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BUREAU OF INDIAN STANDARDS

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भारतीय मानक मसौदा
परीक्षण के तरीके — सेल्यूलोज एसीटेट फ्लेक्स
(आई. एस. 7188 का पहला *पुनरीक्षण*)

Draft *Indian Standard*

METHODS OF TEST — CELLULOSE ACETATE FLAKES

(*first revision of IS 7188*)

(ICS 83.080.20)

Methods of Sampling and Test for Plastics Sectional Committee, PCD 27	Last date for Comments: **12 September 2025**

FOREWORD

(*Formal clauses to be added later*)

This standard was originally published in 1974. This revision (first) has been undertaken to:

— update the cross referred standards; and
— make the editorial corrections.

Original standard was published by taking reference from BS 2880: 1957 “Methods of testing cellulose acetate flakes” issued by the British Standards Institution.

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2: 2022 ‘Rules for rounding off numerical values (*second revision*)’.

1 SCOPE

This standard prescribes the methods of sampling and test for cellulose acetate flakes.

2 REFERENCES

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

IS No.	*Title*
IS 460 (Part 1) : 2020	Test Sieves — Specification : Part 1 Wire cloth test sieves (*fourth revision*)

IS 460 (Part 2) : 2020	Test Sieves — Specification : Part 2 Perforated plate test sieves (*fourth revision*)
IS 1607 : 2013	Methods of test sieving (*second revision*)
IS 2316 : 1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (*second revision*)
IS 2828: 2019 /ISO 472: 2013	Plastics - Vocabulary (*second revision*)
IS 4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 2828 shall apply.

4 SAMPLING

4.1 Lot

In any consignment all the material belonging to one batch of manufacture shall constitute a lot. Each lot shall be tested separately for judging the conformity to the specification.

4.2 Sampling from Bulk

When the supply of cellulose acetate flakes is made in bulk, one lot shall be formed for every 5 tonnes or part thereof. Samples of material from each lot shall be collected at regular intervals when the material is unloaded. The gross sample collected from each lot shall be not less than 5 kg.

4.3 Sampling from Containers

The number of containers to be selected in the sample from each lot shall be as follows:

Table 1 Requirements for Cellulose Acetate Flakes
(*Clause* 4.3)

SI No.	*Lot Size (Number of containers) (N)*	*Sample Size (n)*
(1)	(2)	(3)
1.	Up to 25	5
2.	26 to 50	10
3.	51 to 100	15
4.	101 to 200	20
5.	201 and above	25

4.3.1 These containers shall be selected at random by using random number tables (*see* IS 4905). From each of the selected container equal quality of the material shall be collected and these portions shall be thoroughly mixed to form a composite sample weighing not more than 1 kg.

4.4 Number of Tests and Criteria for Conformity

4.4.1 The gross sample collected under **4.2** or **4.3** shall be packed in airtight containers and shall be used for testing various characteristics. All these tests shall be performed on composite sample unless otherwise agreed to between the purchaser and the supplier.

4.4.2 The lot shall be declared as complying with the requirements of the standard if the test results satisfy the relevant requirements.

5 DETERMINATION OF MOISTURE

5.1 Outline of the Method

The material is heated in an oven at 105 ºC to 110 ºC for a specified period and loss in mass is calculated.

5.2 Apparatus

5.2.1 *Weighing Bottle* — squat type.

5.2.2 *Desiccator* — containing calcium chloride.

5.3 Procedure

Dry a squat-type glass weighing bottle and stopper in an oven at 105°C to 110 °C for 30 min. Cool in a desiccator over calcium chloride for 30 min and weigh accurately when cool. Transfer about 5 g of the sample into the weighing bottle, replace the stopper and weigh again. Dry the sample in an oven at 105°C to 110 °C keeping the lid partially open for a period of 4 h. Replace the cover of the bottle and cool in a desiccator for 30 min. When cool weigh the weighing bottle with contents.

5.4 Calculation

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

where

M_1 = mass, in g, of the weighing bottle;
M_2 = mass, in g, of the weighing bottle with material before drying; and
M_3 = mass, in g, of the weighing bottle with material after drying.

6 DETERMINATION OF ASH

6.1 Apparatus

6.1.1 *Silica Crucible* — capacity 100 ml.

6.1.2 *Muffle Furnace*

6.2 Procedure

6.2.1 Place a 100 ml silica crucible in a muffle furnace at 500 °C to 600 °C for 15 min. Cool the crucible in a desiccator for 30 min and record its mass accurately. Weigh approximately 10 g of the test material into the crucible. Place the crucible over a Bunsen flame until the material ignites. Remove the flame and allow the material to burn on its own. Once fuming ceases, reapply the Bunsen flame until the bottom of the crucible glows dull red. Then, transfer the crucible to a muffle furnace maintained at 500 °C to 600 °C. After complete combustion of all carbonaceous matter, remove the crucible, cool it in a desiccator for 30 min, and weigh it accurately.

6.2.2 Run a moisture determination for the sample simultaneously

6.3 Calculation

$$\text{Ash, percent by mass} = \frac{(M_3 - M_1)}{(M_2 - M_1)} \times 100 \times \frac{100}{(100 - P)}$$

where

M_1 = mass, in g, of empty crucible;
M_2 = mass, in g, of crucible with material taken for test;
M_3 = mass, in g, of crucible with ash; and
P = percent moisture present in the sample.

7 DETERMINATION OF FREE ACETIC ACID

7.1 Outline of the Method

Extract the free acid by shaking with distilled water. Titrate this against standard sodium hydroxide using bromothymol blue indicator.

7.2 Reagents

7.2.1 *Standard Sodium Hydroxide Solution* — 0.01 N.

7.2.2 *Bromothymol Blue Indicator* — 0.4 percent solution *(m/v)* in water.

7.3 Procedure

Accurately weigh 6 g of the sample to the nearest 1 mg in a 250 ml iodine flask. Add 10 ml of distilled water that has been neutralized to bromothymol blue indicator. Allow to stand for 3 h with occasional shakings. Filter through Whatman filter paper No. 1 or equivalent. Wash the residue with 50 ml of distilled water, also neutral to bromothymol blue, and collect the washings together with the filtrate. Titrate the filtrate against standard sodium hydroxide to a blue end-point.

Note — If the sample contains more than 2 percent by mass of the moisture, dry the sample before weighing.

7.4 Calculation

Free acidity (as CH_3COOH), percent by mass = $V \times N$

where

V = volume, in ml, of standard sodium hydroxide used in titration; and
N = normality of standard sodium hydroxide solution.

8 DETERMINATION OF ACETIC ACID YIELD

8.1 Outline of the Method

The finely divided cellulose acetate is allowed to stand in contact with a mixture of acetone and sodium hydroxide solution. The amount of alkali consumed for hydrolysing the material is quantified by titration.

8.2 Reagents

8.2.1 *Sodium Hydroxide solution* — carbon dioxide-free, approximately 1 N.

8.2.2 *Sulphuric Acid* — approximately 1 N.

8.2.3 *Acetone* - 95 percent (*m/m).*

8.2.4 *Phenolphthalein* — 6.5 percent solution (*m/v*) in alcohol

8.3 Procedure

Accurately weigh about 2 g of the finely powdered and dried material into a 250 ml stoppered conical flask. Distribute the test portion evenly over the base of the flask. Without lifting the flask from the workbench, carefully add 80 ml of acetone, and shake the contents for 1 h. While stirring continuously, add 50 ml of sodium hydroxide and continue shaking for 3 h. Rinse down the stopper with water, adding about 50 ml of water to the contents of the flask. Add exactly 50 ml of sulphuric acid followed by about 0.5 ml of phenolphthalein indicator. Allow to stand for 30 min with occasional shaking. Titrate the excess acid against standard sodium hydroxide. Conduct a blank determination using the same reagents and procedure, without the test sample.

8.4 Calculation

$$\text{Acetic acid yield, percent by mass} = \frac{(V_1 - V_2 \times 0.060\,05 \times N \times 100)}{M}$$

where

V_1 = volume, in ml, of sodium hydroxide solution used in the titration;
V_2 = volume, in ml, of sodium hydroxide solution used in blank;
N = normality of sodium hydroxide solution; and
M = mass, in g, of material taken for the test.

9 DETERMINATION OF VISCOSITY

9.1 Outline of the Method

The solution of known strength of the material in acetone is made and viscosity determined using a U-tube Viscometer.

9.2 Apparatus

9.2.1 *Viscometer* — U-tube.

9.3 Reagents

9.3.1 *Acetone* — 95 percent (*m/m*).

9.4 Procedure

Select an appropriate U-tube viscometer and clean it thoroughly by rinsing with suitable solvents, followed by ethyl ether. Remove each solvent by passing a current of dry air through it and ensuring that no moisture remains inside the instrument. Suspend the cleaned viscometer in the bath maintained at (25 ± 0.5) ºC, taking care that the capillary arm is vertical. Accurately weigh 6 g of the dried sample to the nearest 1 mg, and transfer it into a 250 ml stoppered bottle containing 100 ml of acetone. Stopper the bottle and shake in a linear shaker till the solution is completely dissolved. Fill the viscometer with the solution using a long pipette to minimize any wetting of the tube along the filling mark. Allow the viscometer to reach the test temperature and adjust the volume to bring the liquid to within 2 mm of the filling mark. Once the solution has reached the test temperature, apply suction or pressure to bring the liquid level up to a point at least 1 cm above the timing mark. Release the suction of pressure and measure the time required for the bottom of the meniscus to pass from the top edges of time marks.

9.5 Calculation

Viscosity at 25 ± 0.5 ºC, centistokes = $C \times t$

where

C = viscometer constant obtained by calibrating the viscometer using a solution of known viscosity; and
t = time, in sec, for the liquid to fall by gravity between the top edges of time marks.

10 DETERMINATION OF CLARITY

10.1 Procedure

Pour a 6 percent (*m/v*) solution of the material prepared as described in **9.4,** slowly through the sides into a clarity tube made of glass, having an outer diameter of 1.4 cm and wall thickness of 1 mm, closed at one end and graduated in centimetres and having a black spot of 4 mm diameter at the flat base end, taking care to avoid air bubbles. Stop pouring the solution at the point the black spot at the base becomes just invisible when viewed through the liquid column vertically against normal day light. Measure this height of liquid column and report as clarity.

11 DETERMINATION OF HEAT STABILITY

11.1 Procedure

Fill a heat-resistant glass tube, 150 mm long, 12.5 mm diameter and 1 mm wall thickness to about 50 mm with the material. Place the test tube in a bath of glycerine or other suitable mineral oil so that half of the column of the sample is above the level of the liquid in the bath. Heat the bath at a uniform rate. Once the temperature reaches 180 °C, regulate the heating rate to approximately 2 °C per minute. Observe the colour of the sample in the tube. Record the temperature at which cellulose acetate changes colour to brown. Continue heating and record the temperature at which the sample fuses.

12 DETERMINATION OF AUTOCLAVE STABILITY

12.1 Reagent

12.1.1 *Sodium Hydroxide Solution* — 0.1 N.

12.2 Procedure

Weigh (6 ± 0.1) g of the sample and transfer it into a 250 ml flask. Add 100 ml of distilled water and cover the flask with a watch glass. Prepare a blank using 100 ml of distilled water in a similar flask. Place the flask in an autoclave. Heat under a pressure of 1 kgf/cm^2 for 4 h. Cut off the heating and allow the pressure to come to normal on its own accord. Remove the flask and filter the cellulose acetate over Whatman filter paper No. 41 or equivalent. Wash and titrate the filtrate against standard sodium hydroxide solution (0.1 N) using phenolphthalein as indicator. Carry out a blank using the same amount of distilled water as in the test under identical conditions.

12.3 Calculation

Autoclave stability (as CH_3CooH) Percent by mass = $\frac{(V_1 - V_2) \times N \times 0.060\,05 \times 100}{M}$

where

V_1 = volume, in ml, of standard sodium hydroxide used for the sample,
V_2= volume, in ml, of standard sodium hydroxide used for blank,
N = normality of standard sodium hydroxide, and
M = mass, in g, of the sample taken for test.

13 DETERMINATION OF BULK DENSITY

13.1 Apparatus

13.1.1 *Funnel* — of the following dimensions:

- a) Vertical height — 98 ± 1 mm
- b) Diameter of large orifice — 95 ± 1 mm
- c) Diameter of small orifice — 44 ± 1 mm

13.1.2 *Cylindrical Measuring Vessel* — of the following dimensions:

- a) Capacity — 250 ±1 ml
- b) Internal diameter — 54 ± 1 mm
- c) Height — 108 mm approx

13.2 Procedure

Place the funnel in a vertical position and close the small orifice. Place a quantity of the sample exceeding 250 ml in the funnel. Place the measuring vessel so that its open end is coaxial with the funnel and located approximately 25 mm below the orifice. Allow the material to flow into the measuring vessel. Once the measuring vessel is full, draw a straight edge across the top to remove excess material. Weigh the contents of the vessel to the nearest 0.1 g.

13.3 Calculation

$$\text{Bulk density, g/ml} = \frac{M}{250}$$

where

M = mass, in g, of the contents of the vessel.

14 DETERMINATION OF SIEVE ANALYSIS

14.1 Procedure

Carry out the sieve analysis on a suitable portion of the material by dry rate method prescribed in IS 1607 using the following test sieves [*see* IS 460 (Parts 1 and 2)]:

850 micron, 710 micron, 500 micron, 425 micron, 355 micron, 300 micron, 250 micron, 212 micron, and 150 micron

15 DETERMINATION OF IRON AND COPPER

15.1 Iron

15.1.1 *Reagents*

15.1.1.1 *Hydrochloric acid* — 1:1

15.1.1.2 *Ammonium thiocyanate solution* — 5 percent (*m/v).*

15.1.1.3 *Standard iron solution* — *See* IS 2316.

15.1.2 ***Procedure***

Dissolve the ash obtained in **6** in 10 ml of hydrochloric acid, heat the solution to 60 °C and make distinctly ammoniacal and filter. Wash the precipitate free of chloride with ammoniacal water. Collect the filtrate in a 100 ml Nessler cylinder for estimation of copper. Re-dissolve the precipitate using 10 ml of warm Hydrochloric acid (1:1) and transfer into a 100 ml Nessler cylinder. Add 5 ml of ammonium thiocyanate solution and make up to the mark. Match the colour produced against a blank with standard iron solution of known strength.

15.1.3 Calculation

$$\text{Iron (as Fe), ppm} = \frac{V \times 100}{M}$$

where

V = volume, in ml, of standard iron solution used; and
M = mass, in g, of the moisture free sample taken.

15.2 Copper

15.2.1 Reagents

15.2.1.1 *Sodium diethyldithiocarbamate Solution* — 0.1 percent (*m/v*)

15.2.1.2 *Standard copper solution — See* IS 2316

15.2.2 *Procedure*

To the filtrate obtained from **15.1.2** add 5 ml of sodium diethyldithiocarbamate solution. Match the colour produced against a blank using standard copper solution.

15.2.3 *Calculation*

$$\text{Copper (as Cu), ppm} = \frac{V \times 100}{M}$$

where

V= volume, in ml, of standard copper sulphate solution used; and
M = mass, in g, of the moisture free sample taken.