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Jawaharlal Nehru
"Step Out From the Old to the New"

IS 3812-1 (2003): Specification for Pulverized Fuel Ash,
Part 1: For Use as Pozzolana in Cement, Cement Mortar and Concrete [CED 2: Cement and Concrete]
Indian Standard

PULVERIZED FUEL ASH — SPECIFICATION

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

( Second Revision )

ICS 91.100.10

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 5
FOREWORD

This Indian Standard (Part 1) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

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 alone or along with 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, alone or along with 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, autoclaved aerated concrete blocks, etc, have been further established in the country in recent years. Recent 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 recent 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 standard. 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 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 of 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. Though the last revision of the standard pertained only to fly ash, number of important applications of fly ash were left uncovered. The Sectional Committee, therefore, felt necessary that a comprehensive specification should be brought out. Separate Indian Standards for fly ash for use in different end applications such as lime pozzolana mixture applications, sintered applications, geotechnical applications and agricultural applications are also being developed.

This revision intends to bring out the standard for pulverized fuel ash in two parts. The other part is:

Part 2 For use as admixture in cement mortar and concrete

(Continued on third cover)
Indian Standard

PULVERIZED FUEL ASH — SPECIFICATION

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

(Second Revision)

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 the standards indicated in Annex A.

3 TERMINOLOGY

For the purpose of this standard, the definitions given below shall apply and for other terms 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 Pulverized Fuel Ash — Pulverized fuel ash with reactive calcium oxide less than 10 percent, by mass. Such fly ash are normally produced from burning anthracite or bituminous coal and has pozzolanic properties.

3.3 Calcareous Pulverized Fuel Ash — Pulverized fuel ash with reactive calcium oxide not less than 10 percent by mass. Such fly ash are normally produced from lignite or sub-bituminous coal and have 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 aluminates hydrates.

NOTE — To evaluate this fraction, the total calcium oxide contents is to be reduced by the fraction calculated as calcium carbonate (CaCO3), based on the measured carbon dioxide (CO2) content and the fraction calculated as calcium sulphate (CaSO4), based on the measured sulphate (SO4) content, disregarding the SO3 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.

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 electrostatic precipitator are finer than the fly ash collected at initial stages of electrostatic 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 PULVERIZED FUEL ASH

5.1 Fly ash as collected, if does not conform to the requirements of this standard or if required otherwise, may be processed and/or beneficiated and/or segregated to modify its physical or chemical characteristics.

5.2 Appropriate technologies may be applied for beneficiation, segregation and processing of fly ash to improve its properties, such as lime reactivity, loss of ignition, particle size distribution and any of other physical and/or chemical properties. Some of the
technologies that may be used are burning/removal of unburned 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 Pulverized fuel ash, shall conform to the chemical requirements given in Table 1.

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 percent. All tests for the properties specified in 6.1 shall, however, be carried out on oven dry samples.

7 PHYSICAL REQUIREMENTS

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

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.

7.3 Notwithstanding the strength requirements specified in Table 2, mixes in which pulverized fuel ash is incorporated shall show a progressive increase in strength.

8 TESTS

8.1 The sample or samples of pulverized fuel 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 pulverized fuel ash shall be carried out as it is supplied. In case the pulverized fuel 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 pulverized fuel 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 pulverized fuel 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.

Table 1 Chemical Requirements

(Clause 6.1)

<table>
<thead>
<tr>
<th>Sl No.</th>
<th>Characteristic</th>
<th>Required Values</th>
<th>Method of Test, Ref to</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td>(2)</td>
<td>(3)</td>
<td>(4)</td>
</tr>
<tr>
<td>i)</td>
<td>Silicon dioxide (SiO₂) plus aluminium oxide (Al₂O₃) plus iron oxide (Fe₂O₃) in percent by mass, Min</td>
<td>70</td>
<td>50</td>
</tr>
<tr>
<td>ii)</td>
<td>Silicon dioxide (SiO₂) in percent by mass, Min</td>
<td>35</td>
<td>25</td>
</tr>
<tr>
<td>iii)</td>
<td>Reactive silica in percent by mass, Min</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>iv)</td>
<td>Magnesium oxide (MgO) in percent by mass, Max</td>
<td>5.0</td>
<td>5.0</td>
</tr>
<tr>
<td>v)</td>
<td>Total sulphur as sulphur trioxide (SO₃) in percent by mass, Max</td>
<td>3.0</td>
<td>3.0</td>
</tr>
<tr>
<td>vi)</td>
<td>Available alkalis as sodium oxide (Na₂O) in percent by mass, Max</td>
<td>1.5</td>
<td>1.5</td>
</tr>
<tr>
<td>vii)</td>
<td>Total chlorides in percent by mass, Max</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>viii)</td>
<td>Loss on ignition in percent by mass, Max</td>
<td>5.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>

Optional test.

For the purpose of this test, wherever reference to cement has been made, it may be read as pulverized fuel ash.

IS 3812 (Part 1) : 2003
Table 2 Physical Requirements (Clause 7.1)

<table>
<thead>
<tr>
<th>SI No.</th>
<th>Characteristic</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td></td>
<td>(2)</td>
</tr>
<tr>
<td>i)</td>
<td>Fineness-specific surface in m²/kg by Blaine’s permeability method, Min</td>
<td>320</td>
</tr>
<tr>
<td>ii)</td>
<td>Particles retained on 45 micron IS sieve (wet sieving) in percent, Max</td>
<td>34</td>
</tr>
<tr>
<td>iii)</td>
<td>Lime reactivity — Average compressive strength in N/mm², Min</td>
<td>4.5</td>
</tr>
<tr>
<td>iv)</td>
<td>Compressive strength at 28 days in N/mm², Min</td>
<td>Not less than 80 percent of the strength of corresponding plain cement mortar cubes</td>
</tr>
<tr>
<td>v)</td>
<td>Soundness by autoclave test — Expansion of specimen in percent, Max</td>
<td>0.8</td>
</tr>
</tbody>
</table>

NOTE — Fly ash of fineness 250 m²/kg (Min) is also permitted to be used in the 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, meets all the requirements specified in 6 and 7 of this standard.

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 pulverized fuel ash and for subsequent identification of pulverized fuel ash sampled.

10 STORAGE

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

11 DELIVERY

11.1 Supplies of pulverized fuel 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 pulverized fuel ash may also be supplied in bags (jute, jute-laminated, multiple paper or polyethylene lined) bearing the net mass (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 mass of pulverized fuel ash in each bag or consignment shall be as mutually agreed upon between the purchaser and the supplier.

11.2 Tolerance Requirements for the Mass of Pulverized Fuel Ash Packed in Bags

11.2.1 The average net mass of pulverized fuel 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:

<table>
<thead>
<tr>
<th>Batch Size</th>
<th>Sample Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 to 150</td>
<td>20</td>
</tr>
<tr>
<td>151 to 280</td>
<td>32</td>
</tr>
<tr>
<td>281 to 500</td>
<td>50</td>
</tr>
<tr>
<td>501 to 1200</td>
<td>80</td>
</tr>
<tr>
<td>1201 to 3200</td>
<td>125</td>
</tr>
<tr>
<td>3201 and over</td>
<td>200</td>
</tr>
</tbody>
</table>

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 mass 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 mass of pulverized fuel 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 tonne, the overall tolerance on net mass of pulverized fuel ash shall be 0 to + 0.5 percent.

11.4 The consignment may also be marked with the Standard Mark.

12 MANUFACTURER’S CERTIFICATE

The manufacturer shall satisfy himself that the pulverized fuel ash conform to the requirements of this standard, and if requested, shall supply a certificate to this effect to the purchaser or his representative.

13 MARKING

13.1 Each bag/consignment of pulverized fuel ash shall be clearly and permanently marked with the following information:

a) Identification of the manufacturer of pulverized fuel ash;
b) Type of pulverized fuel ash, that is, siliceous or calcareous as applicable;
c) Form of pulverized fuel ash, that is, fly ash and its minimum fineness as per Table 2;
d) Batch/Control unit number;
e) Net mass;
f) Month and year of packing; and
g) Any other identification mark as required by the purchaser.
13.2 BIS Certification Marking

The pulverized fuel 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 thereunder. The details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

14 REJECTION

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

14.2 Pulverized fuel 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

(Clauses 2, 3 and 4)

LIST OF REFERRED INDIAN STANDARDS

<table>
<thead>
<tr>
<th>IS No.</th>
<th>Title</th>
<th>IS No.</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>264 : 1976</td>
<td>Nitric acid (second revision)</td>
<td>4032 : 1985</td>
<td>Method of chemical analysis of hydraulic cement (first revision)</td>
</tr>
<tr>
<td>265 : 1993</td>
<td>Hydrochloric acid (fourth revision)</td>
<td>4082 : 1996</td>
<td>Recommendations on stacking and storage of construction materials and components at site (second revision)</td>
</tr>
<tr>
<td>1070 : 1992</td>
<td>Reagent grade water (third revision)</td>
<td>4305 : 1967</td>
<td>Glossary of terms relating to pozzolana</td>
</tr>
<tr>
<td>1727 : 1967</td>
<td>Methods of test for pozzolanic materials (first revision)</td>
<td>4905 : 1968</td>
<td>Methods for random sampling</td>
</tr>
<tr>
<td>3812 (Part 2) : 2003</td>
<td>Pulverized fuel ash — Specification: Part 2 For use as admixture in cement mortar and concrete (second revision)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

ANNEX B

[Table 1, Sl No. (iii)]

METHOD OF TEST FOR 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-1.1.4 Sodium peroxide — solid.
B-1.1.5 Sodium hydroxide
B-1.1.6 Sodium carbonate — solid.
B-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 1000 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 1000 ml.
B-1.1.6 Potassium Hydroxide Solution — Dissolve 250 g of potassium hydroxide in water and make up to 1000 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 sulfonic acid (C₆H₅NO₅S), 0.7 g of sodium sulphate (anhydrous Na₂SO₄) and 9 g of sodium metabisulphite (Na₂S₂O₅) 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 (NH₂CH₂COOH) and 5.850 g of sodium chloride in water and make up to 1000 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 cement (m₁) 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 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

Evaportate 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). 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 ± 25°C). Check for constant mass m₂.

B-2.2.2 Expression of Results

The impure silica is calculated in percent from the following formula:

$$\text{Impure SiO₂} = \frac{m₂}{m₁} \times 100$$

where

- \( m₁ \) = mass of the test portion, in g; and
- \( m₂ \) = 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 (1175 ± 25°C) for 10 min, leave to cool to room temperature in a desiccator and weigh \(m_1\). 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 following formula:

\[
\text{Pure SiO}_2 = \frac{m_2 - m_1}{m_1} \times 100
\]

where

- \(m_1\) = 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 \((1.15 \pm 0.05)\) 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 from 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 SiO\(_2\) 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 following formula:

\[
\text{Soluble SiO}_2 = \frac{500 \times m_4 \times 100}{20 \times 1000 \times m_1}
\]

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 SiO\(_2\)/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 cement \((m_j)\), 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 cement 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 ± 25°C) then check for constant mass ($m_4$). 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 following formula:

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

where

\[
\begin{align*}
    m_5 &= \text{mass of the test portion, in g}; \\
    m_6 &= \text{mass of the ignited insoluble residue, in g}.
\end{align*}
\]

B-4 REACTIVE SILICON DIOXIDE (SiO₂)

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

(FOREWORD)

**COMMITTEE COMPOSITION**

Cement and Concrete Sectional Committee, CED 2

<table>
<thead>
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<td>Superintendent Engineer (S &amp; S) (Alternate)</td>
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<tr>
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</tbody>
</table>

(Continued on page 9)
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National Test House, Kolkata
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The India Cements Limited, Chennai
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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.

The significant modifications in this revision includes:

a) Instead of earlier designation of fly ash as Grade I and Grade II, this Part will cover pulverized fuel ash for use as pozzolana in cement, cement mortar and concrete.

b) Four forms of pulverized fuel ash, namely, fly ash, bottom ash, pond ash and mound ash have been defined.

c) A new clause on beneficiation, segregation and processing of pulverized fuel ash has been added.

d) The chemical requirements has now been prescribed on the basis of siliceous and calcareous pulverized fuel ash.

e) Requirements for reactive silica (as an optional test) and total chlorides have been added.

f) Limit for moisture content for fly ash in dry condition has been added.

g) Additional requirement of particle retained on 45 micron IS Sieve (wet sieving) has been added under physical requirements as an optional test.

h) Requirement for drying shrinkage has been deleted.

j) A clause on uniformity requirement has also been added.

Considerable assistance has been rendered by Fly Ash Mission, Department of Science & Technology, Government of India in preparation of this standard.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

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.
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This Indian Standard has been developed from Doc : No. CED 2 (5869).

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