Indian Standard

METHODS OF SAMPLING AND TEST FOR CARBON BLACK

(First Revision)

UDC 678·4·046·2 : 620·113

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 6 *June* 1986

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Indian Standard

METHODS OF SAMPLING AND TEST FOR CARBON BLACK

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 11 November 1985, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1974 and is now being revised to include a new test method, namely, toluene discolouration test.

0.3 Carbon blacks play an important role as reinforcing agent in rubber products. For rubber products to perform well in actual use, it is essential that all the ingredients which go into them are of proper quality. To help manufacturers and users of carbon black to evaluate properly the quality, this standard has been formulated.

0.4 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*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the methods of sampling and test for carbon blacks used in the rubber industry.

2. SAMPLING

2.1 General

2.1.1 Packaged shipments of carbon blacks are generally delivered in packages of 25 kg. The packages shall be grouped in unit loads, stacks, or may be loaded in tiers of loose bags. In each case, a shipment involves a given number of packages, units, tiers or a definite total mass.

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*Rules for rounding off numerical values (*revised*).

2.1.2 In a consignment all the packages of carbon black belonging to the same source and single batch of manufacture shall be grouped together to constitute a lot. Samples shall be taken from each lot, either at the point of manufacture or at the place of delivery. The number of packages to be taken from each lot shall be as given in Table 1 and they shall be selected at random with the use of a random number table (*see* IS : 4905-1968*) or in its absence in the following manner:

> Starting from any pack count them as 1, 2, ... *r* where *r* is the integral part of *N*/*n*, *N* being the number of packages in the lot and *n* being the number to be sampled according to Table 1. Every *r*th package will be included in the sample.

TABLE 1 NUMBER OF BAGS TO BE SAMPLED

No. of Bags in the Lot (*N*)	No. of Bags to be Sampled (*n*)
Up to 50	3
51 to 150	5
151 and above	8

2.1.3 The size of the individual sample taken shall be related to the tests to be made on the material. It is recommended that no sample be smaller than 1 kg in mass or 3 litres in volume.

2.2 Apparatus

2.2.1 *Sampling Device* — It shall consist of an open polyethylene tube, of diameter not less than 25 mm, which can be inserted into the package to its approximate geometric centre. The carbon black sample can be poured through this tube from the geometric centre of the package (*see* Fig. 1).

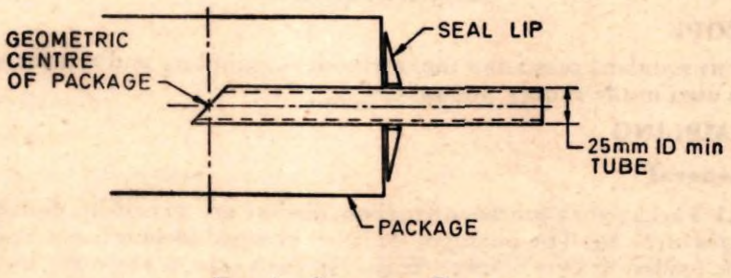

Fig. 1 Sampling Device

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*Methods for random sampling.

2.3 Sampling Procedure — Place the bag of carbon black to be sampled in an upright position. Adjust the seal lip on the tube at a distance from the bevelled and corresponding to the depth required to reach the geometric centre of the bag. Make a slit at the approximate centre of the upper end of the bag. Insert the sampling tube into the cut bag with the bevelled end pointing downward to the required depth. Hold the seal lip against the bag to act as a seal. Turn the bag up-side down to allow the carbon black to flow through the tube into an adequate container. Discard aporoximately the first half litre of sample material. Remove a sufficiently large sample for testing. Finally, open the bag once again, remove the sampling device and seal the bag opening. Take care not to break up the pellets with the sampling device.

2.4 Treatment and Storage of Samples

2.4.1 Sample as given in **2.1.3** shall be collected from all the bags and mixed thoroughly in order to make a composite sample. Prior to testing, pass the composite sample in a single-stage riffle-type sample splitter at least twice in order to prevent stratification. This is particularly important for tests of pellet quality. The composite sample shall be testd for all the requirements.

2.4.2 Handle the samples collected for the determination of the pellet quality with care to avoid breakdown of the pellets.

2.4.3 Store the samples in airtight containers until the tests are completed.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. PELLET SIZE DISTRIBUTION

4.0 This method is essentially based on ASTM D 1511-60 'Pellet size distribution of carbon black' (*Reapproved* 1969), issued by the American Society for Testing and Materials, USA.

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*Specification for water for general laboratory use (*second revision*).

4.1 Apparatus

4.1.1 *Ro-Tap Sieve Shaker* — This is a mechanically operated sieve shaker which imparts a uniform rotary and tapping motion to a stack of 200-mm sieves. The ro-tap should be adjusted to accommodate a stack of five sieves, receiver pan, and cover plate. The bottom stops should be adjusted to give a clearance of 1·55 mm between the bottom plate and the screens so that the screens will be free to rotate. The Ro-tap machine shall be powered with an electric motor producing 1 725 to 1750 rev/min. This will produce 140 to 160 taps per minute and 280 to 320 rotary motions per minute. The cover plate on the Ro-tap shall be fitted with a cork-stopper which shall extend from 3 to 9 mm above the metal recess. At no time shall a rubber, wood or other material than cork be permitted. Alternative machanical shakers may be used as long as they can be shown to give the same results as the Ro-tap.

4.1.2 *Sieves* — IS Sieves [*see* IS 460 (Part 1) -1985*] having openings of 2·00 mm, 1·00 mm, 500 microns, 250 microns and 125 microns.

4.1.3 *Bottom Receiver Pan and Top Sieve Cover*

4.2 Procedure — A representative sample of at least 100 g should be prepared. Prepare the nest of sieves by fitting each sieves one on top of the other, with the largest aperture sieve at the top and the smallest at the bottom. Place a receiver under the bottom sieve. To the top sieve add the sample weighed to the nearst 0·1 g. Cover the top sieve with a lid and instal the nest of sieves in the mechanical shaker. Allow the sieve assembly to shake for 60 to 70 seconds. Weigh to the nearest 0·1 g the contents of each sieve and the receiver.

4.3 Expression of Results — Express the mass retained on each sieve and on the receiver as a percentage of the total sample recovered.

5. IODINE ADSORPTION NUMBER

5.0 This method is essentially based on ISO 1304-1974 'Carbon black for use in the rubber indstury' — Determination of iodine adsorption number'. This method is not suitable for carbon blacks containing over 1·5 percent volatile matter or over 0·25 percent benzene extract.

5.1 Apparatus

5.1.1 *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.

5.1.2 *Oven* — capable of maintaining a temperature of 105 ± 2°C.

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*Specification for test sieves: Part 1 Wire cloth test sieves (*third revision*).

5.1.3 *Burette* — 25 ml, graduated in 0·05 ml; or 10 ml, graduated in 0·02 ml.

5.1.4 *Pipettes* — 25 ml and 20 ml, calibrated to the nearest 0·01 ml.

5.1.5 *Centrifuge* — capable of speed above 1 000 rev/min. If a centrifuge is not available then porcelain filter crucible may be used.

5.1.6 *Balance* — analytical, with a sensitivity of at least 0·1 mg.

5.2 Reagents

5.2.1 *Iodine Solution* — 0·047 3 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.

5.2.2 *Standard Sodium Thiosulphate Solution*—0·039 4 N Dissolve 9·781 0 g of sodium thiosulphate pentahydrate ($Na_2S_2O_3 . 5H_2O$), 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.

5.2.3 *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 minutes. With stirring, cool and store in bottles. A suitable soluble starch indicator may also be used.

5.3 Procedure — Dry an adequate representative sample for 1 hour at 105°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 125 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. Through mixing of the carbon black and iodine solution is necessary. Centrifuge immediately for at least one minute at over 1 000 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 100-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.

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

5.4 Expression of Result

$$I = (B - S)\frac{N}{M} \times 158{\cdot}64$$

where

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

$158{\cdot}64$ = dilution factor × equivalent weight of iodine

$$= \frac{25}{20} \times 126{\cdot}91$$

6. DIBUTYL PHTHALATE ABSORPTION (DBP)

6.0 This method is essentially based on ASTM D 2414-65. 'Dibutyl phthalate absorption number of carbon black'.

6.1 Apparatus

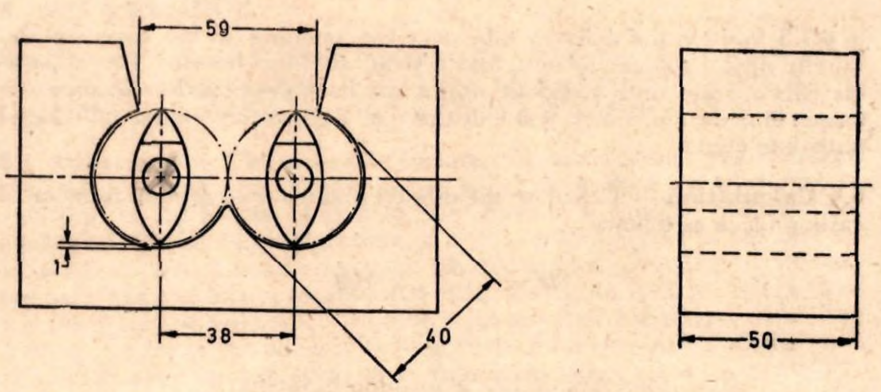
6.1.1 *Absorptometer* — Equipped with a constant-rate burette. The absorptometer consists of three sections: a motor-drive system, a mixing chamber with rotor blades, and torque sensing system. The motor-drive system includes a gear arrangement for a choice of rotor speeds. The essential features of the mixing chamber and rotor blades are shown in Fig. 2. The rotor blades mix the test specimen with a liquid fed at a constant rate from the burette. As mixing approaches the end point, the torque generated is resisted by a spring and a dashpot, having a damper control valve, in the torque-sensing system. The motor and associated burette are automatically cut off at the end point when the torque exceeds a pre-selected limit.

6.1.2 *Oven* — gravity-convection type, capable of temperature regulation of ± 2°C at 105°C.

6.1.3 *Analytical Balance* — sensitivity 0·1 mg.

6.2 Reagent

6.2.1 *Dibutyl Phthalate* — having a specific gravity of 1·045 to 1·050 at 27°C.



All dimensions in millimetres.

FIG. 2 ESSENTIAL FEATURES OF MIXING CHAMBER AND ROTOR BLADES

6.3 Procedure

6.3.1 Adjust the absorptometer to the following conditions:

Spring	Set at tension of 2·68 kgf/cm
Rotor speed	125 rev/min
Torque limit switch	Set at 5
Damper valve	0·150 initial setting

Final adjustments to be made in accordance with the results obtained on the dibutyl phthalate standard blacks.

6.3.2 Adjust and fill the automatic burette with dibutyl phthalate as follows:

> Set the burette selector in the 60 ml/min position. Connect the burette to the dibutyl phthalate supply. Activate the manual switch to draw the plunger into the barrel. When the plunger stops, reverse the stopcock position, place the delivery tube over waste container, and activate the delivery switch. Repeat the procedure until the barrel and the delivery tube have been filled to capacity with the reagent.

6.3.3 Set the burette selector to the 4 ml/min position and the switches to automatic delivery positions.

6.3.4 Dry an adequate sample of black for 1 hour at 105 ± 2°C. Weigh 20 ± 0·01 g of the black and transfer to the absorptometer mixing chamber, and replace the chamber cover.

6.3.5 Position the delivery tube over the opening in the cover, set the burette digital counter to zero, and activate the start button. The apparatus will operate until sufficient torque has been developed to activate the torque limit switch, which will halt the test. Record the volume of dibutyl phthalate used.

6.4 Calculation — Calculate the dibutyl phthalate absorption number of carbon black as follows:

$$D = \frac{A}{B} \times 100$$

where

D = dibutyl phthalate absorption in ml/100 g,
A = volume in ml of dibutyl phthalate used, and
B = mass in g of the sample used.

7. POUR DENSITY

7.0 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 1513-60 'Pour density of pelletid carbon black' (*Reapproved* 1969).

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

7.2 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 thg 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.

7.3 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.

8. SIEVE RESIDUE

8.0 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 D '1514-60 Sieve residue from carbon black' (*Reapproved* 1969).

8.1 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.

8.1.1 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.

8.2 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 futher 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 be first dispersed in water using a high speed stirrer and a wetting agent.*

8.3 Calculation

$$\text{Sieve residue, percent by mass} = \frac{A}{M} \times 100$$

where

A = mass of residue in g, and

M = mass of sample in g.

9. LOSS ON HEATING

9.0 This method is based on ISO/R 1126-1969 'Carbon black for use in the rubber industry — Determination of loss on heating' and ASTM D1509-59 'Heating loss of carbon black' (*Reapproved* 1969).

9.1 Apparatus

9.1.1 *Oven* — capable of temperature regulation of ± 2°C at 105°C.

9.1.2 *Weighing Bottle* — suitable low form with stopper.

9.2 Procedure — Dry the weighing bottle and stopper at 105°C for 30 minutes. 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 105 ± 2°C. Transfer to a desiccator, replace stopper, cool to room temperature and reweigh to the nearest 0·1 mg.

9.3 Precautions

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

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

9.4 Calculation

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

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.

10. ASH CONTENT

10.0 This method is based on 'ISO/R 1125-1969 Carbon black for use in the rubber industry — Determination of ash contens' and ASTM D 1506-59 'Ash content of carbon black' (*Reapproved* 1969).

10.1 Apparatus

10.1.1 *Muffle Furnace* — capable of temperature regulation of ± 25°C at 550°C.

10.1.2 *Silica or Porcelain Crucible* — high form, capacity 15 ml, with cover.

10.2 Procedure — Ignite the crucible and cover in the muffle furnace as 550 ± 25°C for 1 hour. 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 hour at 105°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 hours at 550 ± 25°C. Remove from furnace and replace cover and cool in a desiccator to room temeperature. 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 hours.

10.3 Precautions

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

10.3.2 Exert care in removing ashed sample from furnace to desiccator.

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

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

10.4 Calculation

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

where

D = mass of the crucible and ash,
B = mass of the crucible, and
C = mass of the crucible and sample.

10.4.1 Report the average of at least two determinations.

11. ACETONE EXTRACT

11.1 Apparatus

11.1.1 *Soxhlet Extraction Apparatus* — with 20 to 30 ml cup.

11.1.2 *Flat-Bottomed Flask* — 150 ml.

11.1.3 *Whatman Type Paper Extraction Thimbles* — 25 × 100 mm.

11.2 Reagent

11.2.1 *Acetone*

11.3 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 desiccator and weigh.

11.4 Calculation

$$\text{Acetone extract, percent by mass} = \frac{A}{M} \times 100$$

where

A = mass of residue from the sample determination, and

M = mass of sample.

12. STAINING TEST

12.0 In this test the colour of toluene extract of carbon black is compared with that of tole toluene. It is a measure of the staining tendency.

12.1 Apparatus — two matched Nessler cylinders.

12.2 Reagent — toluene.

12.3 Procedure — Into a Nessler cylinder, weigh 1 g of sample to the nearest 0·01 g. Add 10 ml of toluene and shake for 1 minute. Allow to settle and compare the colour of the supernatant layer with that of toluene alone in the other Nessler cylinder, by viewing against a white background through the side.

12.4 The supernatant liquid should be of the same colour as that of the pure toluene. If it is not, a staining test should be carried out on rubber.

13. FINES CONTENT

13.0 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' (*Reapproved* 1969).

13.1 Apparatus

13.1.1 *Test Sieves* — three, conforming to IS : 460 (Part 1)-1985* with apertures of 125 microns, and three sieve of any size wihch are used only as spacers.

13.1.2 *Five Separator Receivers*

13.1.3 *One Sieve Cover*

13.1.4 *One Bottom Receiver Pan*

13.1.5 *Mechanical Test Sieve Shaker* — Such as that described in **4.1.1.** Alternative test sieve shakers may be used as long as they give the same results as the ro-tap.

13.2 Procedure — Stack the sieves in the following order from bottom to top:

> Bottom receiver, 125-micron sieve, separator receiver, 125-micron sieve, separator receiver, 125-micron 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 min ± 10 seconds. Weigh the contents in each reciever to the nearest 0·1 g.

13.3 Calculation

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

where

A = mass in g of the sample in receiver; and
M = mass in g of the original sample.

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

14. SULPHUR CONTENT

14.0 Two methods for determination of the total sulphur in all types of carbon black for use in the rubber industry are described — Method A, using an oxygen bomb calorimeter; and Method B using a combustion furnace. These methods are based on ISO/R 1138-1969 'Determination of total sulphur content of carbon black for the rubber industry'.

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*Specification for test sieves : Part 1 Wire cloth test sieves (*third revision*).

14.1 Method A — Oxygen Bomb Calorimeter

14.1.0 *Principle of the Method* — A test portion of dried carbon black is weighed and ignited in an oxygen bomb calorimeter. The bomb is opened, the inner surfaces washed with water, and the washings collected in a beaker. The sulphur in the washings is precipitated as barium sulphate which is collected and weighed, and the percentage or sulphur is calculated.

14.1.1 *Reagents*

14.1.1.1 *Barium chloride solution* — 100 g/l. Dissolve 100 g of barium chloride ($BaCl_2 2H_2O$) in water and dilute to 1 litre.

14.1.1.2 *Hydrochloric acid* — relative density 1·155.

14.1.1.3 *Picric acid solution* — saturated. Add an excess of picric acid to water in a glass-stoppered bottle. Shake the bottle well to dissolve enough picric acid to saturate the solution. A few crystals of picric acid should remain in contact with the solution after saturation.

14.1.1.4 *Oxygen* — A supply of oxygen, such as from commercial cylinders, of adequate purity (free from sulphur compounds).

14.1.2 *Apparatus*

14.1.2.1 *Oxygen bomb calorimeter* — complete with ignition transformer, ignition wire, fuel capsule crucibles, oval type bucket, calorimeter jacket, electric stirrer for water calculation around bomb, and calorimetric thermometer covering the range 10 to 35°C in intervals of 0·02°C.

14.1.2.2 *Oxygen cylinder* — with regulator and connector for oxygen bomb calorimeter.

14.1.2.3 *Crucible* — porcelain, 30-ml capacity.

14.1.2.4 *Oven* — preferably gravity convection type, capable of maintaining a temperature of 105 ± 2°C.

14.1.2.5 *Muffle furnace* — capable of maintaining a temperature of 925 ± 25°C.

14.1.2.6 *Filter funnels* — bunsen filtering long-stem, 60° angle fluted.

14.1.3 *Procedure*

14.1.3.1 Dry an adequate amount of the sample for 1 hour at a temperature of 105 ± 2°C. Weigh, to the nearest 0.1 mg, about 0·5 g of the dried sample in the fuel capsule. Place the crucible in the loop electrode and attach a fuse wire as described in the instructions for the oxygen bomb calorimeter. Bend the fuse wire so that the wire extends just below the surface of the carbon black. Pipette 5 ml of water into the bomb cylinder. Assemble the bomb and fill with oxygen (30 kgf/cm^2). Put 2 litres of water, at ambient temperature, in the calorimeter, bucket. Place the bucket in the calorimeter, set the bomb in the bucket, attach the thrust terminal to the bomb, close the calorimeter with the thermometer in place, and start the electric stirrer. Allow the electric stirrer to operate for 2 minutes and then record the water temperature. Close the ignition circuit momentarily to fire the charge. If the ignition system has no pilot light, hold the ignition switch button for 5 seconds before releasing. Observe the rise in water temperature. The temperature should rise about 2°C in approximately 5 minutes (If the temperature fails to rise, the test should be abandoned and a fresh determination begun). Allow the electric stirrer to operate for a total of 10 minutes after ignition. Remove the bomb, and release the pressure gradually over a period of time of at least 1 minute.

14.1.3.2 Open the bomb and wash all the inner surfaces of the bomb with water, catching all the washings in a 250-ml beaker. Filter the solution into a 400-ml beaker, washing the filter paper with three 5 ml portions of water. Add 5 ml of hydrochloric acid, 5 ml of saturated picric acid solution, and 10 ml barium chloride solution to the filtrate. Digest the mixture on an electric hotplate at low temperature for approximately 15 minutes. Filter through a low ash paper and wash the precipitate in the paper with 10 ml portions of boiling water. Continue washing until a test with silver nitrate shows absence of chloride in the filtrate. Transfer the filter paper with the precipitate to a porcelain crucible, weighed to the nearest 0.1 mg. Char and ignite over the low flame of a high-temperature gas burner. Transfer to a muffle furnace at a temperature of 925 ± 25°C for 30 minutes. Remove the crucible and contents to a desiccator, allow to cool to ambient temperature and weigh to the nearest 0·1 mg.

omb

NOTE — Treatment of ash after incineration with sulphuric acid is desirable to convert barium sulphide formed, if any, into barium sulphate.

14.1.4 *Calculation*

$$\text{Total sulphur, percent by mass} = \frac{0{\cdot}137\,3\ (B - A)}{C} \times 100$$

where

B = mass in g of the crucible and precipitate after ignition,

A = mass in g of the crucible, and

C = mass in g of the dried test portion.

14.2 Method B — Combustion Furnace

14.2.0 *Principle of Method* — A test portion of dried carbon black is weighed and heated at a temperature of 1 425°C in a combustion furnace in a stream of oxygen. Sulphur compounds evolved from the test portion are collected in a titrating flask containing hydrochloric acid solution, then titrated with potassium iodate-potassium iodide solution, and the percentage of sulphur is calculated.

14.2.1 *Reagents*

14.2.1.1 *Hydrochloric acid* — Mix 2 volumes of concentrated in hydrochloric acid (relative density 1·155) with 13 volumes of water.

14.2.1.2 *Potassium iodate-potassium iodide solution* — Dissolve 1·112 5 g of potassium iodate (KIO_3), 100 g of potassium iodide (KI), and 5 g of potassium hydroxide in approximately 500 ml of water. Dilute to 1 litre in a volumetric flask.

NOTE — The solution should be stored in a brown or green glass bottle. It is advisable to prepare a fresh solution every 30 days.

14.2.1.3 *Starch indicator* — Dissolve 2·5 g of starch and 5 mg of mercuric iodide ($Hg I_2$) in 1 litre of water.

14.2.1.4 *Oxygen* — A supply of oxygen, such as from commercial cylinders, of adequate purity (free from sulphur compounds).

14.2.2 *Apparatus*

14.2.2.1 *Combustion furnace* — suitable for use at a temperature of 1 425 ± 25°C.

14.2.2.2 *Combustion tube* — about 75 cm in length, with one end tapered.

14.2.2.3 *Combustion boats* — either of fused aluminium oxide (alundum) or of porcelain.

NOTE — All new combustion boats should be fired for 1 hour at 1425 ± 25°C prior to use.

14.2.2.4 *Pressure regulator and flowmeter*

14.2.2.5 *Oxygen purification tube* — consisting of a gas-washing bottle containing concentrated sulphuric acid (relative density 1·834) a water absorbing bottle filled with a mixture of soda asbestos and anhydrous calcium sulphate that has been saturated with carbon dioxide.

14.2.2.6 *Gas dispersion tube* — of coarse fritted glass.

14.2.2.7 *Titrating flasks* — about 500-ml capacity.

14.2.2.8 *Rubber stopper* — equipped with a high-temperature heat reflector or baffle made of either metal or a refractory.

14.2.2.9 *Oven* — preferably gravity convection type, capable of maintaining a temperature of 105 ± 2°C.

14.2.3 *Procedure* — Dry an adequate amount of carbon black for 1 hour at a temperature of 105 ± 2°C. Weigh, to the nearest 1 mg, about 1 g of the dried a sample and transfer to a combustion boat. Fill the titrating flask one-third full with hydrochloric acid, add 2 mlo f the starch indicator and, while agitating the solution, add just enough of the potassium iodate-potassium iodide solution to give a perceptible colour. Place the gas-dispersion tube in the titrating flask. With the combustion furnace at a temperature of 1 425°C and the oxygen flowing at 1 litre per minute (*see* Note), insert the combustion boat containing the carbon black into the hot zone of the combustion tube. Stopper the combustion tube immediately so as to pass the combustion gases evolved through the gas-dispersion tube. Titrate the solution in the titrating flask with the potassium iodate-potassium iodide solution to maintain the blue colour in the titrating flask. Consider the final point to be reached when after 3 minutes of bubbling, it is not necessary to add potassium iodate-potassium iodide solution to maintain the blue colour in the titrating flask.

NOTE — Prior to each series of determinations, the combustion system should be purged for 30 minutes with the oxygen flowing and with the furnace temperature at 1 425 ± 25°C.

14.2.4 *Calculation*

$$\text{Total sulphur, percent by mass} = \frac{A}{20\,B}$$

where

A = volume in ml of potassium iodate-potassium iodide solution required for the titration, and

B = mass in g of the test portion.

15. *p*H VALUE

15.0 This method is essentially based on ASTM D 1512-1960 '*p*H value of carbon black (*reapproved* 1969)'.

15.1 Apparatus

15.1.1 *pH Meter* — equipped with glass and calomel electrodes, having an accuracy of ± 0·05 *p*H.

15.1.2 *Beakers and Watch Glasses* — made of neutral glass and of sufficient size to accommodate the sample used.

15.1.3 *Hot Plate*

15.1.4 *Glass or Porcelain Plate and Spatula or Mortar and Pestle*

15.1.5 *Container* — Stainless steel or copper, for boiling distilled water.

15.2 Procedure

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

15.2.2 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.

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

15.2.4 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.

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

15.2.6 Place the electrodes in the sludge and rotate the beaker gently in alternate direction until a constant *p*H is obtained.

15.2.7 Repeat the procedure on a second sample.

15.3 Precautions

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

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

15.3.3 The distilled water used in the test should be as pure as possible. The *p*H of the freshly boiled distilled water shall be 6·9 to 7·1.

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

16. TOLUENE DISCOLOURATION

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

16.1 Apparatus

16.1.1 *Spectrophotometer* — 20 nm maximum spectral band pass capable of measuring transmittance in the 425 ± 5 nm range.

16.1.2 *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.

16.1.3 *Analytical Balance* — Sensitivity 0·01 mg.

16.1.4 *Oven* — Gravity convection type capable of temperature regulation of ± 1° at 105°C.

16.1.5 *Filter Paper* — Whatman No. 41 or equivalent, diameter 150 mm.

16.1.6 *Mechanical Shaker* — Capable of 240 strokes/minute.

16.1.7 *Mortar and Pestle*

16.1.8 *Glass Filtering Funnel*

16.1.9 *Beaker*

16.1.10 *Erlenmayer Flask*

16.2 Reagent

16.2.1 *Toluene* — Analytical grade.

16.3 Procedure

16.3.1 *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.

16.3.2 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.

16.3.3 Allow sample to cool to room temperature in a closed container. Weigh 2·00 ± 0 01 g of sample and transfer to a 125-cm^3 Erlenmeyer flask Add 20 cm^3 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 spectrophotemeter 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.

(*Continued from page* 2)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	*Pa*	1 Pa = 1 N/m^2