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Indian Standard

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

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METHODS OF TEST FOR POLYETHYLENE MOULDING MATERIALS AND POLYETHYLENE COMPOUNDS

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Indian Standard

METHODS OF TEST FOR POLYETHYLENE MOULDING MATERIALS AND POLYETHYLENE COMPOUNDS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 26 August 1963, after the draft finalized by the Plastics Sectional Committee had been approved by the Chemical Division Council.

0.2 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.

0.3 The International Organization for Standardization (ISO) has published the following recommendations in the field of testing polyethylene moulding materials:

ISO/R 292-1963 PLASTICS. DETERMINATION OF MELT FLOW INDEX OF POLYETHYLENE AND POLYETHYLENE COMPOUNDS.

ISO/R 306-1963 PLASTICS. DETERMINATION OF VICAT SOFTENING POINT.

The corresponding methods of test given in this standard agree with these ISO Recommendations.

0.4 Wherever a reference to any Indian Standard appears in this standard, it shall be taken as a reference to the latest version of the standard.

0.5 Metric system has been adopted in India and all quantities and dimensions in this standard have been given in this system. Non-metric values to which the industry has been accustomed are also given in brackets, wherever necessary, for the sake of smooth changeover by December 1966.

0.6 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 : 2-1960 Rules for Rounding Off Numerical Values (*Revised*).

1. SCOPE

1.1 This standard prescribes the methods of test for polyethylene moulding materials and polyethylene compounds.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

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

2.2 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.

2.3 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.

2.4 Elongation at Break — The percentage strain produced in a test specimen stretched to its breaking point.

2.5 Melt Flow Index — The weight in grams of the material extruded in ten minutes through a standard die at a specified pressure at $190.0^\circ \pm 0.5^\circ\text{C}$.

2.6 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.

2.7 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.

2.8 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.

2.9 Vicat Softening Point — The temperature at which a flat-ended needle of cross-sectional area one square millimetre loaded with one kilogram weight penetrates one millimetre into a polyethylene sample.

2.10 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.

3. SAMPLING

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

4. DETERMINATION OF APPARENT DENSITY

4.1 Carry out the determination in duplicate.

4.2 Apparatus

4.2.1 Funnel — with 15 cm maximum diameter and the stem removed, producing a hole of 15-mm diameter at the bottom.

4.2.2 Measuring Cylinder — 500 ml capacity.

4.3 Procedure — Weigh, to the nearest 0.1 g, about 200 g of the material and pour it at a slow and steady rate into the measuring cylinder through the funnel which shall be resting on the top of the cylinder. Read off from the graduations on the measuring cylinder the volume in millilitres taken up by the material.

4.4 Calculation and Reporting — Calculate the apparent density by dividing the weight of the material by the volume occupied. The mean of two determinations shall be taken as the representative value of apparent density of the material.

5. DETERMINATION OF DENSITY AND BULK FACTOR

5.1 Density

5.1.1 Carry out the determination on two specimens prepared from the same test sample material.

5.1.2 Reagents

5.1.2.1 Dilute alcohol — having a density of 0.905 to 0.910 at 23°C, and prepared by diluting ethyl alcohol with distilled water.

5.1.3 Preparation of Test Specimens — Mould a sheet as prescribed under 6.2.1 and anneal in boiling water for one hour in a 2-litre beaker. Cool slowly in the water till the temperature falls to 40°C. Cut from the sheet two specimens of volume 10 to 15 mm³ from different parts of the sheet, using a sharp blade.

5.1.4 Procedure — Pipette 100 ml of dilute alcohol into a clean Drechsel bottle kept immersed in a water-bath maintained at $23.0^{\circ} \pm 0.1^{\circ}\text{C}$. After temperature equilibrium has been attained, lower one test specimen carefully into the dilute solution, avoiding any adhering air bubbles. Add distilled water from a burette (conforming to Class A of *IS: 1997-1961 Specification for Burettes) 0.2 ml at a time, with stirring. When the specimen remains just suspended in the solution well away from the glass surface, the density of the solution and the specimen is taken to be the same. Prepare a calibration curve for the density of dilute alcohol by adding increasing amounts of water and determining the density. Read off the density of the specimen from the calibration curve. For an accurate result, density of solution at the end may also be obtained with a pycnometer or specific gravity bottle at $23.0^{\circ} \pm 0.1^{\circ}\text{C}$. Carry out the test in a similar manner on the second specimen.

5.1.5 Calculation — Calculate the density of the two specimens to the fourth decimal place. If the two values vary by more than 0.000 5, reject the readings and repeat the results until the difference between the two values is not greater than 0.000 5. Report the mean of the two values as the density.

5.2 Bulk Factor — Calculate the bulk factor by dividing the value of density (see 5.1.5) by the value of apparent density (see 4.4).

6. DETERMINATION OF TENSILE STRENGTH, YIELD STRESS AND ELONGATION AT BREAK

6.1 Carry out the determination on six test specimens prepared from the same test sample material.

6.2 Preparation of Test Specimens

6.2.1 Milling and Moulding — The recommended milling and moulding temperatures are given in Table I. Milling shall be done on a friction ratio of 1 : 1.1 to 1.2. Homogenize the polyethylene granules in a mixing mill for a total time of ten minutes; when the plastic stage is reached, cross-mix the sample at two-minute intervals. The nip of the mixing

*Since revised.

mill shall be between 0.5 mm and 1.5 mm. A sheet shall then be compression-moulded between highly polished stainless steel plates from the homogenized material. A suitable spacer shall be used between the plates according to the required thickness of the polyethylene sheet. The moulding shall be carried out under pressure steadily increasing for five minutes and reaching a maximum of 20 kg/cm² on the sample. Maintain the material under these conditions for five more minutes and then cool under pressure till the temperature reaches 40°C. Release the pressure and withdraw the sample.

TABLE I RECOMMENDED TEMPERATURES FOR MILLING AND MOULDING

(Clause 6.2.1)

MELT FLOW INDEX	MILLING TEMPERATURE (°C) <i>Max</i>	MOULDING TEMPERATURE (°C)
(1)	(2)	(3)
0.2	160	160
0.3	150	160
2.0	140	150
7.0	130	145
20.0	120	140

6.2.2 Annealing — Anneal the sheet by immersing in boiling distilled water for ten minutes followed by cooling in air.

6.2.3 Cut the test specimens from the sheet by a suitable punch or with a sharp knife.

6.3 Apparatus

6.3.1 Tensile Testing Machine — capable of producing a specified rate of grip separation and indicating the tensile load applied on the test piece. The loading range of the tensile machine shall be such that the breaking load lies between 85 percent and 15 percent of the maximum of the scale.

6.3.2 Punch Cutting Machine — provided with an accurately machined die for cutting dumb-bell shaped test specimens as shown in Fig. 1.

6.3.3 Compression Moulding Machine — The mould plates shall be flat, evenly ground and well finished stainless steel or nickel chrome steel plates.

apart at the narrow central part of the dumb-bell. Measure the thickness of the specimen at three places between the gauge marks. Use the minimum thickness for calculating the cross-sectional area of the specimen.

6.5 Procedure

6.5.1 Adjust the tensile testing machine to give a constant rate of separation of grips of 45.0 ± 5.0 cm/min. Test the load-indicating device for calibration or index error. Stretch the test specimen at the specified rate in the tensile testing machine. Record the steady load and calculate the yield stress. With increase in strain, the specimen breaks. Record the load at break for the computation of tensile strength. Any specimen which breaks outside the gauge mark shall be disregarded. A sliding marker with graduations shall be used to record the separation of the gauge marks (up to the nearest 2 mm) when the specimen breaks. The percent increase in length compared to the original gauge mark gives the percent elongation.

6.5.1.1 Record the temperature close to the tensile testing machine.

6.6 Calculation and Reporting

6.6.1 Determine the average of the six specimens tested. Record the ambient temperature of testing.

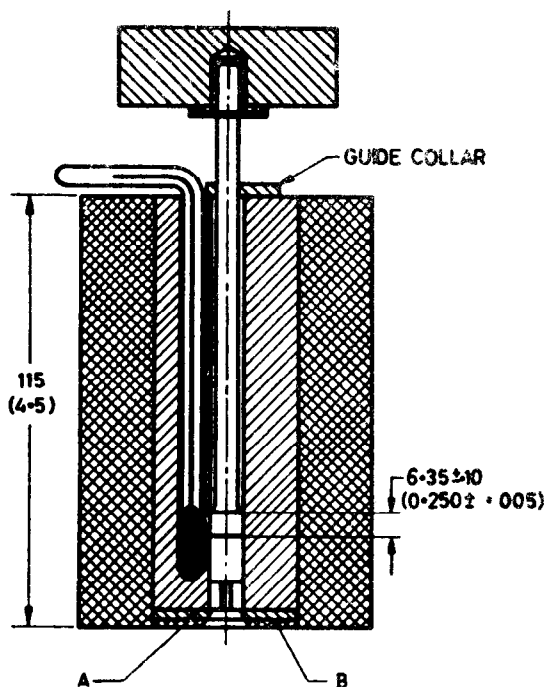
6.6.2 Report the results for tensile strength and yield stress as corrected for 27°C to the nearest kg/cm², after applying a specified temperature correction factor, where necessary.

6.6.3 No temperature correction is applied for elongation at break which is expressed to the nearest 10 percent.

7. DETERMINATION OF MELT FLOW INDEX

7.1 Apparatus — The apparatus is basically an extrusion plastometer. The general design is as shown in Fig. 2. Polyethylene, which is contained in a vertical metal cylinder, is extruded through a jet by a loaded piston, under controlled temperature conditions. The essential parts of the apparatus are as given in 7.1.1 to 7.1.8.

7.1.1 Cylinder — The cylinder of steel (see Note) shall be fixed in a vertical position and suitably lagged for operation at 190°C. The cylinder shall be at least 115 mm (or 4.5 in) long. The internal diameter shall be between 9.500 mm (or 0.374 in) and 10.000 mm (or 0.394 in.), and shall meet the requirements of 7.1.2. The base of the cylinder shall be thermally insulated, if the area of the exposed metal exceeds 4 cm² (or 0.6 in²). It is recommended that the



All dimensions are in millimetres and the dimensions given in parentheses are in inches.

FIG. 2 APPARATUS FOR DETERMINING MELT FLOW INDEX

(Showing Large External Diameter Cylinder, Jet Retaining Plate A and Insulating Plate B)

insulating material used shall be polytetrafluoroethylene [thickness about 3 mm (or 0.12 in)] in order to avoid sticking of the extrudate.

NOTE — To ensure satisfactory operation of the apparatus, the cylinder and piston are made of steel of different hardness. It is convenient, for ease of maintenance and renewal, to make the cylinder of the harder steel.

7.1.2 Piston — A hollow piston of steel (see Note under 7.1.1) shall have a length which is at least as long as the cylinder. The axes of the cylinder and the piston shall coincide, and the effective length of the piston shall be 135 mm (or 5.32 in) maximum. The piston shall have a head of length 6.35 ± 0.10 mm (or 0.250 ± 0.005 in). The diameter of the head shall be less than the internal diameter of the cylinder at all points along the working length of the cylinder by 0.075 ± 0.015 mm

(or 0.0030 ± 0.0006 in.). In addition, for calculating the load (see 7.1.3), this diameter shall be known within ± 0.025 mm (or ± 0.001 in.). The lower edge of the head shall have a radius of 0.4 mm (or 0.016 in.), and the upper edge shall have its sharp edge removed. Above the head the piston shall be relieved to about 9 mm (or 0.35 in.) diameter. A stud may be added at the top of the piston to support the removable load, but the piston shall be thermally insulated from this load.

7.1.3 Removable Load on Top of the Piston — The force P , in grams, exerted by the combined mass of the load and the piston, shall be calculated as given below:

$$\text{For Jet A:} \quad P = 464 \frac{D^2}{d^4}$$

$$\text{For Jet B:} \quad P = 46.4 \frac{D^2}{d^4}$$

where

D = diameter of the piston, measured to an accuracy of ± 0.025 mm (or ± 0.001 in.), and

d = internal diameter of the jet, measured to an accuracy of ± 0.005 mm (or ± 0.0002 in.).

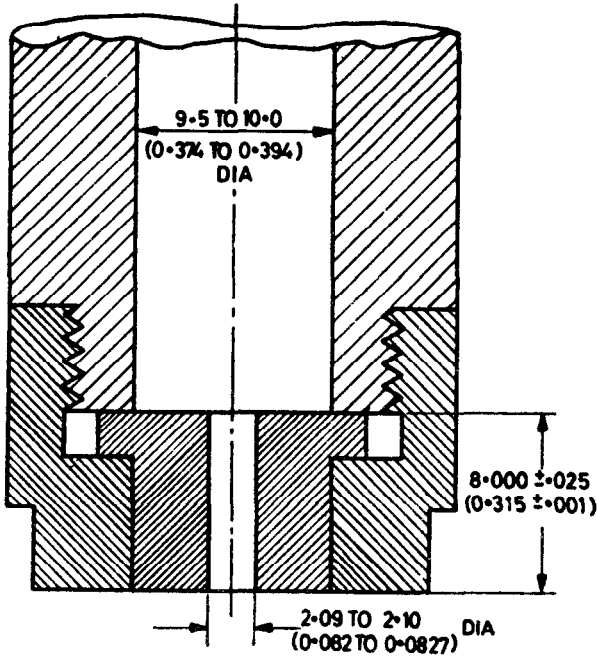
7.1.3.1 The force applied shall be calculated to an accuracy of ± 10 gf.

7.1.4 Heater — The heater shall maintain the polyethylene in the cylinder at a temperature of $190^\circ \pm 0.5^\circ\text{C}$. An automatic temperature control is recommended.

7.1.5 Temperature-Measuring Device — This device shall be located as close as possible to the jet, but situated within the body of the cylinder. This measuring device shall be calibrated to permit temperature measurement to an accuracy of $\pm 0.1^\circ\text{C}$.

7.1.6 Jet A — as shown in Fig. 3. It shall be 8.000 ± 0.025 mm (or 0.315 ± 0.001 in.) long and shall be made of hardened steel. The mean internal diameter shall be between 2.090 mm (or 0.0823 in.) and 2.100 mm (or 0.0827 in.) and shall be uniform along its length within ± 0.005 mm (or ± 0.0002 in.). The jet shall not project beyond the base of the cylinder.

7.1.7 Jet B — It shall be 8.000 ± 0.025 mm (or 0.315 ± 0.001 in.) long and shall be made of hardened steel. The mean internal diameter shall be between 1.160 mm (or 0.0457 in.) and 1.200 mm (or 0.0472 in.) and shall be uniform along its length within ± 0.005 mm (or ± 0.0002 in.). The jet shall not project beyond the base of the cylinder.



All dimensions are in millimetres and the dimensions given in parentheses are in inches.

FIG. 3 JET A (SHOWING SMALL EXTERNAL DIAMETER CYLINDER WITH AN ALTERNATIVE METHOD OF RETAINING THE JET)

7.1.8 All surfaces of the apparatus in contact with the material under test shall be highly polished.

7.2 Cleaning and Maintenance of the Apparatus

7.2.1 The apparatus shall be cleaned after each test. On no account shall abrasives or materials likely to damage the surface of the piston, cylinder or jet be used in removing superficial polyethylene or in manipulating any part of the apparatus.

7.2.2 Suitable solvents for cleaning the apparatus are xylene or tetrahydronaphthalene. The piston shall be cleaned, while still hot, with a cloth dipped in the solvent; and the cylinder, also while still hot, with a swab dipped in the solvent. The jet is cleaned with a closely fitting brass reamer or wooden peg, followed by immersion in boiling solvent.

7.2.3 It is recommended that at fairly frequent intervals, for instance about once a week, the instruments in constant use, the insulating plate and the jet retaining plate, if fitted shall be removed and the cylinder cleaned throughout.

7.3 Test Sample — Homogenize the material in a two-roll mixing mill for ten minutes as prescribed in 6.2.1 and cut the homogenized crepe into pieces of suitable size.

7.4 Procedure — Two test methods have been prescribed in this standard, *Method A* shall apply to materials of melt flow index 0.15 to 25 and *Method B* shall apply to materials of melt flow index 25 to 250.

7.4.1 Method A

7.4.1.1 Clean the apparatus as prescribed in 7.2. Raise the temperature of the cylinder and piston to $190^{\circ} \pm 0.5^{\circ}\text{C}$ for 15 minutes and maintain this temperature during the extrusion of the polyethylene. It is recommended that the temperature-measuring device (*see* 7.1.5) be a mercury-in-glass thermometer located permanently within the mass of the cylinder (*see* Note). A low melting alloy such as Wood's metal improves the thermal contact, and its use is recommended.

NOTE — If any other temperature-measuring device is used, then it should be calibrated at $190^{\circ} \pm 0.5^{\circ}\text{C}$, before the commencement of each series of tests, against a mercury-in-glass thermometer conforming to 7.1.5, placed within the cylinder and immersed in polyethylene to its appropriate depth of immersion.

7.4.1.2 Charge the cylinder with a quantity of the sample as given below and reinsert the unloaded piston into the top of the cylinder. Allow the material to attain temperature equilibrium for a total period of 4 ± 0.5 minutes, after which place the load on the top of the piston to extrude the polyethylene through the jet. Measure the rate of extrusion by cutting the extrudate at regular intervals of time at the jet with a suitable sharp edged instrument, to give short lengths of extruded material, which shall be called 'cut-offs'. The time intervals at which each cut-off shall be taken are as given below:

MELT FLOW INDEX	AMOUNT OF TEST PORTION IN THE CYLINDER	TIME INTERVAL
	grams	seconds
0.15 to 1.0	3 to 4	240
1.0 to 3.5	4 to 5	60
3.5 to 10	6 to 8	30
10 to 25	6 to 8	20

7.4.1.3 Take several cut-offs within 20 minutes of the introduction of the test portion into the cylinder. Discard the first cut-off and any

others which contain air bubbles. Weigh the remaining successive cut-offs, which number at least three, individually to the nearest milligram and determine the average weight. If the difference between the maximum and the minimum values of the individual weighings exceeds 10 percent of the average value, discard the test results and repeat the test on a fresh test portion.

7.4.2 Method B

7.4.2.1 For this method, jet *B* shall be used. This jet is designed to give a rate of extrusion approximately one-tenth the rate of jet *A*.

7.4.2.2 The exact conversion factor shall be determined by measuring the melt flow index of an extruded sample of melt flow index between 7 and 10, first using jet *A* and then using jet *B*. The conversion factor is defined as the ratio of the average weight of cut-offs using jet *A* with a 30-second time interval to the average weight of cut-offs using jet *B* with a 30-second time interval.

7.4.2.3 The test specimen used in determining the conversion factor shall be hot homogenized, for example, on mixing rolls, and shall contain an anti-oxidant (*see* Note).

NOTE — The melt flow index of polyethylene may be affected by previous thermal and mechanical treatment, and in particular oxidation will tend to reduce the melt flow index. Oxidation occurring during the test will usually cause a systematic reduction in the masses of successive cut-offs. This phenomenon is not exhibited by polyethylene compounds containing an anti-oxidant.

7.4.2.4 The test procedure shall be the same as prescribed in Method *A*. The time interval between cut-offs shall be 30 seconds, and the weight of the cylinder charge shall be 6 to 8 g.

7.5 Calculation and Reporting

7.5.1 Method A

$$\text{Melt flow index} = \frac{600 \times \text{average weight of cut-offs, in grams}}{\text{time interval, in seconds}}$$

7.5.1.1 Express the melt flow index to two significant places.

7.5.2 Method B

$$\text{Melt flow index} = \frac{600 \times \text{average weight of cut-offs, in grams}}{\text{time interval, in seconds}} \times \text{conversion factor}$$

7.5.2.1 Express the melt flow index to two significant places.

8. DETERMINATION OF POWER FACTOR AND PERMITTIVITY

8.1 Test Specimens

8.1.1 Carry out the test on at least two test specimens.

8.1.2 Homogenize the material in a mixing mill for ten minutes as prescribed in 6.2.1. 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 ± 0.025 mm from the mean value.

8.1.3 Punch out from the sheet with punch cutter, discs of diameter 50 ± 1 mm (or 2.00 ± 0.04 in).

8.2 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 : 1998-1962 Methods of Test for Thermosetting Synthetic Resin Bonded Laminated Sheets, 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 : 1998-1962 shall be taken.

8.3 Reporting — Report the power factor to four places of decimal. Report the permittivity to two places of decimal,

9. DETERMINATION OF VICAT SOFTENING POINT

9.1 Apparatus — The apparatus as shown in Fig. 4 consists of the parts given below. The construction of the apparatus shall be such that the dial gauge reading caused by differential thermal expansion over the intended temperature range does not exceed 0.02 mm when the test specimen is replaced by a piece of borosilicate glass or low expansion alloy steel. It is recommended that the apparatus be constructed of low expansion alloy.

9.1.1 Weighted Rod — It is held in a rigid metal frame so that it can move freely and vertically; the base of the frame serving to support the test specimen under the indenter at the end of the rod (see Fig. 4).

9.1.2 Indenter — The rod has a cylindrical *indenting tip*, preferably of hardened steel, 3 mm long, of circular or square cross-sectional area 1.000 ± 0.015 mm². The lower surface of the tip is square to the axis of the rod and free from burrs.

9.1.3 Micrometer — The penetration of the indenting tip into the test specimen is measured by means of a micrometer dial gauge graduated in divisions of 0.01 mm. The thrust of the dial gauge, which contributes to the thrust on the test specimen, shall be known and shall comply with the requirements laid down in 9.1.4.

9.1.4 Weight Pan — It is fitted to the rod and a removable cylindrical slotted midway is provided such that the total thrust applied to the test specimen can be made between 1 000 g and 1 050 g. The combined weight of the rod, indenter and weight pan shall not exceed 100 g.

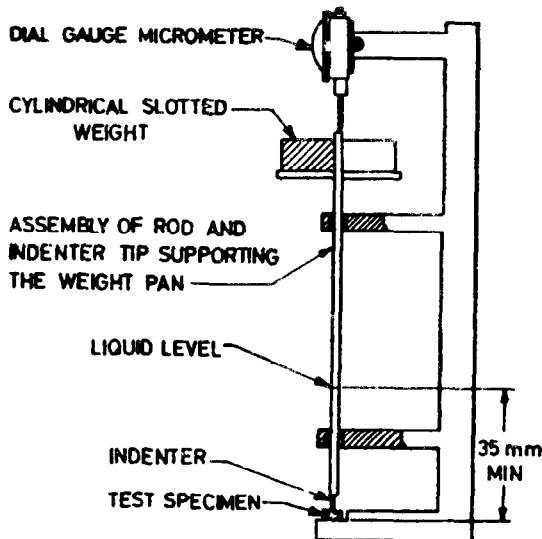


FIG. 4 APPARATUS FOR THE DETERMINATION OF THE VICAT SOFTENING POINT

9.1.5 Heating Bath—containing a suitable liquid (see Note 1), in which the apparatus shall be placed so that the test specimen is at least 35 mm below the surface of the liquid. An efficient stirrer shall be provided. The bath shall be equipped with means of control so that the temperature can be raised at a uniform rate of $50^{\circ} \pm 5^{\circ}\text{C}$ per hour (see Note 2).

NOTE 1—Liquid paraffin, transformer oil, glycerol and silicone oils may be suitable liquid heat-transfer media, but other liquids may also be used. In all cases, it should be established that the liquid chosen is stable at the temperature used and does not affect the material under test.

NOTE 2—A uniform rate of temperature rise can be obtained by controlling the heat input either manually or automatically. One procedure found to be satisfactory is to provide an immersion heater adjusted to give the correct rate of temperature rise at the starting temperature of the test, and then to increase the power input (either in the same heater or in a subsidiary heater) by adjustment of a rheostat or variable transformer.

9.1.6 Thermometer, Mercury-in-Glass Type—The thermometer shall be of appropriate range with minimum 0.5 degree C graduations. The scale error at any reading shall not exceed 0.5 degree C.

9.2 Test Specimens

9.2.1 Carry out the test on two test specimens.

9.2.2 The specimens shall be 3 to 6.4 mm in thickness, with an area of at least 10×10 mm and with their surfaces flat and parallel, and shall be prepared as prescribed in 6.2.1.

9.3 Annealing — The test specimens shall be annealed as prescribed in 6.2.2.

9.4 Procedure

9.4.1 Mount the test specimen horizontally under the indenter of the unloaded micrometer as shown in Fig. 4; the tip of the indenter shall at no point be nearer to the edge of the test specimen than 3 mm. The surface of the test specimen in contact with the base of the apparatus shall be flat.

9.4.2 Immerse the assembly in the heating bath, the temperature of which shall be constant and at least 50°C below the expected softening point of the material (*see Note*). The bulb of the thermometer should be at the same level as, and as close as is practical to, the test specimen.

NOTE — It is desirable to have a cooling coil in the liquid bath in order to reduce the time required to lower the temperature after previous tests. This is removed or drained before starting another test, as boiling of coolant can affect the rate of temperature rise.

9.4.3 After five minutes, with the micrometer indenter in position, note the reading of the dial gauge or set to zero; then add the cylindrical slotted weight on to the weight pan so that the total thrust on the test specimen is between 1 000 and 1 050 g.

9.4.4 Raise the temperature of the bath at a uniform rate of $50^{\circ} \pm 5^{\circ}\text{C}$ per hour. Stir the liquid well during the test.

9.4.5 Note the temperature of the bath at which the indenting tip has penetrated one millimeter into the test specimen.

9.5 Reporting — Report to the nearest 0.5°C the mean of the Vicat softening points of two test specimens, which shall not differ between themselves by more than 2°C .

10. DETERMINATION OF CARBON BLACK CONTENT

10.1 Apparatus

10.1.1 Combustion Boat — made of porcelain or silica, having minimum dimensions of 75 mm length, 9 mm width and 8 mm height.

10.1.2 Combustion Tube — made of hard glass, of approximately 30 mm diameter and 400 ± 50 mm length.

10.1.3 Gas Flow Meter — for measuring and controlling the rate of flow of nitrogen within 1.7 ± 0.3 litres per minute.

10.1.4 Thermometer — in the range 250° to 550°C.

10.1.5 Furnace — to accommodate the combustion tube and to give temperatures up to at least 500°C.

10.2 Reagents

10.2.1 Nitrogen — gas (see *IS: 1747-1960 Specification for Nitrogen, Technical).

10.2.2 Trichloroethylene [see *IS: 245-1962 Specification for Trichloroethylene, Technical (Revised)].

10.3 Procedure

10.3.1 Heat the combustion boat to red heat, allow it to cool in a desiccator for at least 30 minutes and weigh to the nearest 0.0001 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 ± 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.

10.3.2 Heat the furnace to $500^\circ \pm 5^\circ\text{C}$ and maintain this temperature for 10 minutes. Disconnect the outlet tube from the cold traps. Withdraw the combustion tube containing the boat from the furnace and allow to cool for 5 minutes, 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 to 30 minutes and weigh to the nearest 0.0001 g (W_1). Heat the boat strongly in air to constant weight (W_2).

10.4 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.

*Since revised.

11. DETERMINATION OF BUTYL RUBBER CONTENT**11.1 Reagents****11.1.1 Methyl Isobutyl Ketone****11.1.2 Petroleum Ether** -- boiling range 80° to 100°C.**11.2 Procedure**

11.2.1 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° 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.

11.2.2 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° to 120°C for 45 min. Cool in a desiccator and weigh. Repeat the drying and weighing until constant weight is obtained.

11.3 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.

12. DETERMINATION OF COLOUR BLEEDING

12.1 Test Specimen — Homogenize the material in a two-roll mixing mill for ten minutes as prescribed in 6.2.1. Cut homogenized sheet into pieces of suitable size.

12.2 Procedure — 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 $50^{\circ} \pm 1^{\circ}\text{C}$ for 72 ± 1 hours. 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.

12.3 Reporting — If either the filter paper or the polyethylene sheet is stained, it shall be reported that colour bleeding has occurred.

13. DETERMINATION OF COLOUR FASTNESS TO WATER

13.1 Procedure — Cut a small piece from a sheet prepared as prescribed in 6.2.1 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 $70^{\circ} \pm 1^{\circ}\text{C}$ for 24 ± 1 hours. Examine the water for discolouration by comparing it, against a white background, with a sample of distilled water.

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

14. DETERMINATION OF pH, WATER-SOLUBLE SULPHATES AND WATER-SOLUBLE CHLORIDES

14.1 Preparation of Water Extract — Reduce the sample of the material to a fine powder so that the major portion passes through 1.18 mm IS Sieve [see IS : 460-1962 Specification for Test Sieves (Revised)]. Take about 4 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 one hour 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 14.2, 14.3 and 14.4.

14.2 Determination of pH — 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 minutes of the preparation of the extract.

*Whatman No. 1 is suitable.

14.3 Determination of Water-Soluble Sulphates**14.3.1 Reagents**

14.3.1.1 Concentrated hydrochloric acid — conforming to IS : 265-1962 Specification for Hydrochloric Acid (*Revised*).

14.3.1.2 Barium chloride solution — 10 percent.

14.3.2 Procedure — 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_2SO_4)]. Place the beaker on a steam-bath and allow the precipitate to settle for about 4 hours. 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.

14.3.3 Calculation

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

where

W_1 = weight in g of the precipitate, and

W_2 = weight in g of the sieved material taken for the test in 14.1.

14.4 Determination of Water-Soluble Chlorides**14.4.1 Reagents**

14.4.1.1 Dilute nitric acid — approximately 5 N.

14.4.1.2 Standard silver nitrate solution — 0.1 N.

14.4.1.3 Standard ammonium thiocyanate solution — 0.1 N.

14.4.1.4 Ferric ammonium sulphate solution — saturated solution in distilled water.

14.4.2 Procedure — 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 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.

14.4.3 Calculation

$$\text{Chlorides (as NaCl), percent by weight} = \frac{58.46 (10 N_1 - V N_2)}{W}$$

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 14.1.

15. DETERMINATION OF COLOUR FASTNESS TO DAYLIGHT

15.1 Apparatus

15.1.1 Exposure Case — shall consist of a box $90 \times 60 \times 5$ 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.

15.2 Procedure

15.2.1 Prepare a sheet of the material as prescribed in 6.2.1. Cut out from the sheet sufficient number of test pieces of 50×25 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.

15.2.2 A set of eight wool standards, as specified in IS : 686-1957 Method for Determination of Colour Fastness of Textile Materials to Daylight, of size 30×5 mm, shall be pinned to another piece of cardboard and half of the portion of each standard covered with black paper.

15.2.3 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

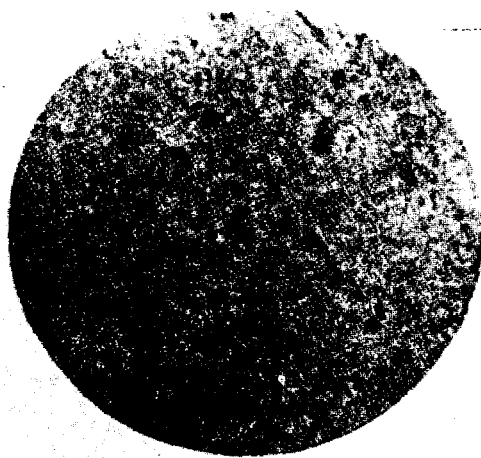
Geometric Grey Scale. Compare the exposed and unexposed portions of test specimens with the wool standard No. 5.

16. DETERMINATION OF CARBON BLACK DISPERSION IN POLYETHYLENE MATERIALS

16.1 Procedure

16.1.1 Place two clean microscope slides on a hot plate maintained at 170° 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 $1\frac{1}{2}$ to 2 minutes, 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 minutes.

16.1.2 When the slides are cool enough to be handled, examine the three specimens through a microscope at a magnification of 200 ± 10 with a field of view of 1 ± 0.1 mm diameter. Compare each specimen with the photomicrograph shown in Fig. 5 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. 5 A PORTION OF CARBON BLACK DISPERSION AS SEEN WITH A FIELD OF VIEW OF 1 mm UNDER 200 MAGNIFICATION

16.2 Reporting — 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. 5.

17. DETERMINATION OF ANTIOXIDANTS

17.0 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.

17.1 Determination of N-N'-di-2-Naphthyl-*p*-Phenylenediamine (DNPD)

17.1.1 Reagents

17.1.1.1 Toluene — conforming to *IS : 537-1955 Specification for Toluene, Pure, Nitration Grade.

17.1.1.2 Ethyl alcohol — 95 percent.

17.1.1.3 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.

17.1.2 Procedure

17.1.2.1 Absorptiometer method

- a) **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 17.1.2.1 (c). 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.
- b) 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.

*Since revised.

Heat the contents on a boiling water-bath and reflux it for one to one and a half hours. Wash the condenser with 15 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.

- c) 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 μ . 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 17.1.2.1 (a), relating optical density to concentration of DNPD.

NOTE—The maximum optical density is usually reached in 25 to 45 minutes.

- d) *Calculation*

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

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.

17.1.2.2 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.

17.2 Determination of Phenolic Antioxidant Content (Diazo Method)

17.2.1 Reagents

17.2.1.1 Toluene—same as in 17.1.1.1.

17.2.1.2 Ethyl alcohol—same as in 17.1.1.2.

17.2.1.3 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 cool the tube in ice. 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 minutes. Now add with constant shaking 11 ml of freshly prepared sodium hydroxide solution (2N), also previously cooled in ice.

17.2.2 Procedure

17.2.2.1 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, 2, 4,20 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 17.2.2.2. Plot a graph of optical density against mg of phenolic antioxidant/42 ml of the final solution. This graph covers the range of 0 to 0.2 mg of phenolic antioxidant/42 ml of the final solution.

17.2.2.2 Prepare the solution of the polyethylene compound as given in 17.1.2.1 (b). 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 dicresylol propane and 4, 4'-thiobis (3-methyl-6-tert-butylphenol), light of wavelength approximately 430 m μ shall be used.

17.2.2.3 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 17.2.2.1, relating optical density to concentration of antioxidant.

17.2.3 Calculation

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

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.

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