

BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

(Not to be reproduced without the permission of BIS or used as an Indian Standard)

Draft Indian Standard

PULVERIZED FUEL ASH – SPECIFICATION

PART 1 FOR USE AS POZZOLANA IN CEMENT, CEMENT MORTAR AND CONCRETE

[*Third Revision* of IS 3812 (Part 1)]

ICS No.: 91.100.10

**Cement and Concrete
Sectional Committee, CED 2**

**Last Date for Receipt of
Comments is 15 September 2009**

FOREWORD

(Formal clauses of the foreword will be added later)

Pulverized fuel ash is a residue resulting from the combustion of ground or powdered or crushed bituminous coal or sub-bituminous coal (lignite). About 80 percent of the total ash is finely divided and get out of boiler along with flue gases and is collected by suitable technologies. This ash generally and in this standard is termed as fly ash. It is sometimes referred as chimney ash and hopper ash. The balance about 20 percent of ash gets collected at the bottom of the boiler and is taken out by suitable technologies and is referred as bottom ash. Fly ash is collected and stored in dry condition. When fly ash and/or bottom ash is carried to storage or deposition lagoon or pond in the form of water slurry and deposited, it is termed as pond ash. Whereas if fly ash and/or bottom ash is carried to a storage or deposition site in dry form and deposited, it is termed as mound ash.

Pulverized fuel ash is available in large quantities in the country as a waste product from a number of thermal power stations and industrial plants using pulverized or crushed or ground coal or lignite as fuel for boilers. The effective use of pulverized fuel ash as a pozzolana in the manufacture of and for part replacement of cement, as an admixture in cement, cement mortar and concrete, lime pozzolana mixture and products such as fly ash lime bricks, concrete blocks, asbestos cement products, etc, have been established in the country. Investigations of Indian pulverized fuel ashes have indicated greater scope for their utilization as a construction material. Greater utilization of pulverized fuel ash will lead to not only saving of scarce construction materials but also assist in solving the problem of disposal of this waste product. The investigations have also indicated the necessity to provide proper collection methods for fly ash so as to yield fly ash of quality and uniformity which are prime requirements of fly ash for use as a construction material.

This standard was first published in 1966 in three parts to cater to the requirements of fly ash for three specific uses: Part 1 covering use of fly ash as a pozzolana, Part 2 covering use of fly ash as an admixture for concrete, and Part 3 covering use of fly ash as fine aggregate for mortar and concrete. The Sectional Committee responsible for the formulation of this standard subsequently felt that the performance of fly ash as a pozzolana or an admixture or a fine aggregate, was complementary and not separable and hence requirements of fly ash for these uses should be covered by a single standard. This standard was, therefore, revised in 1981 by combining the three parts into a single standard, also incorporating the modifications found necessary based on the experience gained with the use of earlier standards. This revision classified fly ash in two grade, Grade 1 for incorporation in cement, mortar and concrete and in lime pozzolana mixture, and for manufacture of Portland pozzolana cement, and Grade 2 for incorporation in cement mortar and concrete and in lime pozzolana mixture.

Improvements have taken place over time in combustion technologies and ash collection technologies. These technological developments have resulted in improvement in ash qualities; specially the fineness and loss on ignition. Technologies have also been developed for a large number of utilizations of wide range of pulverized fuel ash. Application of technologies in the collection, transportation and deposition of ash have also resulted in availability of pulverized fuel ash in four forms, namely fly ash, bottom ash, pond ash and mound ash. The second revision was brought out in 2003 as 'Specification for Pulverized Fuel Ash' in two parts. Part 1 covering its use as pozzolana in cement, cement mortar and concrete and Part 2 covering its use as admixture in cement mortar and concrete; these parts replaced the specifications of erstwhile Grade I and Grade II. The significant modifications in the last revision were: four forms of pulverized fuel ash namely fly ash, bottom ash, pond ash and mound ash defined, new clause on beneficiation, segregation and processing of pulverized fuel ash added, chemical requirements prescribed on the basis of siliceous and calcareous pulverized fuel ash, requirements for reactive silica (as an optional test) and total chlorides added, limit for moisture content for fly ash in dry condition added, additional requirement of particle retained on 45 micron IS Sieve (wet sieving) added under physical requirements as an optional test, requirement for drying shrinkage deleted and a clause on uniformity requirement added.

This standard (Part1) covers the extraction and the physical and chemical requirements of pulverized fuel ash for use as pozzolana for manufacture of cement and for part replacement of cement in cement mortar and concrete. It clarifies that only fly ash qualifies as the only type of pulverized fuel ash suitable for above pozzolanic applications. The other part being Part 2 'For Use as admixture in cement mortar and concrete'. This third revision of the standard has been brought out to incorporate modifications found necessary in light of the use of last version of the standard and various feedback received thereon.

The significant modifications in this revision include:

- a) Definition of reactive silica along with explanatory notes has been included and the same has been specified as part of necessary requirements for the purpose of compliance to this standard.
- b) It has been further clarified through use of the term 'fly ash' in all appropriate clauses that only fly ash qualifies as the pulverized fuel ash suitable for pozzolanic purposes in accordance with this standard.
- c) It has been clarified that fly ash coarser than that given in Table 2 or its note (as applicable) shall not be beneficiated by grinding to improve its fineness for use for the purpose of this standard,
- d) Method for determination of available alkalies has been added.
- e) Method for determination of moisture content has been added.
- f) Provisions on manufacturer's certificate and marking have been modified to reflect the beneficiating agency, when involved.
- g) References to the cross-referred Indian Standards have been updated.

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.

Draft Indian Standard

PULVERIZED FUEL ASH – SPECIFICATION

PART 1 FOR USE AS POZZOLANA IN CEMENT, CEMENT MORTAR AND CONCRETE

[Third Revision of IS 3812 (Part 1)]

1 SCOPE

1.1 This standard (Part 1) covers the extraction and the physical and chemical requirements of pulverized fuel ash for use as pozzolana for manufacture of cement and for part replacement of cement in cement mortar and concrete.

1.2 Pulverized fuel ash to be used as pozzolana in cement, cement mortar and concrete in accordance with this standard shall be fly ash only which may be either in as collected condition or beneficiated, segregated or processed.

2 REFERENCES

The standards given in Annex A 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 these standards indicated in Annex A.

3 TERMINOLOGY

For the purpose of this standard, the definitions given below shall apply and for other terms, the definitions given in IS 4305 shall apply.

3.1 Pulverized Fuel Ash – Ash generated by burning of ground or pulverized or crushed coal or lignite fired boilers. It can be fly ash, bottom ash, pond ash or mound ash.

3.2 Siliceous Fly Ash – Fly ash with reactive calcium oxide less than 10 percent, by mass. Such fly ash is normally produced from burning anthracite or bituminous coal and has pozzolanic properties.

3.3 Calcareous Fly Ash – Fly ash with reactive calcium oxide not less than 10 percent by mass. Such fly ash is normally produced from lignite or sub-bituminous coal and has both pozzolanic and hydraulic properties.

3.4 Reactive Calcium Oxide (CaO) – That fraction of the calcium oxide which under normal hardening condition can form calcium silicate hydrates or calcium aluminate hydrates.

NOTE – To evaluate this fraction, the total calcium oxide content is to be reduced by the fraction calculated as calcium carbonate (CaCO_3), based on the measured carbon dioxide (CO_2) content and the fraction calculated as calcium sulphate (CaSO_4), based on the measured sulphate (SO_3) content, disregarding the SO_3 taken up by alkalis.

3.5 Fly Ash – Pulverized fuel ash extracted from flue gases by any suitable process such as by cyclone separator or electro-static precipitator.

3.6 Bottom Ash – Pulverized fuel ash collected from the bottom of boilers by any suitable process.

3.7 Pond Ash – Fly ash or bottom ash or both mixed in any proportion and conveyed in the form of water slurry and deposited in pond or lagoon.

3.8 Mound Ash – Fly ash or bottom ash or both mixed in any proportion and conveyed or carried in dry form and deposited dry.

3.9 Reactive Silica – That fraction of silicon dioxide which is soluble after treatment with hydrochloric acid (HCl) and with boiling potassium hydroxide (KOH) solution.

NOTES

1. Reactive silica is one of the parameters that determine the pozzolanic potential of a fly ash, that is its tendency to react with available calcium hydroxide to form cementitious hydration products.
2. The quantity of reactive silica is determined by subtracting from the total silicon dioxide content that fraction contained in the residue insoluble in hydrochloric acid and potassium hydroxide, both on a dry basis.

4 EXTRACTION OF FLY ASH

4.1 Fly ash may be extracted from flue gases of ground or pulverized or crushed coal or lignite fired boilers by any suitable process; such as, by cyclone separation or electrostatic precipitation; bottom ash from the boilers shall not be added to the fly ash. Fly ash collected at later stages of electro static precipitator are finer than the fly ash collected at initial stages of electro static precipitator.

NOTE – For extraction details for other types of Pulverized Fuel Ash such as bottom ash, pond ash and mound ash refer to IS 3812(Part 2).

5 BENEFICIATION, SEGREGATION AND PROCESSING OF FLY ASH

5.1 Fly ash as collected, if does not conform to the requirements given in Table 1 and Table 2 of this standard or if required otherwise, may be processed and/or beneficiated and/or segregated to modify its physical or chemical characteristics to meet the requirements given in Table 1 and Table 2, except that the fly ash coarser than that given in Table 2 or its note (as may be the case) shall not be beneficiated by grinding to improve its fineness for use for the purpose of this standard

5.2 Appropriate technologies may be applied for beneficiation, segregation and processing of fly ash to improve its properties, such as lime reactivity, loss on ignition, particle size distribution and any of other physical and/or chemical properties (see *also* 5.1). Some of the technologies presently in use are burning/removal of unburnt carbon, sieving/grading of fineness, grinding/attrition

for reducing particle size, thermal treatment and blending of fly ash of different qualities.

6 CHEMICAL REQUIREMENTS

6.1 Fly ash, shall conform to the chemical requirements given in Table 1.

Table 1 Chemical Requirements
(Clause 6.1)

SI No.	Characteristic	Requirements		Method of Test, Ref to IS	
		Siliceous fly ash	Calcareous fly ash	Annex	IS No.
(1)	(2)	(3)	(4)	(5)	(6)
i)	Silicon dioxide (SiO ₂) plus aluminium oxide (Al ₂ O ₃) plus iron oxide (Fe ₂ O ₃) in percent by mass, <i>Min</i>	70	50	-	IS 1727
ii)	Silicon dioxide (SiO ₂) in percent by mass, <i>Min</i>	35	25	-	IS 1727
iii)	¹⁾ Reactive silica in percent by mass, <i>Min</i>	20	20	B	-
iv)	Magnesium oxide (MgO) in percent by mass, <i>Max</i>	5.0	5.0	-	IS 1727
v)	Total sulphur as sulphur trioxide (SO ₃) in percent by mass, <i>Max</i>	3.0	3.0	-	IS 1727
vi)	Available alkalis as sodium oxide (Na ₂ O) in percent by mass, <i>Max</i>	1.5	1.5	C	-
vii)	Total chlorides in percent by mass, <i>Max</i>	0.05	0.05	-	IS 12423 ¹⁾
viii)	Loss on ignition in percent by mass, <i>Max</i>	5.0	5.0	-	IS 1727

1) For the purpose of this test, wherever reference to 'cement' has been made in IS 12423, it may be read as 'fly ash'

6.2 The fly ash may be supplied in dry or moist condition as mutually agreed. However, in case of dry condition, the moisture content shall not exceed 2.0 percent, when determined in accordance with Annex D. All tests for the properties specified in 6.1 shall, however, be carried out on oven dry samples.

7 PHYSICAL REQUIREMENTS

7.1 Fly ash, when tested in accordance with the methods of test specified in IS 1727, shall conform to the physical requirements given in Table 2.

Table 2 Physical Requirements
(Clause 7.1)

SI No. (1)	Characteristic (2)	Requirements (3)
i)	Fineness – specific surface in m ² /kg by Blaine's permeability method, <i>Min</i>	320
ii)	¹⁾ Particles retained on 45 micron IS sieve (wet sieving) in percent, <i>Max</i>	34
iii)	Lime reactivity – Average compressive strength in N/mm ² , <i>Min</i>	4.5
iv)	Compressive strength at 28 days in N/mm ² , <i>Min</i>	Not less than 80% of the strength of corresponding plain cement mortar cubes
v)	Soundness by autoclave test – Expansion of specimen in percent, <i>Max</i>	0.8

NOTE – Fly ash of fineness 250 m²/kg (*min*) is also permitted to be used in manufacture of Portland Pozzolana Cement by intergrinding it with Portland Cement Clinker if the fly ash when ground to fineness of 320 m²/kg or to the fineness of the resultant Portland Pozzolana Cement whichever is lower, conforms to all the requirements specified in **6** and **7** of this Standard.

1) Optional test

7.2 Uniformity Requirements

In tests on individual samples, the specific surface, particles retained on 45 micron IS Sieve (wet sieving) and lime reactivity value shall not vary more than 15 percent from the average established from the tests on the 10 preceding samples or of all preceding samples if less than 10.

8 TESTS

8.1 The sample or samples of fly ash for test shall be taken as described in **9** and shall be tested in accordance with **6** and **7**.

8.2 All tests for the properties of the fly ash shall be carried out in 'as supplied' condition. In case the fly ash supplied is to be beneficiated or segregated or processed, the tests shall be carried out only after beneficiation, segregation or processing as applicable.

8.3 Independent Testing

8.3.1 If the purchaser or his representative requires independent test, the samples shall be taken before or immediately after delivery at the option of the purchaser or his representative, and the tests shall be carried out/arranged by the purchaser in accordance with this standard. The supplier shall make available, free of charge, the fly ash required for testing.

8.3.2 After a representative sample has been drawn, tests on the sample shall be carried out as expeditiously as possible.

9 SAMPLING

9.1 Samples for Testing and by Whom to be Taken

A sample or samples for testing may be taken by the purchaser or his representative, or by any person appointed to superintend the work for purpose of which the fly ash is required or by the latter's representative.

9.2 In addition to the requirements of **9.1**, the methods and procedure of sampling shall be in accordance with IS 6491.

9.3 Facilities for Sampling and Identifying

The supplier shall afford every facility, and shall provide all labour and materials for taking and packing the samples for testing the fly ash and for subsequent identification of fly ash sampled.

10 STORAGE

Fly ash may be stored in accordance with the recommendation given in IS 4082 for cement.

11 DELIVERY

Supplies of fly ash may be made in bulk in suitable quantities mutually agreed upon between the purchaser and the supplier. Where so required by the purchaser, the fly ash may be supplied in bags (jute, jute-laminated, multiple paper or polyethylene lined) bearing the net quantity (may be 15 kg, 30 kg, 300 kg, 600 kg as agreed to between that purchaser and the supplier), supplier's name or registered trade-mark, if any. The tolerance on the quantity of fly ash in each bag or consignment shall be as per **11.2** unless mutually agreed upon between the purchaser and the supplier.

11.2 Tolerance Requirements for the Quantity of Fly Ash Packed in Bags

11.2.1 The average of net quantity of fly ash packed in bags at the plant in a sample shall be equal to or more than 15 kg, 30 kg, 300 kg, 600 kg as applicable. The number of bags in a sample shall be as given below:

<i>Batch Size</i>	<i>Sample Size</i>
100 to 150	20
151 to 280	32
281 to 500	50
501 to 1 200	80
1 201 to 3 200	125
3 201 and over	200

The bags in a sample shall be selected at random (see IS 4905).

11.2.2 The number of bags in a sample showing a minus error greater than 2 percent of the specified net quantity shall be not more than 5 percent of the bags in the sample. Also the minus error in none of such bags in the sample shall exceed 4 percent of the specified net quantity of fly ash in the bag.

NOTE – The matter given in **11.2.1** and **11.2.2** are extracts based on the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977* to which reference shall be made for full details. Any modification made in these Rules and other related Acts and Rules would apply automatically.

11.2.3 In case of a wagon or truck load of 5 to 25 t, the overall tolerance on net quantity of fly ash shall be 0 to + 0.5 percent.

12 MANUFACTURER'S CERTIFICATE

The manufacturer (the beneficiating agency, if any, otherwise source thermal power station) shall satisfy himself that the fly ash conforms to the requirements of this standard, and if requested, shall supply a certificate to this effect giving actual results.

13 MARKING

13.1 Each bag/consignment of fly ash shall be legibly and indelibly marked with the following information :

- a) Indication of the source of producer (source thermal power station);
- b) Name of beneficiating agency, if any;
- c) Name of packaging/ supplying agency;
- d) The words 'Fly Ash' and type of fly ash that is, Siliceous or Calcareous, as applicable;
- e) Net quantity in kg;
- f) Fineness (*Min*);
- g) Batch/control unit number; and
- h) Month and year of packing.

13.2 BIS Certification Marking

The fly ash may also be marked with the Standard Mark.

13.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made there under. The details of conditions under which a licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

14 REJECTION

14.1 Fly ash may be rejected if it does not comply with any of the requirements of this standard.

14.2 Fly ash in bulk storage for more than 6 months or in bags for more than 3 months after completion of tests, may be retested before use and may be rejected, if it fails to conform to any requirements of this standard.

ANNEX A
(Clause 2)**LIST OF REFERRED INDIAN STANDARDS**

<i>IS No.</i>	<i>Title</i>
264:1976	Nitric acid (<i>second revision</i>)
265:1993	Hydrochloric acid (<i>fourth revision</i>)
1070:1992	Reagent grade water (<i>third revision</i>)
1727:1967	Methods of test for pozzolanic materials (<i>first revision</i>)
2263:1979	Methods of preparation of indicator solutions (<i>first revision</i>)
2316:1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (<i>second revision</i>)
3812(Part 2):xxxx	Pulverized fuel ash – Specification: Part 2 For use as admixture in cement mortar and concrete (<i>third revision</i> <i>under preparation</i>)
4032:1985	Method of chemical analysis of hydraulic cement (<i>first</i> <i>revision</i>)
4082:1996	Recommendations on stacking and storage of construction materials and components at site (<i>second revision</i>)
4305:1967	Glossary of terms relating to pozzolana
4905:1968	Methods for random sampling
6491:1972	Methods for sampling fly ash
12423:1988	Methods for colorimetric analysis of hydraulic cement

ANNEX B
(Table 1)**METHOD FOR DETERMINATION OF REACTIVE SILICA****B-1 Reagents and Special Solutions**

B-1.1 Unless specified otherwise, pure chemicals of analytical reagent grade shall be employed in tests, and distilled water (see IS 1070) shall be used where the use of water as a reagent is intended. The following reagents and special solutions of analytical reagent grades are required (see IS 2263 and IS 2316).

B-1.1.1 Reagents

B-1.1.1.1 Hydrochloric acid – Sp gr 1.16 (conforming to IS 265).

B-1.1.1.2 Sulphuric acid – Sp gr 1.42 (conforming to IS 264).

B-1.1.1.3 Hydrofluoric acid – 40 percent.

B-1.1.1.4 Sodium peroxide – solid.

B-1.1.1.5 Sodium hydroxide

B-1.1.1.6 Sodium carbonate – solid.

B-1.1.1.7 Sodium chloride – solid.

B-1.1.2 Dilute Solutions of Reagents – Prepare the following dilute solutions by diluting the reagent with distilled water.

B-1.1.2.1 Hydrochloric acid – 1:2, 1:9, 1:19 (by volume).

B-1.1.2.2 Sulphuric acid – 1:1 (by volume).

B-1.1.2.3 Hydrofluoric acid – 1:3 (by volume).

B-1.1.3 Sodium Hydroxide Solution 4 mol/l – Dissolve 160 g of sodium hydroxide in water and make up to 1 000 ml. Store in a polyethylene flask.

B-1.1.4 Polyethylene Oxide Solution – Dissolve 0.25 g of polyethylene oxide of average modular mass 200 000 to 600 000, in 100 ml water while stirring vigorously. This solution is stable for approximately two weeks.

B-1.1.5 Boric Acid Solution Saturated – Dissolve approximately 50 g of boric acid in water and make up to 1 000 ml.

B-1.1.6 Potassium Hydroxide Solution – Dissolve 250 g of potassium hydroxide in water and make up to 1 000 ml.

B-1.1.7 Citric Acid Solution – Dissolve 10 g of citric acid in water and make up to 100 ml.

B-1.1.8 Ammonium Molybdate Solution – Dissolve 10 g of ammonium molybdate in water and make up to 100 ml.

B-1.1.9 Reducing Solution – Dissolve in water 0.15 g of 1-amino-2-hydroxy naphthalene-4 sulphonic acid ($C_{10}H_9NO_4S$), 0.7 g of sodium sulphite (anhydrous Na_2SO_3) and 9 g of sodium metabisulphite ($Na_2S_2O_5$) and make up to 100 ml. This solution will keep for a maximum of one week.

B-1.1.10 Buffer solution of pH 1.40 – Dissolve 7.505 g of amino-acetic acid (NH_2CH_2COOH) and 5.850 g of sodium chloride in water and make up to 1 000 ml. Dilute 300 ml of this solution to 1000 ml with dilute hydrochloric acid (1:99).

B-2 Determination of Silica

B-2.1 Decomposition with Sodium Peroxide

Weigh (1 ± 0.05 g) of fly ash (m_1) and 2 g of sodium peroxide into a platinum crucible and mix thoroughly with a spatula. Brush back into the mixture any particles adhering to the spatula. Cover the mixture with 1 g of sodium peroxide. Carefully preheat the covered crucible for about 2 min at the opening of the furnace before placing it in the heated zone controlled at a uniform temperature of (500 ± 10 °C). After 30 min, remove the crucible from the furnace and allow it to cool to room temperature. The sintered mass should not stick to the sides of the crucible. If it does, then repeat the decomposition at a temperature 10 °C lower than that was first used. Transfer the sintered mass to a 400 ml beaker and rinse the crucible with 150 ml cold water. Cover the beaker with a watch glass and heat until the solid is completely dissolved. Then add cautiously 50 ml of hydrochloric acid. The solution obtained shall be perfectly clear. If not, reject it and repeat the decomposition by peroxide at a temperature increased by 10 °C or for double the time in the furnace. Add to the solution 1 ml of sulphuric acid (1:1). Bring the solution to the boil and boil for 30 min.

B-2.2 Precipitation and Determination of Silica

B-2.2.1 Procedure

Evaporate to dryness the solution and allow the beaker to cool. Treat the residue with 5 ml of water and 10 ml of hydrochloric acid. While stirring, add some ashless filter paper pulp to the mixture and then 5 ml of the polyethylene oxide solution ensuring that the precipitate and the polyethylene oxide are thoroughly mixed, especially the precipitate adhering to the sides of the beaker. Stir the mixture thoroughly then add 10 ml of water, stirring briefly and leave to stand for 5 min. Then filter through a medium filter paper into a 500 ml volumetric flask and rinse with hot hydrochloric acid (1:19). Remove any precipitate adhering to the sides of the beaker using a rubber scraper. Wash the filter and precipitate at least five times with hot hydrochloric acid (1:19), then rinse with hot water, ensuring that the residue in the filter is broken up thoroughly during washing, until free from Chloride ions. Collect the washings in the same 500 ml volumetric flask. Ignite the filter and the precipitate in a platinum crucible ($1\ 175 \pm 25$ °C). Check for constant mass m_2 .

B-2.2.2 Expression of Results

The impure silica is calculated in percent from the formula:

$$\text{Impure SiO}_2 = \frac{m_2}{m_1} \times 100$$

where

- m_1 = Mass of the test portion, in g; and
 m_2 = Mass determined in accordance with **B-2.2.1**, in g.

B-2.3 Determination of Pure Silica**B-2.3.1 Procedure**

Moisten the residue obtained with about 0.5 ml to 1 ml of water. Add approximately 10 ml of hydrofluoric acid and then two drops of sulphuric acid. Evaporate in a fume cupboard over a sand bath or hot plate, then continue to heat until free from white sulphuric acid fumes. Ignite the crucible with the evaporation residue in an electric furnace ($1\ 175 \pm 25$ °C) for 10 min, leave to cool to room temperature in a desiccator and weigh (m_3). The evaporation residue is decomposed as described in **B-2.4**. If the residue obtained by this method exceeds 0.5 percent, the analysis shall be restarted and decomposition with sodium peroxide used.

B-2.3.2 Expression of Results

The pure silica content is calculated in percent from the formula:

$$\text{Pure SiO}_2 = \frac{m_2 - m_3}{m_1} \times 100$$

- m_1 = Mass of the test portion, in g,
 m_2 = Mass determined in accordance with **B-2.2.1**, in g, and
 m_3 = Mass determined in accordance with **B-2.3.1**, in g.

B-2.4 Decomposition of the Evaporation Residue

To the evaporation residue, obtained in accordance with **B-2.2.1**, add 2 g of the sodium carbonate and sodium chloride mixture and fuse to a bright red heat using a gas burner. Swirl the melt frequently until the residue is completely dissolved. Check visually that no part of the residue remains at the base of the crucible. Allow the crucible and its contents to cool, transfer to a 250 ml beaker, add about 100 ml water and acidify with a few ml of concentrated hydrochloric acid. When the decomposed mass is completely dissolved, remove the platinum crucible from the solution and rinse it with water. The solution shall be perfectly clear. If not, filter through a medium filter paper, wash, burn off the paper, ignite and then repeat the decomposition as above. Transfer the solution to the 500 ml

volumetric flask containing the filtrate and washings from the precipitation of silica in accordance with **B-2.2.1**. Fill the flask up to the mark with water. After stirring, this solution is ready for use. It is used in the photometric determination of the silica remaining in solution (see **B-2.5**).

B-2.5 Determination of Soluble Silica

B-2.5.1 Procedure

Pipette 20 ml of the solution prepared in accordance with **B-2.4** from the 500 ml volumetric flask into a polyethylene beaker already containing a magnetic stirrer bar and add 20 ml water. While stirring with the magnetic stirrer, add 15 drops of hydrofluoric acid (1:3). Stir again for at least 1 min. Then pipette 15 ml of the boric acid solution. Adjust the pH of the solution to (1.15 ± 0.05) by adding, drop by drop, sodium hydroxide or hydrochloric acid (1:2), using a pH meter calibrated with a buffer solution of similar pH value (for example 1.40). Add by a pipette 5 ml of the ammonium molybdate solution to the solution (time 0). Adjust the pH of the solution to 1.60 by adding, drop by drop, the sodium hydroxide solution or hydrochloric acid (1:2). Transfer the solution to a 100 ml volumetric flask and rinse the beaker with hydrochloric acid of pH 1.60. After 20 min, add from a pipette 5 ml of the citric acid solution, stir and leave to stand for 5 min. Then add from a pipette 2 ml of the reducing solution. Make up to volume with dilute hydrochloric acid of pH 1.60 and mix. At time 0 + 30 min measure the optical density with the photometer against a blank solution prepared in a similar way and using the same wavelength of a cell of the same optical length as used for the construction of the calibration graph. The silica concentration in mg SiO₂ per 100 ml is read from the calibration graph (m_4).

B-2.5.2 Expression of Results

The soluble silica content is calculated in percent from the formula:

$$\text{Soluble SiO}_2 = \frac{500 \times m_4 \times 100}{20 \times 1\,000 \times m_1}$$

where

m_1 = Mass of the test portion, in g; and
 m_4 = Silica content of the solution in accordance with **B-2.5** in mg SiO₂/100 ml.

B-2.6 Total Silica

B-2.6.1 Expression of Results

The total silica content is equal to the sum of the pure silica content (see **B-2.3**) and the soluble silica content (see **B-2.5**).

B-3 Determination of Insoluble Residue

B-3.1 Procedure

To (1 + 0.05 g) of fly ash (m_5), placed in a porcelain dish, add 25 ml of cold water and disperse using a glass stirring rod. Add 40 ml of hydrochloric acid. Heat the solution gently and crush the sample with the flattened end of a glass stirring rod until decomposition of the fly ash is complete. Evaporate to dryness on a water bath. Repeat the operation twice more with 20 ml hydrochloric acid. Treat the residue from the last evaporation with 100 ml of dilute hydrochloric acid (1:3). Re-heat filter through a medium filter paper and wash with almost boiling water at least ten times until free from Chloride ions. Transfer the filter and its contents to a 250 ml conical flask fitted with a bulb condenser and add 100 ml of the potassium hydroxide solution. Leave to stand for 16 h at room temperature and then boil the solution under reflux for 4 h. Filter on a medium filter paper and wash with water then with 100 ml of hydrochloric acid (1:9) and finally with almost boiling water until free from Chloride ions. Ignite at ($975 \pm 25^\circ\text{C}$) then check for constant mass (m_6). In general, an ignition period of 30 min is sufficient for obtaining constant mass.

B-3.2 Expression of Results

The insoluble residue is calculated in percent from the formula:

$$\text{Insoluble residue} = \frac{m_6}{m_5} \times 100$$

where

m_5 = Mass of the test portion, in g; and

m_6 = Mass of the ignited insoluble residue, in g.

B-4 Reactive Silicon Dioxide (SiO_2)

This is determined by subtracting from total silicon dioxide content (see **B-2.6**) that fraction contained in the insoluble residue (see **B-3**) both on dry sides.

ANNEX C
(Table 1)**METHOD FOR DETERMINATION OF AVAILABLE ALKALIES****C-1 PROCEDURE**

C-1.1 Weigh 5.0 g of the sample and 2.0 g of hydrated lime on a piece of weighing paper, carefully mix using a metal spatula, and transfer to a small plastic vial of approximately 25 ml capacity. Add 10.0 ml of water to this mixture, seal the vial by securing the cap or lid to the vial with tape (see Note), blend by shaking until the mixture is uniform, and store at $38 \pm 2^{\circ}\text{C}$.

NOTE – To ensure that moisture loss from the paste does not occur, place the sealed vial in a sealable container (such as a small sample or mason jar), add sufficient water to cover the bottom of the container, and seal.

C-1.2 Open the vial at the age of 28 days and transfer the contents to a 250 ml casserole. Break up and grind the cake with a pestle, adding a small amount of water, if necessary, so that a uniform slurry containing no lumps is obtained (see Note). Add sufficient water to make the total volume 200 ml. Let stand 1 h at room temperature with frequent stirring. Filter through a medium-textured filter paper onto a 500 ml volumetric flask. Wash thoroughly with hot water (eight to ten times).

NOTE – At times it may be necessary to break the vial and peel off the plastic from the solid cake. In such cases, care should be exercised to avoid the loss of material and to remove all solid material from the fragments of the vial. If the cake is too hard to break up and grind in the casserole, a mortar should be used.

C-1.3 Neutralize the filtrate with dilute HCl (1+3), using 1 to 2 drops of phenolphthalein solution as the indicator. Add exactly 5 ml of dilute HCl (1+3) in excess. Cool the solution to room temperature and fill the flask to the mark with distilled water. Determine the amount of sodium and potassium oxides in the solution using the flame photometric procedure, described in IS 4032, except that the standard solutions shall be made up, to contain 8 ml of calcium chloride (CaCl_2) stock solution per litre of standard solution, and the solution as prepared shall be used in place of the solution of cement.

NOTE – The standard solutions made up with 8 ml of calcium chloride (CaCl_2) stock solution contain the equivalent to 504 ppm of CaO. Tests have shown that this amount closely approximates the amount of calcium dissolved in the test solution.

C-2 CALCULATION AND REPORTING OF RESULTS

Calculate the results as percent by mass of the original sample material. Report as equivalent percentage of sodium oxide (Na_2O), calculated as follows:

$$\text{Equivalent Na}_2\text{O, \%} = \text{Na}_2\text{O, \%} + 0.658 \times \text{K}_2\text{O, \%}$$

ANNEX D
(Clause 6.2)

METHOD OF TEST FOR DETERMINATION OF MOISTURE CONTENT

D-1 PROCEDURE

D-1.1 Dry the clean empty Petri dish (approximately 100 mm diameter) at a temperature of 105 °C to 110°C and weigh it, after cooling in a desiccator. Spread uniformly, not less than 2.5±0.5g, fly ash sample, as received basis, in this Petri dish and weigh. Heat this uncovered Petri dish with fly ash in a drying oven at a temperature of 105 °C to 110°C for one hour. Cool the Petri dish with heated fly ash in a desiccator and weigh. Repeat the process until there is no further loss in mass.

D-2 CALCULATION AND REPORTING OF RESULTS

D-2.1 Calculate the percentage of moisture to the nearest 0.1% as follows:

$$\text{Moisture content, \%} = (x/y) \times 100$$

where,

x = loss in mass of fly ash during drying

y = mass of fly ash taken, as received basis