

**BUREAU OF INDIAN STANDARDS**

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परीक्षण पद्धतियाँ — पोलिइथाइलीन मोल्डिंग सामग्री और यौगिक  
(IS 2530 का पहला पुनरीक्षण)

*Draft Indian Standard*

**METHODS OF TEST — POLYETHYLENE MOULDING MATERIALS AND  
COMPOUNDS**

*(First Revision of IS 2530)*

(ICS 83.080.20)

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Methods of Sampling and Test for Plastics  
Sectional Committee, PCD 27

Last date for receipt of comment is  
**06 January 2025**

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**FOREWORD**

*(Formal clauses to be added later)*

This standard was originally published in 1963. This revision has been brought out to update the test methods for different parameters as separate Indian Standards are already published for these parameters.

Polyethylene is the second most important material used in the manufacture of plastic goods. In recent years, the demand for polyethylene goods has been growing at a very fast rate. All the varieties of polyethylene, namely, low density, intermediate density and high density, can be moulded or extruded into a wide variety of useful products like bottles, beakers, pipes, tubes, containers and films of various thicknesses. In these products, the inertness combined with lower weight compared to that of glass, is the major advantage. Thick walled polyethylene pipes are used for transport of water due to ease of laying, resistance to corrosive soil conditions and light weight. Polyethylene also has excellent electrical properties. Its low power loss and dimensional stability, together with its relative inertness to ozone, make it an outstanding material for high frequency insulation. The chemical inertness, along with toughness of polyethylene, has led to its use as corrosion-resistant coating for tanks, drums and reaction vessels.

In reporting the result of a test or analysis 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 test for polyethylene moulding materials and polyethylene compounds.

## 2 REFERENCES

The standards listed 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 listed in Annex A.

## 3 TERMINOLOGY

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

**3.1 Apparent Density (or Bulk Density)** – The weight per unit volume of the granular material expressed as g/ml.

**3.2 Bulk Factor** – The ratio of the volume of a certain weight of the loose material to its volume after moulding under prescribed conditions. It is also the ratio of the density of the material to its apparent density.

**3.3 Density** – The density at 23 °C of a specimen prepared as prescribed and annealed in boiling water for one hour and then cooled in water to 40 °C or below.

**3.4 Elongation at Break** – The percentage strain produced in a test specimen stretched to its breaking point.

**3.5 Melt Flow Index** – The weight in grams of the material extruded in ten minutes through a standard die at a specified pressure at  $(190.0 \pm 0.5)$  °C.

**3.6 Permittivity or Dielectric Constant** – The ratio of the capacitance of a capacitor, the dielectric of which consists entirely of the material under test, to the capacitance of the same configuration of electrodes in vacuum or (practically) in air.

**3.7 Power Factor** – The ratio of power loss, in watts, of a capacitor, the dielectric of which consists entirely of the material under test, to the apparent power, in volt-amperes.

**3.8 Tensile Strength at Break** – The maximum tensile load sustained by the test specimen under prescribed conditions divided by the original cross-sectional area of the specimen at the breaking point.

**3.9 Vicat Softening Point** – The temperature at which a flat-ended needle of cross-sectional area one square millimetre loaded with 1 kg weight penetrates one millimetre into a polyethylene sample.

**3.10 Yield Stress** – The lowest stress, referred to the minimum original cross-sectional area, after which the measured stress remains sensibly constant, or the stress at the first point of inflexion in the stress-strain curve whichever is greater.

## 4 SAMPLING

**4.1** Representative samples of the material shall be obtained as prescribed in the relevant material specification.

## 5 TEST METHODS

For the measurement of properties given in col (2) of Table 1, the methods given in col (3) of Table 1 shall be used.

**Table 1**  
(Clause 5)

SI No.	Parameter	Methods of Test, Ref to IS/Annex/Other Standards	Test Condition And Supplementary Instructions
(1)	(2)	(3)	(4)
<b>Rheological Properties</b>			
1.	Melt mass-flow rate	IS 13360 (Part 4/Sec 1/Subsec 1) and IS 13360 (Part 4/Sec 1/Subsec 2)/ASTM D1238	Refer IS 7328
2.	Melt volume-flow rate		
3.	Moulding shrinkage	ISO 294-4	Parallel/Normal
4.	Density	IS 13360 (Part 3/Sec 10) and IS 13360 (Part 3/Sec 11) and IS 13360 (Part 3/Sec 12)	-
<b>Mechanical Properties</b>			
5.	Tensile modulus	IS 13360 (Part 5/Sec 2)	Test speed 1 mm/min.
6.	Yield stress		Failure with yielding.
7.	Yield strain		Test speed 50 mm/min.
8.	Nominal strain at break		Failure without yielding. Strain at break ≤ 10 percent: test speed 5 mm/min. Strain at break > 10 percent: test speed 50 mm/min.
9.	Stress at 50 percent strain		
10.	Stress at break		
11.	Strain at break		
12.	Tensile strength at yield	IS 13360 (Part 5/Sec 1)	-
13.	Tensile strength at break		-
14.	Elongation at yield		-
15.	Elongation at break		-
16.	Tensile creep modulus	ISO 899-1	Strain ≤ 0.5 percent

17.	Flexural modulus	IS 13360 (Part 5/Sec 7)	Test speed 2 mm/min.
18.	Izod impact strength	IS 13360 (Part 5/Sec 4)	-
19.	Charpy notched impact strength	IS 13360 (Part 5/Sec 5)	Edgewise impact, method 1eA. Also record type of failure.
20.	Tensile notched impact strength	ISO 8256	Only to be quoted if fracture cannot be obtained with notched Charpy test.
21.	Puncture energy	ISO 6603-2	Striker velocity 4.4 m/s. Striker diameter 20 mm. Support ring diameter 40 mm. Lubricate the striker. Clamp the specimen sufficiently to prevent any out of plane movement of its outer regions.
22.	Maximum puncture force		
23.	Shore A or D hardness	IS 13360 (Part 5/Sec 11)	-
24.	Coefficient of friction (COF)	IS 13360 (Part 11/Sec 1)	-
<b>Thermal Properties</b>			
25.	Melting temperature	ISO 11357-3	Record peak melting temperature. Use 10 °C/min heating/cooling rate.
26.	Glass transition temperature	ISO 11357-2	Record midpoint temperature. Use 10 °C/min heating/cooling rate.
27.	Vicat softening point	IS 13360 (Part 6/Sec 1)	-
28.	Temperature of deflection under load	ISO 75-2	-
29.	Coefficient of linear thermal expansion	ISO 11359-2	-
30.	Burning behaviour	IS/IEC 60695-11-10	Record one of the classifications V-0, V-1, V-2, HB, HB40 or HB75
31.	Oxygen index	ISO 4589-2	Use procedure A (top surface ignition).
32.	Heat deflection temperature	IS 13360 (Part 6/Sec 3) and IS 13360 (Part 6/Sec 17)	-
33.	Brittleness temperature	IS 13360 (Part 6/Sec 11)	-
34.	Oxidation induction time	Annex B of IS 4984	-
<b>Electrical Properties</b>			
35.	Relative permittivity	IS 4486/ IEC 62631-2-1	-
36.	Dissipation factor		-
37.	Volume resistivity	IS 3396/ IEC 62631-3-2	Measure value at 1 min.
38.	Surface resistivity		Use contacting line electrodes 1 mm to 2 mm wide, 50 mm long and 5 mm apart.

39.	Electric strength	IEC 60243 – 1	Use 20 mm diameter spherical electrodes. Immerse in transformer oil in accordance with IEC 60296. Use a voltage
40.	Comparative tracking index	IEC 60112	Use solution A.
41.	Dielectric strength	IS 2584	-
42.	Power factor and permittivity	Annex K	-
<b>Permeation Properties</b>			
43.	Water absorption	IS 13360 (Part 8/Sec 1)	-
44.	Environmental stress crack resistance	IS 13360 (Part 8/Sec 9)	-
45.	Volatile content	Annex C of IS 4984	-
46.	Gas permeability	IS 13360 (Part 8/Sec 6)	-
47.	Oxygen gas transmission rate	ISO 15105-2	-
48.	Water vapour transmission rate	ISO 2528	-
<b>Ageing Properties</b>			
49.	Oven ageing	4 of IS 7016 (Part 8)	-
50.	Natural/artificial weathering	IS 15827	-
<b>Optical Properties</b>			
51.	Haze and/or luminous transmittance	IS 13360 (Part 9/Sec 5)	-
52.	Specular gloss	IS 13360 (Part 9/Sec 7)	-
53.	Transparency	IS 13360 (Part 9/Sec 8)	-
54.	Yellowness index and/or whiteness index	IS 13360 (Part 9/Sec 9)/ASTM E313	-
55.	Colour fastness to daylight	Annex B	-
56.	Colour bleeding	IS 13360 (Part 9/Sec 10)/Annex C	-
<b>Other Properties</b>			
57.	Apparent density	IS 13360 (Part 3/Sec 2)	-
58.	Bulk factor	IS 13360 (Part 3/Sec 4)	-
59.	Carbon black content	Annex D	-
60.	Butyl rubber content	Annex E	-
61.	Colour fastness to water	Annex F	-
62.	pH, Water-soluble sulphates and water-soluble chlorides	Annex G	-

63.	Carbon black dispersion in polyethylene materials	Annex H	-
64.	Antioxidants	Annex J	-

ANNEX A  
(Clause 2)  
LIST OF REFERRED STANDARDS

<i>IS No./Other Standards</i>	<i>Title</i>
IS/ISO 105-B01 : 2014	Textiles — Tests for colour fastness Part B01 Colour fastness to light : Daylight
IS 1998 : 1962	Methods of test for thermosetting synthetic resin bonded laminated sheets
IS 2584 : 1963	Method of test for electric strength of solid insulating materials at power frequencies
IS 2824 : 2007/IEC 60112 : 2003	Method for the determination of the proof and the comparative tracking indices of solid insulating materials ( <i>second revision</i> )
IS 2828 : 2019/ISO 472 : 2013	Plastics — Vocabulary ( <i>second revision</i> )
IS 3396 : 1979	Methods of test for volume and surface resistivity of solid electrical insulating materials ( <i>first revision</i> )
IS 4486 : 1967	Recommended methods for the determination of the permittivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including metre wavelengths
IS 4984 : 2016	Polyethylene pipes for water supply — Specification ( <i>fifth revision</i> )
IS 7016 (Part 8) : 2023/ISO 1419 : 2019	Methods of test for rubber or plastics coated fabrics: Part 8 Accelerated ageing ( <i>first revision</i> )
IS 7328 : 2020	Specification for polyethylene material for moulding and extrusion ( <i>third revision</i> )
IS 13360 (Part 3/Sec 2) : 1997/ISO 60 : 1977	Plastics — Methods of testing: Part 3 Physical and dimensional properties Section 2 Determination of apparent density of material that can be poured from a specified funnel
IS 13360 (Part 3/Sec 4) : 1995	Plastics — Methods of testing: Part 3 Physical and dimensional properties Section 4 Determination of bulk factor of moulding materials
IS 13360 (Part 3/Sec 10) : 2021/ISO 1183-1 : 2019	Plastics — Methods of testing: Part 3 Physical and dimensional properties Section 10 Determination of density of non-cellular plastics — Immersion method, liquid pycnometer method and titration method ( <i>first revision</i> )
IS 13360 (Part 3/Sec 11) : 2021/ISO 1183-2 : 2019	Plastics — Methods of testing: Part 3 Physical and dimensional properties Section 11 Determination of density of non-cellular plastics — Density gradient column method ( <i>first revision</i> )
IS 13360 (Part 3/Sec 12) : 2016/ISO 1183-3 : 1999	Plastics — Methods of testing: Part 3 Physical and dimensional properties Section 12 Determination of density of non-cellular plastics — Gas pycnometer method
IS 13360 (Part 4/Sec 1/Subsec 1) : 2018/ISO 1133-1 : 2011	Plastics — Methods of testing: Part 4 Rheological properties Section 1 Determination of melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of

- thermoplastics Subsection 1 Standard method (*first revision*)
- IS 13360 (Part 4/Sec 1/Subsec 2) : 2018/ISO 1133-2 : 2011 Plastics — Methods of testing: Part 4 Rheological properties Section 1 Determination of melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics Subsection 2 Method for materials sensitive to time-temperature history and/or moisture (*first revision*)
- IS 13360 (Part 5/Sec 1) : 2021/ISO 527-1 : 2019 Plastics — Methods of testing: Part 5 Mechanical properties Section 1 Determination of tensile properties — General requirements (*second revision*)
- IS 13360 (Part 5/Sec 2) : 2017/ISO 527-2 : 2012 Plastics — Methods of testing: Part 5 Mechanical properties Section 2 Determination of tensile properties — Test conditions for moulding and extrusion plastics (*first revision*)
- IS 13360 (Part 5/Sec 4) : 2021/ISO 180 : 2019 Plastics — Methods of testing: Part 5 Mechanical properties Section 4 Determination of izod impact strength (*second revision*)
- IS 13360 (Part 5/Sec 5) : 2017/ISO 179-1 : 2010 Plastics — Methods of testing: Part 5 Mechanical properties Section 5 Determination of charpy impact properties — Non-instrumented impact test (*first revision*)
- IS 13360 (Part 5/Sec 7) : 2022/ISO 178 : 2019 Plastics — Method of testing: Part 5 Mechanical properties Section 7 Determination of flexural properties (*second revision*)
- IS 13360 (Part 5/Sec 11) : 2013/ISO 868 : 2003 Plastics — Methods of testing: Part 5 Mechanical properties Section 11 Determination of indentation hardness by means of durometer (shore hardness) (*first revision*)
- IS 13360 (Part 6/Sec 1) : 2018/ISO 306 : 2013 Plastics — Methods of testing: Part 6 Thermal properties Section 1 Determination of vicat softening temperature of thermoplastic materials (*second revision*)
- IS 13360 (Part 6/Sec 3) : 2022/ISO 75-1 : 2020 Plastics — Method of testing: Part 6 Thermal properties Section 3 Determination of temperature of deflection under load — General test method (*third revision*)
- IS 13360 (Part 6/Sec 11) : 2004/ISO 974 : 2000 Plastics — Methods of testing: Part 6 Thermal properties Section 11 Determination of the brittleness temperature by impact
- IS 13360 (Part 6/Sec 17) : 2017/ISO 75-2 : 2013 Plastics — Methods of testing: Part 6 Thermal properties Section 17 Determination of temperature of deflection under load — Plastics and ebonite (*second revision*)
- IS 13360 (Part 8/Sec 1) : 2022/ISO 62 : 2008 Plastics — Methods of testing: Part 8 Permanence/chemical properties Section 1 Determination of water absorption (*first revision*)
- IS 13360 (Part 8/Sec 6) : 1997/ISO 2556 : 1974 Plastics — Methods of testing: Part 8 Permanence/chemical properties Section 6 Determination of the gas transmission rate of films and thin sheets under atmospheric pressure — Manometric method
- IS 13360 (Part 8/Sec 9) : 2022/ISO 22088-3 : 2006 Plastics — Methods of testing: Part 8 Permanence/chemical properties Section 9 Determination



	of resistance to environmental stress cracking (ESC) — Bent strip method ( <i>first revision</i> )
IS 13360 (Part 9/Sec 5) : 1999	Plastics — Methods of testing: Part 9 Optical properties Section 5 Determination of haze and luminous transmittance of transparent plastics
IS 13360 (Part 9/Sec 7) : 2023	Plastics — Methods of test: Part 9 Optical properties Section 7 Determination of specular gloss of plastic films and solid plastics ( <i>first revision</i> )
IS 13360 (Part 9/Sec 8) : 2023	Plastics — Methods of test: Part 9 Optical properties Section 8 Determination of transparency of plastic sheeting ( <i>first revision</i> )
IS 13360 (Part 9/Sec 9) : 2001	Plastics — Methods of testing: Part 9 Optical properties Section 9 Determination of yellow index of plastics
IS 13360 (Part 9/Sec 10) : 2018	Plastics — Methods of testing: Part 9 Optical properties Section 10 Qualitative evaluation of the bleeding of colorants
IS 13360 (Part 11/Sec 1) : 1999/ISO 8295 : 1995	Plastics — Methods of testing: Part 11 Special properties Section 1 Film and sheeting — Determination of coefficients of friction
IS 15827 : 2019	Cladding films for greenhouse/polyhouse — Specification ( <i>first revision</i> )
IS/IEC 60695-11-10 : 2013	Fire hazard testing: Part 11 Test flames Section 10 50 W Horizontal and vertical flame test methods
ISO 294-4 : 2018	Plastics — Injection moulding of test specimens of thermoplastic materials Part 4: Determination of moulding shrinkage
ISO 899-1 : 2017	Plastics — Determination of creep behaviour Part 1: Tensile creep
ISO 2528 : 2017	Sheet materials — Determination of water vapour transmission rate (WVTR) — Gravimetric (dish) method
ISO 4589-2 : 2017	Plastics — Determination of burning behaviour by oxygen index Part 2: Ambient-temperature test
ISO 6603-2 : 2023	Plastics — Determination of puncture impact behaviour of rigid plastics Part 2: Instrumented impact testing
ISO 8256 : 2023	Plastics — Determination of tensile-impact strength
ISO 11357-2 : 2020	Plastics — Differential scanning calorimetry (DSC) Part 2: Determination of glass transition temperature and step height
ISO 11357-3 : 2018	Plastics — Differential scanning calorimetry (DSC) Part 3: Determination of temperature and enthalpy of melting and crystallization
ISO 11359-2 : 2021	Plastics — Thermomechanical analysis (TMA) Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature
ISO 15105-2 : 2003	Plastics — Film and sheeting — Determination of gas-transmission rate — Part 2: Equal-pressure method
IEC 60243-1 : 2013	Electric strength of insulating materials - Test methods - Part 1: Tests at power frequencies
IEC 60296 : 2020	Fluids for electrotechnical applications – Mineral insulating oils for electrical equipment

IEC 62631-2-1 : 2018	Dielectric and resistive properties of solid insulating materials - Part 2-1: Relative permittivity and dissipation factor - Technical frequencies (0.1 Hz - 10 MHz) - AC Methods
IEC 62631-3-2 : 2015	Dielectric and resistive properties of solid insulating materials - Part 3-2: Determination of resistive properties (DC methods) - Surface resistance and surface resistivity
ASTM D1238	Standard test method for melt flow rates of thermoplastics by extrusion plastometer
ASTM E313	Standard practice for calculating yellowness and whiteness indices from instrumentally measured color coordinates

## ANNEX B

(Table 1, Sl No. 55)

### DETERMINATION OF COLOUR FASTNESS TO DAYLIGHT

#### B-1 APPARATUS

**B-1.1 Exposure Case** — Shall consist of a box 90 cm × 60 cm × 5 cm covered with 3 mm window glass. The bottom of the case shall be fitted with wire mesh to allow ventilation. A wooden rack shall be fitted in the box. Battens 10 cm wide shall be placed so that the distance between any two battens is about 5 cm. The battens shall be equally spaced to allow ventilation in the box. The case shall be situated in the open, facing south in the northern hemisphere. It shall be supported on legs about 17 cm high and the case inclined to horizontal at 45 °. The case should be so placed that there is no obstruction in any direction and the test pieces should be so placed in the case that no shadows are cast on them during daytime.

#### B-2 PROCEDURE

**B-2.1** Prepare a sheet of the material as prescribed in 6.2 of IS 13360 (Part 5/Sec 2). Cut out from the sheet sufficient number of test pieces of 50 mm × 25 mm size. Cover half the portion of each test piece in a manner to protect the covered area from light. This could be done by covering the half portion with black paper. The whole set of test pieces shall be attached to a piece of cardboard.

**B-2.2** A set of eight wool standards, as specified in IS/ISO 105-B01 of size 30 mm × 5 mm, shall be pinned to another piece of cardboard and half of the portion of each standard covered with black paper.

**B-2.3** The test pieces and the wool standards shall be exposed in the exposure case. The cover of glass shall be wiped daily to avoid accumulation of dust. Observe the test pieces and standards at suitable intervals till wool standard No. 5 shows fading equivalent to Grade 4 on Geometric Grey Scale. Compare the exposed and unexposed portions of test specimens with the wool standard No. 5.

## ANNEX C

(Table 1, Sl No. 56)

## DETERMINATION OF COLOUR BLEEDING

### C-1 TEST SPECIMEN

Homogenize the material in a two-roll mixing mill for 10 min as prescribed in 6.2 of IS 13360 (Part 5/Sec 2). Cut homogenized sheet into pieces of suitable size.

### C-2 PROCEDURE

**C-2.1** Place the test specimen on a piece of colourless polyethylene sheet 75 mm square and containing no antioxidant or other additive. Cover the specimen with a 75 mm square dry filter paper. In order to ensure good contact between the filter paper, the specimen and the polyethylene sheet on which it rests, these shall be sandwiched between two pieces of glass plate 75 mm square and 5 mm thick. Keep this assembly in an air oven maintained at a temperature of  $(50 \pm 1)$  °C for  $(72 \pm 1)$  h. Remove the assembly from the oven, and separate the parts. Examine the piece of polyethylene sheet and filter paper for staining, first against a white and then against a black background.

### C-3 REPORTING

**C-3.1** If either the filter paper or the polyethylene sheet is stained, it shall be reported that colour bleeding has occurred.

## ANNEX D

(Table 1, Sl No. 59)

## DETERMINATION OF CARBON BLACK CONTENT

### D-1 APPARATUS

**D-1.1 Combustion Boat** – Made of porcelain or silica, having minimum dimensions of 75 mm length, 9 mm width and 8 mm height.

**D-1.2 Combustion Tube** – Made of hard glass, of approximately 30 mm diameter and  $(400 \pm 50)$  mm length.

**D-1.3 Gas Flow Meter** – For measuring and controlling the rate of flow of nitrogen within  $(1.7 \pm 0.3)$  litres per minute.

**D-1.4 Thermometer** – In the range 250 °C to 550 °C

**D-1.5 Furnace** – To accommodate the combustion tube and to give temperatures up to at least 500 °C.

### D-2 REAGENTS

**D-2.1 Nitrogen** – Gas (*see* IS 1747)

**D-2.2 Trichloroethylene** (*see* IS 245)

### D-3 PROCEDURE

**D-3.1** Heat the combustion boat to red heat, allow it to cool in a desiccator for at least 30 min and weigh to the nearest 0.000 1 g. Place about 1 g of the sample of the polyethylene compound, accurately weighed, in the boat. Place the boat with the sample in the middle of the combustion tube. Insert a stopper carrying a thermometer and a tube for the admission of nitrogen into one end of the combustion tube. The thermometer shall be so adjusted that its bulb is in contact with the boat. Pass nitrogen through the combustion tube at a rate of  $(1.7 \pm 0.3)$  litres per minute and maintain the same rate of flow during the subsequent heating. Place the combustion tube in the furnace and connect its outlet to two cold traps in series, both containing trichloroethylene and the first being cooled with solid carbon dioxide. Put the outlet tube from the second trap to a fume hood or to the outside atmosphere.

**D-3.2** Heat the furnace to  $(500 \pm 5)$  °C and maintain this temperature for 10 min. Disconnect the outlet tube from the cold traps. Withdraw the combustion tube containing the boat from the furnace and allow to cool for 5 min, maintaining the flow of nitrogen at the same rate as before. Remove the boat from the nitrogen inlet side of the combustion tube, allow it to cool in the desiccator for 20 min to 30 min and weigh to the nearest 0.000 1 g ( $W_1$ ). Heat the boat strongly in air to constant weight ( $W_2$ ).

### D-4 CALCULATION

$$\text{Carbon black content, percent by weight} = 100 \frac{W_1 - W_2}{W_3}$$

where

$W_1$  = weight in g of the boat before heating in air;  
 $W_2$  = weight in g of the boat after heating in air; and  
 $W_3$  = weight in g of the material taken for the test.

## ANNEX E

(Table 1, Sl No. 60)

### DETERMINATION OF BUTYL RUBBER CONTENT

#### E-1 REAGENTS

##### E-1.1 Methyl Isobutyl Ketone

**E-1.2 Petroleum Ether** – Boiling range 80 °C to 100 °C

#### E-2 PROCEDURE

**E-2.1** Weigh about one gram of the polyethylene compound to the nearest 0.001 g. Add the weighed compound to 30 ml of petroleum ether contained in a 150 ml beaker. Mark the level of liquid in the beaker and heat on a water-bath until the polyethylene compound is completely dissolved. Make the volume up to the mark with petroleum ether. Heat the contents to boiling on the water-bath, remove from the water-bath and add rapidly 30 ml of methyl isobutyl ketone with stirring, followed immediately by 30 ml of a mixture of equal parts of petroleum ether and

methyl isobutyl ketone. Cover the beaker with a watch-glass and allow to cool in a bath of water maintained at 20 °C to 25 °C for (30 ± 5) min. Filter the mixture under moderate vacuum, Wash the beaker and the residue with 25 ml of a mixture of equal parts of petroleum ether and methyl isobutyl ketone in small portions of approximately 5 ml. Do not allow the residue to dry out during the washing operations.

**E-2.2** Transfer the filtrate and washings to a weighed 150 ml dish and wash the filter flask with 10 ml of the mixed solvent. Evaporate the mixture to dryness on a water-bath. Place the dish in an oven at a temperature of 110 °C to 120 °C for 45 min. Cool in a desiccator and weigh. Repeat the drying and weighing until constant weight is obtained.

### E-3 CALCULATION

$$\text{Butyl rubber content, percent by weight} = 100 \frac{W_1}{W_2}$$

where

$W_1$  = weight in g of the residue; and

$W_2$  = weight in g of the material taken for the test.

## ANNEX F

(Table 1, Sl No. 61)

### DETERMINATION OF COLOUR FASTNESS TO WATER

#### F-1 PROCEDURE

**F-1.1** Cut a small piece from a sheet prepared as prescribed in **6.2** of IS 13360 (Part 5/Sec 2) and place it in a 100 ml beaker. Cover the specimen with approximately ten times its weight of distilled water. Keep the beaker with the contents in an air oven maintained at (70 ± 1) °C for (24 ± 1) h. Examine the water for discolouration by comparing it, against a white background, with a sample of distilled water.

#### F-2 REPORTING

If the water is free from colour, it shall be reported that the colour fastness of the material to water is satisfactory.

## ANNEX G

(Table 1, Sl No. 62)

### DETERMINATION OF pH, WATER-SOLUBLE SULPHATES AND WATER-SOLUBLE CHLORIDES

#### G-1 PREPARATION OF WATER EXTRACT

**G-1.1** Reduce the sample of the material to a fine powder so that the major portion passes through 1.18 mm IS Sieve [see IS 460 (Part 1)]. Take about 4 g to 5 g of the sieved material in a 250 ml glass stoppered flask and cover with ten times its weight of boiling demineralized

water (or properly prepared distilled water). Stopper the flask and shake so that the material is thoroughly wetted. Allow it to cool for 1 h with occasional shaking. Decant the water extract and make the volume to 500 ml. Determine the pH, water-soluble sulphates and water-soluble chlorides as prescribed in **G-2**, **G-3** and **G-4**.

## **G-2 DETERMINATION OF pH**

**G-2.1** Determine the pH of the water extract to the nearest 0.1 unit without delay by any suitable method, provided the determination is completed within 30 min of the preparation of the extract.

## **G-3 DETERMINATION OF WATER-SOLUBLE SULPHATES**

### **G-3.1 Reagents**

**G-3.1.1** *Concentrated Hydrochloric Acid* – Conforming to IS 265

**G-3.1.2** *Barium Chloride Solution* – 10 percent

### **G-3.2 Procedure**

**G-3.2.1** Take 100 ml of the water extract and make it acidic with concentrated hydrochloric acid adding 4 ml of the acid in excess. Dilute in a beaker to about 200 ml with water and heat to boiling. Remove the solution from the source of heat and add barium chloride solution in a fine stream. Add the reagent in slight excess [10 ml will precipitate about 0.6 g of sulphates (as Na<sub>2</sub>SO<sub>4</sub>)]. Place the beaker on a steam-bath and allow the precipitate to settle for about 4 h. Filter the precipitate through a weighed Gooch crucible. Wash the precipitate with hot water until it is free from chloride ions. Dry and ignite over a burner or in a muffle furnace for half an hour. Cool in a desiccator and weigh.

### **G-3.3 Calculation**

$$\text{Sulphates (as Na}_2\text{SO}_4\text{), percent by weight} = \frac{304.3 W_1}{W_2}$$

where

$W_1$  = weight in g of the precipitate; and

$W_2$  = weight in g of the sieved material taken for the test in **G-1**.

## **G-4 DETERMINATION OF WATER-SOLUBLE CHLORIDES**

### **G-4.1 Reagents**

**G-4.1.1** *Dilute Nitric Acid* – Approximately 5 N

**G-4.1.2** *Standard Silver Nitrate Solution* – 0.1 N

**G-4.1.3** *Standard Ammonium Thiocyanate Solution* – 0.1 N

**G-4.1.4 Ferric Ammonium Sulphate Solution** – Saturated solution in distilled water

#### **G-4.2 Procedure**

**G-4.2.1** Take 50 ml of the water extract in a beaker and dilute to 100 ml. Add 5 ml of dilute nitric acid and then add 10 ml of standard silver nitrate solution. Stir to coagulate the precipitate. Filter, and wash the precipitate with dilute nitric acid. Add to the combined filtrate and washings 2 ml to 3 ml of ferric ammonium sulphate solution and titrate the excess of silver nitrate with standard ammonium thiocyanate solution. Carry out a blank titration at the same time.

#### **G-4.3 Calculation**

$$\text{Chlorides (as NaCl), percent by weight} = \frac{58.46 (10 N_1 - VN_2)}{W}$$

where

$N_1$  = normality of standard silver nitrate solution;  
 $V$  = volume in ml of standard ammonium thiocyanate solution used in the titration;  
 $N_2$  = normality of standard ammonium thiocyanate solution; and  
 $W$  = weight in g of the sieved material taken for the test in **G-1**.

### **ANNEX H**

(Table 1, Sl No. 63)

#### **DETERMINATION OF CARBON BLACK DISPERSION IN POLYETHYLENE MATERIALS**

##### **H-1 PROCEDURE**

**H-1.1** Place two clean microscope slides on a hot plate maintained at 170 °C to 210 °C. Place three specimens of pin-head size, each weighing about 5 mg, and each one cut from a separate granule, on one of the hot microscope slides, about 20 mm apart. Place a piece of metal shim 40 mm long, 20 mm wide and 0.03 mm thick at each end and cover the whole with the other hot microscope slide. Press the specimens by applying even pressure for  $1\frac{1}{2}$  min to 2 min, to the whole area of the face of the upper slide. After the specimens have been placed on the slides, these shall not remain on the hot plate for more than 3 min.

**H-1.2** When the slides are cool enough to be handled, examine the three specimens through a microscope at a magnification of  $(200 \pm 10)$  with a field of view of  $(1 \pm 0.1)$  mm diameter. Compare each specimen with the photomicrograph shown in Fig. 1 in respect of number and size of agglomerates. Note shall also be made of any lack of uniformity of the background.



*Courtesy: British Standards Institution*

FIG. 1 A PORTION OF CARBON BLACK DISPERSION AS SEEN WITH A FIELD OF VIEW OF 1 mm UNDER 200 MAGNIFICATION

## H-2 REPORTING

**H-2.1** The carbon black dispersion in the material under test shall be considered to be satisfactory, if the specimens show a uniform background free from white streaks and if the number and size of agglomerates in the specimens are not greater than those shown in Fig. 1.

## ANNEX J

(Table 1, Sl No. 64)

### DETERMINATION OF ANTIOXIDANTS

**J-1** In this standard, methods for determination of N-N'-di-2-naphthyl-*p*-phenylenediamine (DNPd) and phenolic antioxidants have been given. These antioxidants are generally used in polyethylene compounds.

### J-2 DETERMINATION OF N-N'-DI-2-NAPHTHYL-*p*-PHENYLENEDIAMINE (DNPd)

#### J-2.1 Reagents

**J-2.1.1** *Toluene* – Conforming to IS 537

**J-2.1.2** *Ethyl Alcohol* – 95 percent

**J-2.1.3** *Hydrogen Peroxide-sulphuric Acid Mixture*

Add 25 ml of sulphuric acid (20 percent v/v) to 4 ml of hydrogen peroxide (100 volume strength), or to an equivalent volume of hydrogen peroxide of lower concentration and dilute to 100 ml with distilled water.

#### J-2.2 Procedure



### J-2.2.1 Absorptiometer Method

#### J-2.2.1.1 Preparation of Calibration Curve

Weigh accurately 0.1 g of DNPD in a 100 ml graduated flask. Add 10 ml of Toluene and make the volume to 100 ml with Ethyl alcohol. Shake the flask till all the antioxidant is dissolved (Solution A). Prepare a solution approximately 300 ml of ethyl alcohol and toluene in the ratio of 9 : 1 by volume (Solution B). Pipette 10 ml of solution A into a 100-ml flask and make the volume to 100 ml with solution B (Solution C). Pipette 0, 1, 2, ..... 8 ml of solution C into a clean test tube and make up the volume to 20 ml accurately with solution B. Add 2 ml of Hydrogen peroxide-sulphuric acid mixture to all test tubes. Measure the optical density of the resulting green solutions as given in J-2.2.1.3. Plot a graph of optical density against mg of DNPD/22 ml of solution. This graph covers the range 0 to 0.8 mg of DNPD/22 ml of the final solution.

**J-2.2.1.2** Weigh about one gram of the polyethylene compound in the form of small pieces or thin shavings to the nearest 0.01 g in a clean 50 ml round bottom flask containing 2 g of glass beads. Add 10 ml of toluene to the flask and fit it with a condenser. Heat the contents on a boiling water-bath and reflux it for one to one and a half hour. Wash the condenser with 15 ml to 20 ml of ethyl alcohol, remove the flask, stopper it with a glass stopper, and shake vigorously to precipitate any dissolved polymer. Cool and filter the solution into a 100 ml graduated flask. Wash the flask and the filter paper with ethyl alcohol until the total volume of filtrate is 100 ml.

**J-2.2.1.3** Take 20 ml of the above solution in a test tube, add 2.0 ml of hydrogen peroxide-sulphuric acid mixture, mix well and allow to stand. Measure the optical density of the resulting green solution at intervals against a blank of the reagents, using light of wavelength approximately 430 m $\mu$ . Record the maximum reading as the optical density (*see Note*). Convert the optical density to weight in grams of DNPD per 20 ml of original solution by means of a calibration curve prepared as given in J-2.2.1.1, relating optical density to concentration of DNPD.

NOTE — The maximum optical density is usually reached in 28 min to 45 min.

#### J-2.2.1.4 Calculation

$$\text{DNPD antioxidant content, percent by weight} = \frac{500A}{W}$$

where

A = amount in g of DNPD present in 20 ml of the original solution as read from the calibration curve; and

W = weight in g of the material taken for the test.

### J-2.2.2 Visual Comparator Method

As an alternative to the use of the absorptiometer method, it is permissible to use a colour comparator for making a visual comparison of the colour of the sample solution with that of a series of solutions of known and varied concentrations of antioxidant to which has been added

sulphuric acid-hydrogen peroxide mixture. In this alternative procedure, the standard solutions may conveniently be replaced by standard coloured glass discs.

### **J-3 DETERMINATION OF PHENOLIC ANTIOXIDANT CONTENT (DIAZO METHOD)**

#### **J-3.1 Reagents**

**J-3.1.1 Toluene** – Same as in **J-1.1.1**

**J-3.1.2 Ethyl Alcohol** – Same as in **J-1.1.2**

**J-3.1.3 Diazotized Sulphanilic Acid Solution** – Dissolve 1.91 g of colourless recrystallized sulphanilic acid in distilled water and dilute the solution to 250 ml. Dissolve 0.85 g of sodium nitrite in distilled water and dilute the solution to 250 ml. Dilute one volume of concentrated sulphuric acid with three volumes of distilled water. Mix 10 ml of sulphanilic acid solution and 2 ml of dilute sulphuric acid prepared as above in a stoppered glass test tube and Place 10 ml of sodium nitrite solution in a similar tube and cool in ice. Mix the contents of both the tubes and allow to stand in the ice-bath for 10 min. Now add with constant shaking 11 ml of freshly prepared Sodium hydroxide solution (2 N), also previously cooled in ice.

#### **J-3.2 Procedure**

##### **J-3.2.1 Preparation of Calibration Curve**

Weigh accurately 0.1 g of phenolic antioxidant in a 100 ml graduated flask and make the volume to 100 ml with ethyl alcohol. Take one millilitre of this solution in another 100-ml graduated flask and make the volume to 100 ml with ethyl alcohol. Take 0 ml, 2 ml, 4 ml, ..... 20 ml of this solution in test tubes and make the volume up to 29 ml with ethyl alcohol in all test tubes. Add one ml of toluene, 10 ml of distilled water and 2 ml of diazotized sulphanilic acid solution in all test tubes and mix well. Measure the optical density of the resulting solution as given in **J-3.2.2**. Plot a graph of optical density against mg of phenolic antioxidant/42 ml of the final solution. This graph covers the range of 0 mg to 0.2 mg of phenolic antioxidant / 42 ml of the final solution.

**J-3.2.2** Prepare the solution of the polyethylene compound as given in **J-2.2.1.2**. Take 10 ml of the solution in a test tube and make the volume to 29 ml with ethyl alcohol. Add one millilitre of toluene, 10 ml of distilled water and mix the contents well. Add 2 ml of diazotized sulphanilic acid solution, mix the contents again and allow to stand for ten minutes Measure the optical density of the resulting coloured solution. When the colour developed is orange-yellow as is the case with Dicrosylol propane and 4,4'-thiobis (3-methyl-6-tert-butylphenol), light of wavelength approximately 430 m $\mu$  shall be used.

**J-3.2.3** The optical density shall be converted to weight in grams of antioxidant per 10 ml of the original solution by means of a calibration curve prepared as given in **J-3.2.1**, relating optical density to concentration of antioxidant.

#### **J-3.3 Calculation**

$$\text{Phenolic antioxidant content, percent by weight} = \frac{1000A}{W}$$

where

$A$  = amount in g of the antioxidant present in 10 ml of the original solution as read from the calibration curve; and

$W$  = weight in g of the polyethylene compound taken for the test.

**ANNEX K**  
(Table 1, Sl No. 42)

**DETERMINATION OF POWER FACTOR AND PERMITTIVITY**

**K-1 TEST SPECIMENS**

**K-1.1** Carry out the test on at least two test specimens.

**K-1.2** Homogenize the material in a mixing mill for ten minutes. Mould a sheet of 1.0 mm thickness in the hydraulic press. The surface of the sample shall be flat and parallel and free from flaws, voids, sink marks, etc. The thickness of the sheet shall not vary by more than  $\pm 0.025$  mm from the mean value.

**K-1.3** Punch out from the sheet with punch cutter, discs of diameter  $(50 \pm 1)$  mm.

**K-2 PROCEDURE**

Determine the power factor and permittivity of the specimen at a frequency of 16 Mc/s by the method prescribed for determination of power factor and permittivity in IS 1998 or by any other method which gives the same results. But in the case of a dispute, the values obtained by the method prescribed in IS 1998 shall be taken.

**K-3 REPORTING**

Report the power factor to four places of decimal. Report the permittivity to two place of decimal.