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Draft Indian Standard

GLASS MAKING SANDS - SPECIFICATION

(*Third Revision*)

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ICS 81.040.30

Glass, Glassware & Laboratoryware Sectional Committee, CHD 10

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FOREWORD

This standard was originally issued as a tentative standard in 1953 and revised in 1963. However, in view of the progress made by the glass industry in the country and demands made on it by buyers, need was felt to revise the standard again to provide useful guidance in the selection of silica sand required for the production of quality glassware. In the second revision in 1980, improved methods of analysis were incorporated. Limits of colouring oxides of manganese, copper and chromium and their methods of test which were not given earlier, had also been included to make the standard comprehensive.

In this third revision, instrumental test methods are added and new requirements for Na₂O and K₂O have been added along with several editorial changes such as the inclusion of ICS No., Reference Clause, etc. have also been incorporated.

Free iron which is sometimes present in silica sand as a contamination results in poor appearance of the surface of finished glassware. It is, therefore, advisable to remove it by magnetic separation before silica sand is used in a batch.

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 : 2022 'Rules for rounding off numerical values (second revision)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

GLASS MAKING SANDS - SPECIFICATION

(*Third Revision*)

1 SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for silica sand suitable for the manufacture of some of the important types of glassware.

1.2 This standard does not cover silica sand for optical and other special glasses.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of and necessary adjuncts to 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 indicated.

<i>IS No.</i>	<i>Title</i>
264 : 2005	Nitric acid - Specification (<i>third revision</i>)
266 : 1993	Sulphuric acid - Specification (<i>third revision</i>)
1070 : 1992	Reagent Grade Water – Specification (<i>third revision</i>)
1382 : 1981	Glossary of terms relating to glass and glassware (<i>first revision</i>)

3 TERMINOLOGY

3.1 For the purpose of this standard the definitions given in IS 1382 shall apply.

4 GRADES

4.1 Glass making sands shall be of the following four grades:

- a) *Special Grade* – intended for the manufacture of high grade colourless glass, such as crystal glass, tableware and decorated ware;
- b) *Grade 1* – intended for the manufacture of decolourized glassware, such as container ware, lamp ware, etc.;
- c) *Grade 2* – intended for the manufacture of glassware where a slight tint is permissible; and
- d) *Grade 3* – intended for the manufacture of some coloured glasses.

5 REQUIREMENTS

5.1 Description – Silica sand shall be fairly free from contamination like clay material, pebbles and other extraneous matter.

5.2 Size Grading – The grain size constituting the material, when tested according to the method prescribed in Annex A, shall lie within the following limits:

Retained on 1-mm IS Sieve	Nil
Retained on 600-micron IS Sieve, percent by mass, <i>Max</i>	1.0
Passing through 600-micron IS Sieve, but retained in 300 micron IS Sieve, percent by mass, <i>Max</i>	50.0

Passing through 125-micron IS Sieve, percent by mass, <i>Max</i>	5.0
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5.3 Moisture Content - Unless otherwise specified and agreed to between the purchaser and the supplier, the sand as received shall not contain more than 4 percent by mass of moisture, when determined by the method prescribed in Annex B.

5.4 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Annex C. Reference to the relevant clauses of Annex C is given in col 7 of the table.

TABLE 1 REQUIREMENTS FOR GLASS MAKING SANDS

(Clause 5.4)

SL. No.	CHARACTERISTICS	Requirements				Methods Of Test (Ref to CL No. in Appendix C)
		Special Grade	Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Loss on ignition, percent by mass, <i>Max</i>	0.5	0.5	0.5	0.5	C-3
ii)	Silica (as SiO ₂), percent by mass, <i>Max</i>	99.0	98.5	98.0	97.0	C-4
iii)	Iron oxide (as F ₂ O ₃), percent by mass, <i>Max</i>	0.020	0.04	0.06	0.10	C-5
iv)	Aluminium oxide (as Al ₂ O ₃), percent by mass, <i>Max</i>	*	*	*	1.5	C-6
v)	Titanium dioxide (as TiO ₂), percent by mass, <i>Max</i>	0.10	0.10	0.10	*	C-7
vi)	Manganese oxide (as MnO)	To pass the test	To pass the test	To pass the test	*	C-8
vii)	Copper Oxide (as CuO)	To pass the test	To pass the test	To pass the test	*	C-9
viii)	Chromium Oxide (as Cr ₂ O ₃)	To pass the test	To pass the test	To pass the test	*	C-10

* These requirements shall be as agreed to between the purchaser and the manufacturer

6 SAMPLING

6.1 Representative samples of the material shall be drawn and conformity of the material to the requirements of the specification determined according to the procedure prescribed in Annex D.

6.2 The supplier shall submit the tender sample packed in three different containers, each containing not less than 500 g of the material.

6.3 The purchaser may select as approved sample, the sample submitted by the supplier or any other agreed sample. He shall return to the supplier one suitably sealed container of the approved sample.

ANNEX A

(Clause 5.2)

METHOD OF SIZE GRADING OF GLASS MAKING SANDS

A-1 PREPARATION OF TEST SAMPLE

A-1.1 Break aggregates, if any, in a large, clean and dry mortar. Repeat the process of coning and quartering as described under D-2.2 until a prepared sample not less than 150 g is obtained. Reserve the rest of the material for tests prescribed in Appendix B and Annex C.

A-2 PROCEDURE

A-2.1 Dry about 150 g of the prepared sample to constant mass at 110°C in a porcelain dish, and sieve in three portions of about 50 g each, accurately weighed, through 1-mm, 600-micron, 300-micron and 125-micron IS Sieves. Shake the prepared sample well (but do not rub on the sieves) for a period of at least 3 minutes. Weigh the material retained on each sieve, and express the masses as percentage of the mass of the prepared sample taken for the test.

A-2.2 In the case of the test with 125-micron IS Sieve, calculate the percentage of material passing through the sieve by deducting from 100 the percentage of the material retained on it and other sieves.

A-2.3 Carry out the test in duplicate and report the average of the test results for each sieve.

ANNEX B

(Clause 5.3)

DETERMINATION OF MOISTURE CONTENT

B-1 PREPARATION OF TEST SAMPLE

B-1.1 Take the material reserved in A-1.1 and repeat the process of coning and quartering as described under D-2.2 until about 50 g of the sample is obtained.

B-2 PROCEDURE

B-2.1 Weigh accurately about 20 g of the prepared sample in a porcelain dish previously dried at 105 to 110°C and weighed to the nearest 0.01 g. Place the dish with the sample in an air-oven maintained at 105 to 110°C for half an hour, cool in a desiccator and weigh again. Repeat heating and cooling till constant mass is obtained.

B-3 CALCULATION

B-3.1 Calculate moisture content in the sample as follows:

$$\text{Moisture content, percent by mass} = \frac{(M - M_1)}{M} \times 100$$

Where,

M = mass in g of the sample taken for the test, and

M₁ = mass in g of the sample after heating.

ANNEX C

(Clause 5.4)

CHEMICAL ANALYSIS OF GLASS MAKING SANDS

C-1 QUALITY OF REAGENTS

C-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

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

C-2 PREPARATION OF TEST SAMPLE

C-2.1 Take the material reserved in **A-1.1**, and spray water so that the material is uniformly wetted and moves freely without balling. Then mix the wetted material thoroughly on a flat, non-absorbent slate or table, and spread out in a flat layer about 1 cm thick. Take portions of about 2 g of the wetted material from different parts of this flat layer so as to obtain a sample of total mass of about 50 g of the material. Retain the remainder of the flat layer in a tapered bottle.

NOTES

1 The properly wetted material generally contains 1 to 5 percent moisture, though it may vary according to the type of sand.

2 Sufficient time should be allowed for the sand to be thoroughly wetted so that it does not tend to segregate on being disturbed.

C-2.2 For the complete range of tests for chemical analysis, mix the sample obtained in C-2.1, grind finely in an agate mortar, then dry to constant mass at 110°C and store in a weighing bottle.

C-3 DETERMINATION OF LOSS IN IGNITION

C-3.1 Weigh accurately about 1 g of the test sample (*see C-2.2*) in a tared platinum crucible, introduce it into a muffle furnace and slowly raise the temperature to 950 to 1 000°C. After about 30 minutes, remove the crucible, cool in a desiccator and weigh. Repeat the ignition till constant mass is reached.

C-3.2 Calculation

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

Where,

M_1 = loss in mass in g on ignition, and

M = mass in g of the sample taken for the test.

C-4 DETERMINATION OF SILICA

C-4.0 Outline of the Method - The sample is decomposed by fusion with sodium carbonate and extracted with acid. Silica is then determined by dehydration and baking followed by hydrofluorization as usual. The residual silica is determined photometrically.

C-4.1 Reagents

C-4.1.1 Sodium Carbonate – anhydrous.

C-4.1.2 Dilute Hydrochloric Acid – 1:1, 1:3 and 1:19(v/v).

C-4.1.3 Dilute Sulphuric Acid – 1:1(v/v).

C-4.1.4 Hydrofluoric Acid – 40 percent (m/m).

C-4.1.5 Potassium Pyrosulphate – Solid.

C-4.1.6 EDTA Solution – 0.025 M approximately. Dissolve 9.31 g of disodium ethylenediaminetetraacetate dihydrate ($C_{10}H_{14}N_2O_8Na_2 \cdot 2H_2O$) in water and dilute to 1 litre.

C-4.1.7 Phenolphthalein Solution – Dissolve 1 g of phenolphthalein in 100 ml of rectified spirit.

C-4.1.8 Dilute Ammonia Solution – 1:3 (v/v).

C-4.1.9 Ferric Ammonium Sulphate Solution – Dissolve 4.82 g of ferric ammonium sulphate [$NH_4Fe(SO_4)_2 \cdot 12H_2O$] in water containing 80 ml of dilute hydrochloric acid and dilute to 500 ml in a volumetric flask.

C-4.1.10 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 litre.

C-4.1.11 Oxalic Acid Solution – Dissolve 100 g of oxalic acid in water and dilute to 1 litre.

C-4.1.12 Ascorbic Acid Solution – 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, may be used up to 5 days only.

C-4.1.13 Standard Silica Solution – 0.04 mg/ml. Prepare as follows:

- a) First prepare a stock solution with 0.4 g/l; for this purpose 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 about 10 g of sodium hydroxide, using a polyethylene beaker. Cool and dilute with water in a volumetric flask to 500 ml. Store in a polyethylene bottle.
- b) Prepare immediately before use a standard solution containing 0.04 mg/ml by diluting 10 ml of the above solution to 100 ml.

C-4.2 Procedure

C-4.2.1 Decomposition of the Sample – Weigh accurately about 0.5 g of the finely ground test sample (*see* C-2.2) into a 100-ml platinum dish. 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 until the mixture fuses. Keep at this temperature for 10 minutes and finally heat till a clear melt is obtained. Remove from the burner and cool.

C-4.2.1.1 Add about 30 ml of dilute hydrochloric acid and 0.5 ml of dilute sulphuric acid. Allow the covered dish to stand on a steam bath until disintegration of the melt is complete. Crush any large flakes with a flattened end of glass rod.

C-4.2.2 Gravimetric Determination of Insoluble Silica

C-4.2.2.1 Evaporate the solution in the dish to dryness on a steam bath and allow the dish to remain on it for some time. The evaporation may be facilitated by occasional use of a glass rod to break up the crust of salt that covers the liquid and tends to prevent further evaporation.

Transfer the dish to an air-oven maintained between 105 to 110°C and bake it for 1 hour. Cool the dish and its contents slightly, then add 20 ml of dilute hydrochloric acid (1:1). After about 1 minute, add 30 ml of hot water and stir the mixture until solution of soluble salts appears to be complete. Cover the dish and allow the mixture to digest on steam-bath for 10 minutes without stirring. Filter (Whatman No. 40) is suitable and rinse the dish with dilute hydrochloric acid (1:19) scrubbing the dish with a rubber-tipped glass rod. Wash the precipitate with hot water until free from chlorides. Reserve the filtrate and washings for determination of residual silica (*see* C-4.2.3).

C-4.2.2.2 Transfer both the filter paper and precipitate to an ignited and weighed platinum crucible. Ignite at a low temperature until the precipitate is free from carbonaceous matter and then heat in the furnace at 1050°C to constant mass (1 hour being normally sufficient). Cool and weigh (M_1).

C-4.2.2.3 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. Heat the crucible and residue, cautiously at first, over a gas flame until the disappearance of fumes and finally for 5 minutes in the furnace at 1050°C; cool and weigh. If the residue weighs more than 10 mg, repeat the treatment with dilute sulphuric and hydrofluoric acids and ignite again. Cool in a desiccator and weigh (M_2).

C-4.2.3 Photometric Determination of Residual Silica – Transfer the filtrate reserved in **C-4.2.2.1** to a 500-ml volumetric flask, make up to the mark with water and mix.

C-4.2.3.1 Transfer 20 ml of the solution prepared in **C-4.2.3** to a 100-ml volumetric flask, Add 5 ml of EDTA solution, a drop of phenolphthalein solution followed by dilute ammonia solution dropwise till the colour changes to red. Add 5 ml ferric ammonium sulphate solution and dilute to about 40 ml with water.

NOTE – pH of the solution at this stage should be from 10 to 10.5.

C-4.2.3.2 Add to the above solution 5 ml of ammonium molybdate solution, allow to stand for 10 minutes and then add 5 ml of oxalic acid solution followed immediately by 5 ml of ascorbic acid solution. Dilute to the mark with water and allow the solution to stand for 30 minutes for development of full colour.

C-4.2.3.3 Calibration curve – Transfer 0, 1, 2, 3 and 4 ml respectively of the standard silica solution to five 100-ml volumetric flasks. Dilute each to 10 ml with water. Add a drop of phenolphthalein solution, acidify with a few drops of dilute hydrochloric acid (1:3) and proceed as prescribed in **C-4.2.3.1** and **C-4.2.3.2** omitting the sample solution prepared in **C-4.2.3**. Take the required aliquots of these solution in the absorption cell, one at a time, and plot a curve showing the optical densities of solutions at 810 nm against the amount of silica in mg present in each solution.

C-4.2.3.4 Next take appropriate aliquot of the solution prepared in **C-4.2.3.1** and **C-4.2.3.2** in the absorption cell and measure its optical density at 810 nm and note from the calibration curve the amount of silica in milligrams present in it. Finally compute mass of silica (M_3) (as SiO_2) in grams in 500 ml of the solution (*see* C-4.2.3).

C-4.3 Calculation

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

Where,

M_1 = mass in g of platinum dish and contents before hydrofluoric acid treatment (**C-4.2.2.2**);

M_2 = mass in g of platinum dish and residue after hydrofluoric acid treatment (C4.2.2.3);

M_3 = mass in g silica present in 500 ml of solution (C-4.2.3.4); and

M = mass in g of sample taken for the test (C-4.2.1).

C-5 DETERMINATION OF IRON OXIDE

C-5.0 Outline of the Method – Determination of iron oxide (as Fe_2O_3) is carried out photometrically using o-phenanthroline.

C-5.1 Apparatus

C-5.1.1 Photoelectric Calorimeter

C-5.2 Reagents

C-5.2.1 Hydrofluoric Acid - 40 percent (*m/m*).

C-5.2.2 Concentrated Sulphuric Acid – see IS 266;

C-5.2.3 Sodium Carbonate – anhydrous.

C-5.2.4 Concentrated, Nitric Acid – see IS 264;

C-5.2.5 Ammonia Solution – Relative density 0.90.

C-5.2.6 Dilute Hydrochloric Acid – (1:1) (v/v).

C-5.2.7 Tartaric Acid – 10 percent (m/v) in water.

C-5.2.8 p-Nitrophenol – 1 percent (m/v), in absolute alcohol.

C-5.2.9 Hydroxylamine Hydrochloride Solution – 10 percent (m/v) in water.

C-5.2.10 o-Phenanthroline Solution – Dissolve 1 g of the reagent in 100 ml of hot water. Cool, store in a polyethylene bottle.

C-5.2.11 Standard Iron Solution – Dissolve 0.2 g of pure iron in concentrated hydrochloric acid. Dilute the solution to 100 ml. Transfer the solution to a 1000-ml volumetric flask and dilute up to the mark and mix thoroughly. Transfer 100 ml of this solution to a 1 000-ml volumetric flask and dilute with water up to the mark. One millilitre of this solution is equivalent to 0.02 mg of iron (as Fe).

C-5.3 Preparation of Sample Solution – Weigh accurately about 2.5 g of the prepared sample (C-2.2) into a clean platinum crucible. Add 15 ml of hydrofluoric acid and 6 to 8 drops of concentrated sulphuric acid to the crucible and evaporate on a sand-bath to fume. Cool, wash down the sides of the crucible with water and add further 10 ml of hydrofluoric acid. Repeat the evaporation to dryness. Heat the residue at first cautiously, then strongly and finally fuse the residue with 0.5 g of anhydrous sodium carbonate. Extract the fused mass with water and acidify with 10 ml of dilute hydrochloric acid. Wash the crucible with a 5 ml of concentrated hydrochloric acid and add to the main solution. Add 2 to 3 drops of concentrated nitric acid and boil the solution for a few minutes. Cool, transfer the solution to a 250-ml volumetric flask, make up the volume to the mark with water and mix.

C-5.3.1 Test Blank Solution – Prepare a test blank solution as above omitting the sample but using the same quantities of reagents as in C-5.3. Cool, transfer the solution to a 250-ml volumetric flask, make up the volume to the mark with water and mix.

C-5.4 Procedure for Iron Oxide – Transfer 25 ml of the solution prepared in (C-5.3) to a 100-ml volumetric flask. Add 2 drops of p-nitrophenol and 10 ml of tartaric acid solution. Neutralize with ammonia solution to yellow colour of the indicator. Acidify with a few drops of hydrochloric acid (1:1), add 2 ml of hydroxylamine hydrochloride solution and 10 ml of o-phenanthroline solution. Cool, make up to the mark with water and mix. Allow the solution to stand for about 30 minutes for full development of colour.

C-5.4.1 Calibration Curve – Transfer 25 ml each of the test blank solution (C-5.3.1) to six 100-ml volumetric flasks and add 0, 1, 2, 3, 4 and 5 ml respectively of standard iron solution. Using the same quantities of reagents and procedure as in C-5.4 make up the solutions for preparation of calibration curve. Take the required aliquots of these solutions in the

absorption cell, one at a time, and plot a curve showing the optical densities of solutions at 510 nm against the amount of iron in milligrams present in each solution.

C-5.4.2 Next take appropriate aliquot of the solution prepared in **C-5.4** in the absorption cell and measure its optical density at 510 nm and note from the calibration curve the amount of iron in milligrams present in it.

C-5.4.3 Calculation

Iron oxide (as Fe₂O₃), percent by mass = $\frac{M_1}{M} \times 0.1429$

Where,

M = mass in mg of iron content present in the aliquot taken for the test (**C-5.4.2**), and

M = mass in g of the material present in the aliquot taken for the test.

C-6 DETERMINATION OF ALUMINIUM OXIDE

C-6.0 Outline of the Method – Aluminium (also iron and titanium when present) is complexed quantitatively with excess EDTA by boiling. The excess EDTA is back-titrated with standard zinc solution at about pH 5.2 using xylenol orange indicator. The aluminium EDTA complex (also titanium EDTA complex, if present) on boiling with ammonium fluoride liberates EDTA which when titrated with standard zinc solution gives the amount of aluminium (plus titanium if present) in the solution. Due correction for titanium is carried out.

C-6.1 Reagents

C-6.1.1 Dilute Hydrochloric Acid – 1:1 (v/v), 1: 9 (v/v).

C-6.1.2 EDTA Solution – 0.025M. Dissolve 9.31 g of disodium ethylenediamine tetra acetate dihydrate (C₁₀H₁₄N₂O₈Na₂, 2H₂O) in water and dilute to 1 litre.

C-6.1.3 Methyl Orange – Dissolve 0.01 g of methyl orange in water.

C-6.1.4 Dilute Ammonia – 1: 5. Dilute 25 ml of concentrated ammonia (relative density 0.90) to 150 ml.

C-6.1.5 Acetate Buffer – pH 5.2 Dissolve 21.5 g of sodium acetate (CH₃COONa, 3H₂O) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1litre.

C-6.1.6 Standard Aluminium Solution – Weigh 0.5293 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 till solution is complete. Cool, transfer the solution to 1000-ml volumetric flask and make up to the mark with water. One millilitre of this solution is equivalent to 1.0 mg of aluminium (as Al₂O₃).

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

C-6.1.7 Standard Zinc Solution – 0.01 M Dissolve 2.193 8 g of zinc acetate [Zn(CH₃COO)₂,2H₂O] in 50 ml of water, add 0.5 ml of glacial acetic acid and 25 ml of

acetate buffer (**C-6.1.5**) and dilute to 1 litre in a volumetric flask. Find out the equivalent Al_2O_3 in g ml from the procedure given in **C-6.2** taking 20 ml of standard aluminium solution (*see C-6.1.6*).

C-6.1.8 Xylenol Orange – Dissolve 0.1 g of xylenol orange in 100 ml of water containing two drops of dilute hydrochloric acid (1 : 1).

C-6.1.9 Ammonium Fluoride – 10 percent (m/v). Dissolve 10 g of ammonium, fluoride in 100 ml of water. Add 3 drops of xylenol orange (**C-6.1.8**) and then dilute hydrochloric acid (1 : 9) dropwise till the colour changes to yellow.

C-6.2 Procedure – Transfer 50 ml aliquot of the solution (**C-5.3**) to a 250-ml conical flask. Add 10 ml of EDTA (for Al_2O_3 up to 1 percent) solution to provide an excess of at least 5 ml over the expected amount of alumina and iron (1 ml of 0.025 M EDTA = 1.25 mg Al_2O_3 approximately). Add 2 to 3 drops of methyl orange and then dilute ammonia dropwise 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 minutes. 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 minutes. Cool, add 5 ml of buffer solution and titrate the liberated EDTA with standard zinc solution.

C-6.3 Calculation

$$\text{Aluminium oxide, percent by mass} = \frac{V \times M_1}{M} \times 100 - 0.6378$$

Where,

V = volume in ml of standard zinc solution required for the titration of liberated EDTA,

M_1 = equivalent Al_2O_3 in g per ml of standard zinc solution,

M = mass in g of the material present in the aliquot taken for the test, and

D = percentage of TiO_2 present in the sample. The value is obtained from photometric determination (*see C-7.2.2*).

C-7 DETERMINATION OF TITANIUM DIOXIDE

C-7.0 Outline of the Method – The yellow coloured complex produced in acid solution of the sample by the addition of hydrogen peroxide is determined photometrically.

C-7.1 Reagents

C-7.1.1 Dilute Sulphuric Acid – 1:1 (v/v).

C-7.1.2 Phosphoric Acid – Add 400-ml of phosphoric acid (RD 1.75) to 500 ml of water, cool and dilute to 1 litre.

C-7.1.3 Hydrogen Peroxide – 20 volumes.

C-7.1.4 Standard Titanium Dioxide Solution – Weigh accurately 3.680 g of potassium titanyl oxalate [$\text{K}_2\text{TiO}(\text{C}_2\text{O}_4)_3 \cdot 2\text{H}_2\text{O}$]. 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 continue for 10 minutes. Cool, pour the solution into 750 ml of water and dilute to 1000 ml in a volumetric flask. One millilitre of this solution is equivalent to 0.5 mg of titanium dioxide (as TiO_3).

C-7.2 Procedure – Transfer 25 ml of the solution prepared in **C-5.3** to a 50-ml volumetric flask. Add 2.5 ml of dilute sulphuric acid, 5 ml of phosphoric acid and 5 ml of hydrogen peroxide. Make up the volume to the mark with water and mix.

C-7.2.1 Calibration Curve – Transfer 25 ml each of the test blank solution (**C-5.3.1**) to five 50-ml volumetric flasks and add 0, 1, 2, 4 and 6 ml respectively of the standard titanium dioxide solution, using the same quantities of reagents and procedure as in **C-7.2**. Make up the solutions for preparation of calibration curve. Take the required aliquot 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 milligrams present in each.

C-7.2.2 Take appropriate aliquot of the solution prepared in **C-7.2** 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-7.3 Calculation

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

Where,

M_1 = mass in mg of titanium dioxide content present in the aliquot taken for the test (**C-7.2.2**), and

M = mass in g of the material present in the aliquot taken for the test (**C-7.2**).

C-8 TEST FOR MANGANESE OXIDE

C-8.1 Reagents

C-8.1.1 Sulphuric Acid – 1:1 (v/v).

C-8.1.2 Potassium Periodate

C-8.1.3 Phosphoric Acid – (RD 1.75)

C-8.2 Procedure – Transfer 50 ml of solution prepared in **C-5.3** to a 250-ml beaker. Add 15 ml of sulphuric acid (1:1) and evaporate on a sand-bath. When the solution starts fuming, cool, dilute to 75 ml with water and add 5 drops of phosphoric acid. Add 0.5 g of potassium periodate and boil the solution for 10 minutes to develop the colour of permanganate. Cool, transfer to a 100-ml Nessler cylinder; make up to the mark with water and mix. Carry out a control test with 50 ml of test blank solution (**C-5.3.1**) using the same quantities of reagents and following the same procedure as with the sample solution.

C-8.2.1 The material shall be taken as having satisfied the requirement of the test if the colour produced with the sample solution is not deeper than that produced with the test blank

solution.

C-9 TEST FOR COPPER OXIDE

C-9.1 Reagents

C-9.1.1 Ammonium Citrate – Dissolve 100 g of citric acid in 150 ml of water and 210 ml of ammonia. Add a pinch of sodium diethyldithiocarbamate and extract the traces of copper with chloroform. Dilute the solution to 500 ml.

C-9.1.2 EDTA (Disodium Salt) – 10 percent (*m/v*).

C-9.1.3 Ammonium Hydroxide – 1:1 (*v/v*).

C-9.1.4 Cresol Red – 0.1 percent (*m/v*) in 50 percent rectified spirit.

C-9.1.5 Sodium Diethyldithiocarbamate – 0.1 percent (*m/v*) solution in water

C-9.1.6 Chloroform

C-9.2 Procedure – Transfer 50 ml of solution prepared in **C-5.3** to 250-ml beaker. Add 5 ml of ammonium citrate, 5 ml of EDTA solution and 2 drops of cresol red. Add ammonia dropwise until the indicator shows its alkaline colour (*pH* 8.5). Transfer the solution to a separating funnel and add 5 ml of sodium diethyldithiocarbamate solution. Shake vigorously with 10 ml of chloroform for about 2 minutes. Collect the chloroform extract into a 50-ml Nessler cylinder, make up to the mark with chloroform and mix. Carry out a control test with 50 ml of test blank solution (**C-5.3.1**) using the same quantities of reagents and following the same procedure as with the sample solution.

C-9.2.1 The material shall be taken as having satisfied the requirement of the test if the colour produced with the sample solution is not deeper than that produced with the test blank solution.

C-10 TEST FOR CHROMIUM TRIOXIDE

C-10.1 Reagents

C-10.1.1 Sodium Peroxide

C-10.1.2 Sulphuric Acid – (1:1) (*v/v*)

C-10.1.3 Sulphuric Acid – Approximately 2 N.

C-10.1.4 Diphenylcarbazide – 0.25 percent (*m/v*) solution in acetone.

C-10.1.5 Acetone

C-10.2 Procedure – Transfer 50 ml of solution prepared in **C-5.3** to a 250-ml beaker and add 5 g of sodium peroxide to render the solution strongly alkaline. Boil the solution to expel hydrogen peroxide. Filter through a filter paper (Whatman No. 41 is suitable) and wash the residue with hot water. Acidify the filtrate with sulphuric acid (1:1) using an indicator paper. Add 10 ml of 2 N sulphuric acid and boil the solution for a few minutes. Cool and transfer to a 100-ml Nessler cylinder. Add 4 ml of diphenylcarbazide solution, make up to the mark with water and mix. Carry out a control test with 50 ml of test blank solution (**C-5.3.1**) using the same quantities of reagents and following the same procedure as with the sample solution.

C-10.2.1 The material shall be taken as having satisfied the requirement of the test if the colour produced with the sample solution is not deeper than that produced with the test blank solution.

ANNEX D

(Clause 6.1)

SAMPLING OF GLASS MAKING SANDS

D-1 GENERAL REQUIREMENTS OF SAMPLING

D-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

D-1.1 Samples shall not be taken in an exposed place.

D-1.2 The sampling instrument shall be clean and dry.

D-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

D-1.4 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or any other containers on which the material has no action.

D-1.5 Each sample container, after filling, shall be sealed airtight with a suitable stopper, and marked with full details of the sample.

D-2 SCALE OF SAMPLING

D-2.1 Sampling of Bags –Whenever the material is supplied in bags, following procedure shall be followed.

D-2.1.1 Lot – All the bags containing material of the same grade and supplied at the same time shall constitute a lot.

D-2.1.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, samples shall be taken from each lot separately.

D-2.1.3 The number of bags to be selected from the lot for this purpose shall depend on the size of the lot and shall be according to Table 2.

TABLE 2 SCALE OF SAMPLING

(Clause D-2.1.3)

No. of Bags in the <i>LOT</i>	Sample size
<i>N</i>	<i>n</i>
(1)	(2)
Up to 100	3
101 to 300	4
301 to 500	5
501 to 1000	6

1001 and above	7
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D-2.1.3.1 These bags shall be selected at random from the lot. In order to ensure the randomness of selection, reference may be made to IS 4905. In case this standard is not readily available, following procedure may be adopted:

Starting from any bag in the lot, count them in one order as 1, 2, 3)
. . . . up to r and so on where r is the integral part of N/n. Every rth bag thus counted shall be withdrawn till the requisite number of bags is obtained.

D-2.1.4 From each of the bags thus selected, a small quantity of material shall be taken from different parts of the bag and shall be thoroughly mixed. The quantity of material so selected from each bag shall be sufficient to make triplicate determinations for all the requirements given in the specification.

D-2.1.5 From each of the portions representing selected bags, a small but approximately equal quantity of material shall be taken and thoroughly mixed so as to prepare a composite sample of the quantity sufficient to make triplicate determinations for all the characteristics tested on the composite sample (*see D-2.1.9*). The composite sample shall be divided into three equal parts, one for the purchaser, the other for the supplier and the third to be used as a referee sample.

D-2.1.6 The remaining portion of the material from each bag shall be divided into three equal parts each forming an individual sample. One set of individual samples shall be marked for the purchaser, the other for the supplier and the third to be used as referee samples.

D-2.1.7 All the individual and composite samples shall be transferred immediately to thoroughly dried bottles which shall be sealed air-tight with glass stoppers and labelled with all the particulars of sample.

D-2.1.8 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier to be used in case of any dispute between the two.

D-2.1.9 Tests for size grading and determination of silica and moisture contents shall be carried out on each of the individual samples. Tests for determination of all the remaining requirements given in Table 1 shall be conducted on the composite sample.

D-2.1.10 The average (\bar{X}) and range (\bar{R}), being defined as the difference between maximum and minimum values of the tests results shall be computed for the test results of silica and moisture contents. The lot shall be considered to have met the requirements for silica if the expression $(\bar{X} - 0.6 \bar{R})$ is greater than or equal to the minimum value specified in Table 1.

D-2.1.10.1 The lot shall be considered to have met the requirement for size grading if the test results on individual samples satisfy the corresponding requirements given in 4.2.

D-2.1.10.2 The lot shall be considered to have met the requirement for moisture content if the expression $(\bar{X} + 0.6 \bar{R})$ is less than or equal to the maximum value specified in **4.3**.

D-2.1.11 For declaring the conformity of the lot to the requirements of all other tests on the composite sample, the test results shall satisfy the relevant requirements given in Table 1.

D-2.1.12 The lot shall be declared as conforming to the requirements of the specification if **D-2.1.10** and **D-2.1.11** are satisfied.

D-2.2 Sampling from Bulk Heaps or Wagons

D-2.2.1 Unless otherwise agreed to between the purchaser and the supplier, draw several samples from one heap or wagon by means of a scoop of about 0.5 kg capacity from different parts namely, the front, middle and back, and at different depths. Place the separate samples thus collected in a covered cask. To obtain a representative sample for the heap or wagon, empty the cask on a level, clean and hard surface; spread the sample flat and scoop the mass together into a cone. Flatten the cone and divide into four equal parts. Remove two diagonally opposite parts. Mix the two remaining parts together and form a cone out of them again. Repeat this operation of coning and quartering till finally at least 1.5 kg of the representative sample is left.

D-2.2.1.1 Repeat this process of sampling for each heap or wagon to obtain a set of representative samples.

D-2.2.1.2 In the event of the bulk in a heap or wagon exceeding 10 tonnes, the final weight of the representative sample shall be at least 0.15 kg for each tonne of the bulk.

D-2.2.2 Test Samples and Referee Sample

D-2.2.2.1 Test samples – Divide each representative sample, as obtained under D-2.2 into three parts. Take one part of each representative sample to obtain a set of individual test samples.

D-2.2.2.2 Three sets of individual test samples, not less than 500 g each, shall be transferred immediately to thoroughly dried bottles, which shall be sealed air-tight with glass stoppers. These shall be labeled with all the particulars on samples. One set of the test samples shall be for the purchaser and the other for the supplier.

D-2.2.2.3 Referee sample – The third set of the individual test samples, bearing the seals of the purchaser and the supplier, shall constitute the referee sample, to be used in case of dispute between the purchaser and the supplier, and shall be kept at a place agreed to between the two.

D-2.2.3 Timber of Tests – All the characteristics shall be tested on representative samples.

D-2.2.4 The material in a heap or wagon shall be considered as conforming to the requirements of the specification if all the test results on the representative sample satisfy the relevant requirements given in 4.