BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

Draft Indian Standard

Ultrafine Fly Ash – Specification

Foreword

(Formal clauses will be added later)

Ultrafine fly ash (UFFA) is a very fine pozzolanic material (pulverized fuel ash) consisting of ultrafine, glassy, spherical particles (of average size 3 to 5 micron) produced through multistage classification of selected fly ash conforming to IS 3812 (Part 1): 2013 'Pulverized fuel ash — Part 1 For use as pozzolana in cement, cement mortar and concrete (*third revision*)'. Due to the fine size of ultrafine fly ash particles, it has more reactive surface area, which helps to achieve higher early strength and lower permeability to the concrete mix due to mechanical packing effect of well graded fine particles.

The use of finer pozzolanic materials in production of concrete especially of higher grades (M 60 and above) is progressively increasing in India. Keeping this trend in view and to meet the requirement of the construction industry, the standard on silica fume, IS 15388 : 2003 'Silica Fume – Specification' was formulated earlier. The standard for another ultrafine material, metakaolin was also formulated as IS 16354 : 2015 'Metakaolin for Use in Cement, Cement Mortar and Concrete – Specification'. The advancements made during the last decade in grinding and classification technologies, aided in yet another standard on ultrafine slag namely IS 16715 : 2018 'Ultrafine GroundGranulated Blast Furnace Slag – Specification'. This new standard is also in line with the above.

The ultrafine slag as well as ultrafine fly ash, both are indigenously produced in India while majority of silica fume is imported. The performance of these ultrafine materials when used as part replacement of cement in concrete or mortars in the range of 5 to 10 percent is comparable and found to enhance the properties and durability of concrete especially its permeability.

This standard contains clauses **10.2**, **10.2.2**, **12** and **13** which call for agreement between the purchaser and the supplier.

This standard contributes to the following Sustainable Development Goal: Goal 9 'Industry, Innovation and Infrastructure' towards building resilient infrastructure, promote inclusive and sustainable industrialization and foster innovation', Goal 11 'Sustainable Cities and Communities' and Gold 13 'Climate Action'.

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, 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

ULTRAFINE FLY ASH – SPECIFICATION

Cement and Concrete Sectional Committee, CED 02	*Last Date for comments 25 December 2022*

1 SCOPE

This standard covers the chemical and physical requirements of ultrafine fly ash for use in concrete, mortar and other systems containing hydraulic cement.

2 REFERENCES

The Indian standards listed in Annex A contain provisions which through references 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 given in Annex A.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 4305, for pozzolanic materials and fly ash and the following shall apply.

3.1 Ultrafine Fly Ash

A very fine pozzolanic material mainly consisting of pulverized fuel ash (fly ash) extracted from flue gases resulting from combustion of ground or pulverized coal and collected by cyclone separator or electrostatic precipitator [*see* IS 3812 (Part 1)] and further processed and air classified to required fineness as per this standard.

3.2 Raw Material

Fly ash used in the manufacture of ultrafine fly ash shall conform to IS 3812 (Part 1).

4 MANUFACTURE

4.1 The ultrafine fly ash shall be produced by multi-stage classification of fly ash conforming to IS 3812 (Part 1), either by cyclone separators or other suitable methods so that the resulting product conforms to the specifications laid down in this standard. The residue (coarser part of fly ash) shall be suitably disposed of by the producer either for the manufacture of fly ash bricks or any other fly ash based (cement matrix) products

as per published Indian Standards or in any other standard form to avoid environmental pollution.

4.2 Ultrafine fly ash shall not be prepared/manufactured by grinding pulverized fuel ash, but only through classification of the fly ash obtained from the cyclone separator.

5 CHEMICAL REQUIREMENTS

Ultrafine fly ash shall conform to the chemical requirements given in Table 1.

Table 1 Chemical Requirements
(*Clause* 5)

Sl No	Characteristic	Requirement	Test Method
(1)	(2)	(3)	(4)
i)	Silicon dioxide (SiO_2) + Aluminium oxide (Al_2O_3) + Iron oxide (Fe_2O_3), in percent by mass, *Min*	70	IS 1727
ii)	SiO_2, in percent by mass, *Min*	35	IS 1727
iii)	Magnesium oxide, in percent by mass, *Max*	5	IS 1727
iv)	Total sulphur as sulphur anhydride (SO_3), *Max*	3	IS 1727
v)	Total chlorides, in percent by mass, *Max*	0.05	IS 4032
vi)	Loss on ignition, in percent by mass, *Max*	4	IS 1727
vii)	Moisture content, in percent by mass, *Max*	2	Annex B
viii)	Total alkalies equivalent to sodium oxide (Na_2O), in percent by mass, *Max*	1.5	Annex C

6 PHYSICAL REQUIREMENTS

Ultrafine fly ash shall conform to the physical requirements given in Table 2.

Table 2 Physical Requirements
(*Clause* 6)

Sl No (1)	Characteristic (2)	Requirement (3)	Test Method Ref to (4)
i)	Specific surface m^2/kg (by BET method), *Min*	1 500	IS 11578
ii)	Oversize percent retained on 45 micronIS sieve, *Max* (*see* Note 1)	5	IS 1727
iii)	Oversize percent retained on 45 micronIS sieve, variation from average percent, *Max* (*see* Note 1)	5	IS 1727
iv)	Compressive strength at 7 days as percent of control sample, *Min* (*see* Note 2)	95	IS 1727
v)	Compressive strength at 28 days as percent of control sample, *Min* (*see* Note 2)	110	IS 1727
vi)	Lime reactivity measured, MPa, *Min* (*see Note* 3*)*	7	IS 1727
vii)	Soundness, by autoclave test method,percent, *Max*	0.8	IS 4031 (Part 3)

NOTES

1 The average shall consist of the ten preceding tests or all of the preceding tests if the number is less than ten.

2 The determination of compressive strength of Ultrafine fly ash cement mortar shall be done as per **10** of IS 1727 with the following modifications:

a The dry materials for the standard test mortar shall be UFFA : cement : standard sand in proportion 0.1 : 0.9 : 3.0 by weight, blended intimately.

b The following quantities of materials are suggested for preparation of the mortar:
50 g: UFFA
450 g : cement
1500 g : standard sand

3 Optional method of determination of reactive silica using X-ray diffraction (XRD) may also be employed. For more details, National Council for Cement and Building Materials, Ballabgarh's established method as in their Monograph (MS-14-2010) may be referred.

7 PARTICLE SIZE

7.1 Ultrafine fly ash shall have the following particle size distribution (PSD) as determined by using laser diffraction PSD analyser.

d_{10} < 2 µm
d_{50} < 7 µm
d_{90} < 15 µm

where d_n indicates that n percent of the particles on a mass basis are below a given size (diameter).

8 SAMPLING AND CRITERIA FOR CONFORMITY

8.1 Sampling

8.1.1 The method and procedure of sampling of ultrafine fly ash shall be the same as the method given for fly ash in IS 6491. All samples whether grab or composite shall have a mass of at least 1 kg. Two grab/ composite samples shall be taken from the first 100 t of ultrafine fly ash. For each subsequent 100 t from a lot of ultrafine fly ash, one sample shall be taken. However, not less than two samples shall be taken in any sampling process.

8.1.2 The sample or samples for testing may be taken by the purchaser or his representative or by any person appointed to supervise the work for which the ultrafine fly ash is required or by the latter's representative.

8.2 Criteria for Conformity

8.2.1 The samples of ultrafine fly ash drawn as per **8.1** and then prepared as per **9** and shall be tested for chemical and physical properties as per **5** and **6** respectively.

8.2.2 Samples representing each 100 t of ultrafine fly ash shall be tested for moisture content, loss on ignition, and oversize of material in accordance with Annex B and Tables 1 and 2 respectively.

8.2.3 Testing for all other physical and chemical characteristics (except those mentioned in **8.2.2**) shall be carried out on complete samples representing not more than 400 t material each. The composite sample shall be prepared by combining portions equally from each of the 100 t samples.

8.2.4 The lot shall be considered passing if the sample meets all the requirements. The ultrafine fly ash may be rejected if it fails to meet any of the requirements of this standard. In case of dissatisfaction with the result of tests, the producer or supplier may request re-testing of the failed consignment on terms and conditions as mutually agreed between the supplier and the purchaser.

9 SAMPLE PREPARATION

9.1 The grab or composite sample drawn in accordance with **8.1** shall be mixed

thoroughly. The mixing may be done in a clean and dry laboratory mixer. The amount of ultrafine fly ash shall be 40 to 50 percent of the volume capacity of the mixer. The mixing time shall be 5 ± 1 min. It shall be ensured that no material is thrown out from the mixer during mixing. A polyethene sheet or any other suitable arrangements shall be made to keep the material in the mixer during mixing.

9.2 A sampling device of appropriative size shall be used to take material from the thoroughly mixed sample for purpose of making the test specimen. At least six random sub-samples shall be taken to prepare the test specimen.

10 PACKING

10.1 The ultrafine fly ash shall be packed in any of the following bags:

a) PP woven laminated block bottom valve sacks conforming to IS 16709,
b) Multi-wall paper sacks conforming to IS 11761,
c) HDPE/ PP woven sacks with liner conforming to IS 11652,
d) Laminated jute sacking bag or
e) Any other approved composite bag.

Bags shall be in good condition at the time of packing and inspection.

10.2 The net quantity of ultrafine fly ash per bag shall be 50 kg subject to tolerance as agreed to between the supplier and the purchaser.

10.2.1 The net quantity of ultrafine fly ash per bag may also be 25 kg, 10 kg, 5 kg, 2 kg, or 1 kg and packed in suitable bags as agreed to between the purchaser and the manufacturer.

10.2.2 Supplies of ultrafine fly ash may be made in drums or bulk by arrangement between the purchaser and the manufacturer/supplier.

11 STORAGE AND INSPECTION

11.1 The ultrafine fly ash shall be stored in such a manner so as to permit easy accessfor proper inspection and identification of each consignment.

11.2 Adequate facilities shall be provided to the purchaser for careful sampling and inspection, either at the source or at the site of the work, as may be specified by the purchaser. IS 4082 may be referred for guidance in general. Ultrafine fly ash shall be stored similar to cement/ fly ash/silica-fume/ metakaolin, depending upon the storage requirement in bags/bulk form.

12 DELIVERY

The supply of ultrafine fly ash shall be made in suitable quantities mutually agreed between the purchaser and the supplier. Where so required by the purchaser, the material shall be supplied in bags (jute laminated, multiply paper, or polyethene liner). The ultrafine fly ash may also be delivered in drum/bulk as agreed upon between the supplier and the purchaser.

13 MANUFACTURER'S CERTIFICATE

The supplier/manufacturer shall satisfy himself that the ultrafine fly ash conforms to the requirements of this standard and, if requested by the purchaser, shall furnish a

certificate to this effect, indicating the results of the tests carried out on the samples of the ultrafine fly ash.

14 MARKING

14.1 Each bag/consignments of ultrafine fly ash shall be clearly and permanently marked with the following information.

a) Manufacturer's name and his registered trade-mark;
b) The words 'ULTRAFINE FLY ASH';
c) Net quantity, in kg;
d) The words 'USE NO HOOKS' on the bags;
e) Batch/control unit number in terms of week, month and year of packing; and
f) Address of the manufacturer.

14.2 BIS Certification Marking

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

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

15 HEALTH AND SAFETY

15.1 Ultrafine fly ash is very fine powdery material, and when handled, some dust may become suspended in the air in the working area. It is therefore advisable to take all necessary precautions while handling and using the material.

15.2 Every attempt should be made to keep airborne ultrafine fly ash particles to a minimum to avoid irritation of eyes, nose and throat. Should this not be practicable, then the use of goggles, dust masks and suitable respiratory protective devices shall be made. *See* IS 14352 and IS 9473 for reference.

15.3 Similar information shall be provided in the shipping invoices accompanying the shipment of bulk ultrafine fly ash.

15.4 The ultrafine fly ash consignment shall be in good condition at the time of inspection.

ANNEX A
(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
1727 : 1967	Methods of test for pozzolanic material (*first revision*)
3812 (Part 1) : 2013	Pulverized fuel ash – Part 1: For use as pozzolana in cement, cement mortar and concrete (*third revision*)
4031 (Part 3) : 1988	Methods of physical tests for hydraulic cement: Part 3 Determination of soundness (*first revision*)
4032 : 1985	Methods of chemical analysis of hydraulic cement (*first revision*)
4082 : 1996	Stacking and storage of construction materials and components at site – Recommendations (*second revision*)
4305 : 1967	Glossary of terms relating to pozzolana
6491 : 1972	Methods of sampling fly ash
9473 : 2002 / ISO 3864 (Part 1):2002	Respiratory protective devices – Filtering half masks to protect against particles – Specification (*first revision*)
11578 : 1996	Method for determination of specific surface area of powder and porous particle using low temperature gas adsorption techniques
11652 : 2017	Textiles- high density polyethylene (HDPE)/Polypropylene (PP) woven sacks for packaging of 50 kg cement - Specification (*third revision*)
11761 : 1997	Multi- wall paper sacks for cement — Specification (*first revision*)
12174 : 1987	Specification for jute synthetic union bags for packing cement
14352 : 1996	Miners safety goggles – Specification
16709 : 2017	Textiles – Polypropylene (PP) woven, laminated, block bottom valve sacks for packaging of 50 kg cement – Specification

ANNEX B

(*Table* 1 and *Clause* 8.2.2)

METHOD OF TEST FOR DETERMINATION OF MOISTURE CONTENT

B-1 PROCEDURE

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 ± 0.5 g, ultrafine fly ash sample as received basis, in this Petri dish and weigh. Heat this uncovered Petri dish with ultrafine fly ash in a drying oven at a temperature of 105 °C to 110 °C for 1 h. Cool the Petri dish with heated ultrafine fly ash in a desiccator and weigh. Repeat the process until there is no further loss in mass.

B-2 CALCULATION AND REPORTING OF RESULTS

Calculate the percentage of moisture to the nearest 0.1 percent as follows:

$$\text{Moisture content, percent} = \frac{x}{y} \times 100$$

where

x = loss in mass of the ultrafine fly ash during drying, g, and

y = mass of ultrafine fly ash, as received basis, g.

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 ± 2 °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 to 200 ml. Let it stand for 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 an 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 containing the equivalent of 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:

$$\text{Equivalent } Na_2O, \text{percent} = Na_2O, \text{percent} + 0.658 \times K_2O, \text{percent}$$

Annex D

(Committee composition will be added after finalization)

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