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CEYLON STANDARD 160 : 1972

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SPECIFICATION FOR BALL CLAY FOR CERAMIC INDUSTRY

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BUREAU OF CEYLON STANDARDS

SPECIFICATION FOR
BALL CLAY FOR CERAMIC INDUSTRY

C. S. 160 : 1972

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CEYLON STANDARD SPECIFICATION FOR BALL CLAY FOR CERAMIC INDUSTRY

FOREWORD

This Ceylon Standard has been prepared by the Drafting Committee on Ceramics. It was approved by the Civil Engineering Divisional Committee of the Bureau of Ceylon Standards and was authorised for adoption and publication by the Council of the Bureau on 3rd October 1972.

The term ball clay is applied to a large group of sedimentary highly plastic clays, usually dark in colour but burning to a white or creamy body. Ball clay resembles the secondary kaolins in that kaolinite is the chief constituent, although the content of impurities is higher. Ball clay differs from China clay by its greater toughness and plasticity, better bonding power and lower refractoriness.

Ball clay is widely used in the ceramic industry. It is chiefly used for china and earthenware and as a bonding material for harder and less plastic clays in refractories and stoneware. In whiteware bodies it plays a dual role in increasing the plasticity of the raw mix and the content of flux.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated expressing the result of a test or observation shall be rounded off in accordance with C. S. 102: Ceylon Standard on Presentation of Numerical Values. The number of figures to be retained in the rounded off values shall be the same as that of the specified value in this standard.

In the preparation of this standard considerable assistance has been derived from the Geological Survey Department, Ceylon Ceramics Corporation, and the publications of the Indian Standards Institution.

1. SCOPE

This standard prescribes the requirements, the methods of test and sampling of ball clay used in ceramic industry.

2. REQUIREMENTS

2·1 The material shall be in the form of shredded clay, lump or powder and shall be highly plastic when tempered with water. The material shall slake in water and form a smooth suspension when dispersed.

2·2 **Particle size distribution** - The particle size distribution of the material shall be as follows when determined according to the method prescribed in Appendix A.

Sieve aperture	Percent material
Coarser than 53μm, max.	5
Finer than 2μm, min.	70

2·3 **Grit** - Not more than 5% by mass shall remain on a 53μm C. S. Sieve* when determined according to the method prescribed in Appendix B.

2·4 **Fired colour** - The colour of the material when fired to 1,350° C shall be light grey, or cream and free from any specks or patches.

2·5 **Plasticity** - The optimum water content of the clay in its workable plastic state shall not be less than 30% by mass when determined according to the method prescribed in Appendix C.

2·6 **Dry linear shrinkage** - The dry linear shrinkage of the material shall be not more than 10% when determined according to the method described in Appendix D.

2·7 **Maturity** - The material fired at 1,250° C shall not show a water absorption of more than 3% by mass when determined according to the method described in Appendix E.

2·8 **Strength** - The material shall have a dry modulus of rupture of not less than 0·211 kgf/mm^2 when determined according to the method described in Appendix F.

2·9 **Chemical analysis** - The material shall also conform to the requirements given in Table 1 when analysed according to the method shown against each characteristic.

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* C. S.124. Ceylon Standard Specification for Test Sieves.

TABLE 1

CHEMICAL ANALYSIS OF BALL CLAY FOR CERAMIC INDUSTRY

Serial No.	Characteristic	Requirement % by mass	Method of Test
(i)	Ferric oxide (as Fe_2O_3) max.	1·5	G-2
(ii)	Titanium dioxide (as TiO_2) max.	1·5	G-3
(iii)	Alumina (as Al_2O_3)	35±3	G-4
(iv)	Ferric oxide and Titanium dioxide together (Fe_2O_3 + TiO_2) max.	2·5	—
(v)	Loss on ignition, max.	15	G-5

3. SAMPLING

3·1 General Requirements of Sampling

3·1·1 In drawing, preparing, storing and handling test samples the following precautions and directions shall be observed.

3·1·2 Samples shall not be taken in an exposed place.

3·1·3 Sampling instrument shall be clean and dry when used.

3·1·4 Precautions shall be taken to protect the samples, the material being sampled the sampling instrument and the containers for samples from adventitious contamination.

3·1·5 The samples shall be placed in clean dry air-tight glass or other suitable containers.

3·1·6 The sample containers shall be of such a size that they are almost completely filled by the sample.

3·1·7 Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sample, the date of sampling and other identification particulars.

3·1·8 The sample shall be stored in such a manner that during storage the properties of the material do not get affected.

3·1·9 The sampling shall be done by a person or persons mutually agreed to between the purchaser and the supplier.

3·2 Methods of Sampling

3·2·1 **Lot** – In a single consignment all the material coming from the same supplier or source shall constitute a lot.

3·2·2 **Sub-lot** – For the purpose of sampling all the wagons in a lot shall be divided into a suitable number of homogenous sub-lots of approximately equal mass in accordance with Table 2.

3·2·3 A minimum of 25 percent of wagons shall be selected at random from the sub-lot. The corresponding number of increments to be taken from a sub-lot shall be distributed over the selected wagons with a view to determining the number of increments that should be taken from each of the selected wagons in the sub-lot.

3·2·4 All the increments taken from the selected wagons in a sub-lot shall be pooled together to constitute a gross sample.

TABLE 2

SCALE OF SAMPLING FOR WAGONS

Weight in the bulk	No. of sub-lots	No. of increments from each sub-lot
(1)	(2)	(3)
Up to 5 tonnes	1	10
6 to 10 tonnes	2	20
11 to 50 tonnes	3	30
51 to 100 tonnes	4	40
101 and over	5	50

3·3 Preparation of samples

3·3·1 All the increments from the same sub-lot shall be thoroughly mixed to constitute a gross sample representing the sub-lot. The minimum size of the gross sample shall be about 3kg. If the gross sample is less than this, additional number of increments shall be drawn so as to make up the required quantity.

3·3·2 The gross sample shall be suitably reduced by the method of coning and quartering or any other suitable method to about 750 g which shall be divided into three portions called laboratory samples, one for the purchaser, another for the supplier and the third as the referee sample, each duly placed in the sample container.

4. CRITERIA FOR CONFORMITY

4·1 One laboratory sample from each sub-lot shall be tested individually for all the requirements of this specification and the sub-lot shall be considered satisfactory if the laboratory sample satisfies all the requirements of this specification.

4·2 The lot shall be declared to conform to the requirements of this specification if all the sub-lots (see 4·1) are found satisfactory.

APPENDIX A

DETERMINATION OF PARTICLE SIZE DISTRIBUTION

A-0 Principle

A-0·1 The progress of sedimentation of a clay suspension in a tall cylinder is followed by withdrawing sample portions at a known depth based on the temperature of the suspension below the surface, with a pipette and weighing the sediment. Stokes' law is then applied to find out the grain size.

A-1 Apparatus

A-1·1 **The Andreasen Pipette** – The pipette (See Fig. 1) consists of a graduated cylinder (W) not less than 60mm in diameter and having a capacity of 550ml when filled to the upper mark on the scale. It is provided with a ground glass stopper through which passes the stem of a pipette which extends 200mm beneath the surface of the suspension and about 40mm from the bottom, the tip being at the level of the zero mark on the scale while the original surface of the suspension is at the 200mm mark. The one mark pipette (P) has a capacity of 10ml, and is provided with a three-way stopcock just above the ground glass joint. The stopcock regulates flow in the tube inside the cylinder, the pipette (P) and a draining tube for drawing the clay suspension in a weighed porcelain basin or silica dish.

A-1·2 **Basin** – Porcelain, or silica dish.

A-2 Reagents

A-2·1 **Sodium pyrophosphate solution** – Dissolve 24·55g of sodium pyrophosphate ($Na_4\ P_2O_7 \cdot 10H_2O$) in 500ml of water.

A-3 Procedure

A-3·1 **Preparation of sample** – Crush the clay sample to pass through a 4·75mm C. S. Sieve. Determine the moisture by drying a portion of the sample at 110° C.

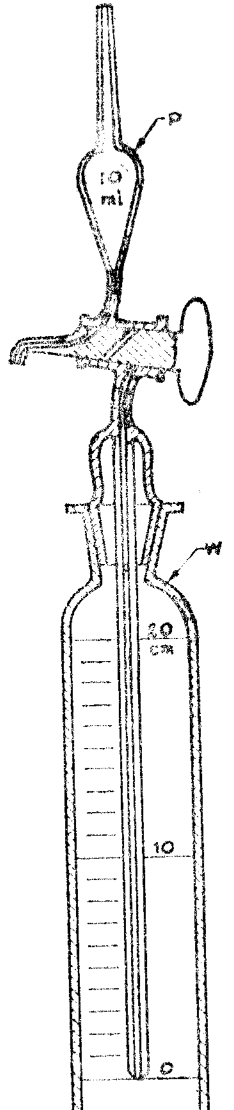

FIG. 1 – Andreasen Pipette

A-3·2 Place 5·50g of clay sample (calculated, dry basis) in a 250ml wide-mouth bottle and add 200ml of distilled water. Allow to stand for 24 hours. Add 10ml of the sodium pyrophosphate solution. Agitate the bottle with its contents by rotating end-over-end for 17 hours.

NOTE:- This is best done by packing the bottle in the jar of a ball mill so as to turn end-over-end as the mill rotates.

A-3·3 Transfer the dispersed sample to the Andreasen pipette and add distilled water to bring the level of the suspension to the upper mark on the scale, making the total volume 550ml.

NOTE:- This produces a 1 percent suspension, and the concentration of the electrolyte is 0·002Mol/l (gram molecule per litre).

A-3·4 Place the Andreasen pipette in a constant temperature bath at a temperature of 30° C, for 15 to 30 minutes to permit it to attain the temperature of the bath. Remove it, shake by hand and replace immediately in the bath. Withdraw 10ml of suspension at increasing intervals of time, at a uniform rate of about 20 seconds. The samples are drawn at the interval of 3 minutes and 30 minutes, and then at 1, 2, 3, 5, 8, 24 and 96 hours.

NOTE:- These intervals are chosen arbitrarily, but are adhered to in order to save time in calculating the grain size.

A-3·5 Take 10ml aliquots in small, weighed porcelain basins, and evaporate to dryness at 110° C. Weigh the residue accurately to 0·1mg.

A-4 Calculations

A-4·1 Calculate the percentage of the dried residue as follows:-
Mass of dried residue,

$$M_1 \text{ percent} = \frac{M_1}{M_2} \times 100$$

where,

M_1 = mass of the dried residue, and
M_2 = mass of the clay sample.

NOTE:- Make a correction for the sodium pyrophosphate percent in the mass of the dried residue, M_1.

A-4·2 **Calculation of size distribution** – The grain sizes are calculated from Stokes' law which may be expressed as follows:-

$$r = \sqrt{\frac{9 h \eta}{2 (D_1 - D_2) gt}}$$

where,

r = radius of spherical particle in mm.

h = distance in mm between liquid surface and pipette tip when suspension is drawn.

η = viscosity of suspending medium (g/cm. sec)

D_1 = Density of the particle.

D_2 = Density of suspending medium.

g = gravitation constant ($9 \cdot 81 m/s^2$) and,

t = time in seconds from start of the test.

A-4·2·1 When the test is conducted at 30° C, the clay having a density of 2·65 and distilled water as the suspending medium, the above formula may be expressed as follows:

$$r = 1 \cdot 923 \sqrt{\frac{h}{t}}$$

where r is expressed in µm.

A-4·2·2 Stokes' law is applicable to angular or cubical particles of the same weight as spherical particles. By calculating the grain size as the edge length, or width, of a cube of the same volume as a sphere of radius r, the width of the cube may be expressed as follows:

width of the cube, C = 1·612 r

NOTE:- This width of the cube shall be adopted for expressing the results of test.

A-4·2·3 Plot a smooth graph with (C) as abscissae and the percentage by mass (m) finer than the corresponding size as ordinate. From the curve find out the particle size distribution for the specified sizes.

A-4·2·4 Duplicate tests shall be made on each sample. If the mass percentages vary more than one percent, the test shall be repeated.

APPENDIX B

DETERMINATION OF GRIT CONTENT

B-0 Outline of the method

B-0·1 The clay ground to pass completely 250μm C. S. Sieve is wet sieved on a 53μm C. S. Sieve, and grit content is calculated from the amount of residue remaining on the sieve.

B-1 Apparatus

B-1·1 **Balances** – A physical balance sensitive to 0·1g and an analytical balance sensitive to 0·001g.

B-1·2 **Mechanical stirrer** – The stirrer shall essentially be a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 1 000 r. p. m., without load. The shaft shall be fitted with a replaceable stirring paddle, made of non-rusting metal, plastic or hard rubber. A special dispersion cup fitted with wire baffles shall be provided to hold the sample.

B-1·3 **Sieve** – 250μm and 53μm C. S. Sieves, conforming to C. S. 124.*

B-2 Preparation of sample

B-2·1 Brush the clay sample to pass completely 250μm C. S. Sieve and place in an air oven. Dry at 105° to 110° C for not less than 5 hours and then cool in a desiccator.

B-3 Procedure

B-3·1 Weigh about 250g of the sample prepared as in B-2 to nearest 0·1g and transfer to a glass container of 2 litre capacity. Wet the clay with 1 litre of distilled water and allow it to slake for 2 hours. If a free flowing slurry is not produced by this treatment, add another 500ml of water. To ensure complete separation or dispersion of clay, agitate the slurry by means of a mechanical stirrer rotating at a speed of 1 000 r. p. m. for 30 minutes.

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* C. S. 124. Ceylon Standard Specification for Test Sieves.

B-3·2 Transfer the slaked and stirred clay sample, without loss to a 53µm C. S. Sieve, and wash by means of a small jet of water from a wash bottle. Continue washing until the water passing through the sieve shows only traces of sample. Should any lumpy material remain on the screen, return the residue to the container with the stirrer by careful washing with a gentle jet of water, and agitate in about 1 litre of water for 30 minutes, then wash the slurry as already described. Then again sieve it.

B-3·3 Nest the sieve on a pan containing about 3ml of water. Wash the residue by holding the pan and sieve firmly in hands, and by sidewise movements, causing the water to raise up through the sieve and into the residue. The movement coupled with interspersed circular motions allows thorough washing. Carefully blot the sieve on its underside with a soft damp sponge when no more material pass through and place the sieve in an air oven. Dry at 110° ± 5°C for two hours.

B-3·4 Carefully brush the residue on the sieve to a glazed, weighing paper, and weigh to the nearest 0·001g on an analytical balance.

B-3·5 Perform a duplicate analysis.

B-4 Calculation

B-4·1 Calculate the grit content of the clay on the dry mass basis as follows:-

$$\text{Grit content, percent} = \frac{M_1}{M_2} \times 100$$

where,

M_1 = mass of the residue on 53µm C. S. Sieve.,

and M_2 = mass of the dry clay sample.

NOTE:- Round off the calculated value to the second place of decimal.

B-4·2 Report the average of three specimens.

APPENDIX C

DETERMINATION OF WATER OF PLASTICITY

C-0 Outline of the method

C-0·1 The water content of the clay at the point of maximum workability is determined. This when expressed as a percentage on oven dried clay gives the value for water of plasticity.

C-1 Apparatus

C-1·1 Spatula.

C-1·2 Porcelain basin.

C-1·3 Burette–50ml capacity.

C-1·4 Balance – Accurate up to 0·01g

C-1·5 250µm C. S. Sieve.

C-2 Procedure

C-2·1 Dry thoroughly about 500g of clay at a temperature of 70° C and crush to pass a 250µm C. S. Sieve. Work up the sieved clay with water, by adding small quantities of water from the burette, to a soft plastic consistency and thoroughly wedge and knead by hand. Allow it to age for 24 hours keeping it covered by wet cloth during the period. After 24 hours knead the plastic mass again and knead it well by further small additions of water until proper working consistency for either extrusion or moulding into bars, is obtained. Take three equal portions, the edges and corners rounded to avoid any handling loss, and weigh immediately to the nearest 0·01g.

C-2·2 Dry the three clay masses in the room temperature for 24 hours, then dry in an air oven at 70° C for 24 hours and finally dry at 110° ± 5°C for 12 hours. Cool them in a desiccator and weigh to the nearest 0·1g.

C-3 Calculation

$$\text{Water of plasticity, percent} = \frac{M_1 - M_2}{M_2} \times 100$$

Where,

M_1 = mass in g of plastic clay mass, and

M_2 = mass in g of the dried clay mass.

NOTE:– Round off the calculated value to the first place of decimal.

C-3·1 Report the average of three specimens.

APPENDIX D

DETERMINATION OF DRY LINEAR SHRINKAGE

D-0 Outline of the method

D-0·1 The dry linear shrinkage of a clay is determined by measuring the decrease in one dimension of a clay mass, when the water content is reduced from the plastic state to the dry state at 110° C.

D-1 Apparatus

D-1·1 **Mould** – A collapsible wooden or any other suitable mould with internal dimension of 25 x 25 x 120mm, suitable for forming the test specimen of that size.

D-1·2 **Micrometer** – Graduated to at least 0·02mm, to an accuracy of ± 0·005mm.

D-2 Procedure

D-2·1 Take a portion of clay as prepared in C-2·1 and again knead thoroughly. Coat evenly the inside of the mould with a thin layer of kerosene oil or light machine oil. Press a lump of the plastic clay mass into the mould. Take care to avoid entrapping any air. Strip off any excess clay with a straight edged spatula from the top of the mould and finally wipe off any adhering clay. Make the surface of clay smooth and mark two reference points approximately 100mm apart. Measure the distance between the two points accurately to second place of decimal with the micrometer. Prepare 5 such test specimens.

D-2·2 Carefully release the test specimens from the mould and allow to dry slowly under a wet cloth for 48 hours. Air dry the specimen for another 48 hours at room temperature, occasionally turning sides of the test specimens to expose new surfaces for evaporation. Place the test specimens in an air oven, with automatic temperature controls, and dry them at 110° ± 5° C for 24 hours. Remove the test specimens from the oven and keep in a desiccator to cool. Measure accurately to second place of decimal the distance between the two reference points.

D-3 Calculation

D-3·1 Calculate the linear dry shrinkage as follows:—

$$\text{Linear dry shrinkage, percent} = \frac{L_p - L_d}{L_d} \times 100$$

Where,

L_p = length of the plastic clay mass in mm, and

L_d = length of the dry clay mass in mm.

NOTE:- Round off the calculated value to the second place of decimal.

D-3·2 Report the average of the five test specimens.

APPENDIX E

DETERMINATION OF MATURITY

E-0 Outline of the method

E-0·1 Buttons of plastic clay prepared, are fired to a temperature of 1,350° C and the water absorption is determined by soaking the fired buttons in water and measuring the amount of water absorbed.

E-1 Apparatus

E-1·1 **Balance** – Accurate to 0·01g.

E-1·2 **Oven** – Capable of maintaining a temperature of 105° to 110° C.

E-1·3 **Furnace** – Muffle furnace, capable of maintaining a temperature of 1,350° C.

E-1·4 **Standard Pyrometric Cone** – Any standard pyrometric cone equivalent to 1,350° C like seger cone 12 shall be used.

E-2 Preparation of Test Sample

E-2·1 Mix sufficient quantity of clay with water carefully to form a soft mass and allow to stand, covered with a wet cloth, for 24 hours. Prepare about 10mm thick discs having a diameter of 25mm by hand pressing the soft clay mass in steel moulds. Cover the discs with wet cloth and allow to dry slowly at room temperature to avoid warpage or cracking. Place the air dried discs in an air-oven and gradually raise the temperature in about 6 hours at 105° to 110° C and maintain overnight at that temperature. Take the samples out and cool in a desiccator. The individual dry test pieces shall weigh about 50g.

E-2·2 Place the test specimens prepared as in F-2·1 in an electric furnace with the standard pyrometric cone. Raise the temperature by about 75°C per hour. After the pyrometric cone bends indicating attainment of the specified temperature, hold it for 30 minutes and then allow the furnace to cool to about 100° to 150° C, take the discs out and cool in a desiccator.

E-3 Procedure

E-3·1 Weigh individually 5 test pieces. Immerse them, in a beaker, in distilled water and boil for 5 hours, taking care that the specimens are covered with water at all times. Use setter pins or some similar device to separate the specimens from the bottom and sides of the pan and from each other. After 5 hours, allow the pieces to soak in water for 24 hours.

E-3·2 Blot each piece lightly with a moistened, lint free linen or cotton cloth to remove all excess water from the surface. Avoid excessive blotting as that will remove water from the pores. Weigh immediately after blotting to nearest 0·05g.

E-4 Calculation

E-4·1 Calculate the water absorption as follows:

$$\text{Water absorption, percent,} = \frac{M - M_1}{M_1} \times 100$$

Where,

M = mass of saturated test piece, and

M_1 = mass of the dry test piece.

E-4·2 Report average of the values obtained with the five test pieces.

APPENDIX F

DETERMINATION OF STRENGTH

F-0 Outline of the method

F-0·1 The modulus of rupture of dry clay bodies containing 50 percent quartz is determined by using a single beam apparatus with centre point loading.

F-1 Apparatus

F-1·1 A suitable apparatus consisting of the following essential features shall be used.

F-1·1·1 **The** load shall be applied at the centre point of the span, normal to the loaded surface of the beam, employing bearing blocks designed to ensure that forces applied to the beam will be vertical only and applied without eccentricity.

F-1·1·2 The load shall be applied at a uniform rate and in such a manner as to avoid shock.

F-1·1·3 The edges of the load applying block and of the supports shall not depart from a plane by more than 0·05mm.

F-2 Preparation of Test pieces

F-2·1 Mix sufficient quantity of clay with equal amount of quartz. The quartz shall be so ground that 30 percent shall be of a size between 150 microns and 75 microns and 70 percent shall pass 75µm C. S. Sieve. Pass the clay and quartz mixture three times through a 250µm O. S. Sieve. Then mix with sufficient quantity of water and knead with hand into a soft, plastic and homogenous mass. Allow the plastic mass to age for 48 hours. After ageing, pass the plastic mass three times through de-airing pug mills, with a vacuum of about 500mm of mercury. Extrude bars of 25mm diameter from the air dried plastic mass and cut in bars of 180mm length. Place bars on the glass plates very lightly oiled and put marks exactly 100mm apart on each test bar. Cover the test bars with a piece of wet cloth and allow to dry in air at room temperature. After five days of drying at the room temperature with occasional turning around the axis, dry them finally in an air oven slowly raising the temperature to $110^\circ \pm 2^\circ C$. Cool the test bars in a desiccator. The test bars shall be straight free from warpage.

F-2·2 Prepare at least 10 such test bars.

F-3 Procedure

F-3·1 Place a test bar on two of the bearing edges, in such a way that the bearing edges coincide with the mark made on the test bar. Apply the load uniformly at the centre point at a rate of 5kg/s approximately. Note the load which just breaks the test bar.

F-3·1·1 Repeat the test with 9 other test bars.

F-4 Calculation

F-4·1 Calculate the dry modulus of rupture as follows:

$$\text{Modulus of rupture} = \frac{80PL}{100\,\pi\,d^3} \quad (\text{Kg/mm}^2)$$

Where,

P = total load in kg,

L = span of the bar in mm and

d = diameter of the test bar in mm.

F-4·2 Report the average of the values, not differing individually by more than 5 percent.

APPENDIX G

CHEMICAL ANALYSIS OF BALL CLAY

G-0 Quality of Reagents

G-0·1 Unless otherwise specified, pure chemicals and distilled water shall be used.

NOTE:– 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

G-1 Determination of oxides of iron, titanium and aluminium

G-1·1 **Principle** – Ball clay is decomposed by sintering with fusion mixture and the fired mass is extracted with hydrochloric acid. Silica is dehydrated by baking and is filtered. From the filtrate hydroxide of iron, titanium and aluminium are precipitated by ammonia, filtered, washed and ignited. From the ignited mixed oxide, ferric oxide and titanium dioxide are estimated and alumina is found out by difference.

G-1·2 **Reagents**

G-1·2·1 **Fusion mixture** – Mix carbonates of sodium and potassium in equal proportions.

G-1·2·2 **Dilute hydrochloric acid** – 1 : 1 and 1 : 4 (by volume).

G-1·2·3 **Concentrated sulphuric acid** – sp.gr. 1·84.

G-1·2·4 **Hydrofluoric acid** – 40 percent.

G-1·2·5 **Potassium bisulphate** – solid.

G-1·2·6 **Ammonium chloride** – solid.

G-1·2·7 **Dilute nitric acid** – 1 : 1 (by volume).

G-1·2·8 **Methyl red indicator solution** – Dissolve 0·15g of methyl red in 500ml of distilled water.

G-1·2·9 **Concentrated ammonium hydroxide** – sp.gr. 0.90

G-1·2·10 **Ammoniacal ammonium nitrate solution** – Dissolve one gram of ammonium nitrate in 100ml of water and make it slightly alkaline to methyl red with dilute ammonia.

G-1·3 Procedure

G-1·3·1 Mix thoroughly about 0·5g of the accurately weighed test sample with 5g of fusion mixture in a covered platinum crucible and cover the mixture with a layer of the fusion mixture. Raise the temperature gradually until frothing ceases and then complete the fusion by maintaining the temperature at 900° to 950° C for about 30 minutes.

G-1·3·2 Cool, place the crucible and lid in a porcelain basin and add 30ml of dilute hydrochloric acid (1:1). Warm until the melt has disintegrated, wash the crucible and lid with dilute hydrochloric acid (1:4), taking care to remove all silica. Break all lumps with a glass rod and evaporate slowly to dryness over a steam bath or on an electric hot plate breaking occasionally the crust that may hinder the evaporation. Bake for an hour at 110° C, preferably on a sand bath to drive off hydrochloric acid. Add 20 to 30ml of dilute hydrochloric acid (1:1) and 50ml of hot water and heat to boiling to dissolve the soluble salts. Allow to settle for a few minutes and then filter through filter paper (Whatman No. 41 or equivalent is suitable). Wash the silica first with dilute hydrochloric acid (1:4) and then with hot water until completely free from chloride. Preserve the paper and the residue (A).

G-1·3·3 Transfer the filtrate back to the dish Again evaporate the filtrate and re-bake for dehydration of silica. Moisten the residue with a few drops of dilute hydrochloric acid solution (1:1) add hot water, filter, wash and collect the silica residue (b) as under G-1·3·2. Preserve the filtrate for the determination of mixed oxides.

G-1·3·4 Place silica residues (A) and (B) without drying in a platinum crucible and heat cautiously to dry the residue and char the papers. Burn off the carbon and ignite at 1 100° C. Cool. Moisten the contents with 1ml of water, add three to four drops of concentrated sulphuric acid and 15ml of hydrofluoric acid. Evaporate the solution to dryness on a sand bath or under an infra red lamp, heat again slowly at first, and then on the Blast Meker for 1-2 minutes. Preserve the residue (C) for determination of mixed oxides.

G-1·3·5 Fuse the residue (C) preserved under G-1·3·4 in the crucible with 1g of potassium bisulphate, cool and extract with a small amount of water acidulated with concentrated sulphuric acid. Add to the filtrate reserved under G-1·3·3. Add about 5g of ammonium chloride and a few drops of dilute nitric acid and heat almost to boiling. Add three drops of methyl red indicator and add slowly 1:1 ammonium hydroxide until the colour of the solution changes to yellow. Boil the alkaline solution for one to two minutes to remove the excess ammonia; allow to settle for about 5 minutes and filter through filter paper (Whatman No. 41 or equivalent is suitable).

G-1·3·6 Puncture the bottom of the filter paper, wash down the precipitate completely into the original beaker first with water and then with 10ml of concentrated hydrochloric acid and 50ml of hot water. Place the filter paper inside the beaker and macerate well. Place the covered beaker on the steam bath for about 15 minutes for complete solution of the hydroxides. Dilute to 200ml, precipitate and filter as described under G-1·3·5 and wash with hot ammoniacal ammonium nitrate solution.

G-1·3·7 Wash the precipitate thoroughly till it is free from chloride, place it in a weighed platinum crucible, burn filter paper slowly and ignite at 1 100° C to constant weight. This ignited residue of mixed oxides consists of ferric oxide, titania and alumina.

Percentage of mixed oxides =

$$\frac{\text{Difference in mass of platinum crucible}}{\text{mass of sample}} \times 100$$

G-2 Determination of Ferric Oxide

G-2·1 **Principle** – The ignited residue of mixed oxides is fused with an alkaline bisulphate and the fused mass extracted with sulphuric acid. After removal of silica, iron in the filtrate is determined by the colour developed with 1:10 orthophenanthroline and the optical density of the solution is measured with the help of an absorptiometer and compared with the standard.

G-2·2 Reagents

G-2·2·1 **Potassium bisulphate** – solid.

G-2·2·2 **Sodium bisulphate** – solid.

G-2·2·3 **Dilute sulphuric acid** – 1:9 (by volume).

G-2·2·4 **1:10 Orthophenanthroline** – 0·1% solution.

G-2·2·5 **Hydroxylamine hydrochloride** – 10% solution.

G-2·2·6 **Sodium citrate** – 10% solution.

G-2·2·7 **Standard iron solution** – (1ml = 0·0001g of Fe_2O_3). Dissolve 0·4911g of ferrous ammonium sulphate in water and add 10ml of dilute sulphuric acid (1:1) and 5ml of hydrogen peroxide (20%). Boil for 15 minutes to decompose the excess of hydrogen peroxide. Cool and dilute to 1,000ml in a measuring flask.

G-2·3 Procedure

G-2·3·1 Fuse the residue obtained under G-1·3·7 with potassium or sodium bisulphate in a porcelain crucible. Disssolve the melt in dilute sulphuric acid (1:9) and evaporate to fumes. Cool, dilute with water and filter off any silica. Wash the residue with hot water. Make up the volume of the filtrate to a standard (say 100ml) with dilute sulphuric acid.

G-2·3·2 To a set of three 100ml volumetric flasks, add nothing to the first (to serve as a blank). Pipette suitable aliquots of the standard and sample solution to the other two, depending on the amount of iron present in the sample solution. Add 5ml of hydroxylamine hydrochloride solution to each flask with a graduated cylinder and allow to stand for 10 minutes. Add 10ml of orthophenanthroline solution followed by 10ml of sodium citrate solution and stir well. Dilute to the mark with distilled water, mix and keep for one hour. Measure the optical density at 560μm (or appropriate filter) of the sample and the standard solutions, using reagent blank as the reference blank solution. Calculate the concentration of iron in the sample solution by comparing with the standard and hence the percentage of iron in the original solution.

G-3 Determination of Titania

G-3·1 **Outline of the method** – To the sulphuric acid solution of the ignited mixed oxides hydrogen peroxide is added and the colour due to iron is supressed by means of phosphoric acid. The titanium in the solution is determined colorimetrically with the help of an absorptiometer and compared with the standard.

G-3·2 Reagents

G-3·2·1 **Dilute sulphuric acid** – 1:20 (by volume).

G-3·2·2 **Phosphoric acid** – 85%.

G-3·2·3 **Hydrogen peroxide** – 30%.

G-3·2·4 **Standard titanium solution** – (1ml=0·0001g of TiO_2).

Weigh to the nearest milligramme 0·500g of pure calcined titania previously dried at 110° C. Fuse with 8–10g of potassium bisulphate in a clean platinum crucible, keep at a temperature as low as possible to maintain fluidity. Cool the fused mass and dissolve in about 300ml of dilute sulphuric acid (1:5), filter and transfer to a 500ml graduated flask, and make up to the mark with dilute sulphuric acid (1:20), mix thoroughly. Take 10ml of this solution in a 100ml graduated flask and dilute with hot water to the mark.

G-3·3 **Procedure** – To a set of three 100ml volumetric flasks, add nothing to the first (to serve as a blank). Pipette suitable aliquots of the sample and standard solutions to the other two flasks, depending on the titanium present in the sample solution. Add 50ml of dilute sulphuric acid to each of the flasks with a graduated cylinder and sufficient phosphoric acid to the flask containing sample solution to decolourise the iron. Add the same amount of phosphoric acid to the other two flasks. Add 5ml of hydrogen peroxide solution to all three flasks, mix well, dilute to the mark with distilled water. Mix, keep for half an hour and measure the optical density of the sample and the standard solutions at 410μm, (or appropriate filter) using reagent blank as the reference blank solution. Calculate the concentration of titanium in the sample solution by comparing with the standard and hence the amount of titania in the original solution.

G-4 Determination of Alumina

G-4·1 **Principle** – Alumina is estimated by difference.

G-4·2 **Calculation**

$$\text{Alumina (as } Al_2O_3\text{), percent by mass} = \frac{M - M_1}{M_2} \times 100$$

Where,

M = mass in g of the mixed oxides as obtained in G–1·3·7.

M_1 = mass in g of the ferric oxide and titanium dioxide together as calculated from G–2·4 and G–3·4 respectively.

M_2 = Mass in g of the sample taken.

G–5 Determination of loss on ignition

G–5·1 **Procedure** – Take about 50g of the material in a porcelain basin and heat to constant weight at 105° ± 2° C. Cool in a desiccator.

G–5·1·1 Heat 1g of the above dried material in a platinum crucible for 15 minutes over a small flame, then for 30 minutes over a good Meker burner, with the lid on for the last 10 minutes. Cool the crucible and contents in a desiccator, weigh again, heat for 10 minutes over the Meker burner, and then weigh again after cooling. The loss in mass denotes loss on ignition.

G–5·1·2 **Calculation** –

Calculate the loss on ignition as follows:—

$$\text{Loss on ignition, percent by mass} = 100 \times \frac{M_1 - M_2}{M_1}$$

Where,

M_1 = mass in g of the dry material and

M_2 = mass in g of the material after ignition.

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