भारतीय मानक
Indian Standard

IS 17440 : 2020

कार्बन ब्लैक — विशिष्टि

Carbon Black — Specification

ICS 83.040.20

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Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Carbon black is a paracrystalline form of carbon. It has high surface area to volume ratio. However, its surface area is lower than that of activated carbon. Carbon black is a material generally commercially produced by incomplete combustion of heavy petroleum products such as coal tar, FCC tar or ethylene cracking tar. It is majorly used as a coloured pigment in plastics, inks, paints and as a reinforcing filler in tyres and other rubber products.

Furnace black method is one of the most widely used method for producing carbon black. There are two methods for producing carbon black that is, furnace black or thermal black. Carbon black in its finished form is > 99 percent pure and this differentiates it from a commonly known “soot” (solid material formed from the incomplete combustion of diesel and wood).

The list of experts who had made significant contribution to the formulation of this standard is given at Annex O.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*).’ The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CARBON BLACK — SPECIFICATION

1 SCOPE

This standard prescribes requirements for different varieties of carbon black for applications in paints, plastics, inks, tyres and rubber products. It also describes various test methods for the determination of Ash content, *p*H value, acetone extract, tinting strength, pour density, iodine adsorption number, particle size determination, total and external surface area by nitrogen adsorption.

2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision 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.

IS No.	*Title*
460 (Part 1) : 1985	Test sieves: Part 1 Wire cloth test sieves (*third revision)*
9788 : 1981	Specification for titanium dioxide, rutile for paints

3 REQUIREMENTS

The material shall be of such a composition as to satisfy the requirement of this standard. The material shall be free from foreign matter like wood, metal and fibres. Carbon black shall also comply with the requirements prescribed in Table 1.

4 PACKAGING AND MARKING

4.1 Packaging

The material shall be supplied in bags. The net mass of each bag shall be 25 ± 0.5 Kg. The bags shall be shaped to facilitate stacking in pellets by slight ironing.

4.2 Marking

4.2.1 Each package shall bear legibly and indelibly the following information:

a) Name and grade of the material,
b) Indication of the source of manufacture,
c) Mass of the material in the container,
d) Date of packing, and
e) Batch number.

4.2.2 *BIS Certification Marking*

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

5 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled H_2O shall be employed in the tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities.

Table 1 Requirements for Carbon Black

(*Clause* 3)

Sl No.	Name of the Test	Paints	Inks	Plastics	Tyres	Abrasion	Annex
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Ash content (percent by mass), *Max*	0.2	0.1	0.5	0.75	0.75	A
ii)	*p*H value	4.0-6.0	6-8	7-10	6.5-7.5	6.5-7.5	B
iii)	Acetone extract, percent by mass, *Max*	0.5	2.0				C
iv)	Tinting strength (percent)	58-60	110-120	98-130	104	120 ± 5	D
v)	Pour density(kg/m^3)	355 ± 40	340 ± 40	420 ± 40	376 ± 30	316-380	E
vi)	Iodine adsorption Number (mg/g)	121 ± 5	30-85	-	140-160	143-158	F
vii)	Particle size determination (millimicrons), *Max*	-	75	-	-	-	G
viii)	Nitrogen surface area m^2/kg	112-126 × 10^3	114-124 × 10^3	-	78-88 × 10^3	-	H
ix)	Sieve residue						
	45 micron IS sieve, percent by mass, *Max*	0.05	0.1	0.1	0.1	0.1	J
	500 micron IS sieve, percent by mass, *Max*	0.05	0.001	0.001	0.001	0.001	
x)	Loss on Heating, percent by mass, *Max*	2.5	2.0	2.0	2.0	2.5	K
xi)	Toluene Discolouration, percent by mass, *Min*	90	85	85	80	80	M
xii)	Fines content, percent by mass, *Min*	15.0	15.0	15.0	15.0	15.0	N

ANNEX A

(*Clause* 3, *Table* 1)

A-1 ASH CONTENT

A-2 PURPOSE

Ash content in carbon black is determined to know the amount of non-carbon compounds present after combustion.

A-3 APPARATUS

A-3.1 Muffle Furnace — Capable of temperature regulation of ±25°C at 550°C.

A-3.2 Silica or Porcelain Crucible — High form, capacity 15 ml, with cover.

A-4 PROCEDURE

Ignite the crucible and cover in the muffle furnace as 550 ± 25°C for 1 h. Place the crucible and cover in a desiccator and allow to cool at room temperature and then weigh to nearest 0.1 mg. Dry an adequate sample of carbon black for 1 h at 125°C and weigh accurately approximately 2 g of the dried sample into previously ignited crucible. Place the crucible with the cover removed into furnace and leave for 16 h at 550 ± 25°C. Remove from furnace and replace cover and cool in a desiccator to room temperature. Weigh to the nearest 0.1 mg.

NOTE — In the case of channel blacks the heating shall be done at 950 ± 25°C for a period of 4 to 5 h.

A-5 PRECAUTIONS

A-5.1 Keep the door of the furnace open about 6 mm to admit air to support the combustion of organic matter.

A-5.2 Take care in removing ashed sample from furnace to desiccator.

A-5.3 Always keep the cover on the crucible when transferring it to and from the desiccator to prevent loss of ash due to air currents.

A-5.4 After the sample has cooled in the desiccator, admit air slowly to avoid loss of ash from the crucible.

A-6 CALCULATION

$$\text{Ash, percent by mass} = \frac{D - B}{C - B} \times 100$$

where

D = *mass* of the crucible and ash,

B = mass of the crucible, and

C = mass of the crucible and sample.

Report the average of at least two determinations.

ANNEX B

(*Clause* 3, *Table* 1)

B-1 *p*H VALUE

B-2 APPARATUS

B-2.1 *p*H Meter — Equipped with glass and calomel electrodes, having an accuracy of 0.05 *p*H.

B-2.2 Beakers and Watch Glasses — Made of neutral glass and of sufficient size to accommodate the sample used.

B-2.3 Hot Plate

B-2.4 Glass or Porcelain Plate and Spatula or Mortar and Pestle

B-2.5 Container — Stainless steel or copper, for boiling distilled water.

B-3 PROCEDURE

Crush any pelleted or lumpy carbon black to a fine powder, using either the spatula and plate or mortar and pestle.

Add 10 ml of boiling distilled water to each gram of carbon black weighed into the beakers. To facilitate wetting of unpelletized carbon black, add a few drops of pure ethanol or acetone.

For large samples of 1 g or more of carbon black, boil the mixture for 15 min, but do not allow to go to dryness.

For small samples of 0.1 to 0.5 g of carbon black, use 50 ml beakers and boil the mixture on the hot plate until a sludge remains, but do not allow to go to dryness.

Let the mixture cool to room temperature in an atmosphere free from chemical fumes which might contaminate the samples. Decant off any supernatant liquid.

Place the electrodes in the sludge and rotate the beaker gently in alternate direction until a constant pH is obtained.

Repeat the procedure on a second sample.

B-4 PRECAUTIONS

B-4.1 To prevent contamination of the sample during boiling, a clean watch-glass may be used over the beaker.

B-4.2 Standardize the *p*H meter with a reliable buffer in pH range of the carbon black samples to be tested.

B-4.3 The distilled water used in the test should be as pure as possible.

B-4.4 The *p*H of the freshly boiled distilled water shall be 6.9 to 7.l.

B-5 REPORT

The report shall include the results obtained from the two individual determinations and their average.

ANNEX C

(*Clause* 3, *Table* 1)

C-1 ACETONE EXTRACT

C-2 APPARATUS

C-2.1 Soxhlet Extraction Apparatus — With 20 to 30 ml cup.

C-2.2 Flat-Bottomed Flask — 150 ml.

C-2.3 Whatman Type Paper Extraction Thimbles — 25 x 100 mm.

C-3 REAGENT

C-3.1 Acetone

C-4 PROCEDURE

Weigh the cleaned and dried 150 ml flask. Weigh into a paper extraction thimble accurately to nearest 0.001 g, about **5 g** of the sample. Cut six pieces of filter paper slightly larger than the internal diameter of the thimble and press each one into place on top of the sample. Place the thimble in a soxhlet extractor and reflux for four hours with 100 ml of acetone in the flask. After four hours remove the thimble and distil off most of the acetone from the flask. Evaporate the remaining solvent with compressed air, dry the flask at 95°C in an oven, cool in a desiccators and weigh.

C-5 CALCULATION

Acetone extract, percent by mass = A/M × 100

where

A = mass of residue from the sample determination, and

M = mass of sample.

ANNEX D

(*Clause* 3, *Table* 1)

D-1 RELATIVE TINTING STRENGTH

D-1.1 Principle

A dispersion of the coloured pigment under test, prepared under known conditions on an automatic muller, is mixed in a known ratio with a dispersion of white pigment. The intensity of colour and hue of the resulting paste is compared with that of a similar paste made under the same conditions from the approved sample of coloured pigment and the same dispersion of white pigment.

D-1.2 The degree of development of tinting strength of a coloured pigment is dependent on the amount of work done in the preparation of the dispersion. Therefore, in determining the relative tinting strengths of two coloured pigments it is necessary for the comparison to be carried out at the level of maximum development of colour. In this method, which uses an automatic muller, the development of tinting strength is influenced by the force applied, the number of revolutions, the medium, the volume of the mix, and the rheology of the mix. By following the procedure described in **D.4.1**, the

conditions are determined under which a practical maximum of tinting strength may be obtained on the automatic muller. When these conditions are known for a particular pigment, there is no need to carry out the procedure described, in **D-4.1.**

D-1.3 Of the factors listed in **D-1.2**, the following can be specified without difficulty:

a) The force applied, which shall be the maximum available;

b) The medium to be used, which is linseed stand oil; and

c) The volume of the mix of pigment and medium which shall be about 2 ml.

D-1.4 The remaining factors to be determined which shall be chosen according to the level of

tinting strength of the coloured pigment under test, are the following:

a) The ratio of pigment to medium,

b) The number of revolutions to be used, and

c) The ratio of coloured pigment to white pigment.

D-1.5 The appropriate ratio of pigment to medium, by mass, depends not only on the oil

absorption of the pigment but also on the viscosity of the mix during the mulling operation. As a first step, all pigments can initially be allocated to one of the following three groups:

a) Pigment of low medium requirements-average mulling concentration 66.7 percent of pigment by mass;

b) Pigments of intermediate medium requirement-average mulling concentration 40 percent of pigment by mass; and

c) Pigments of high medium requirement-average mulling concentration 25 percent by mass.

The quantities to be used in the test for the three groups defined above shall be the following:

1) 3.0 g of pigment and 1.5 g of the linseed stand oil.

2) 1.0 g of pigment and 1.5 g of linseed stand oil.

3) 0.5 g of the pigment and 1.5 g of the linseed stand oil.

NOTES

1 If the mixture chosen is found to be unsuitably stiff or fluid for use on the muller, one of the other ratios should be used as appropriate.

2 If the diameter of the muller plates is at the top end of the range specified in **D-1.1**, it may be necessary to increase the amounts specified in order to reduce wear on the plates.

D-2 APPARATUS

D-2.1 Automatic Muller

With ground glass plates, preferably water cooled, of diameter 180 to 250 mm to which a variable but known force of up to about 1 000 N may be applied. The driving glass plate shall have a speed of rotation of between 70 and 120 revolutions per minute and the apparatus shall have an arrangement for presetting the number of revolutions in multiples of 25.

D-2.2 Palette Knife or Spatula — Of steel or plastic material.

D-2.3 Glass Slides — Slides of glass or other transparent, colourless, non absorbent substance.

D-2.4 Film of Plastic Material — Transparent and colourless

D-2.5 Film Application — Suitable for applying two or three films side by side of wet paste, thickness 50 to 100 μm.

D-2.6 Balance — Accurate to ±0.001 g

D-3 REAGENTS

D.3.1 Linseed Stand Oil

D-3.2 White pigment Paste-with the following composition: 40 parts by mass of titanium dioxide rutile (*see* IS 9788) and 60 parts by mass of linseed oil. Mix well so as to achieve preliminary wetting of the solids, then grind on a triple roll mill, until the particle size is less than 5μm when tested on a fineness of grind gauge. Store in airtight containers, preferably tubes with screw caps.

D-4 PROCEDURE

D-4.1 Determination of Conditions for Preparing Dispersion of Coloured Pigments

D-4.1.1 Weigh an appropriate quantity of medium and transfer it to the lower plate of the muller: weigh the corresponding quantity of the approved sample of the coloured pigment and sprinkle it carefully on the medium. Mix the pigment with the medium by means of the spatula, but do not grind. Spread the mixture in approximately circular patch, of diameter about 100 nm, in the centre of the lower plate and clean the spatula as much as possible by wiping it on the upper plate of the muller. Close the plates and carry out the grinding in stages of 50 revolutions; after each stage collect the paste from both plates with the spatula and spread it out in an approximately circular patch on the centre of the lower plate, wiping the spatula on the

upper plate as before. After a total of 200 revolutions, remove a small portion (about one-quarter of the total volume) of the collected paste, set this aside and continue grinding the main bulk. Remove similar small portions after totals of 300 and 400 revolutions: set these also aside and then clean the muller plates and spatula.

D-4.1.2 Place on the lower muller plate 3 ± 0.01 g of white pigment paste and an amount of the coloured pigment paste which has been ground for 200 revolutions which contains 0.12 g of coloured pigment (*see* **D-3.1.4**) and mix the two pastes with the spatula as homogeneously as possible, without grinding. Spread the mixture in an approximately circular patch in the centre of the lower plate of the matter, wipe the spatula on the upper plate, close the plates and grind for four stages of 25 revolutions each with the minimum applied force; after each stage collect the paste in the centre of the lower plate as described previously. Then remove the paste from the muller and set aside for later assessment.

Repeat this operation with the same mass of each of the portions of coloured paste which have been ground for 300 and 400 revolutions, respectively.

D-4.1.3 Apply each of these reduction pastes in order, side by side, with touching edges on a sheet of glass or transparent film of plastics material so that the intensity of colour can be judged visually. Record the minimum number of revolutions necessary to obtain the sample of coloured pigment paste which produces the maximum intensity of colour and use the number of revolutions for the test itself.

D-4.1.4 The amount of coloured pigment paste which contains 0.12 g of coloured pigment will, when mixed with 3 g of white pigment paste, **give** a reduction ratio of 1 : 10. This ratio should be modified to for example, 1 : 5 or 1 : 20 (to suit weak pigments or strong pigments, respectively) in order to produce a colour on reduction of intensity which is suitable for assessing the intensity of colour and hue of the reduction -pastes.

D-4.2 Preparation of Dispersion of Coloured Sample

Prepare a dispersion on the automatic muller of the approved sample of the coloured pigment as described in **D-3.1**, by carrying out the grinding in stages of 50 revolutions to the full number of revolutions previously decided without removing any of the paste, but gathering and spreading the paste after each stage. When the grinding has been completed, collect the paste and store. Clean the muller and spatula, and repeat the operation with the same quantities of the test sample of pigment and oil and using the same procedure on the same muller. Collect the coloured pigment paste from this sample and store. Clean the muller and spatula.

D-4.3 Mixing of Dispersions of coloured Pigment and White Pigment

Place on the lower muller plate 3 ±0.01 g of white pigment paste and an amount of the coloured pigment paste which has been ground for 200 revolutions and which contains the mass of coloured pigment to give the chosen reduction ratio, and mix the two pastes with the spatulas homogeneously as possible, without grinding. Spread the mixture in an approximately circular patch in the centre of the lower plate, wipe the spatula on the upper plate, close the plates and grind for four stages of 25 revolutions each with the minimum applied force; after each stage collect the paste in the centre of the lower plate as described previously. Then remove the paste from the muller for later assessment and store in a suitable receptacle. Repeat the operation using the coloured pigment dispersion paste prepared from the test sample of coloured pigment, thus producing a comparative reduction paste.

D-4.4 Comparison of Colour on Reduction and Determination of Relative Tinting Strength

D-4.4.1 P1ace a quantity of each of the two reduction pastes obtained as described in **D-4.3** side by side on a glass plate or plastics film, and with the film applicator draw them down to form two uniformly thick strips not less than 25 mm wide with touching edges not less than‘40 mm long. Compare for intensity of colour and for hue the two strips immediately after application by examining them in diffused daylight through the glass or film of plastics material.

Where good daylight is not available; make the comparison in artificial light.

If the intensities of colour are equal and the hues are the same, the colours on reduction are the same, and the relative tinting strength of the sample under test is 100 percent. However, if the intensities of colour are equal but the hues are not the same, and there is also a difference in colour on reduction; note the difference and its nature.

D-4.4.2 If the intensities of colour are not considered to be equal, and, therefore, the colours on reduction are not the same, repeat the operations of **D-4.3** and **D-4.4.1**, but weighing and using a quantity of the coloured pigment dispersion of the test sample which is estimated to give an intensity of colour equal to that of the original mass taken of the coloured pigment dispersion of the approved sample.

For example, if it is considered that the, sample under test is 15 percent stronger than the approved sample, in the repeat test, the mass of coloured test Pigment dispersion used shall be15 percent less than that taken for the first test, but the mass of coloured pigment dispersion of the approved sample shall be the same as

that originally used. If there is also 9 difference in hue, note the difference and its nature.

D-4.5 Calculation

Relative tinting strength = $\frac{b \times 100}{a}$ gives percent of the approved sample

where

a = parts of the test sample that produce the same intensity as approved sample, and

b = parts of the approved sample.

NOTE — It is important to include the percentage sign (percent), and to note that the tinting strength is relative to that of the approved sample as 100 percent.

ANNEX E

(*Clause* 3, *Table* 1)

E-1 POUR DENSITY

This method is essentially based on ISO/R 1306-1970 'Pelletized carbon black for use in the rubber industry. Determination of pour density' and ASTM D 15 13-60 'Pour density of pelletid carbon black' .

E-2 APPARATUS

Cylindrical container without any pouring lip or deformation of wall and of 1 000 ml capacity.

E-3 PROCEDURE

Pour the carbon black into the centre of the tared container from a height of not more than 50 mm, from the rim. A large excess should be used to form a cone above the rim of the cylindrical container, Level the surface with a single sweep of a straight edged spatula, held perpendicular to and in firm contact with the lip of the container. Determine the mass of carbon black in the container to the nearest gram. Repeat the procedure on two additional samples.

E-4 CALCULATION

$$D = M_2 - M_1$$

where

D = pour density in g/ l;

M_2 = mass in g, of the sample and the container; and

M_1 = mass in g, of the container.

ANNEX F

(*Clause* 3, *Table* 1)

F-1 IODINE ADSORPTION NUMBER

F-1.1 Principle

Iodine number is defined as the milligrams of iodine adsorbed by one gram of the material when the iodine residual concentration of the filterate is 0.02 N. IAN is related to the surface area of carbon black and therefore it measures the quality of the base material. If high contents of volatile or solvent extractable substance is present, IAN decreases. As carbon black is used as a filler material in plastics, rubber and inks, it requires certain IAN in order to reach and maintain the physical properties of the end product.

F-2 APPARATUS

F-2.1 Vials — Optically clear type with polyethylene or ground-glass stopper of 45 ml capacity. Alternatively, tall form weighing bottles of 30 ml capacity with ground-glass stoppers may be used.

F-2.2 Oven — capable of maintaining a temperature of 105 ± 2°C.

F-2.3 Burette — 25 ml, graduated upto 0.05 ml; or 10 ml, graduated upto 0.02 ml.

F-2.4 Pipettes — 25 ml and 20 ml, calibrated to the nearest 0.01 ml.

F-2.5 Centrifuge — Capable of speed above 1000 rev/ min. If a centrifuge is not available then porcelain filter crucible may be used.

F-2.6 Balance — Analytical, with a sensitivity of at least 0.1 mg.

F-3 REAGENTS

F-3.1 Iodine Solution — 0.0473 N — Dissolve 6 g of iodine and 57 g of potassium iodide (KI) weighed to the nearest 0.1 mg in 30 ml water, dilute to 1 litre in a volumetric flask when solution is complete and standardize against sodium thiosulphate solution.

F-3.2 Standard Sodium Thiosulphate Solution-0.0394 N — Dissolve 9.781 0 g of sodium thiosulphate pentahydrate ($Na_2S_2O_5$. $5H_2O$), weighed to the nearest 0.1 mg in 500 ml of water in a 1 litre volumetric flask. Add 5 ml of n-pentanol and shake to mix thoroughly. Dilute to 1 litre and standardize with 0.1 N potassium dichromate solution.

F-3.3 Starch Indicator Solution — Stir 2-5 g of starch and 2 mg of mercuric iodide in 25 ml of water. Add the suspension immediately to 1 litre of boiling water, boil for 5 min. With stirring, cool and store in bottles. A suitable soluble starch indicator may also be used.

F-4 PROCEDURE

Dry an adequate representative sample for 1 hour at 105 deg C. The pellet shall be crushed before drying. Weigh accurately into a glass vial or bottle approximately 0.5 g of dried sample for expected iodine numbers under 135 and 0.25 g for ,iodine numbers from 135 to 500, all weights being to the nearest 0.1 mg. Pipette 25 ml of the iodine solution into the glass vail or bottle and stopper immediately. Shake the mixture for one minute at not less than 120 strokes per minute. If a mechanical shaker is used, 240 vigorous strokes per minute are recommended. Thorough mixing

of the carbon black and iodine solution is necessary. Centrifuge immediately for at least one minute at over 1000 rev/min after shaking. Immediately after the centrifuge has come to rest, decant the iodine solution in one smooth motion into a 50 ml beaker, leaving the black at the bottom of the vial. Then immediately pipette 20 ml of this solution into a l00 ml conical flask and titrate with the standard sodium thiosulphate solution until a pale yellow colour remains. Add approximately 5 ml starch indicator or approximately 0.1 g soluble starch indicator and continue the titration until one drop of thiosulphate causes the blue colour to change to colourless. Read the burette to the nearest 0.01 ml.

Carry out a blank determination using **20** ml of iodine solution following the above procedure.

F-5 EXPRESSION OF RESULT

$$I = (B - S)\frac{N}{M} \times 158.64$$

I = iodine absorption number in mg of iodine per gram of carbon black;

B = volume in ml of sodium thiosulphate solution required for titration of blank (the average of two determinations);

S = volume in ml, of sodium thiosulphate solution required for titration of sample (the average of two determinations);

N = normality of standard sodium thiosulphate solution;

M = mass in g, of the sample taken for the test; and 158.64 = dilution factor x equivalent weight of iodine. $\frac{25}{20} \times 126.91$

ANNEX G

(*Clause* 3, *Table* 1)

G-1 PARTICLE SIZE DETERMINATION (ELECTRON MICROSCOPY)

G-1.1 Significance

The morphology of carbon black should significantly affects the transient and end use properties of systems as carbon black loaded polymer. One of the most important property of carbon black is its particle size distribution. It relates to the degree of blackness and reinforcement. Size of the aggregate and its shape also affect end use performance of a carbon black.

The preferred method to determine the particle size is Transmission Electron Microscopy (TEM) preferably using ‘lacey carbon grid’.

G-2 APPARATUS

Transmission type electron microscope with point to point resolution of 1 nm or better. Desired resolution is achieved at high operating voltage. Voltage range recommended is around 60 to 120 KV. For better resolution, 300 kV instrument can also be used.

G-2.1 Image Analysis System

It consists of a TEM-interfaced camera which is capable of 640 x 480 pixel or better resolution. The computer is equipped with a hardware to capture TEM images digitally, and software in order to perform morphological operations.

G-2.2 Two Roll Mill

G-2.3 Vacuum Evaporator — For preparation of carbon films as substrates for electron microscopy.

G-2.4 It should be capable of reducing the absolute pressure to 1.3 MPa. It should also contain the necessary apparatus for a-c glow discharge.

G-2.5 Ultrasonic Generator — Probe which provides sufficient energy for sufficient dispersion.

G-2.6 Dry Box

G-2.7 Analytical Balance — With an accuracy of 0.5 mg.

G-2.8 Electrically Heated Tube Furnace — It should have the capability to heat to 800-900 °C under an inert environment. It should have a ability to introduce and remove the sample without allowing oxygen intrusion.

G-2.9 Pyroprobe — Capable of being heated from 150 to 1 000 deg C in an inert environment.

G-2.10 Carbon Rods and Carbon Rod Sharpener

G-2.11 Glass Microscope Slides, 25 by 75 mm

G-2.12 Test tube, transfer pipette, rubber bulbs, glass vials, buchner funnel, filter paper, carbon coated electron microscope, wire screening with openings approximately 1 mm^2, wire screening, tweezers, spatulas, fluorocarbon duster, lens tissue, porcelain boats, centrifuge, beakers.

G-3 REAGENTS AND MATERIALS

G-3.1 Chloroform, reagent grade

G-3.2 Tetrahydrofuran, R.G

G-3.3 1,2-Dichloroethane, R.G

G-3.4 Ethyl Acetate, R.G.

G-3.5 Poly (Vinyl Formal) Resin

G-3.6 Cellulose Acetate Butyrate Resin

G-3.7 Pthalate Type Plasticizer (such as sanitizer)

G-4 SAMPLE PREPARATION-DISPERSION PROCEDURES

G-4.1 Dry Carbon Black (Sonic Bath)

Weigh carbon black (8 to 10 mg) into a test tube containing 1 cm^3 of solvent (preferably isopropyl alcohol). Adjust the ultrasonic bath power for maximum agitation. This requires adjustment of water level. As the water is heated by ultrasonic energy, ice can be added to control the temperature in order to maintain maximum dispersive capability. Place this test tube containing carbon black and solvent mixture into the most intense part of ultrasonic field and allow the mixture to agitate for 3 to 5 min. Hold the test tube with tongs and mount it in a simple wire holder. Transfer a small portion of carbon black solvent mixture in some another test tube containing 1 cm^3 of solvent. If the particle size increases, the amount of concentrate required also increases. Sample is transferred between the transfer pipette and the test tube. Cork the test tube and ultrasonic dispersion is done. The concentration of the diluted dispersion is checked by collecting a small amount in a pipette. The sample is viewed against a white background.

For carbon black of tread grade, the dispersions shall be transparent becoming somewhat darker with the size of the particle. The diluted dispersions of very coarse carbon black will be on the threshold of complete opacity. Adjust the concentration by adding more solvent or concentrate if required. Permit the sample for ultrasonic agitation. The volume of carbon black solvent mixture should be maintained at 1 cm^3 In case the dilution is required and the excess of volume above 1 cm^3 should be discarded. Place a specimen grid along with the substrate. Remove diluted dispersion with the help of fresh pipette and place one drop on the grid close to the centre at a height of 12 mm. Dry the specimen for 1 min on a filter paper. In case the relative humidity in the room exceeds by 30 percent, the above specimen preparation procedure should be performed. For TEM grids, place them in a sample holder and then place it in a pyrolysis chamber. In order to prevent the oxidation of the sample, purge the chamber with inert gas. Pyrolyse the specimen grid at a temperature around 550 °C.

G-4.2 Dry Carbon Black (Ultrasonic Probe)

Weigh carbon black (5 to 10 mg) into a 30 cm^3 glass vial. Add to it 20 cm^3 of solvent. Keep the vial containing solvent and carbon black in an ice bath. Insert the probe into a vial at a depth of around 2.5 cm. Ultrasonicate at 40 to 50 watts for 10 min. Transfer small amount of carbon black/solvent mixture into another vial and add 20 cm^3 of fresh solvent. Sonicate for 3 min additionally. Check concentration by extracting small amount into a pipette and place one drop on filter paper. Dispersions should be transparent for tread grade carbon black, becoming darker with particle size. Add more solvent or concentrate in order to adjust the concentration. Maintain the volume of carbon black solvent to around 20 cm^3.

Place the specimen grid along with a thin carbon substrate when the final dispersion drop on the filter paper is in an acceptable colour range. Remove small amount of final dispersion with the help of fresh pipette and place the specimen grid with carbon substrate on a piece of filter paper. Now take a small amount of dispersion with the help of pipette and place a drop on the grid close to the center. Allow the grid to dry for 1 min on the filter paper and place it under the microscope and examine the dispersion.

G-5 TEM ANALYSIS PROCEDURE

Set the beam intensity to proper level for the analysis of the image (with the specimen removed). Correct for any shading in the blank image. Place the carbon black specimen under an electron microscope, locate it at the center of the stage of microscope and adjust to encentric height. In order to acquire the good microscopy

practices, focusing and stigmating of the images, higher magnification is recommended. Near one side of a grid opening, start the measurements. Continue to select and measure adjoining fields of carbon black aggregates in a straight line pattern of tracking, moving towards the centre. Repeat if necessary until data of 2 000 aggregates is recorded.

G-5.1 Image Analysis

Each image analysis system has its own method for processing the images it receive from the camera. Thresholding can be accomplished with the help of an automated process, manually for each field and with a use of preset threshold if thickness of carbon film and morphology are consistent and other imaging conditions are not altered. Image analysis with the help of software should be configured to perform noise reduction and edge smoothing. Erosion followed by dilution can help in edge smoothing. It can smooth the "noise", which is associated with particle boundaries without the alteration of the real data. Attempts for noise reduction in order to eliminate the measurements of objects which are not carbon black are made. In many cases, software may be configured to reject objects which are less than 50 pixels in area. The results will tend to be bias in case all aggregates that touch image or guard frame boundaries are rejected. Such effects can be minimized in case appropriate magnification/ resolution is done. Analysis for standard carbon black sample is repeated with a similar particle size suitable for appropriate magnification. Perimeter (P) and Area (A) of the projections of aggregate are to be measured for use in morphological property calculations.

G-6 CALCULATIONS

Single Aggregate:

Parameters Measured:

P = aggregate perimeter

A = aggregate area (nm^2)

Parameters Derived:

V_1 = aggregate volume (unit = nm^3) = $(8/3)\ A^2/P$

D = Area equivalent aggregate diameter (nm) = $4\ (A/\Pi)^{1/2}$

α is the aggregation factor = $13.092(P^2/A)^{-0.92}$

dp = average particle size for single aggregate (nm) = $(\alpha\Pi A)/P$

V_2 = Volume of a particle (nm^3) = $\Pi dp\ ^3/_6$

n = particles present in an aggregate = $\frac{V_1}{V_2}$

Distributional Properties for Particles and Aggregates

n_t = Σn for all aggregates measured or total number of particles

m = mean particle size (nm)

S_d = standard deviation in the size of particle (nm) = $[\ \Sigma(n*dp^4)/(n_t-1)]^{1/2}$

W_m = weight of the mean particle size (in nm) = $[\Sigma(n*dp^4)]/[\Sigma n*dp^3)]$

hi = Heterogenity index of the particle size

d_{sm} = Surface mean diameter of the particle size (nm)= $[\Sigma(n*dp^3)]/[(\Sigma n*dp^2)]$

EMSA = electron microscope surface area (m^2/g) = $6000/(\rho*d_{sm})$ (where ρ = 1.8 g/cm^3, assume density of carbon black),

Nt = number of aggregates measured

M = mean aggregate size (nm)=$\Sigma D/N_t$

SD = Standard deviation of aggregate size (nm) = $[\Sigma\ (D-M)^2/\ (N_t-1)]^{1/2}$

WM = Weight of mean aggregate size (nm) = $\Sigma D^4/\Sigma D^3$

HI = Heterogeneity index of aggregate size = WM/M

ANNEX H

(*Clause* 3, *Table* 1)

H-1 TOTAL AND EXTERNAL SURFACE AREA BY NITROGEN ADSORPTION

H-1.1 Summary

This method is used to determine the total and external surface area of carbon black based on nitrogen adsorption. The nitrogen surface area measurement is based on the B.E.T theory.

H-2 APPARATUS

H-2.1 Multipoint Static Volumetric Gas Adsorption Apparatus with Dewar flasks and Accessories Required

H-2.2 Sample Cells

H-2.3 Analytical Balance

H-2.4 Heating Mantle

H-2.5 Oven

H-3 REAGENTS

H-3.1 Liquid Nitrogen, 98 Percent or Higher Purity

H-3.2 Ultra High purity Nitrogen Gas

H-3.3 Ultra High Purity Helium Gas

H-4 SAMPLE PREPARATION PROCEDURE

Dry carbon black sample at a temperature of 125°C for about 1 h. This step can be omitted in case the sample is subsequently free from moisture. Empty sample is conditioned for 10 min at conditions which are intended for degassing the sample. Weigh the cell to nearest 0.1 mg and record the mass. Weigh 0.4 g of carbon black into the sample cell.

H-4.1 Flow Degassing

Open gas control valve and insert delivery tube into the sample tube. Allow purging with nitrogen or helium for a minute. Place some heat source around sample cell. Degas the sample at 300 ± 10 degree for half an hour or longer in order to ensure that moisture traces are absent. Degassing time that gives a stable surface area may be used for degassing. Once the degassing time has been determined , samples can be degassed on time basis alone. Less than half an hour is required by some sample in case the exposure to moisture is minimum. Minimum time required in order to give a stable surface area is used for the process of degassing. After the degassing process, the tube containing the sample may be directly moved to the analyzer. Else, remove the tube from the source of heat and continue the purging gas flow till it is ready for analysis.

H-4.2 Vacuum Degassing

Keeping the apparatus at atmospheric pressure, keep the sample cell containing carbon black with degassing apparatus. Start the process of degassing as appropriate for the apparatus. Place a heating mantle or some other heat source around sample cell. Degas the sample at 300±10 degree C for about half an hour or more as required. Hold a pressure at ≤1.4 Pa (10 µm Hg). Once degassing times are determined, future samples can be degassed on time basis alone. Allow sufficient margin of excess time. Some samples requires ≤1/2 h in case the moisture exposure is minimal. Minimum amount of time that gives a stable Surface Area may be used for degassing.

H-5 PROCEDURE

Refer user's manual and instructions for multipoint gas adsorption analyzer so that one can become familiar with the procedures. Number of instruments are available that offer a variety of saturation vapour pressure (P_o) measurement options and Dewar size. Dewar flask is filled with liquid nitrogen and allow it to reach temperature equilibrium. At every hour fill the Dewar flask. This should also be done between each analysis.

H-5.1 Small Dewar

Cover the Dewar after filling it for a minimum of 2 h. Dewar should be dry and clean at the end of each day.

H-5.2 Large Dewar (≥1L)

Cover the Dewar for 16 h prior to use, unless continuous P_o measurements are employed. For continuous Po, use Dewar equilibration for 2 h. Once we have reached equilibration, a large Dewar can maintain this equilibration for several days if it is kept covered and filled. The frequency of cleaning is left to the direction of the operator, but is not to exceed once per week.

List of P_o measurement options:

Continuous P_o (measurement at each relative pressure point): The method is one of the best practice, however, the method generally increases analysis time.

H-5.3 Single P_o per Analysis

This value can be measured before, during, or after the run, a P_o value measured at the end of the analysis is preferred since STSA (specific surface area that is accessible to rubber) is calculated from last data points. This is significantly influenced by P_o values. P_o value in this method is determined prior to initiating measurements to ensure equilibrium of the Dewar.

H-5.4 Daily P_o

The method is used in case the evidence of stable Dewar is present and no changes in atmospheric pressure that is, greater than 0.13 kPa (1mm Hg) occur.

H-5.5 Calculated P_o

Value of P_o is calculated by atmospheric pressure measurement and adding a value between 1.3 and 2.6 kPa (10 mm and 20 mm Hg). In order to determine the constant, operator is responsible, most commonly used pressure is 15 mm Hg.

With exceptions to continuous P_o measurements, it is recommended that P_o value be determined prior to NSA/STSA analysis. P_o value between 10 to 20 mm Hg which is above atmospheric pressure and two consecutive P_o values differing not more than 0.13 kPa (1mm Hg) over 10 min time period are indications of stable Dewar.

Expected difference in P_o and atmospheric pressure in lab is taught to the operator by experience. Free space of the sample cell is determined by measurement with helium or by calculating assumed carbon density of 1.9 g/cm^3.

Obtain around five data points evenly spaced in relative pressure (P/P_o) range of 0.1 to 0.5. In order to increase the accuracy of measurement, it is recommended to take two additional data points.

Mass of the cell is determined with dry sample to nearest 0.1 mg. This is done before or after measuring nitrogen adsorption.

Avoid use of helium inconsistently, as a buoyancy error of 1 mg/cm^3 of cell volume can occur. Alternatively, mass of carbon black may be determined directly by pouring it into weighing pan from the sample cell.

H-6 CALCULATION

At the completion of the analysis, most automated instruments will perform the following computation. Verification of the internal computations by the user must be verified conforming to the following method.

H-6.1 Mass of the Sample

Dried Sample Mass

(Mass of the cell + sample)-(Mass of the cell)

Record the mass to nearest 0.1 mg

Nitrogen Adsorbed (Volume)

Volume of adsorbed Nitrogen per gram of sample-

V_a= Volume of Nitrogen (in cm^3)/Mass of the sample in (g)

H-6.2 Surface Area

Using B.E.T plot, Nitrogen surface Area can be determined using Brunauer, Emmett and Teller equation:

$$P/V_a(P_a\text{-}P) = 1/ V_mC + C\text{-}1/V_mC \times P/P_o$$

where

P = manometer pressure in kPa;

P_o = standard vapour pressure of nitrogen (kPa);

V_m = Nitrogen volume in gram which covers one monomolecular layer in standard cm^3/g; and

C = B.E.T constant. Numerical value depends on heat of adsorption of monomolecular layer.

Plot $P/V_a(P_o\text{-}P)$ on the Y-axis and P/P_o on X axis for data sets with P/P_o in 0.05 to 0.30 range.

Three or more data points which gives best straight line are used to calculate the slope and Y-intercept. The Y-intercept and the slope are used to calculate the surface area.

Negative Y-intercept in B.E.T plot, may indicate the presence of micropores (≤ 2 nm diameter), but other factors can also produce negative intercept.

Calculate Nitrogen Surface Area to the nearest 0.1 m^2/g as follows:

$$\text{Surface Area (m}^2\text{/g)} = Vm \times 4.35$$

where

$V_m = \dfrac{1}{B + M}$

B = Y axis intercept, $\pm 10^{-5}$

M = slope of straight line, $\pm 10^{-5}$ and

4.35 = Area which 1 cm^3 of nitrogen occupy = (6.02×10^{23}) $(16.2 \times 10^{-20})/22\,400$

6.023×10^{23} = Avogadro's Number

16.2×10^{-20} = Nitrogen molecule area (in m^2)

22400 = Number of cm^3 occupied by one mole of gas at STP

H-6.3 Statistical Thickness Surface Area (STSA)

Determine STSA of carbon black using a plot of Nitrogen gas adsorbed volume per gram of the sample at STP (V_a) versus the statistical layer thickness (t).

Prepare V_a-t plot by plotting t (nm) on the X-axis versus V_a (dm^3/kg at STP) on the Y-axis with data sets having P/P_o equally spaced in the range of 0.2 to 0.5.

where

t = statistical layer thickness of carbon black = $088\ (P/P_o)^2 + 0.645\ (P/P_o) + 0.298$

Slope can be determined by the use of standard linear regression.

Calculate STSA to nearest 0.1 m^2/g as follows:

$$\text{STSA} = M \times 15.47$$

where

M = slope of V_a-t plot; and

15.47 = a constant for the nitrogen gas conversion to liquid volume and conversion of units to m^2/g.

ANNEX J

(*Clause* 3, *Table* 1)

J-1 SIEVE RESIDUE

This method is essentially based on ISO/R 1437-1970 Carbon black for use in the rubber industry.Method of test for sieve residue' and ASTM D15 14-60 Sieve residue from carbon black'.

J-2 APPARATUS

The apparatus consists of a metal funnel terminating at the foot in a short cylindrical outlet in which is inserted a shallow removable cup. To the bottom of the cup, wire mesh of a suitable aperture size is soldered. Water under a pressure of 2.10 ± 0.35 kgf/cm^2 is supplied by a tube fitted with a nozzle designed to discharge a spreading jet through the sieve and the tube is so arranged that the distance of the orifice from the sieve can be adjusted. The tube is provided with a mesh (37 micron aperture) filter to exclude any solid particle from the water. The area of this filter should be sufficiently large to prevent loss of water pressure.

A similar arrangement is required for another tube which is used to supply a gentler stream of water for wetting the powder and washing the sample off the sides of the funnel.

J-3 PROCEDURE

Fit a suitable size sieve to the base of the funnel. Allow water from the jet to pass through the sieve cup at full pressure for 3 minutes. Examine the sieve for particles and if there are none the apparatus is ready for use. Turn on the main jet and add 100 ± 1 g of sample slowly to the funnel, taking care to prevent plugging of the sieve. Using a gentle stream of water from the subsidiary supply, wash down any of the sample adhering to the sides. Continue the flow of water until the water leaving the sieve is clear. Turn over off the water, remove the sieve cup and rub the residue lightly with the finger to break up any agglomerates. Replace the sieve cup and wash for a further two minutes with the subsidiary supply. Any grit remaining on the sieve should be transferred to a watch-glass dried at 105 °C, and weighed.

NOTE — If necessary the sample can he first dispersed in water using a high speed stirrer and a wetting agent.

J-4 CALCULATION

Sieve residue, percent by mass = A/M × 100

where

A = mass of residue, in g; and

M = mass of sample in g.

ANNEX K

(*Clause* 3, *Table* 1)

K-1 LOSS ON HEATING

This method is based on NO/R 1126-1969 'Carbon black for use in the rubber industry -Determination of loss on heating' and ASTM Dl509-'Heating loss of carbon black'.

K-2 APPARATUS

K-2.1 Oven — Capable of temperature regulation of ± 2°C at 105°C.

K-2.2 Weighing Bottle — Suitable low form with stopper.

K-3 PROCEDURE

Dry the weighing bottle and stopper at 105°C for 30 min. Cool in a desiccator and weigh to the nearest 0.1 mg. Weigh accurately approximately 2 g of sample into weighing bottle with stopper on. Place the weighing bottle in the oven, remove the stopper and leave for 1 hour at 10.5 ± 2°C. Transfer to a dessiccator, replace stopper, cool to room temperature and reweigh to the nearest 0.1 mg.

K-4 PRECAUTIONS

Take the sample of carbon black in a tightly stoppered glass bottle. Allow the closed container to reach room temperature before starting the test.

Keep the stopper on the weighing bottle when transferring to and from the desiccator to prevent loss of carbon black due to air currents.

K-5 CALCULATION

Loss on heating, percent by mass = $\frac{(B-C)}{(B-A)} \times 100$

where

B = mass of weighing bottle with stopper and sample before heating,

C = mass of weighing bottle with stopper and sample after heating, and

A = mass of weighing bottle with stopper.

ANNEX M

(*Clause* 3, *Table* 1)

M-1 TOLUENE DISCOLOURATION

This method covers the determination of the degree of discolouration of toluene by carbon back by means of a spectrophotometer. This method is not applicable to high extract thermal type blacks. This method is essentially based on ASTM D 1618-81 'Carbon black extractable'.

M-2 APPARATUS

M-2.1 Spectrophotometer — 20 nm maximum spectral band pass capable of measuring transmittance in the 425 nm range.

M-2.2 Absorption Cells — With an optical light path of 10 mm (light path of the toluene and *not* the outside of the cell) for the spectrophotometer.

M-2.3 Analytical Balance — Sensitivity 0.01 mg.

M-2.4 Oven — Gravity convection type capable of temperature regulation of ±1 deg at 105°C.

M-2.5 Filter Paper — Whatman No. 41 or equivalent, diameter 150 mm.

M-2.6 Mechanical Shaker — Capable of 240 strokes/ minute.

M-2.7 Mortar and Pestle

M-2.8 Glass Filtering Funnel

M-2.9 Beaker

M-2.10 Erlenmayer Flask

M-3 REAGENT

M-3.1 Toluene — Analytical grade

M-4 PROCEDURE

M-4.1 Standardization of Apparatus

Clean the absorption cell with the lense tissue. Allow the sepectrophotometer to warm up at least 10 minutes before standardization. Rinse the cell twice with clean toluene, wipe the outside surface with lens tissue. Adjust the transmission value to 100 percent on the spectrophotometer, using the wave length of 425 nm.

Crush pelleted samples using mortar and pestle or equivalent. Dry an adequate amount of crushed carbon black sample at 105°C for 60 minutes using the oven.

NOTE — An infra-red lamp must not be used for drying sample as it could vapourize some of the extractible materials.

Allow sample to cool to room temperature in a closed container. Weigh 2.0 g of sample and transfer to a 125 ml Erlenmeyer flask. Add 20 ml of toluene to the sample in the flask and stopper. Without delay, begin shaking the mixture in the mechanical shaker for 60 seconds. Immediately pour as much of the mixture as possible into the glass funnel with filter paper which has previously been prepared and inserted into a 125-cm^3 Erlenmeyer flask. Check standardization of spectrophotometer at 425 nm. Using the same cell as used to standardize before testing, rinse the cell twice with the same filtrate to be tested. Fill the absorption cell with the filtrate and determine the percentage transmission on the spectrophotometer at 425 nm. If necessary larger quantities of sample and toluene may be used, keeping the ratio 10 ml of toluene per gram of back unchanged, to a maximum of 5 g/50 ml of toluene.

ANNEX N

(*Clause* 3, *Table* 1)

N-1 FINES CONTENT

This method is based on ISO/R 1435-1970 'Pelletized carbon black for use in the rubber industry - Determination of fines content', and ASTM D 1508-60 "Fines content of pelletid carbon black".

N-2 APPARATUS

N-2.1 Test Sieves — Three, conforming to IS 460 (Part 1) with apertures of 125 microns, and three sieve of any size wihch are used only as spacers.

N-2.2 Five Separator Receivers

N-2.3 One Sieve Cover

N-2.4 One Bottom Receiver Pan

N-2.5 Mechanical Test Sieve Shaker

N-2.6 Alternative Test Sieve Shakers — may be used as long as they give the same results as the ro-tap.

N-3 PROCEDURE

Stack the sieves in the following order from bottom to top : Bottom receiver- 125 micron sieve, separator receiver - 125micron sieve, separator receiver- 125micron sieve and three additional separator receivers and sieves placed alternately on top. The top sieve is covered with the sieve cover. Three 25 g representative samples shall be weighed to the nearest 0.1 g and transferred to each of the 125 micron sieves. Transfer the assembly to the mechanical shaker and allow to shake for 5 mins. Weigh the contents in each receiver to the nearest 0.1 g.

N-4 CALCULATION

$$\text{Fines content, percent by mass} = A/M \times 100$$

where

A = mass in g, of the sample in receiver; and

M = mass in g, of the original sample;

Report — The report shall include the results obtained from the three individual determinations and their average.

ANNEX O

(*Foreword*)

EXPERTS WHO MADE SIGNIFICANT CONTRIBUTION TO THE DEVELOPMENT OF THIS STANDARD

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