking care to retain the precipitate in the beaker, wash several times with warm water by decantation, and then wash the paper until free from soluble salts. Reserve the filtrate for the MgO determination (Section 15). Return the paper to the beaker containing the precipitate, add 100 ml of H_2SO_4 solution (5 per cent), warm, and titrate to a faint pink end point with standard 0.04 N $KMnO_4$ solution. A blank should be previously determined for the effect of the paper.

NOTE. For greater accuracy, a double precipitation should be made, in which case, after precipitating the calcium oxalate as described above, decant the liquid and wash the beaker and paper several times with warm water. Dissolve the precipitate on the paper with warm HCl (1:4), allowing it to run into the beaker containing the major portion of the calcium oxalate. Wash the paper with hot water. To the solution (about 75 to 100 ml in volume) add several milliliters of saturated $(NH_4)_2C_2O_4$ solution and NH_4OH in slight excess. Heat for 2 hr, filter, wash, and titrate as described above.

Magnesia

15. Evaporate the filtrate from the CaO determination (Section 14) to about 150 to 200 ml and add 2 to 3 g of diammonium phosphate ($(NH_4)_2HPO_4$), stir until dissolved, and then add NH_4OH until alkaline and then 20 ml in excess. Allow the solution to stand overnight. Filter and wash with NH_4OH (5 per cent). Dissolve the precipitate on the paper with hot HCl (1:4), allowing it to run into the beaker containing the precipitate. Wash the paper with hot water. To the solution, which should be not more than 100 ml in volume, add 0.1 to 0.2 g of $(NH_4)_2HPO_4$. Make ammoniacal, and then add a slight excess while stirring constantly until the precipitate is well formed. Then add 10 ml more of NH_4OH and allow to stand overnight or at least for 4 hr. Filter through a No. 40 Whatman paper or equivalent. Transfer the precipitate to the paper and wash well with NH_4OH (5 per cen Place the paper in a weighed platin or porcelain crucible, burn off the pa at a low temperature (below 900 C and ignite to constant weight at 1050 1100 C (15 to 30 min is sufficient).

Alkalies[7]

16. (a) Weigh 1.00 g of the moistu free (105 to 110 C) sample (ground to impalpable powder) and 1.0 g of NH_4 into an agate mortar and mix well. A 7 to 8 g of $CaCO_3$ (Note) and again m intimately. Place a ¼-in. layer of CaC in the bottom of a platinum crucible, a then add the above mixture, tapping t crucible occasionally to obtain a de mass. Place a ¼-in. layer of $CaCO_3$ the top. Heat the crucible over a l flame until ammonia fumes are no longe given off, then increase the heat so th the bottom half of the crucible is a d red and maintain this temperature f about 1 hr. Cool, fill the crucible thre fourths full of water, and heat until t contents can be taken out and crushe an agate mortar. Transfer to a pla num or porcelain dish by means of a of water. Evaporate to a low volum decant through a No. 40 Whatman fil paper, or equivalent, and wash the m terial in the dish several times by d cantation with warm water. Trans to the paper and wash several times w hot water. Acidify with several m liters of HCl and evaporate to a volu of 150 to 200 ml. Add several millili of NH_4OH and sufficient $(NH_4)_2CO_3$ precipitate the lime, keeping the d covered with a watch glass. Warm u the precipitate settles out. Filter a wash with warm water. Evaporate t solution to a low volume, then add small lump of $(NH_4)_2CO_3$ to determ whether practically all calcium has be precipitated. If no precipitate for

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[7] This procedure for the determination of alk the J. Lawrence Smith method.

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13. 3. 72

3 OF 3

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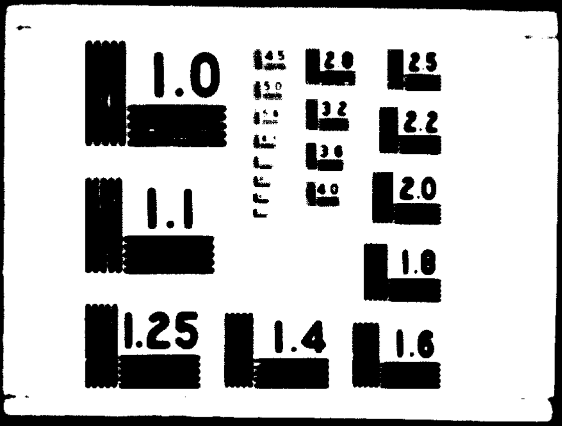

evaporate to dryness, otherwise precipitate and filter as before. Drive off the ammonium salts by heating just short of a dull red. Dissolve the residue in water and add a few milliliters of a saturated solution $(NH_4)_2C_2O_4$ and 1 to 2 ml of NH_4OH to precipitate the last trace of calcium. Heat for 30 to 45 min, filter, and wash with water containing $(NH_4)_2C_2O_4$ (0.1 per cent). Catch the filtrate in a weighed platinum dish. Add several drops of HCl and evaporate to dryness. Ignite gently as [illegible] and weigh as NaCl and KCl.

NOTE.—Calcium carbonate of the ACS grade low in alkalies and heavy metals shall be used.

(b) The separation of the potassium and sodium must be carried out in an atmosphere free from ammonia fumes. Add to the solution of the combined chlorides in a small porcelain dish, sufficient chloroplatinic acid solution to react with all of the sodium and potassium. The necessary amount to use is readily calculated from the known strength of the platinum solution and the weight of mixed chlorides counted as NaCl. The dilution of the resulting solution should be such that when heated on the steam bath any precipitate that may have formed entirely dissolves. This is to prevent inclusion of mother liquor in a mass of crystals suddenly formed. Evaporate until the solution is just sirupy enough to solidify on cooling. Do not evaporate to dryness, as this will dehydrate the sodium salt and render it less soluble in alcohol. Drench the residue with alcohol (80 per cent), filter by decantation through a small paper, wash by decantation with more of the alcohol, crushing the crystals with a small pestle or a widened and rounded end of a short glass rod. Reserve the filtrate and washings if sodium is to be determined directly. The residue should be golden yellow. An orange-red color indicates incomplete removal of the sodium salt. It is unnecessary to bring the mass of the precipitate upon the filter. Dry the dish and paper for a few moments to remove adhering alcohol. Dissolve the precipitate on the filter with hot water, catching the solution in a weighed crucible or small dish of platinum. Evaporate to dryness and add the salt that is still in the porcelain dish. If the salt is at all lumpy, redissolve it in water and again evaporate to dryness. Heat for 1 hr at 130 C in an air oven (100 C suffices for very small amounts of fine-grained precipitate). It is necessary to cover the receptacle at first because the precipitate is prone to decrepitate. When dry, cool and weigh as K_2PtCl_6. Calculate the oxides, as follows:

$$KCl = \text{wt. of } K_2PtCl_6 \times 0.3068$$
$$K_2O = \text{wt. of } K_2PtCl_6 \times 0.1941$$
$$NaCl = \text{total chlorides} - KCl$$
$$Na_2O = NaCl \times 0.5303$$

Standard Method of Test for
WET SIEVE ANALYSIS OF CERAMIC WHITEWARE CLAYS[1]

ASTM Designation: C 325 - 56

Adopted, 1956.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 325; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a procedure for wet sieve analysis of ceramic whiteware clays. The method is intended for use in testing shipments of clay as well as for plant control tests.

Apparatus

2. (*a*) *Stirring Device.*—A mechanical stirrer with a three-bladed propeller 2 in. in diameter and having a speed of approximately 1700 rpm, or the equivalent, shall be provided.

(*b*) *Sieves.*—The sieves shall conform to the Specifications for Sieves for Testing Purposes (ASTM Designation: E 11)[3] and shall include the No. 100 (149-micron), No. 140 (105-micron), No. 200 (74-micron) and No. 325 (44-micron) sieves (Note). The wire cloth for these sieves shall be woven (not twilled) and shall be mounted in circular metal frames 8 in. in diameter, which shall be so constructed as to permit nesting of two or more sieves. A pan and cover for the sieves shall be provided.

Note.—Equivalent sieves from other standard series, such as the Tyler series, may also be used. If results are to be compared with those obtained with sieves from the ASTM series, it is important that the openings of the sieves used fall within the tolerances specified in Specifications E 11 for the corresponding ASTM sieves.

Sample

3. (*a*) The sample shall be obtained in accordance with the Method of Sampling Ceramic Whiteware Clays (ASTM Designation: C 322).[3]

(*b*) The sample as received shall be placed in a drying oven at 100 to 110 C for a period of not less than 5 hr prior to testing.

Procedure

4. (*a*) Transfer duplicate portions, of approximately 250 g of the dried clay sample, weighed to the nearest 0.1 g, to containers of at least 2-liter capacity. Wet the clay with 1 liter of water and allow to slake for 2 hr. If a free-flowing slurry

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the ASTM Committee C-21 on Ceramic Whiteware.

[2] Prior to adoption as standard, this method was published as tentative from 1953 to 1956.

[3] Appears in this publication, see Contents in Numeric Sequence of ASTM Designations at front of book.

is not produced by this treatment, add another 500 ml of water.

(*b*) To insure complete separation of clay from nonplastic impurities, agitate the slurry by means of a mechanical stirrer (Section 2 (*a*)). Continue the stirring between 5 and 10 min.

(*c*) Transfer the slaked and stirred sample, without loss, to the finest sieve to be employed in the test, and wash by means of a small jet of water from a ¼ in. soft rubber hose attached to a water supply having a pressure not in excess of that of an ordinary city main. The force of the jet may be controlled by compressing the end of the hose between the thumb and forefinger. Take care to avoid loss of sample from splashing. Continue washing until water passing through the sieve shows only traces of sample. Should lumpy material remain on the screen, return the residue to the stirrer container by careful washing with a gentle jet of water, and agitate in approximately 1 liter of water for 10 min, then wash the slurry as previously described.

(*d*) Wash the residue remaining on the finest sieve into the pan. Thoroughly wet the remaining sieves to be used in the test with clear water, and nest them in the proper sequence on the finest sieve. Wash the residue in the pan quantitatively onto the top sieve, and give the stack a preliminary washing.

(*e*) Nest the top sieve on the pan, which shall contain about ½ in. of clear water. Wash the residue by holding the pan and sieve firmly in the hands, and by a sidewise movement, causing water to splash up through the sieve and into the residue. This movement, coupled with interspersed circular motions, allows thorough washing. Wash the residue and water remaining in the pan onto the top sieve of the stack.

(*f*) Again fill the pan with the proper amount of water, nest the top sieve and its residue on the pan, and repeat the operation. Continue this until the finest sieve has been washed. Carefully blot each sieve on its underside with a soft, damp sponge, and place the sieve either in a drying oven at 100 to 110 C or under infrared lamps until thoroughly dry. Approximately 2 hr is required with a drying oven, but only about 30 min with an infrared lamp set 12 in. above the sieve.

(*g*) Nest the dried residues and sieves in the proper order, with due care to prevent dusting of the residues. Close the stack of sieves with a dry pan and cover, and tap the assembly lightly for 1 min on a table top.

(*h*) Separate the nested sieves and carefully brush the residue from each onto a weighing paper. Weigh the residues to the nearest 0.001 g on an analytical balance.

Calculations and Report

5. Calculate the sieve analysis for test sample on the dry weight basis, and report the results to the nearest 0.01 per cent of the material retained on each sieve. Report the percentage passing the finest sieve as the difference between 100 per cent and the sum of the percentages retained on the various sieves.

Standard Method for

DETERMINATION OF DRYING AND FIRING SHRINKAGES OF CERAMIC WHITEWARE CLAYS[1]

ASTM Designation: C 326 - 56

Adopted, 1956.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 326; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method covers the determination of linear shrinkage of ceramic whiteware clays, both unfired and fired.

Test Specimens

2. (*a*) Test specimens shall be either round bars approximately 2.5 cm in diameter by 11.5 cm in length, or bars of square cross-section approximately 2.5 by 2.5 by 11.5 cm in dimensions.

(*b*) Test specimens may be prepared either by casting or plastic forming, as circumstances require. At least five specimens shall be prepared. For cast specimens the molds may be either one-piece, or two- or more-part molds; in either instance sufficient space shall be provided to allow solid casting without [illegible]g. Where plastic-forming is employed, the clay-water mass shall be brought to a consistency that permits the making of specimens rigid enough to allow careful handling without distortion immediately after the test piece is made. Plastic-formed test specimens shall be made either by extrusion or by pressing in a suitable metal mold. Where a vacuum pug-mill is used, a vacuum of not less than 25 in. of mercury shall be maintained during the forming operation. Where no vacuum attachment is employed for extrusion, or a metal mold is used, the plastic clay shall be thoroughly hand-wedged to eliminate entrapped air as a preliminary to forming test pieces.

(*c*) The test specimens, cast or plastic-formed, shall be suitably identified and marked with shrinkage reference lines 10.00 cm apart on the long axis of the specimen.

(*d*) The marked specimens shall then be placed on a lightly oiled pallet and allowed to dry at 20 to 40 C for 24 hr. During this preliminary drying period, bars of square cross-section shall be turned 90 deg every 2 hr to eliminate possible warping. After the initial drying period, the specimens shall be placed in a drying oven at 100 to 110 C and further dried for 24 hr.

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the ASTM Committee C-21 on Ceramic Whitewares.

[2] Prior to adoption as standard, this method was published as tentative from [illegible] to 1956.

Shrinkage Measurement

3. Measure the distance between shrinkage reference marks on dried or fired specimens to the closest 0.01 cm with vernier calipers. Record the average of at least five measurements (one measurement on each of five specimens).

Calculations

4. (*a*) Calculate the linear drying shrinkage as a percentage of dry length, as follows:

$$S_d = \frac{L_p - L_d}{L_d} \times 100$$

where:

S_d = linear drying shrinkage, in per cent,
L_p = plastic length of test specimen, and
L_d = dry length of test specimen.

(*b*) Calculate the linear firing shrinkage of clay shrinkage specimens as a percentage of fired length, as follows:

$$S_f = \frac{L_d - L_f}{L_f} \times 100$$

where:

S_f = linear firing shrinkage, in per cent,
L_d = dry length of test specimen, and
L_f = fired length of test specimen.

(*c*) When desired, volume shrinkage may be calculated from linear shrinkage, as follows:

Volume shrinkage, per cent =

$$\left[1 - \left(1 - \frac{S}{100}\right)^3\right]100,$$

where:

S = linear shrinkage, in per cent.

Standard Method of Test for

SPECIFIC GRAVITY OF FIRED CERAMIC WHITEWARE MATERIALS[1]

ASTM Designation: C 329 - 56

Adopted, 1956.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 329; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a procedure for the determination of specific gravity of fired ceramic whiteware materials under prescribed conditions.

NOTE.—This method is not applicable to materials perceptibly attacked by water.

Apparatus and Materials

2. The apparatus shall consist of the following:

(a) *Analytical Balance and Weights.*

(b) *Pycnometers,* of 50-ml capacity, consisting of suitable bottles with capillary tube stoppers.

(c) *Thermometer,* calibrated at 0.5 C intervals in the room temperature range.

(d) *Drying Oven.*

(e) *Weighing Bottle.*

(f) *Desiccator.*

(g) *Vacuum Source.*—A suitable apparatus to produce a vacuum equivalent to an absolute pressure of less than 1 in. of mercury.

(h) *Distilled Water* that has be freshly evacuated, or boiled and coo to remove dissolved air.

Preparation of Sample

3. (a) When possible, the sample test shall consist of at least two piec totaling 100 to 150 g taken from di ferent portions of the material in suc way as to exclude skin surfaces in so as possible. The sample shall be selec so as to be representative of the mater to be tested.

(b) The pieces shall be crushed, necessary, between hardened steel p fices. The specimen shall then be r duced to 25 to 30 g by quartering, a any magnetic material introduced crushing shall be removed. This sam shall be ground in a suitable m so that it will pass a No. 100 (1 micron) ASTM sieve,[3] or its equival

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[1] Under the standardization procedure of the Society, this method is under the jurisdiction of the ASTM Committee C-21 on Ceramic Whitewares.

[2] Prior to adoption as standard, this method was published as tentative from [illegible] to [illegible].

[3] Detailed requirements for this sieve given in the Specifications for Sieves for Te ing Purposes (ASTM Designation: E 1 see p. [illegible].

re shall be taken at all stages of the ing, grinding, and quartering to nize the introduction of impurities, to retain all material even though cult to grind.

cedure

4. (a) Make all determinations in licate. Determine all weights in this cedure to the nearest 0.0001 g.

) Place the ground sample in a s weighing bottle and dry to con- t weight at 105 to 110 C. Close the le with a glass stopper immediately n removal from the oven.

) Dry the pycnometer and stopper 105 to 110 C, cool to room tempera- in a desiccator, weigh on an ana- l balance, and record the weight P. Fill the pycnometer bottle with led water at room temperature, ert the stopper, and remove the water on the tip of the capillary means of filter paper. Weigh the ometer and contents and record weight as W_1. Empty and dry the ometer.

) Place about 8 to 12 g of the dried le in the dry pycnometer; weigh the pycnometer, stopper, and sample and record the weight as W. Add distilled water until the bottle is approximately one-half full, and, to remove entrapped air, first stir the sample and water thoroughly with a glass rod. Then remove the glass rod, using a small quantity of distilled water to wash back into the pycnometer any particles of sample adhering to the rod. Finally subject the sample and water to a reduced air pressure of less than 1.0 in. of mercury (absolute).

TABLE I.—ABSOLUTE DENSITY OF WATER.

[illegible]	Absolute Density, g per cu cm									
	0	1	2	3	4	5	6	7	8	9
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	....	....	....	....	....	....	....	....	....

TABLE II.—ABSOLUTE DENSITY OF DRY AIR AT 760 MM PRESSURE.

[illegible]	Absolute Density, g per cu cm									
	0	1	2	3	4	5	6	7	8	9
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	....	....	....	....	....	....	....	....	....

NOTE.—A suitable method for evacuation of gas is described in Section 4 (c) of the Standard Methods of Test for Specific Gravity of Pigments (ASTM Designation: D 153).[4]

(e) Fill the bottle, after evacuation, with distilled water at room temperature, t_2, insert the stopper, and remove the excess water on the tip of the capillary by means of filter paper. Weigh the pycnometer and contents, and record the weight as W_2.

(f) Temperatures t_1 and t_2 shall be reported to the nearest 0.5 C and shall not differ by more than 5 C.

Calculation

5. (a) Calculate the specific gravity

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[4] 1955 Book of ASTM Standards, Part 8.

with respect to water at 4 C, as follows:

Specific gravity =

$$\frac{d_1 d_2(W - p)}{0.999973[d_2(W_1 - p) - d_1(W_2 - W)]}$$

where:

d_1 = absolute density of water (from Table I) at temperature t_1 (Section 4 (b)),

d_2 = absolute density of water (from Table I) at temperature t_2 (Section 4 (d)),

p = weight of the stoppered pycnometer (Section 4(b)),

W = weight of the stoppered pycnometer and sample (Section 4 (c)),

W_1 = weight of the stoppered pycnometer filled with water (Section 4 (b)), and

W_2 = weight of the stoppered pycnometer, sample, and water (Section 4 (d)).

(b) The absolute density of the sample may be determined by following the directions in Section 4, but making certain that all weighings are made at identical temperatures and in a dry atmosphere. If this precaution is taken, the absolute density may be calculated as follows:

$$G = \frac{W - p}{(W - p) - (W_1 - W_2)}$$

Absolute density = $G(d - a) + a$

where:

G = specific gravity with respect to water at temperature t,

d = absolute density of water (from Table I) at temperature t,

a = absolute density of air (from Table II) at temperature t, and

t = temperature at which all weighings were made.

Report

6. The report shall include the following:

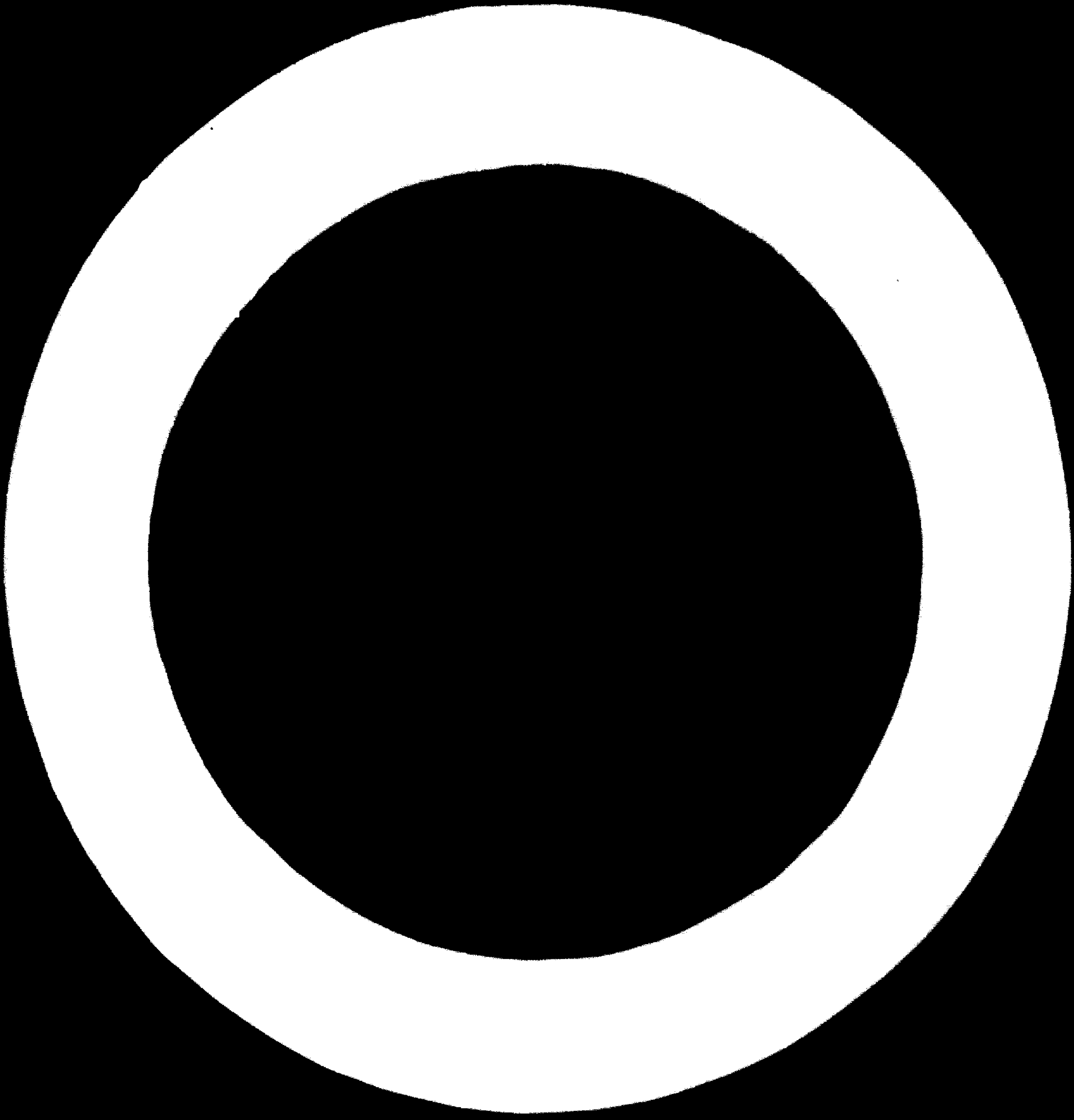
(1) Designation of the material tested.

(2) Data sheet showing all weights and water temperatures. If the absolute density is required, air temperatures shall also be shown.

(3) Specific gravity (or absolute density, if required). Duplicate determinations shall be reported to the nearest 0.001.

Reproducibility of Results

7. Duplicate determinations should not differ by more than 0.005.



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Annex 3

ANNEX 3

British Standards Specification

SPECIFICATION FOR
BRICKS AND BLOCKS OF FIRED BRICKEARTH, CLAY OR SHALE

B.S. 3921 : 1965

Price 15/- net

BRITISH STANDARDS INSTITUTION
INCORPORATED BY ROYAL CHARTER
BRITISH STANDARDS HOUSE, 2 PARK ST., LONDON, W.1
TELEGRAMS: STANDARDS LONDON W1 TELEPHONE: MAYFAIR 9000

is required in resetting the controls of the machine in order to maintain smoothly the specified rate of loading. At the final point of collapse, the indicator needle will continue to fall back rapidly even though every effort is being made to maintain the loading specified. The pattern of final collapse will vary with the type and thickness of the sample being tested. With solid bricks of 2⅝ in thickness for example, the final collapse occurs by shear and is easily recognizable. With highly vitreous vertically perforated specimens, however, final failure is characterized by a complete shattering of the sample.

WATER ABSORPTION TESTS

44. Two alternative standard methods are specified for the determination of water absorption, the 5-hour boiling test (B) and the vacuum test (V). The two forms of test give acceptable agreement with the great majority of bricks, and the choice of method is a matter of convenience. Either method may be used for the purposes of Clauses 10 and 20. A method of test by 24-hour cold immersion (C) is also specified for use as works control test only. The results by this method are always lower than, and are not proportional to, those by the standard methods.

a. **Test specimens.** The test specimens shall normally consist of whole bricks or blocks, but representative portions being approximately a half or a quarter of the brick or block may be used when testing large units. Ten whole specimens, or representative portions from each of them shall be tested.

b. **Accuracy of weighings.** Specimens shall be weighed to an accuracy of 0·1 per cent of the weight of the specimen, using a suitable balance.

c. **Preparation of specimens.** The test specimens shall be dried to constant weight in a ventilated oven at 110–115°C (230–239°F). When cool, each specimen shall be weighed*.

d. **Procedure for 5-hour boiling test. (B).** The specimens shall be placed into a tank of water immediately after weighing so that water can circulate freely on all sides of them. The tank shall be provided with a grid to ensure free circulation of water between bricks and the bottom of the tank. The water shall be heated to boiling in approximately one hour, boiled continuously for 5 hours, and then allowed to cool to room temperature by natural loss of heat for not less than 16 or more than 19 hours. The specimens shall be removed, the surface water wiped off with a damp cloth, and the specimens weighed. When wiping perforated bricks, water that might otherwise be left in the perforations shall be displaced by shaking.

Weighing of any one specimen shall be completed within 2 minutes after its removal from the water.

* It can be assumed that heating for at least 48 hours at 110°C will ensure constant weight, but it should be noted that several hours may be required before the specimens reach 110°C if they are wet when put into the oven. The 48 hours shall be reckoned from the time the specimens reach 110°C. Storage of bricks, unstacked, with spaces between them, in a ventilated room for a period of 4 hours, with a current of air from an electric fan passing over them continuously for at least 2 hours, will cool the specimens to approximately room temperature.

The test may be carried out either on dry bricks, or following the 24-hour cold immersion test if desired, provided that the specimens were in the first instance dried and weighed in accordance with Subclauses *b* and *c*.

e. **Procedure for vacuum test. (V).** The apparatus consists of a cast-iron or other suitable tank capable of holding the required number of specimens connected through stop-cocks to a vacuum pump and water tank (see Fig. 3). Ground ground surfaces on the tank and lid ensure an air-tight fit. The dry specimens, which have previously been weighed, shall be placed on end in the tank, separated from the base by a perforated zinc platform or similar method, and so arranged generally as to allow free access to all surfaces as far as possible.

With stop-cock B closed and stop-cock A open (see Fig. 3) the pump shall be started, continuing until the residual pressure is less than 2 cm of mercury. Stop-cock A is then closed, and stop-cock B opened. After the bricks have become completely immersed and the water has ceased to flow, a period of 10 minutes shall be allowed to ensure that penetration is complete.

The lid of the tank shall then be removed, and the bricks wiped and weighed in the manner prescribed for *d*.

f. **Procedure for 24-hour cold immersion test (for works control). (C).** The dry specimens, which shall be at room temperature, shall be completely immersed, without preliminary partial immersion, in water at room temperature. The water shall have free access to all surfaces as far as possible. After immersion for 24 ± 1 hours the specimens shall be removed and wiped and weighed in the manner prescribed for *d*.

g. **Calculation of water absorption.** The absorption results shall be reported in terms of percentage increase by weight on the dry specimens and shall be calculated to the nearest 0·1 per cent.

The arithmetic mean of the absorptions of the ten specimens is the best unbiased estimate of the true consignment mean and shall be taken as the absorption of the consignment.

SOLUBLE SALTS ANALYSIS

48. *a*. **Preamble.** The preparation of a powdered sample of mineral substance such as brick for chemical analysis is a well-understood technique, as is the analytical determination of the radicals present in an aqueous solution. It is the preparation of an aqueous solution from the powdered bricks as a preliminary to soluble salts analysis that calls for standardization, because widely divergent amounts of soluble salts may be taken into solution depending on the methods of extraction used.

b. **Preparation of sample.** From the bulk sample of 10 bricks or blocks a representative working sample of about 25 g ground to pass No. 100 B.S. sieve shall be prepared. The following are the alternative methods:

(i) Fragments representative of the interior and exterior of the bricks amounting to at least one-tenth of each brick or block are crushed in hardened steel equipment to produce about 5000 g of material passing a sieve with an aperture not greater than B.S. No. 5*. This is mixed and then reduced by coning and quartering or other equivalent method to about 300 g which is then all ground to pass a sieve with an aperture not greater than No. 22. This finer sample is reduced to about 25 g by coning and quartering or other equivalent method and all is ground to pass a No. 100 B.S. sieve*.

A magnet is used to remove any iron that may have contaminated the sample during crushing. The sample shall then be dried at 110°C.

(ii) Holes are drilled in 10 bricks or blocks with a masonry drill not larger than ¼ inch in diameter. The holes are approximately equally spaced over the bed-faces of each brick or the outer surface of the block. They are carried to a depth approximately equal to half the depth of the brick or half the thickness of the web of the block. The number of holes is such as to give a sample of approximately 25 g of powder passing a 100 mesh B.S. sieve. Material from the drillings which does not pass the sieve immediately is ground in a suitable mortar until the whole sample passes through.

A magnet is used to remove any iron which may have contaminated the sample during drilling. The sample shall be dried at 110°C.

c. **Determination of acid-soluble sulphate.** Weigh 2 g of the sample and transfer to a 250 ml beaker and cover with a clock glass. Through the lip of the beaker introduce 150 ml of hydrochloric acid (1 : 9) and heat to boiling, add half a Whatman ashless tablet or equivalent and boil for 10 minutes, stirring to prevent bumping. Cool, filter through a sintered glass buchner funnel and wash thoroughly five or six times with hot distilled water. Add one or two spots of methyl red indicator and ammonia (1 : 1) dropwise till just neutral then add immediately 25 drops hydrochloric acid (sp. gr. 1·18) followed by 3 ml of bromine water (saturated). Heat to boiling, boil for 2 minutes and, while boiling, slowly add from a pipette 10 ml of barium chloride solution (10 per cent). Continue boiling for about 2 minutes, transfer to a steam bath for 1 hour and allow to cool. Stand overnight and filter through a No. 42 Whatman paper (or equivalent). Wash with hot water until free of chlorides. Transfer the precipitate and paper to a weighed platinum crucible, heat gently to dry the residue and char the paper, and finally ignite to 1000°C for 30 minutes, cool and weigh.

$$\text{Weight of } BaSO_4 \times 0{\cdot}4115 = \text{weight of } SO_4$$

NOTE. The 'acid soluble sulphate' may be assumed to correspond fairly closely to the total quantity of sulphate which could be obtained from the brick sample on long continued extraction with water. The quantity is therefore relevant to an assessment of the liability of the brick material to cause sulphate expansion in Portland cement.

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* B.S. 410, 'Test sieves'.

d. **Extraction of water-soluble salts.** The extraction of soluble salts shall be carried out at room temperature, 10 ± 0·05 g of the sample shall be weighed and transferred to a 150 ml polythene bottle, 100 ml of cold distilled water shall be added, the bottle closed with a screw-on polythene top and the bottle shaken for 60 minutes. (A rotary shaker revolving at about 30 revolutions per minute is suitable or alternatively the contents of the bottle may be stirred for 60 minutes by a magnetic stirrer using a polythene covered follower). The suspended sample shall be filtered and the filtrate collected in a clean dry flask. The residue on the filter shall not be washed. Alternatively a centrifuge may be used. The filtering means employed shall be used dry. The alternatives are either:

(i) Sintered glass buchner funnel[*], porosity grade 4, with suction.

(ii) Centrifuge.

(iii) Filter candle with suction.

(iv) Ordinary filter with, e.g., a No. 42 Whatman or equivalent filter paper.

It is essential that the filtrate shall be clear.

e. **Determination of radicles.** Recognized analytical methods shall then be used to determine the following radicles:

Calcium (Ca^{++}), Magnesium (Mg^{++})
Sodium (Na^{+}), Potassium (K^{+})

The following analytical procedure has been found convenient and is recommended though it is not mandatory. The results shall be reported to the nearest 0·01 per cent by weight.

Calcium. Pipette a 10 ml aliquot of the soluble salt extract into a 500 ml conical flask. Add 20 drops of hydrochloric acid (sp. gr. 1·18), followed by 10 ml of potassium hydroxide solution (approximately 4N), and dilute to about 200 ml with water. Add about 0·015 g of calcein indicator[†]. Titrate with standard E.D.T.A. solution from a 10 ml semi-micro burette, the colour change being from fluorescent green to pink.

Magnesium. Pipette a 10 ml aliquot of the soluble salt extract into a 500 ml conical flask. Add 20 drops of hydrochloric acid (sp. gr. 1·18), followed by a 10 ml of ammonia solution (sp. gr. 0·880) and dilute with water to about 200 ml. Add about 0·04 g of methyl thymol blue complexone indicator. Titrate with the standard E.D.T.A. solution from a 10 ml semi-micro burette, the colour change being from blue to colourless.

The volume of E.D.T.A. used for the titration of calcium is subtracted from the volume of E.D.T.A. used for this titration. The remainder represents the volume of E.D.T.A. required for the tiration of the magnesium.

Sodium and potassium. Compare a portion of the soluble salt extract with standard solutions containing 5 p.p.m. of sodium, and 10 p.p.m. of potassium

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[*] B.S. 1752, 'Laboratory sintered or fritted filters'.

[†] Screened Murexide or 2-hydroxyl-1-(2-hydroxy-4-sulpho-1-naphthylazo)-3-naphthoic acid are also suitable indicators.

in a suitable flame photometer. Calculate the sodium and potassium contents by reference to previously prepared calibration graphs.

Standard solutions. Calcium solution (1·0 mg CaO/ml): Dissolve 1·78 g of dried (150°C) calcium carbonate in a slight excess of diluted hydrochloric acid (1/4), boil to expel carbon dioxide, cool and dilute to one litre in a calibrated flask.

Magnesium solution (1·0 mg MgO/ml): Dissolve 0·6032 g of magnesium metal in a slight excess of diluted hydrochloric acid and dilute to one litre in a calibrated flask. Before weighing, etch the metal ribbon or foil in dilute hydrochloric acid and then dry it with alcohol followed by ether. Adjust the weight with scissors.

Standard E.D.T.A. solution (0·5 per cent): Dissolve 5 g of diaminoethane tetra-acetic acid (di-sodium salt dihydrate) in warm water, filter if necessary, cool, and dilute to 1 litre. Store in a polythene bottle. Standardize against the standard calcium and magnesium solutions, calcein and methyl thymol blue complexone, respectively, being used as indicators.

Indicators. Calcein indicator: Mix by grinding together 0·1 g of calcein with 10 g of potassium chloride.

Methyl thymol blue complexone indicator: Mix by grinding together 0·2 g of methyl thymol blue complexone with 20 g of potassium nitrate.

EFFLORESCENCE TEST

46. Ten specimens shall be used for the efflorescence test. Those which have been used for the soluble salts analysis (Clause 37) may be found a convenient sample but where any doubt exists, ten whole bricks or blocks shall be used. Evaporation from faces other than that which will appear as the exposed face in the work shall be prevented by surrounding them with an impermeable sheet, for example, of polythene. Each specimen shall be placed with its exposed face uppermost and allowed to stand in a warm well-ventilated room. A suitable flask containing distilled water shall be inverted and its mouth placed in contact with the exposed face of the specimen (see Fig. 4). A quantity of distilled water capable of saturating the specimen shall be used (see Note). If the distilled water is completely absorbed within 24 hours a further quantity of distilled water shall be used. After a few days, when the water has been absorbed and the specimen appears to be dry, a similar quantity of distilled water shall be used and a further drying period allowed. The specimens shall then be examined for efflorescence.

The liability to efflorescence shall be described as 'nil', 'slight', 'moderate', 'heavy', or 'serious', in accordance with the following definitions:

Nil. No perceptible deposit of efflorescence.

Slight. Not more than 10 per cent of the area of the face covered with a thin deposit of salts.

Moderate. A heavier deposit than 'slight' and covering up to 50 per cent of the area of the face, but unaccompanied by powdering or flaking of the surface.

Heavy. A heavy deposit of salts covering 50 per cent or more of the area of the face but unaccompanied by powdering or flaking of the surface.

Serious. A heavy deposit of salts accompanied by powdering and/or flaking of the surface and tending to increase with repeated wettings of the specimen.

NOTE. It is not possible to specify precisely the quantity of distilled water to be used since this will depend on the size and porosity of the specimen being tested. The object is to use sufficient distilled water to saturate the specimen, dissolve soluble salts, and allow the salts to crystallize at the exposed face.

As a guide, for the 9 × 4½ × 3 format, the quantity of distilled water is about 300 ml. Obviously for larger sizes, the quantity of distilled water will require to be increased in proportion to the volume of solid material.

COMPLIANCE

47. Bricks and blocks shall be deemed to comply with the requirements of this standard when samples taken in accordance with Clauses 37 and 38 and tested in accordance with the appropriate clause for strength, dimensions, soluble salts, efflorescence, or water absorption, satisfy the appropriate requirements of Part 1 and Part 2 of the standard.

PROCEDURE IN THE EVENT OF DISPUTE

48. It may not always be convenient, or even necessary, for both manufacturer and user to be present at the time of sampling and testing. If in such a case a test result is obtained which does not conform to the requirements of this standard such a result may then and only then, lead to a dispute. In this event sampling and testing shall be repeated, in the presence of, and to the satisfaction of representatives of both user and manufacturer, to conform fully with the requirements of this standard. The results of this second test, carried out to resolve the dispute, shall be accepted by both parties as determining whether or not the consignment complies with this standard.

COST OF TESTING

49. Where the goods have been supplied to conform with the requirements of this British Standard, the cost of carrying out the first test of any one consignment shall be borne by the purchaser. If a dispute arises, the cost of the second test, made in accordance with the requirements laid down in Clause 47 and Clause 48 shall be borne by the manufacturer, provided that, if the result of this second test meets the requirements of this standard, the charge shall then be transferred to, and borne by the purchaser.

METHODS FOR THE DETERMINATION OF PARTICLE SIZE OF POWDERS

Part 2.
Liquid sedimentation methods

B.S. 3406 : Part 2 : 1963

Price [illegible] net

BRITISH STANDARDS INSTITUTION
INCORPORATED BY ROYAL CHARTER
BRITISH STANDARDS HOUSE, 2 PARK ST., LONDON, W.1
TELEGRAMS: STANDARDS AUDLEY LONDON   TELEPHONE: MAYFAIR 9000

INTRODUCTION

Terminology. For the purpose of this standard the terms and definitions of B.S. 2955* apply with the addition of the following terms:

Laboratory sample. The portion of the gross sample which is delivered to the laboratory for determination of particle size distribution.

Analysis sample. The portion of the laboratory sample which is used in the size analysis apparatus.

Basic principles. Sedimentation methods are based on the measurement of the rate of settling of the powder particles dispersed in a fluid.

They may be classified as incremental or cumulative. In the former method the concentration of particles is measured at a plane section across the sedimentation vessel and at a known depth below the surface level of the suspension. In the latter method the mean concentration of unsettled particles is measured over the whole distance from the surface level of the suspension to a known depth below the surface, or alternatively the total sediment at a known depth is measured. The weights of particles extracted from the sedimentation column by incremental methods, such as the pipette Methods S1 and S2, determine directly the proportion by weight of the analysis sample that consists of particles having a diameter less than that corresponding to the velocity of fall at the time of sampling. The total sediment weights as determined by the cumulative methods such as the Methods S3 and S4, are not directly proportional as in the incremental methods, and the proportional weights which consist of particles having diameters less than those corresponding to the velocities of fall at the times of sampling must be determined by a difference method, which may be graphical but is preferably arithmetical as described in the preamble to Methods S3 and S4 'Principle of cumulative methods.'

The calculation of particle size is dependent on Stokes's Law, which may conveniently be stated in the following form for size analysis by liquid sedimentation.

$$d = \left(\frac{18\,\eta\,h}{(\rho - \rho_0)\,g\,t}\right)^{\frac{1}{2}} \times 10^4 \qquad (1)$$

where d is interpreted as the Stokes diameter* of the particle in microns, i.e. diameter of a sphere which has the same density and the same free falling velocity in a given fluid as the particle within the range of Stokes's law.

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* B.S. 2955, 'Glossary of terms relating to powders'.

η = absolute viscosity of medium (poises).
ρ = density of medium (g/cm^3).
σ = apparent density of particle* (g/cm^3).
h = distance (cm) through which particle falls in time t (s).
g = acceleration due to gravity, (cm/s^2).

Equation (1) is valid only in the region of viscous flow, which sets an upper limit to the size of particle which can be tested by this means in a given liquid. The limit is determined by the magnitude of the Reynolds number, a dimensionless quantity defined by

$$\frac{v d \rho}{\eta} \times 10^{-4}$$

where $v = h/t$, the free falling velocity.†

The Reynolds number should not exceed 0·2 if the error in using Stokes's law is not to exceed 5 per cent.

For practical purposes the lower size limit is determined by the temperature control over the settling period. As a guide, the methods are applicable to particles having free-falling velocities in water corresponding to those for particles of Stokes diameter 3 microns, and of density 2 g/cm^3. The lower size limit is reduced for particles of greater density.

Suspension and dispersion. The liquid suspending medium must possess suitable characteristics. These are described in Appendix A, which also gives a list of liquids and dispersing agents, and suitable tests.

For the purpose of size-analysis, it is important that the dispersion of the powder in the medium should be complete, unless incomplete dispersion is compatible with the conditions of usage, and that no flocculation should occur during a test. Vigorous stirring of the mixture, together with the addition of a solution of small quantities of a suitable suspending agent, are generally employed to bring about this condition before starting a series of measurements. The choice of a suitable medium and suspending agent can be made empirically. A rapid assessment of the quality of the resulting dispersion is useful in deciding this choice which should, however, always be confirmed by the more reliable methods described in Appendix A before being adopted. It is important to note that the terms 'wetting agent' and 'suspending agent' are not necessarily synonymous. Many inorganic salts can act as efficient suspending agents, but have little effect on interfacial tension. It is also necessary to check that the wetting agent does not increase the solubility of the sample in the suspending fluid.

In the majority of cases the most satisfactory medium is distilled water with the addition of a suitable suspending agent, but organic media are often

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* Equal to the true density for non-porous particles only.
† See B.S. 2955 for definitions of these terms.

employed for materials, such as metal powders, which are not easily wetted by aqueous solutions, or for substances which dissolve in water or react with it. In aqueous media, the effect of pH should not be overlooked, since it can be a deciding factor in determining the degree and stability of dispersion.

Preparation for a sedimentation analysis. The analysis sample is prepared as follows:

Dry the sample.

Sieve on a 75 micron B.S. test sieve* by the dry or wet method†. Record the percentage weight remaining on the sieve.

Subdivide the portion which has passed through the sieve as necessary to provide the analysis sample, using the methods of Part 1‡.

Select a suspending liquid. Advice on this matter is given in Appendix A.

If not already known, determine the viscosity of the suspending liquid at the temperature of the bath by any of the methods described in B.S. 188§ appropriate to the particular suspending liquid. The viscosity of the liquid shall be such that the Reynolds number characterizing the flow round the largest particles of the powder is not greater than 0·2.

If not already known, determine the density of the suspending liquid at the temperature of the bath by the method described in B.S. 733|| or other method giving similar accuracy. Determine the density of the particles by the method described in B.S. 1377 or B.S. 3483¶. If the difference between the density of the particles and that of the suspending liquid is less than 0·5 g/cm³ special care must be taken to avoid convection currents.

Add the dispersing solutions or liquid slowly to the analysis sample** working the paste to a liquid suspension with a suitable implement taking care to avoid grinding of the sample. Then make up the volume to about 150 ml with the suspending liquid, subsequently diluting to the requirements of the method. Stir the suspension for at least 15 minutes by a mechanical stirrer, then de-aerate under reduced pressure using an ordinary water pump.

Apparatus, sample and suspending medium shall be at the same temperature, which shall not vary during the determination by more than ½degC. Closer control will improve the accuracy for small particle sizes.

The apparatus should be located in a draught-free position in the laboratory and all liquids used as suspension media stored in the immediate vicinity so that the particular ambient temperature is attained.

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* B.S. 410, 'Test sieves'.

† B.S. 1796, 'Methods for the use of British Standard fine mesh test sieves'.

‡ B.S. 3406 'The determination of particle size of powders'. Part 1, 'Subdivision of gross sample down to 0.2 ml'.

§ B.S. 188, 'Method for the determination of the viscosity of liquids in c.g.s. units'.

|| B.S. 733, 'Density bottles'.

¶ B.S. 1377, 'Methods of testing soils for civil engineering purposes', B.S. 3483, 'Methods for testing pigments for paints'.

** If the suspending liquid does not readily wet the powder a drop or two of a wetting agent should be added to the dry test portion.

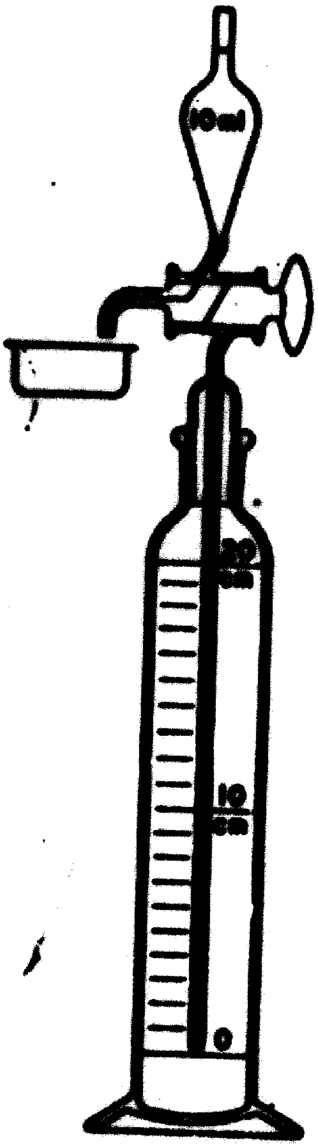

Fig. 22. Fixed position pipette method.

METHOD S2. FIXED POSITION PIPETTE INCREMENTAL METHOD[*]

S2.1 Apparatus

Sedimentation vessel. A glass sedimentation vessel about 5·6 cm internal diameter having a graduated scale 0 to 20 cm marked on the side of the vessel. The zero graduation should not be less than 2·5 cm from the inside base of the vessel, and the capacity of the vessel when filled to the 20 cm mark should be about 550 to 620 ml.

Pipette. A pipette fitted with a two way tap and side discharge tube. The capacity of the pipette to the graduation mark is normally 10 ml. A bell-shaped dome is fused to the pipette with a ground-glass joint to fit the neck of the sedimentation vessel. A small vent hole is made in this dome. The inlet to the pipette stem must be level with the zero mark on the sedimentation vessel. The stem from the pipette bulb to the sampling inlet is constructed of capillary glass tube with a bore not less than 1 mm nor more than 1·3 mm. The tube above the bulb should be 4 mm to 4·5 mm bore.

Ancillary apparatus. As Method S1.

S2.2 Preparation

Calibration of pipette. Clean and dry the pipette. Part fill the sedimentation vessel with distilled water. Set the tap in the sampling position and by means of a rubber tube suck water into the bulb to the level of the graduation mark. Reverse the tap to the discharge position and allow the water to drain into a tared weighing bottle. Apply pressure through the rubber tube to blow any water remaining in the bulb and the discharge tube into the weighing bottle. Weigh the bottle to the nearest 0·001 g and calculate from this weight the internal volume V_p of the pipette.

Temperature. Maintain the requirements stated in the introduction.

Analysis sample. Make up the analysis sample as described in the introduction to give a concentration of about 1 per cent by volume.

Withdrawal time calculation. Calculate the withdrawal time for the first fraction, using Equation (2) (given in S1.3 above).

S2.3 Sedimentation

Procedure. Proceed as Method S1 but when mixing place the finger end over the vent hole in the dome when inverting the sedimentation vessel.

Withdraw and discharge fractions at time intervals as explained in Method S1.

The time to fill the pipette should be about 20 seconds. The fraction is discharged into a tared weighing bottle as described in Method S1. When the

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[*] See A. H. M. Andreasen, 'Zur Kenntnis des Mahlgutes'. *Kolloidchemische Beihefte*, 1928, 27, 349.

pipette bulb has drained, remove the rubber suction tube and run 5 to 7 ml of pure suspending liquid from a normal 10 ml pipette into the bulb of the sedimentation pipette to wash into the weighing bottle any particles adhering to the surface. The stem of the pipette remains filled with suspension.

The depth of immersion of the pipette decreases as each fraction is withdrawn. Determine the exact decrease in depth by experiment, and allow for it in the subsequent calculations. Suppose that the decrease is 0·4 cm for each fraction withdrawn, and that initially the depth of immersion of the pipette was 20 cm, then the depth after the first fraction has been withdrawn will be 19·6 cm, and the mean depth used for calculation of the initial diameter will be 19·8 cm. The mean depth for the second fraction will be 19·4 cm, and so on.

In the original design by Andreasen the volume of the pipette stem to the 20 cm mark is 5 ml. Hence, if the sedimentation vessel is filled to the 20 cm level *without* the stem immersed, then the liquid level will rise to 20·2 cm when the stem is inserted. By this method of operation the mean depth of immersion for the first sample will be 20 cm, for the second sample 19·6 cm, and so on.

Assay of fractions
Calculation of results } As Method S1.
Repeat tests

Report. As Method S1, but report the method used as S2.

Assay of fractions. Each fraction shall be accurately assayed; if by drying a weighing the following procedure shall be adopted:

Evaporate each fraction in its weighing bottle to dryness in an oven maintained at a temperature suitable to the particular suspending liquid and cool in a desiccator. Weigh the weighing bottle and contents to the nearest 0·1 mg and determine the weight W_n of solid material in each of the fractions ($n = 1, 2, \ldots$). Allowance must be made for the weight of dispersing agent retained after evaporation.

The removal of the suspending liquid may be expedited by first centrifuging the fractions collected in centrifuge tubes and decanting the supernatant liquid from the firmly compacted solid material. Then proceed as above.

Fractions may be assayed by other appropriate methods, e.g. chemical or colorimetric.

S1.5 Calculation of results

Calculation of limiting Stokes diameters. If the times of sampling have been other than those specified in S1.4 calculate the limiting Stokes diameters d_n corresponding to each of the time intervals t_n ($n = 1, 2, 3, \ldots$).

$$d_n = \left(\frac{18\eta h}{(\sigma - \rho)\, g t_n}\right)^{½} \times 10^4 \text{ microns} \qquad (3)$$

Calculation of cumulative percentage undersize. Calculate the cumulative percentages P_n by weight of particles smaller than each of the limiting Stokes diameters d_n for each time interval t_n from the weights W_n of the fractions:

$$P_n = \frac{W_n}{W_s} \cdot \frac{V}{V_p} \times 100.$$

where W_n = weight of fraction (g).
W_s = weight of test portion (g).
V = volume of sedimentation vessel to the mark (ml).
V_p = volume of pipette (ml).

S1.3 Preparation

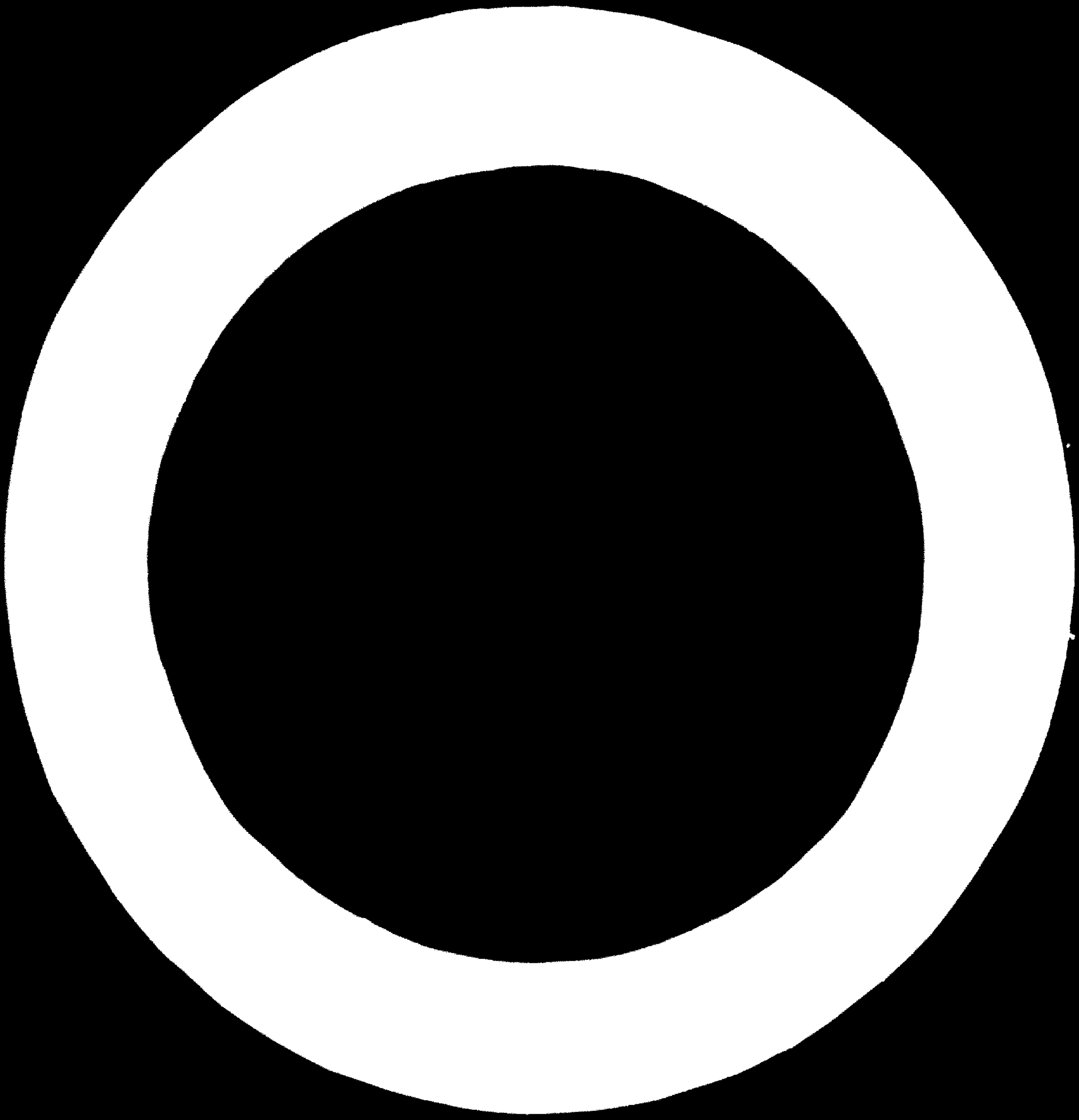
Repeat the procedure on a further representative analysis an from the same laboratory sample. The results of the test shall be if the proportions by weight less than the same limiting Stoke not differ by more than 4 per cent.

S1.7 Report

Plot the results of the analysis with the micron sizes as abs percentages undersize as ordinates. Select from the smooth curve drawn the points cumulative percentages corresponding to the series requ to the nearest 1 per cent.

The report shall indicate that Method S2 has been used and state:

the suspending liquid;
the dispersing agent;
the volume concentration used;
the density of the particles.



Annex 4

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Standard Method of Test for

FREE MOISTURE IN CERAMIC WHITEWARE CLAYS[1]

ASTM Designation: C 324 - 56

Adopted, 1956.[2]

This Standard of the American Society for Testing Materials is issued under the fixed designation C 324; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. This method of test covers a pro-
lure for determination of free moisture
ceramic whiteware clays.

ample

2. The sample shall be obtained in ac-
lance with the Method of Sampling
ramic Whiteware Clays (ASTM Des-
ation: C 322).[3] The sample submitted
testing shall weigh not less than 1000
and shall be kept in an airtight con-
er to prevent loss of water prior
testing.

Procedure

3. Remove the sample of clay from container. Weigh about 500 g of the sample to the nearest 0.1 g. Spread out the weighed portion of the sample in a weighed shallow metal or porcelain container, and dry at 100 to 110 C for 24 hr in a drying oven. Reweigh the dried clay, as quickly as possible, to the nearest 0.1 g.

Calculation

4. Calculate the percentage of free moisture, on the dry-weight basis and to the nearest 0.1 per cent, as follows:

$$\text{Free moisture, per cent} = \frac{A - B}{B} \times 100$$

where:

A = "as-received" weight of the portion of the sample used, and
B = weight of sample after drying.

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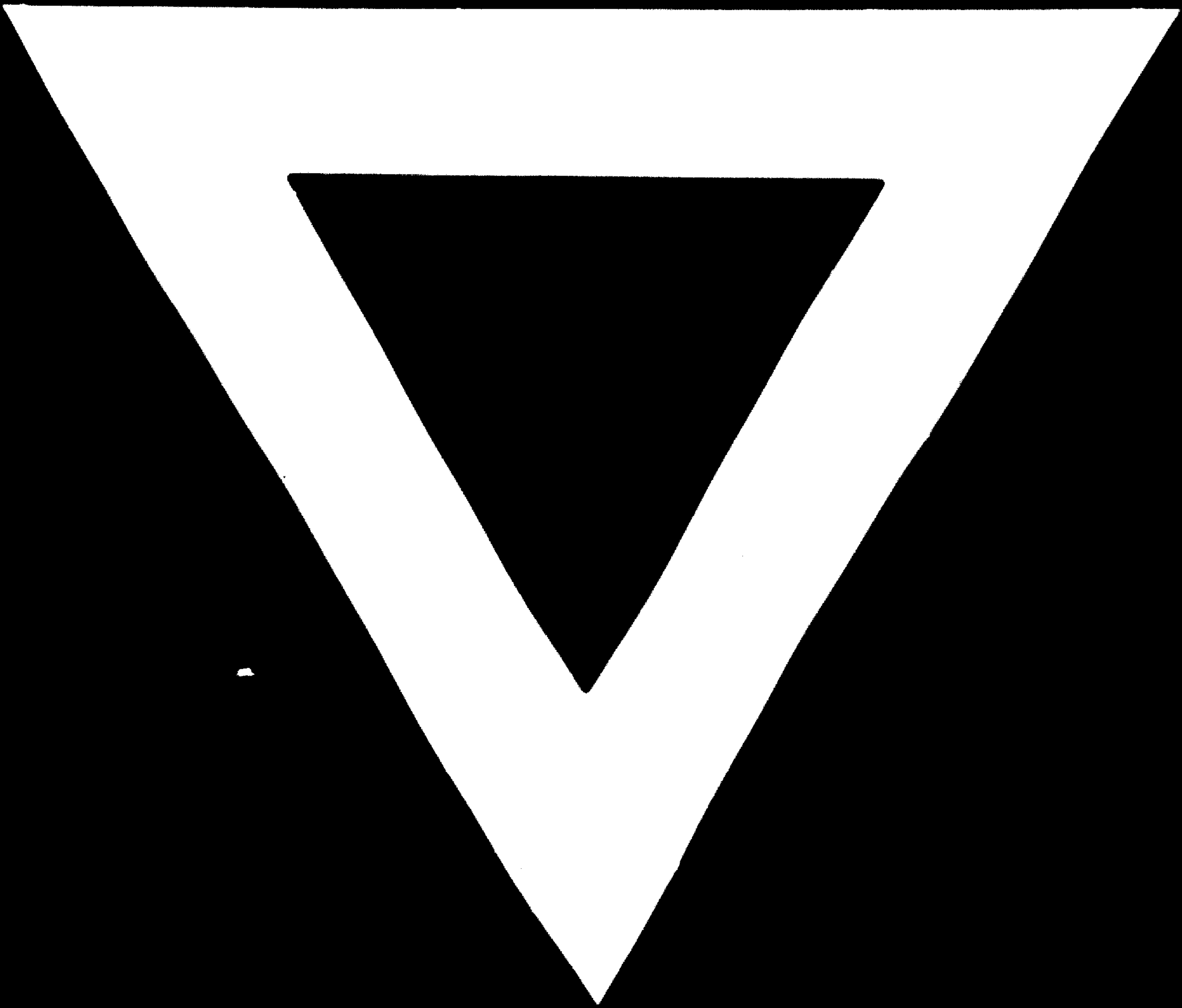
[1] Under the standardization procedure of the Society, this method is under the jurisdiction of ASTM Committee C-21 on Ceramic Whitewares.

[2] Prior to adoption as standard, this method was published as tentative from 1952 to 1956.

[3] See p. 688.



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