भारतीय मानक
Indian Standard

IS 11464 : 2021

सिरैमिक उद्योग के लिए क्वॉर्ट्ज़ — विशीष्टि

(दूसरा पुनरीक्षण)

Quartz for Ceramic Industry — Specification

(Second Revision)

ICS 81.060.10

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Ceramicware Sectional Committee, CHD 09

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Ceramicware Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1985 and revised in 2011. The committee decided to revise this standard in the light of experience gained during these years. Firing temperature has been reduced to 1 200 °C. For packing, plastic bags or polyethylene lined gunny jute bags have been recommended.

Quartz (SiO_2) is natural occurring crystallized silicon dioxide and is widely used in ceramic and its related industries. It is the main constituent of sandstone, one of the two principal constituents of granite, pegmatite, igneous and metamorphic rocks, and is the most frequent gangue minerals in mineral veins. It is very resistant to weathering and is left as quartz sand when rocks containing quartz are disintegrated. It is insoluble in acids except hydrofluoric acid, only slightly attacked by solutions of caustic alkali.

This standard has been formulated with a view to provide guidance to the ceramicware manufacturers for procurement of quartz of proper quality as a suitable raw material. This standard may also serve as a guide to producers of such raw materials to cater to the needs of ceramic industry.

This standard contains clauses **5.2** and **5.3** which call for agreement between the purchaser and the supplier.

The composition of the technical committee responsible for formulation of this standard is given in Annex E.

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 analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

QUARTZ FOR CERAMIC INDUSTRY — SPECIFICATION

(Second revision)

1 SCOPE

1.1 This standard prescribes requirements and the methods of sampling and test for quartz used in ceramic industry.

1.2 This standard does not cover the requirements of quartz used in glass and refractory industries.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

IS No.	*Title*
264 : 2005	Nitric acid — Specification (*third revision*)
265 : 1993	Hydrochloric acid — Specification (*fourth revision*)
266 : 1993	Sulphuric acid — Specification (*third revision*)
799 : 1985	Specification for ammonia, liquor (*second revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
2781 : 2004	Glossary of terms relating to ceramicware (*second revision*)
4905 : 2015	Random sampling and randomization procedures (*first revision*)
8883 (Part 1) : 2005	Methods of sampling chemical and chemical products: Part 1 General requirements and precautions (*first revision*)
10332 : 1982	Specification for hydrofluoric acid, aqueous

3 TERMINOLOGY

For the purpose of this standard the definitions given in IS 2781 shall apply.

4 GRADES

There shall be two grades of the material and are as follows:

a) *Grade* 1 — Suitable for white opaque, transparent glaze and whiteware body with transparent glaze; and

b) *Grade* 2 — Suitable for ceramic body for which fired colour is not of much relevance and for which the body is coated with opaque glaze.

5 REQUIREMENTS

5.1 Description

Quartz shall be in the form of white or light pinkish fine grained powder lumps prepared from natural quartz rock. It shall be free from any other foreign matter.

5.2 Moisture Content

Unless otherwise as agreed to between the purchaser and the supplier, moisture content in quartz as received shall not exceed 2 percent by mass, when determined by the method prescribed in Annex A.

5.3 Grain Size

The grain size of both the grades of the material shall be as agreed to between the purchaser and the supplier.

NOTE — Normally for ceramic industry, the grain size is finer than 75 µm. However, the lumps may also be supplied.

5.4 Specific Gravity

The relative density of the material shall be between 2.5 and 2.7 when tested according to the procedure prescribed in Annex B.

5.5 Fired Colour

The colour of the material may be white and free from any coloured specks when fired at 1 200 °C in an oxidizing condition.

5.6 Chemical Composition

Quartz shall also comply with the requirements prescribed in Table 1 when tested in accordance with the methods prescribed in Annex C.

Table 1 Requirements of Quartz for Ceramic Industry

(*Clauses* 5.6, D-2.4 *and* D-3.3)

Sl No.	Characteristic	Requirement		Method of Test, Ref to
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Loss on ignition, percent by mass, *Max*	0.5	1.0	C-3
ii)	Silica (as SiO_2), percent by mass, *Min*	98	96	C-4
iii)	Alumina (as Al_2O_3), percent by mass, *Max*	1.0	2.0	C-5
iv)	Titanium dioxide (as TiO_2), percent by mass, *Max*	0.1	1.0	C-6
v)	Iron oxide (as Fe_2O_3), percent by mass, *Max*	0.25	1.0	C-7
vi)	Calcium oxide (as CaO), percent by mass, *Max*	0.5	1.0	C-8

6 PACKING AND MARKING

6.1 Packing

Quartz shall be packed in polyethylene lined gunny jute/plastic bags each containing 50 kg.

6.2 Marking

The bags shall be marked with the following:

a) Processor’s name or his recognized trademark, if any;
b) Name and grade of the material;
c) Grain size;
d) Net mass of the content; and
e) Batch number to enable the material to be traced from records.

6.3 BIS Certification Mark

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

7 SAMPLING

7.1 The representative sample shall be drawn as prescribed in Annex D.

7.2 Test Sample

Grind 5 to 10 g of the sample in a steel mortar so that it passes through 75 µm Sieve. Dry to a constant mass at 105 ± 2 °C and use it for purpose of chemical analysis.

ANNEX A

(*Clause* 5.2)

DETERMINATION OF MOISTURE CONTENT

A-1 PROCEDURE

A-1.1 Weigh accurately, nearest to 0.01 g, about 20 g of the sample in a tared platinum dish and dry at 105 to 110 °C in an air-oven for 30 min. Cool in a desiccators and weigh. Repeat heating and cooling till constant mass is obtained.

A-1.2 Calculation

$$\text{Moisture content, percent by mass} = \frac{m - m_1}{m} \times 100$$

where

m = Mass of sample taken for the test, in g; and

m_1 = Mass of sample after drying, in g.

ANNEX B

(*Clause* 5.4)

DETERMINATION OF SPECIFIC GRAVITY

B-1 APPARATUS

B-1.1 Analytical Balance — Accurate up to 0.000 1 g.

B-1.2 Pyknometer — 50 ml capacity, with a ground capillary stopper.

B-1.3 Vacuum Source — To produce a vacuum of 12 to 25 mm of mercury.

B-2 PROCEDURE

B-2.1 The pyknometer shall be dried at room temperature after rinsing it with acetone or any quick-drying solvent and then with hot water. Weigh the pyknometer and record the mass as m. Fill the pyknometer with distilled water and note the water temperature as t. Insert the stopper and wipe the excess water from the tip in such a manner as not to withdraw any water from the capillary. Thoroughly dry any water adhering to the outside of the pyknometer and weigh, recording the mass as m_1.

B-2.2 Empty the pyknometer and dry it again at 105 to 110 °C. Introduce about 10 g of the dried sample and weigh with the stopper in position, recording the mass as m_2. Remove the stopper, fill the pyknometer with distilled water to one-fourth to one-half its capacity and keep the pyknometer in a vacuum desiccator and apply vacuum to remove the entrapped air completely. Release the vacuum and take out the pyknometer from the desiccator and cool it to room temperature.

B-2.3 Fill the pyknometer with water. Insert the stopper, wipe the excess water from the tip and the outside of the pyknometer as above and weigh, recording the mass as m_3.

B-3 CALCULATION

Calculate the relative density as follows:

$$\text{Relative density, } t°/27\ °\text{C} = \frac{(m_2 - m)}{(m_2 - m) - (m_3 - m_1)} \times d$$

where

t = Temperature of water, in °C;

m = Mass of empty stoppered pyknometer, in g;

m_1 = Mass of stoppered pyknometer filled with distilled water, in g;

m_2 = Mass of stoppered pyknometer and sample, in g;

m_3 = Mass of stoppered pyknometer with sample and distilled water, in g; and

d = Density of water at 27 °C.

ANNEX C

(*Clause* 5.6 *and Table* 1)

CHEMICAL ANALYSIS OF QUARTZ

C-1 SAMPLING AND PREPARATION OF SAMPLE

The material shall be sampled and prepared for chemical analysis as given in **7.1** and **7.2**.

C-2 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used.

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

C-3 DETERMINATION OF LOSS ON IGNITION

C-3.1 Procedure

Transfer 1 g of the test sample (*see* **7.2**) to a weighed platinum crucible. Introduce it into a muffle furnace and slowly raise the temperature to 1 000 ± 50 °C. After about 30 min, remove the crucible and cool in a desiccator and weigh. Repeat ignition and cooling to obtain constant mass.

C-3.2 Calculation

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

where

M_1 = Mass of the sample taken for the test, in g; and

M_2 = Mass of the sample after ignition, in g.

C-4 DETERMINATION OF SILICA

C-4.1 Outline of the Method

The sample is decomposed by fusion with sodium carbonate and extracted with acid. Silica is then determined gravimetrically by dehydration and baking followed by hydrofluorization. The residual silica in the filtrate is determined photometrically using molybdenum blue method.

C-4.2 Reagents

C-4.2.1 *Sodium Carbonate* **—** Anhydrous.

C-4.2.2 *Dilute Hydrochloric Acid* **—** (1 : 1), (1 : 3) and (1 : 19) (*v*/*v*) (*see* IS 265).

C-4.2.3 *Dilute Sulphuric Acid* — 1 : 1 (*v*/*v*) (*see* IS 266).

C-4.2.4 *Hydrofluoric Acid* — 40 percent (*m*/*m*) (*see* IS 10332).

C-4.2.5 *Potassium Pyrosulphate*— Solid.

C-4.2.6 *Ethylene Diamine Tetra Acetate* (*EDTA*) *Solution* — 0.025 M approximately. Dissolve 9.31 g of disodium ethylene-diamine tetra acetate, dehydrate ($C_{10}H_{14}N_2Na_2O_8.2H_2O$) in water and dilute to 1 l.

C-4.2.7 *Phenolphthalein Solution* — Dissolve 1 g of phenolphthalein in 100 ml of rectified spirit.

C-4.2.8 *Dilute Ammonia Solution* — 1 : 3 (*v*/*v*) (*see* IS 799).

C-4.2.9 *Ferric Ammonium Sulphate Solution* — Dissolve 40.82 g of ferric ammonium sulphate $[NH_4Fe(SO_4)_2.12H_2O]$ in water containing 80 ml of dilute hydrochloric acid (1 : 1) and dilute to 500 ml in avolumetric flask.

C-4.2.10 *Ammonium Molybdate Solution* — Dissolve 100 g of ammonium molybdate in water containing a few drops of dilute ammonia, filter, if necessary, and dilute to 1 l.

C-4.2.11 *Oxalic Acid Solution* — Dissolve 100 g of oxalic acid in water and dilute to 1 l.

C-4.2.12 *Ascorbic Acid Solution* — 10 percent (*m*/*m*). Dissolve 10 g of the acid in 100 ml of water and filter, if necessary. Preserve in an amber coloured bottle. The solution, thus preserved, could be used up to 5 days.

C-4.2.13 *Standard Silica Solution* — 0.04 mg/ml.

a) Fuse 0.200 g of pure silica (99.8 percent) in 5 g of sodium carbonate in a platinum crucible. Dissolve in 300 ml of water, containing 8 to 10 g of sodium hydroxide, using a polythene beaker. Cool and dilute with water in a volumetric flask to 500 ml. Store in a polythene bottle; and
b) Prepare immediately before use a standard solution containing 0.04 mg/ml by diluting the above stock solution ten times.

C-4.3 Procedure

C-4.3.1 *Decomposition of the Sample*

Weigh accurately about 0.5 g of the finely ground and dried (105 °C to 110 °C) sample into a platinum crucible. Add 3 g of sodium carbonate, after mixing intimately, cover the dish with lid and heat over a Meker burner, slowly increasing the temperature with caution till the mixture fuses. Keep at this temperature for 10 min and finally heat till a clear melt is obtained. Remove from the burner and cool. Extract the melt in a porcelain dish first with water and then with (1 : 1) dilute hydrochloric acid.

C-4.3.1.1 Add about 30 ml of (1 : 1) dilute hydrochloric acid and 0.5 ml of dilute sulphuric acid. Cover the porcelain dish and allow to stand on a steam bath until disintegration of the melt is complete. Crush any large flakes with flattened end of a glass rod.

C-4.3.2 *Gravimetric Determination of Insoluble Silica*

Evaporate the solution in the porcelain dish to dryness and allow it to remain on the steam bath for another hour. Transfer the dish to an air oven maintained between 105 °C and 110 °C and bake for 1 h. Cool the dish and add 20 ml of (1 : 1) dilute hydrochloric acid followed by 30 ml of hot water. Stir the mixture till solution of soluble salts appears to be complete. Digest the mixture on a steam bath for 10 min without stirring. Filter through No. 42 filter paper and rinse the dish with (1 : 19) dilute hydrochloric acid, scrubbing the basin with a rubber tipped glass rod. Wash the precipitate five times with (1 : 19) and hot dilute hydrochloric acid and then with hot water until free from chloride. Reserve the filtrate and washings for photometric determination of residual silica.

C-4.3.2.1 Transfer both the filter paper and precipitate to a tared platinum crucible. Ignite at a low temperature until the precipitate is free from carbonaceous matter and then heat in the muffle furnace at 1 050 °C to constant mass (1 h being normally sufficient). Cool in a desiccator and weigh recording the mass as M_1. Moisten the contents of the cold crucible with water, add five drops of dilute sulphuric acid and 10 ml of hydrofluoric acid. Evaporate to dryness on a hot plate or sand bath, in a fume cupboard, taking care to avoid spurting.

C-4.3.2.2 Heat the crucible and residue, cautiously at first, over a gas flame until the disappearance of white fumes and finally for 5 min in the furnace at 1 050 °C, cool in a desiccator and weigh recording the mass as M_2.

NOTE — If the residue weighs more than 10 mg, repeat the treatment with dilute sulphuric acid and hydrofluoric acid, ignite, cool, and weigh (as M_2). Then fuse any residue with 0.5 g of anhydrous sodium carbonate, dissolve in 10 ml of (1 : 4) dilute hydrochloric acid and transfer quantitatively to the filtrate and washings reserved in **C-4.3.2**.

C-4.3.3 *Photometric Determination of the Residual Silica*

Boil the filtrate and washings reserved in **C-4.3.2** and reduce to about 200 ml. Transfer to a 250-ml volumetric flask and make up to the mark with water.

C-4.3.3.1 *Test blank solution*

Prepare a test blank solution using the same procedure and quantities of reagents as detailed from in **C-4.3.1** to **C-4.3.3**, but excluding the sample and dilute up to 250 ml with water.

NOTE — Fusion may, however, be omitted to avoid undue attack on platinum crucible.

C-4.3.3.2 Transfer 10 ml aliquot of the solution prepared in **C-4.3.3** to a 50 ml volumetric flask. Also, transfer 10 ml of each of the test blank solution prepared in **C-4.3.3.1** to five 50 ml volumetric flasks. Add to the six flasks 0, 1, 2, 3, 4 and 5 ml respectively of the standard silica solution. Then to all the flasks, add 5 ml of EDTA solution, a drop of phenolphthalein solution and then dilute ammonia solution dropwise until the colour changes to red. Then add 5 ml of ferric ammonium sulphate solution and dilute to about 30 ml with water (the *p*H of the solutions should be 1 to 1.5).

C-4.3.3.3 Add 5 ml of ammonium molybdate solution and allow to stand for 10 min. Then add 5 ml of oxalic acid solution followed immediately by 5 ml of ascorbic acid solution and dilute the volume to 50 ml. Allow the solution to stand for 30 min for development of full colour.

C-4.3.3.4 Starting with the blank (‘0’ ml) solution, transfer a suitable portion of the coloured solution one at a time to an absorption cell, record the photometric readings using the light band centred approximately at 810 nm. Prepare the calibration curve from these readings against concentration of silica in mg (as SiO_2).

C-4.3.3.5 Similarly, transfer a suitable aliquot of coloured test solution to the absorption cell and note the photometric reading for the test solution. Convert it to milligrams of silica by means of the calibration curve and calculate the silica content in grams (M_3) in 250 ml of solution prepared in **C-4.3.3**.

C-4.4 Calculation

$$\text{Silica, percent by mass} = \frac{(M_1 - M_2 + M_3)}{M} \times 100$$

where

M = Mass of sample taken for the test in **C-4.3.1**, in g;

M_1 = Mass of crucible and contents before hydrofluoric acid treatment, in g;

M_2 = Mass of crucible and residue after hydrofluoric acid treatment, in g; and

M_3 = Mass of silica present in 250 ml of solution prepared in **C-4.3.3**, in g.

C-5 DETERMINATION OF ALUMINA

C-5.1 Outline of the Method

Aluminium is separated from the aliquot of solution prepared in **C-4.3.3** as sodium aluminate by treatment with sodium hydroxide and is complexed quantitatively with EDTA by boiling with an excess of the reagent. The excess EDTA is back titrated with standard zinc solution at *p*H about 5.2 using xylenol orange indicator. The aluminium EDTA complex on boiling with ammonia fluoride liberates EDTA which when titrated with standard zinc solution gives the amount of aluminium present in the solution.

C-5.2 Reagents

C-5.2.1 *Dilute Sulphuric Acid* — 1 : 1 (*v*/*v*) (*see* IS 266). Add 100 ml of concentrated sulphuric acid (relative density l.84) cautiously with stirring to 100 ml of water and cool.

C-5.2.2 *Hydrofluoric Acid* — 40 percent (*m*/*m*) (*see* IS 10332).

C-5.2.3 *Sodium Hydroxide Solution* — 10 percent (*m*/*v*).

C-5.2.4 *Sodium Carbonate* — Solid.

C-5.2.5 *Dilute Hydrochloric Acid* — 1 : 1 (*v*/*v*) (*see* IS 265).

C-5.2.6 *EDTA Solution* (0.01 M) — Transfer 100 ml of the solution prepared in **C-4.2.6** to a 250-ml volumetric flask and dilute with water up to the mark.

C-5.2.7 *Methyl Orange* — Dissolve 0.05 g of methyl orange in 100 ml of water.

C-5.2.8 *Dilute Ammonia* (1 : 5) — Dilute 25 ml of concentrated ammonia (relative density 0.9) to 150 ml.

C-5.2.9 *Acetate Buffer* (*p*H 5.2) — Dissolve 21.5 g of sodium acetate ($CH_3COONa.3H_2O$) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1 1.

C-5.2.10 *Standard Aluminium Solution* — Weigh 0.529 3 g of aluminium metal (99.9 percent purity) into a beaker containing 40 ml of dilute hydrochloric acid (1 : 1) and small drops of mercury. Heat the beaker over a water bath. Transfer the solution to a 1 000 ml volumetric flask and make up to the mark. One ml of this solution is equivalent to 1.0 mg of alumina (as Al_2O_3).

NOTE — Mercury will not dissolve and may be removed while transferring the solution to 1000 ml volumetric flask.

C-5.2.11 *Standard Zinc Solution* (0.01 M) — Dissolve 5.484 5 g of zinc acetate [$Zn(CH_3COO)_2.2H_2O$] in 50 ml of water. Add 0.5 ml of glacial acetic acid and 25 ml of acetate buffer (*see* **C-5.2.9**) and dilute to 1 l in a volumetric flask. Further dilute 100 ml of this solution to 250 ml in a volumetric flask and find out the equivalent Al_2O_3, in g/ml, of this diluted solution from the procedure given in **C-5.3.3**, taking 20 ml of standard aluminium solution (*see* **C-5.2.10**).

C-5.2.12 *Xylenol Orange Indicator* — Dissolve 0.1 g of xylenol orange in 100 ml of water containing two drops of dilute hydrochloric acid (1 : 1).

C-5.2.13 *Ammonium Fluoride* — Dissolve 20 g of ammonium fluoride in 100 ml of water. Add 3 drops of xylenol orange (*see* **C-5.2.12**) and then add dilute hydrochloric acid (1 : 9) dropwise till the colour changes to yellow.

C-5.3 Procedure

C-5.3.1 *Solution of the Sample*

Weigh accurately about 2 g of the finely ground test sample in a platinum dish. Moisten the sample with 5 ml of water. Add 3 ml of dilute sulphuric acid and 20 ml of hydrofluoric acid and evaporate slowly to dryness on the sand bath or hot plate in a fume cupboard taking care to avoid spurting. Repeat the process with 10 ml of hydrofluoric acid. Cool, add 2 ml of dilute sulphuric acid and evaporate as before. Heat the dry residue cautiously until fumes of sulphur trioxide cease to be evolved. Raise the temperature to 1 000 °C and ignite for 5 min. Cool and fuse the residue with 1 g of sodium carbonate and dissolve in hydrochloric acid, cool and dilute to 250 ml in a volumetric flask.

C-5.3.2 Transfer 100 ml aliquot of the solution prepared in **C-5.3.1** to a 500 ml beaker and nearly neutralize with sodium hydroxide solution. Add with constant stirring another 100 ml of sodium hydroxide solution containing 1 g of sodium carbonate dissolved in it. Allow to stand on a warm water bath for 1 h. Finally cool to room temperature, transfer quantitatively to a 250 ml volumetric flask and dilute up to the mark with water. Mix well and allow to stand for a few minutes. Filter through a filter paper previously washed with sodium hydroxide solution, collecting the filtrate in a beaker, rinsing the beaker first with a little filtrate.

NOTE — Sodium carbonate is used for precipitating calcium and magnesium along with iron and titanium.

C-5.3.3 Transfer a suitable aliquot from the filtrate to a 250 ml conical flask, add two drops of methyl orange indicator followed by 1 : 1 dilute hydrochloric acid drop wise until the colour changes from yellow to red. Add sufficient EDTA solution to provide an excess of at least 5 ml over the expected amount of alumina (1 ml of 0.025 M EDTA= 1.25 mg Al_2O_3 approximately). Add dilute ammonia drop wise until the colour changes from red to yellow. Add 5 ml of acetate buffer and about 25 ml of water and heat to boiling for 5 min. Cool, add 5 ml of buffer solution and titrate with standard zinc solution using 5 to 6 drops of xylenol orange indicator. The end point is indicated by a clear change from yellow to deep orange colour. Add 10 ml of ammonium fluoride solution and heat to boiling for 5 min. Cool, add 5 ml of buffer solution and titrate the liberated EDTA with standard zinc solution.

C-5.4 Calculation

$$\text{Alumina, percent by mass} = \frac{V \times F}{M} \times 100$$

where

V = Volume of standard zinc required for the titration of liberated EDTA, in ml;

F = Equivalent Al_2O_3 of standard zinc acetate solution, mg/ml; and

M = Mass of the sample represented by 100 ml of solution taken in **C-5.3.2**, in g.

C-6 DETERMINATION OF TITANIUM DIOXIDE (TiO_2)

C-6.1 Outline of the Method

The yellow coloured complex, produced in acid solution of the sample by the addition of hydrogen peroxide, is determined photometrically at approximately 410 nm.

C-6.2 Reagents

C-6.2.1 *Dilute Sulphuric Acid* — 1 : 1 (*v*/*v*) (*see* IS 266).

C-6.2.2 *Phosphoric Acid* — Add 400 ml of phosphoric acid (relative density 1.75) to 500 ml of water, cool and dilute to 1 l.

C-6.2.3 *Hydrogen Peroxide* — 20 volumes.

C-6.2.4 *Standard Titanium Dioxide Solution* — Weigh accurately 3.680 g of potassium titanium oxalate [$K_2TiO_2(C_2O_4)_2.2H_2O$]. Transfer to a boiling flask, add 8 g of ammonium sulphate and 100 ml of concentrated sulphuric acid. Gradually heat the mixture to boiling and boil for 10 min. Cool, pour the solution into 750 ml of water, and dilute to 1 000 ml in a volumetric flask. One ml of this solution is equivalent to 0.5 mg of titanium dioxide (as TiO_2).

C-6.3 Procedure

C-6.3.1 *Preparation of the Sample Solution*

C-6.3.1.1 Weigh accurately about 2 g of the dried material in a platinum dish. Moisten the sample with 5 ml of water. Add 3 ml of dilute sulphuric acid and 20 ml of hydrofluoric acid and evaporate to dryness on the sand bath in a farming cupboard, taking care to avoids purting. Cool and repeat the process of evaporation twice with another 10 ml of hydrofluoric acid each time. Cool, add 2 ml of dilute sulphuric acid and evaporate as before. Heat the dry residue cautiously until fumes of sulphur trioxide cease. Ignite the residue at 1 000 °C for 5 min and cool. Fuse the residue with 3 g of anhydrous sodium carbonate, cool the fused mass and dissolve it in 25 ml of dilute hydrochloric acid and dilute with water to 250 ml in a volumetric flask. Reserve the solution for determination of titania.

C-6.3.1.2 Transfer 25 ml of the prepared solution (*see* **C-6.3.1**) to a 100 ml volumetric flask. Add 2.5 ml of dilute sulphuric acid, 5 ml of phosphoric acid and 5 ml of hydrogen peroxide. Make up to the mark with water and mix.

C-6.3.2 *Preparation of the Blank Solution*

Prepare the blank solution following the procedure and using the same quantities of reagents given in **C-6.3.1** but without the material and dilute the solution with water to 250 ml in a volumetric flask.

C-6.3.3 *Calibration Curve*

Transfer 25 ml each of the blank solution (*see* **C-6.3.2**) to six 100 ml volumetric flasks and add 0, 1, 2, 3, 4 and 5 ml respectively of the standard titanium dioxide solution. By using the same quantity of reagents, proceed as given in **C-6.3.1.2** and makeup the solutions up to the mark. Take the required aliquots of these solutions in the absorption cell, one at a time, and plot a curve showing optical densities of solutions at 410 nm against the amount of titanium dioxide, in mg, present in each.

C-6.3.4 Next take appropriate aliquot of the solution prepared in **C-6.3.1.2** in the absorption cell and measure its optical density at 410 nm and note from the calibration curve the amount of titanium dioxide, in milligrams, present in it.

C-6.4 Calculation

$$\text{Titanium dioxide, percent by mass} = \frac{M_1}{M} \times 0.1$$

where

M_1 = Mass of titanium dioxide content, present in the aliquot taken for the test (*see* **C-6.3.4**), in mg; and

M = Mass of the material present in the aliquot taken for the test (*see* **C-6.3.1**), in g.

C-7 DETERMINATION OF IRON OXIDE

C-7.1 Outline of the Method

The orange-red complex produced in acid solution of the sample (*p*H 4 to 6) by the addition of *o*-phenanthroline is determined photometrically at approximately 510 nm.

C-7.2 Reagents

C-7.2.1 *Tartaric Acid Solution* — 10 percent (*m*/*v*).

C-7.2.2 *Hydroxylamine Hydrochloride Solution* — Dissolve 1 g of the reagent in water. Transfer the solution to a 100 ml volumetric flask, dilute to the mark and mix well.

C-7.2.3 *O-Phenanthroline Solution* — Dissolve 1 g of *o*-phenanthroline monohydrate in 90 ml of water with gentle heating and constant stirring. Cool and dilute to 100 ml.

C-7.2.4 *Acetate Buffer* — Dissolve 21.5 g of sodium acetate ($CH_3COONa.3H_2O$) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1 000 ml.

C-7.2.5 *Standard Iron Solution* — Dissolve 0.2 g of pure iron in concentrated hydrochloric acid. Dilute the solution to about 100 ml. Transfer to a 1 000 ml volumetric flask, dilute to the mark and mix well. Dilute 100 ml of this solution to 1 000 ml in a volumetric flask.

One ml of this diluted solution is equivalent to 0.02 mg of iron (as Fe).

C-7.3 Procedure

C-7.3.1 Transfer 5 ml aliquot of the solution prepared in **C-5.3.1** to a 100 ml volumetric flask. Also transfer 0, 1, 2, 4, 6, 8 and 10 ml each of the standard iron solution to seven separate 100 ml volumetric flasks. Then add to each flask 2 ml of tartaric acid solution followed by 2 ml of hydroxylamine hydrochloride solution. Stir well and add 5 ml of *o*-phenanthroline solution and 10 ml of acetate buffer solution. Allow to stand for 15 min and dilute up to the mark with water.

C-7.3.2 Starting with the blank ('0' ml) solution transfer a suitable portion of the coloured solution, one at a time, to an absorption cell; record the photometric readings using the light band centred approximately at 510 nm and prepare the calibration curve from these readings against concentration of iron (as Fe).

C-7.3.3 Similarly transfer a suitable aliquot of the coloured test solution to the absorption cell and note the photometric reading for the test solution. Convert it to milligrams of iron by means of the calibration curve and calculate the iron oxide content in the aliquot taken in **C-7.3.1**.

C-7.4 Calculation

Ferric oxide (as Fe_2O_3), percent by mass =

$$\frac{M}{M_1} \times 0.1429$$

where

M = Mass of iron (as Fe), found in aliquot, in mg; and

M_1 = Mass of the sample presented by the aliquot taken in **C-7.3.1**, in g.

C-8 DETERMINATION OF CALCIUM OXIDE

C-8.1 Outline of the Method

In an aliquot of the sample solution, metallic oxide (R_2O_3) group elements are marked by triethanolamine, and calcium is titrated with EDTA solution using Patton and Reader (P and R) indicator.

C-8.2 Reagents

C-8.2.1 *Concentrated Nitric Acid* — *See* IS 264.

C-8.2.2 *Perchloric Acid* — 70 percent (*m*/*m*).

C-8.2.3 *Hydrofluoric Acid* — 40 percent (*m*/*m*) (*see* IS 10332).

C-8.2.4 *Concentrated Hydrochloric Acid* — *See* IS 265.

C-8.2.5 *Triethanolamine Solution* — 30 percent (*v*/*v*). Dilute 60 ml of triethanolamine to 200 ml.

C-8.2.6 *Sodium Hydroxide Solution* — 5 N approximately. Dissolve 100 g of sodium hydroxide in water, cool and dilute to 500 ml and store in a polythene bottle.

C-8.2.7 *Patton and Reader* (*P and R*) *Indicator* — Mix by grinding together 0.1 g of P and R indicator and 30 g of sodium chloride. About 0.2 g of the mixture is required for each titration.

C-8.2.8 *Standard EDTA Solution* (0.01 M) — Same as in **C-5.2.6**. Standardize against the standard calcium solution (*see* **C-8.2.9**) using P and R indicator.

C-8.2.9 *Standard Calcium Solution* — Dissolve 1.784 8 g of calcium carbonate dried at 150 °C in a slight excess of hydrochloric acid, boil to expel carbon dioxide, cool and dilute to 1 000 ml. 1 ml of this solution is equivalent to 1 mg of calcium oxide (as CaO).

C-8.3 Procedure

C-8.3.1 *Solution of the Sample*

Weigh accurately about 1 g of the test sample in a platinum dish. Add 5 ml of concentrated nitric acid, 5 ml of perchloric acid and about 10 ml of hydrofluoric acid. Cover the dish with a lid. Allow the reaction to proceed for at least 15 min, then remove the lid and evaporate the mixture to dryness on a sand bath in a fume cupboard, taking care to avoid spurting. Cool, add 5 ml of perchloric acid, rinse the lid and sides of the dish with water and again evaporate carefully to dryness. To the cool, dry residue, add 2 ml of concentrated hydrochloric acid and about 15 ml of water. Digest on a steam bath for 10 min, cool, filter if necessary and dilute the solution with distilled water to 250 ml in a volumetric flask. Store the solution in a polythene bottle.

C-8.3.2 *Determination of Calcium Oxide*

Pipette 50 ml of the solution into a 250 ml conical flask. Add 10 ml of triethanolamine solution, 5 ml of sodium hydroxide solution dropwise while shaking the solution, and then add 0.2 g of P and R indicator. Titrate immediately with standard EDTA solution until the colour changes from pink to blue.

NOTE — If sharp end-point is not obtained, add 2 ml of potassium cyanide (10 percent, *m*/*v*) after the addition of triethanolamine.

C-8.4 Calculation

Calcium oxide (as CaO), percent by mass =

$$\frac{V \times F}{M} \times 100$$

where

V = Volume of standard EDTA solution required for titration, in ml;

F = CaO equivalent of the standard EDTA solution, in g/ml; and

M = Mass of the sample represented by the aliquot taken, in g.

ANNEX D

(*Clause* 7.1)

SAMPLING PROCEDURE FOR QUARTZ FOR CERAMIC INDUSTRY

D-1 SCALE OF SAMPLING

D-1.1 Lot

All the bags in a consignment and belonging to the same batch of manufacture and of same grade shall constitute a lot. If the consignment is known to consist of different batches of manufacture, the bags belonging to same batch and variety shall be grouped together and each such group shall constitute a separate lot.

D-1.2 Unless otherwise as agreed to between the buyer and the seller the number of bags to be selected shall depend upon the size of the lot and shall be in accordance with Table 2.

Table 2 Number of Bags to be Selected

(*Clause* D-2.1)

Sl No. (1)	No. of Bags in the Lot (2)	No. of Bags to be Selected (3)
i)	Up to 150	3
ii)	151 – 300	4
iii)	301 – 500	5
iv)	501 – 1 000	7
v)	1 001 and above	10

D-1.3 The bags shall be selected from the lot at random and in order to ensure randomness of selection, the method given in IS 4905 may be followed.

D-2 NUMBER OF TESTS

D-2.1 The bags shall be visually examined for defects as given in **5.1**.

D-2.2 Test for specific gravity shall be conducted on individual sample.

D-2.3 For fired colour, test shall be carried out on all the bags selected as per **D-1.2**.

D-2.4 Tests for all other characteristics in table 1 shall be conducted on composite sample.

D-2.5 For preparation of composite sample and general requirements of sampling methods given in IS 8883 (Part 1) may be followed.

D-3 CRITERIA FOR CONFORMITY

D-3.1 For individual test, from the test results average ($\overline{X}$) and range (R) shall be calculated, range being the difference between the maximum and minimum of the test results and:

$$\text{Average } (\overline{X}) = \frac{\text{Sum of the test results}}{\text{Number of tests}}$$

The lot shall be declared as conforming to the specification with respect to specific gravity, if the expression:

$$R\,(U - L) \leq 0.9$$

$$\overline{X} + 0.6R \leq U$$

$$\overline{X} - 0.6R \geq L$$

Where U is upper specification limit and L is the lower specification limit.

D-3.2 All the bags tested for fired colour shall pass.

D-3.3 All the composite samples shall meet ther elevant requirement given in Table 1.

D-3.4 The lot shall be declared as conforming to the requirements of this specification, if the requirements of **D-3.2** and **D-3.3** are satisfied.

NOTE — In case the number of test results in the sample is 10, they shall be divided into two sub-groups of five test results each taking them consecutively as obtained. The range of each sub-group shall then be determined and mean range shall be calculated by dividing the sum of the ranges by two. In the expression in **D-3.1**, R shall then be replaced by $\overline{R}$.

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Ceramicware Sectional Committee, CHD 09

Organization	*Representative(s)*
CSIR - Central Glass and Ceramic Research Institute, Kolkata	Dr K Muraleedharan (***Chairman***)
All India Institute of Hygiene and Public Health, Kolkata	Prof R. C. Srivastava Dr Nibedita Das (*Alternate*)
All India Pottery Manufacturers' Association, Kolkata	Nomination Awaited
Associated Soapstone Distributing Co Pvt Ltd, Jaipur	Shri Dilip Jha Shri Rajendra Pareek (*Alternate* I) Shri Amit Sharma (*Alternate* II)
Aditya Birla Insulator, West Bengal	Nomination Awaited
Asian Granito India Ltd, Gujarat	Nomination Awaited
Ahmedabad Pottery Manufacturers Association, Naroda	Nomination Awaited
IIT-BHU, Varanasi	Prof Devendar Kumar Prof Om Prakash (*Alternate*)
BHEL, Bengaluru	Nomination Awaited
Bhabha Atomic Research Center, Mumbai	Dr Deep Prakash Dr Abhijit Ghosh(*Alternate*)
C-met, Thrissur, Kerala	Nomination Awaited
CSIR-Central Glass and Ceramic Research Institute, Kolkata	Dr H. S. Tripathi
Cera Sanitary ware Ltd, Kadi	Shri Vivek Narayan Shri Pramod Chauhan (*Alternate*)
Chem. & Allied Products Export Insp. Council, Kolkata	Shri S. K. Ghosh
Controller of Quality Assurance, Kanpur	Shri V. R. Chaudhary Dr K. C. Gupta (*Alternate*)
Govt College of Engg & Ceramic Technology, Kolkata	Prof Ram Chandra Das Prof Rituparno Sen (*Alternate*)
Grindwell Norton Limited, Chennai	Nomination Awaited
HSIL Ltd, Bahadurgarh	Shri R. K. Somany Shri T. K. Raha (*Alternate*)
Industries Commissioner, Gandhinagar	Shri R. D. Barhatt
Johnson Tiles, Mumbai	Nomination Awaited
NTB Hi Tech Ceramics, Pune	Nomination Awaited
National Test House, Kolkata	Dr S. K. Kulshreshtha Shri V. Kumar (*Alternate*)
National Institute of Technology, Rourkela	Nomination Awaited
Office of Dev Commissioner (MSME), New Delhi	Shri Suresh Yadav

Organization	*Representative(s)*
Roca Bathroom Products Pvt Ltd, Tamilnadu	Shri C. Kannan Shri S. Aravazhi (*Alternate*)
Porcelain Enamelers Association, Kolkata	Shri B. S. Ganguly Shri S. K. Mukherjee (*Alternate*)
Rajniklal Ceramic Pvt Ltd, Kolkata	Shri Prabodh M. Sanghve Shri Pulak Chakraborty (*Alternate*)
SPL Ltd, Jhajjar, Haryana	Dr Ashutosh Goyal Shri T. Shridhar (*Alternate*)
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Member Secretary

Shri Sachin ‘S’ Menon
Scientist ‘B‘, CHD, BIS

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