INTERNATIONAL STANDARD

ISO 6810

Second edition
1995-10-01

Rubber compounding ingredients — Carbon black — Determination of surface area — CTAB adsorption methods

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de la surface spécifique — Méthodes par adsorption de CTAB

Reference number
ISO 6810:1995(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 6810 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 6810:1985), which has been technically revised.

Annexes A and B of this International Standard are for information only.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Rubber compounding ingredients — Carbon black — Determination of surface area — CTAB adsorption methods

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies methods for the determination of the surface area of carbon blacks, excluding the area of micropores that are too small to admit molecules of hexadecyltrimethylammonium bromide (cetyltrimethylammonium bromide, commonly referred to as CTAB).

The methods are suitable for characterizing rubber grade carbon blacks of all types.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1126:1992, *Rubber compounding ingredients — Carbon black — Determination of loss on heating.*

ISO 1304:1985, *Rubber compounding ingredients — Carbon black — Determination of iodine adsorption number — Titrimetric method.*

ISO 4652-1:1994, *Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Part 1: Single-point procedures.*

ISO 6809:1989, *Rubber compounding ingredients — Carbon black — Standard reference blacks.*

3 Principle

3.1 The isotherm for adsorption on carbon black of CTAB from an aqueous solution has a long horizontal plateau corresponding to monomolecular coverage of the substrate surface from which the adsorbate is not sterically excluded. The CTAB adsorption by carbon black is not affected by tarry materials and functional groups containing hydrogen, oxygen, etc. Rapid equilibrium is achieved by using mechanical stirring and ultrasonic vibration. Titration with sodium di(2-ethylhexyl) sulfosuccinate solution to a maximum turbidity or colour end-point is used to determine the unadsorbed CTAB after removal of the colloidally dispersed carbon black by filtration. All results are determined relative to a reference black, the CTAB surface area of which is assumed to be exactly as specified in ISO 6809.

3.2 Titration of the unadsorbed CTAB is carried out using one of the following methods:

a) method 1, using sodium di(2-ethylhexyl) sulfosuccinate solution by automatic titrimeter to a maximum-turbidity end-point;

b) method 2, using sodium di(2-ethylhexyl) sulfosuccinate solution by manual titration to a maximum-turbidity end-point;

c) method 3, using sodium di(2-ethylhexyl) sulfosuccinate solution by manual titration to a specified colour end-point;

d) method 4, using sodium dodecyl sulfate (SDS) solution by manual titration to a specified colour end-point.

4 Reagents

All reagents shall be of recognized analytical grade. Distilled water, or water of equivalent purity prepared by passing it through a fixed bed of ion-exchange materials, shall be used. The purified water shall be stored in suitable vessels, and transfer tubing shall be made of polytetrafluoroethylene, polyethylene, quartz, or other materials resistant to chemical attack.

4.1 Buffer solution, of pH 7.

Dissolve 2,722 g of potassium dihydrogen orthophosphate (KH_2PO_4), 4,260 g of disodium hydrogen orthophosphate (Na_2HPO_4) and 1,169 g of sodium chloride (NaCl) in water and dilute to 1 dm^3.

NOTE 1 Further details concerning the preparation are given in BATES *et al.*, *J. Res. NBS*, **29** (1942), p.183. An equivalent buffer solution is available commercially.

4.2 Hexadecyltrimethyl ammonium bromide (CTAB) solution.

Dissolve 3,64 g (0,01 mol) of hexadecyltrimethyl ammonium bromide (CTAB) in 900 cm^3 of purified water in a suitable container. Add 100 cm^3 of the buffer solution (4.1) and warm the solution to a temperature of 27 °C to 37 °C to facilitate dissolution. Cool to a temperature between 22 °C and 25 °C before use.

The temperature of this solution shall not be allowed to fall below 22 °C at any time or slow crystallization will result.

4.3 Formaldehyde, 37 % (*m/m*) solution.

4.4 Sodium di(2-ethylhexyl) sulfosuccinate solution (for methods 1, 2 and 3).

Dissolve 1,00 g of solid 100 % sodium di(2-ethylhexyl) sulfosuccinate in purified water containing 2,5 cm^3 of formaldehyde solution (4.3), using the magnetic stirrer (5.5). Dilute to 1 dm^3 in a suitable polyethylene container, stirring vigorously by means of the magnetic stirrer for 48 h. Allow to stand for 12 days before standardization and use.

Stopper the container tightly and store in a cool place.

Once the container has been opened, store the solid 100 % reagent in a desiccator (5.19).

The reagent solution may be subject to slow biodegradation in the absence of formaldehyde. It shall be used within 6 months of preparation.

4.5 Octylphenoxy polyethoxyethanol, 0,15 % (*m/m*) solution (for method 1).

Dissolve 1,5 g of liquid 100 % octylphenoxy polyethoxyethanol in purified water and dilute to 1 dm^3 in a suitable container, stirring vigorously by means of a magnetic stirrer (5.5) until the solution is homogeneous.

4.6 Sodium dodecylsulfate (SDS) solution (for method 4).

Dissolve 0,606 g of sodium dodecylsulfate (SDS) in water containing 2,5 cm^3 of formaldehyde solution (4.3) and dilute to 1 dm^3 in a suitable container. Allow to stand for at least 24 h.

The purity of the solid reagent is a critical factor. If the solution is not clear (i.e. if it is cloudy or contains precipitate), the reagent is not sufficiently pure and is unsuitable for this test.

4.7 Dichlorofluorescein, ethanolic solution (indicator, pH 4 to 6, for method 4).

Dissolve 0,20 g of solid 2,7-dichlorofluorescein in 70 cm^3 of ethanol and store in a dropping bottle (5.17).

4.8 Bromophenol blue, aqueous ethanolic solution (indicator, pH 3,0 to 3,6, for method 3).

Dissolve 0,10 g of solid bromophenol blue in 10 g of ethanol in an amber dropping bottle (5.17) of capacity 60 cm^3 and add 40 cm^3 of water.

4.9 Standard reference black (see ISO 6809). Use ITRB.

5 Apparatus[1)2)]

5.1 Analytical balance, accurate to 0,1 mg.

5.2 Oven, capable of being maintained at 105 °C ± 2 °C or 125 °C ± 2 °C.

5.3 Ultrasonic bath, modified to include a magnetic stirrer and vial holder. A separate shaker/stirrer apparatus may be used.

5.4 Magnetic stirring bars, polytetrafluoroethylene coated.

diameter: 6 mm; length: 22 mm (all methods);

diameter: 10 mm; length: 32 mm (for methods 3 and 4);

diameter: 10 mm; length: 41 mm (for method 1).

5.5 Magnetic stirrer.

5.6 Dry compressed air or **dry nitrogen** (from either a line or a cylinder with regulator).

5.7 Pressure manifold, connected to the dry compressed air or dry nitrogen supply, regulated at 0,4 MPa to 0,7 MPa.

A typical pressure filtration manifold is shown schematically in figure 1.

5.8 Pressure cell, capacity 30 cm^3, of stainless steel, suitable for 0,7 MPa pressure.

It is essential that this is thoroughly cleaned after use.

5.9 Plastic membrane filters, of diameter 47 mm and with openings of aperture size 0,1 µm.

5.10 Filter holder.

It is essential that this is thoroughly cleaned after use.

5.11 Glass funnel, small.

5.12 Glass vial, of capacity 30 cm^3, with a screw cap.

5.13 Burette (for methods 2, 3 and 4), of capacity 50 cm^3, graduated in 0,1 cm^3 divisions, preferably of the automatic refilling and zeroing type with reagent reservoir, complying with the requirements of ISO 385-1, class A, or calibrated such that the proper corrections can be applied to achieve the required accuracy.

5.14 Dispenser-type pipette, capable of delivering 30 cm^3, complying with the requirements of ISO 648, class A, attached to a suitable reservoir of CTAB solution (4.2).

5.15 Pipettes, of capacity 5,00 cm^3 and 10,00 cm^3, complying with the requirements of ISO 648, class A.

---

1) The following automatic titration equipment has been found suitable for method 1:

- METTLER Memotitrator DL 20, 25 or 40 RC, available from sales offices in most countries;
- BRINKMAN Dosimat 665 burette, used with the Probe Colorimeter, available from Brinkman Instruments, Cantiague Rd, Westbury, NY, USA;
- ATMAST, available from L.A. King Manufacturing Corp., LAKO Division, P.O. Box 2415, Tulsa, OK 74101, USA.

This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the equipment listed.

2) The following filters have been found suitable:

- Gelman HT 200, Gelman No. 66199, Baxter Scientific Cat. No. F 2988-2, available from Baxter Scientific Products, 1430 Wankegan Road, McGraw Park, IL 60085, USA;
- Microfiltration Systems Cat. No. A-010A047A, available from Microfiltration Systems, 6800 Sierra Court, Dublin, CA 94566, USA;
- Millipore Cat. No. SAIJ 076 H7 filters, available from Millipore Corp., Bedford, MA 01730, USA;
- Sartorius SM 11358-047N, available from Sartorius GmbH, Weender Landstr. 94/108, D-37075 Göttingen, Germany;
- Schleicher & Schüll PH 79 (47 mm), available from Schleicher & Schüll GmbH, Hahnstr. 3, D-37586 Kassel, Germany.

This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the equipment listed.

5.16 Flat-bottom conical flasks, of capacity 100 cm^3, with ground-glass stoppers.

5.17 Dropping bottles (for methods 3 and 4).

5.18 Containers, suitable for the preparation and storage of reagent solutions.

5.19 Desiccators.

5.20 Microscope illuminator light source, or similar high-intensity incandescent spot light (for methods 2 and 3).

NOTE 2 A small single-filament clear-glass 10 W light bulb is recommended.

5.21 Automatic titration apparatus (for method 1).

5.22 Beakers, of capacity 100 cm^3, tall form.

5.23 Variable resistance, for use with the light source (5.20).

6 Preparation of the sample

Dry an adequate amount of the sample for 1 h at a temperature of 105 °C ± 2 °C or 125 °C ± 2 °C as described in ISO 1126. Allow to cool to ambient temperature in a desiccator (5.19). Keep the dried sample in the desiccator until ready for testing.

7 Test conditions

The test should preferably be carried out in a room having ambient conditions of either 23 °C ± 2 °C and (50 ± 5) % relative humidity or 27 °C ± 2 °C and (65 ± 5) % relative humidity.

It is recommended that the reagents and the apparatus be allowed to attain temperature equilibrium in the room for at least 2 h before being used.

NOTE 3 Storage of the CTAB solution (4.2) at temperatures below 22 °C will result in slow crystallization.

The test room shall be free from fumes or vapours which could contaminate the reagents and test equipment used and thus affect the results.

8 Verification of filters

Evaluate new batches of filters (5.9) to ensure normal filtration times. The maximum time taken to avoid errors shall be 8 min.

9 Procedure

9.1 Standardization of reagents

9.1.1 Dry an adequate amount of the standard reference black (4.9) as indicated in clause 6.

9.1.2 Weigh, to the nearest 0,1 mg, five test portions of this dried standard reference black to cover the range 0,20 g to 0,60 g in intervals of 0,10 g.

9.1.3 Place each test portion in a 100 cm^3 conical flask (5.16) containing a 22 mm magnetic stirring bar (5.4) and stopper the flasks. By means of an adjustable automatic dispenser-type pipette (5.14) affixed to the CTAB stock solution reservoir add 30,00 cm^3 of CTAB solution to the flask and place the stopper in position. Immerse the flask to a depth of at least 5 cm in an ultrasonic cleaning bath (5.3) modified to provide concurrent stirring, and agitate for 6 min. The water temperature in the bath shall be kept between 22 °C and 27 °C throughout the equilibration procedure, otherwise variations in adsorption equilibrium can occur. It is usual for the water temperature to rise during this operation.

This can be overcome in various ways. For example, water can be replaced if it becomes too warm, small pieces of ice can be dropped into the water or a cooling coil can be installed. However, the bath temperature shall not be allowed to fall below 22 °C. If a separate shaker/stirring apparatus is used, the following sequence is recommended:

1 min ultrasonic agitation;

1 min stirring;

1 min ultrasonic agitation;

1 min stirring;

1 min ultrasonic agitation;

1 min stirring.

9.1.4 Attach the top (threaded) part of the filter holder (5.10) to the stainless-steel pressure cell (5.8) and hand-tighten sufficiently to avoid leakage. (Polytetrafluoroethylene sealing tape can be used if necessary.) Install the filter disc (5.9) in the filter-holder base with the shiny surface facing the inlet, in accordance with the instructions furnished with the filter holder.

Pour the equilibrated carbon black suspension (see 9.1.3) through a small funnel (5.11) into the pressure cell. Connect the cell to the dry compressed air or dry nitrogen source regulated at 0,4 MPa to 0,7 MPa. Discard the first 5 cm^3 of filtrate and then collect the remainder in a clean glass vial (5.12), replacing the screw cap immediately. Gently agitate the filtrate collected to ensure homogeneity but without creating foam. If the filtrate contains any black, discard and do not re-filter.

Titrate the CTAB filtrate by one of the four methods specified in 9.1.5 to 9.1.8.

NOTE 4 Proper seating of the filter may be aided by applying suction to the bottom part of the filter holder during assembly. Care should be taken not to damage the filter by creasing or folding. Proper filter seating can be checked by pressure-testing the assembly before the suspension is added. Absence of gas flow, detectable by placing a finger over the outlet, indicates proper seating.

It is not generally practicable to titrate immediately after filtration; filtrate collection vials shall, therefore, be capable of being sealed until required.

9.1.5 Method 1 — Automatic titration of CTAB filtrate with sodium di(2-ethylhexyl) sulfosuccinate solution to a maximum turbidity end-point (see also annex B).

9.1.5.1 Prepare the automatic titration apparatus (5.21) in accordance with the manufacturer's instructions. Ascertain that the titrant reservoir contains sufficient sodium di(2-ethylhexyl) sulfosuccinate solution (4.4) and that the fluid lines and the pump head are free from air bubbles and have been sufficiently flushed with titrant. The power shall be on and the titrant reservoir stopper loosened to admit air as liquid flows out. Adjust the titrant flow rate to 10 cm^3/min or, if ATMAST automatic titration equipment is employed, to 6 cm^3/min in accordance with the manufacturer's recommendation.

9.1.5.2 Place 45 cm^3 of purified water in a tall-form beaker (5.22) containing the 41 mm magnetic stirring bar (5.4). Add 5 cm^3 of the octylphenoxy polyethoxyethanol solution (4.5).

Transfer, by means of a pipette (5.15), a 10,00 cm^3 portion of the CTAB filtrate (see 9.1.4) into the beaker, taking care to avoid formation of excessive foam. Place the beaker in the sample well of the titration apparatus and adjust the magnetic-stirrer speed control so that the vortex generated by the stirring action is just at the top of the light beam which passes through the beaker.

Lower the titrant (solution 4.4) delivery assembly so that the delivery needle is just below the surface of the liquid; open the titrant stopcock, reset the counter, set the pump control switch to the "titrate" position, and press the "start" button.

Wait for the pump and counter to cut off at maximum turbidity.

Record the counter (volume) reading to the nearest 0,01 cm^3.

Raise the delivery tube clear of the beaker. Move the pump control to "flush" and allow a few drops of titrant to clear the needle. As the last drop of reagent leaves the needle, move the pump control to "off". After the pump stops, close the stopcock and move the needle out of the way of the beaker. Remove the beaker from the well.

Wipe the needle with a clean tissue (do not use solvent). The apparatus is now ready for another determination.

9.1.5.3 Repeat the operations described 9.1.5.1 and 9.1.5.2 for the other four test portions (see 9.1.2).

9.1.5.4 Proceed as specified in 9.1.9.

9.1.6 Method 2 — Manual titration of CTAB filtrate with sodium di(2-ethylhexyl) sulfosuccinate solution to a maximum-turbidity end-point.

9.1.6.1 Preparation of titration assembly line

9.1.6.1.1 Before any titration is carried out, it is necessary to set up the titration assembly line so that the end-point is correctly detected, as described in 9.1.6.1.2 to 9.1.6.1.6.

9.1.6.1.2 Place 55 cm^3 of water in a 100 cm^3 beaker (5.22) containing a 22 mm magnetic stirring bar (5.4). Transfer, by means of a pipette (5.15), 5,00 cm^3 of CTAB solution (4.2) into the beaker, taking care to avoid formation of excessive foam.

Place the beaker on a magnetic stirrer (5.5) and adjust the rotational speed to approximately 21 rad/s (200 r/min).

9.1.6.1.3 Connect the variable resistance (5.23) in series with the light source (5.20) and place the latter directly behind the beaker, approximately midway between the bottom of the beaker and the level of the liquid in it.

Adjust the variable resistance so that the light source filament has an orange-red colour when observed horizontally through the solution in the beaker.

9.1.6.1.4 Add the sodium (di(2-ethylhexyl) sulfosuccinate solution (4.4) from a burette (5.13) at a fast rate until the mixture becomes cloudy; at this point the filament will appear more red.

Proceed with the titrant addition slowly, drop by drop, allowing 15 s between drops. Just before the end-point, a rapid increase in turbidity is observed. Stop the titrant addition and keep on stirring for about 10 s. The filament is just visible when observed through the mixture. The end-point is reached when, by addition of a further drop of titrant, the filament will be no longer visible.

NOTE 5 Addition of one drop of titrant after the end-point has been reached produces flocculation and slow reappearance of the filament.

9.1.6.1.5 If the filament does not disappear at the end-point or if it disappears before the end-point is reached, adjust the resistance setting in order to decrease or increase the filament light intensity and repeat the procedure.

9.1.6.1.6 Note the resistance setting so that calibration and titration are carried out with the same positioning of the variable resistance.

9.1.6.2 Titration

Place 50 cm^3 of water in a 100 cm^3 beaker (5.22) containing a 22 mm magnetic stirring bar (5.4). Transfer, by means of a pipette (5.15), a 10,00 cm^3 portion of CTAB filtrate (9.1.4) into the beaker, taking care to avoid formation of excessive foaming.

Place the beaker on a magnetic stirrer (5.5) and just in front of the light source connected with the variable resistance kept at the same setting noted after the preparation of the titration assembly line.

Titrate as specified in 9.1.6.1.4.

Read the burette to the nearest 0,05 cm^3 and record the volume of titrant used.

Wash the beaker with acetone, followed by water, before reusing it.

9.1.6.3 Other test portions

Repeat the operations specified in 9.1.6.2 for the other four test portions (see 9.1.2).

9.1.6.4 Calculation of standardization factors

Proceed as specified in 9.1.9.

9.1.7 Method 3 — Manual titration of CTAB filtrate with sodium di(2-ethylhexyl) sulfosuccinate solution to a specified colour end-point.

9.1.7.1 Transfer, by means of a pipette (5.15), a 10,00 cm^3 portion of the CTAB filtrate (see 9.1.4) into a 100 cm^3 beaker (5.22) containing a 32 mm magnetic stirring bar (5.4). Add approximately 0,15 cm^3 (3 drops) of bromophenol blue indicator solution (4.8). The amount of indicator added is critical. Be sure to use the same amount for all titrations. Place the beaker on the magnetic stirrer (5.5) and adjust to moderate speed.

9.1.7.2 Place the light source (5.20) directly behind and slightly higher than the bottom of the beaker so that the light beam is reflected off the bottom of the beaker. (An angle of inclination of 30° to 45° from the horizontal is recommended.)

Adjust the apparatus so that the reflections in the bottom of the beaker can be seen at eye level.

9.1.7.3 Add the sodium di(2-ethylhexyl) sulfosuccinate solution (4.4) from a burette (5.13) at a fast rate until the mixture becomes cloudy. Adjust the magnetic stirrer to moderately fast and continue adding titrant, drop by drop, at a fast rate until an orange cast is seen in the reflected light and the mixture is a definite cloudy blue. Proceed with the addition slowly, drop by drop, allowing 1 s between each addition and stopping the stirrer after each addition. Just before the end-point, a sudden cloudiness is observed. Continue the addition at the rate of 1 drop per second until 1 drop causes the cloudy blue mixture to separate, noted by a decrease in the blue haze with most of the blue indicator going into the floc. The floc will float to the top when stirring is stopped.

Record the volume of sodium di(2-ethylhexyl) sulfosuccinate solution used to the nearest 0,05 cm^3.

Wash the beaker with acetone, followed by water, before reusing it.

9.1.7.4 Repeat the operations described in 9.1.7.1 to 9.1.7.3 for the other four test portions (9.1.2).

9.1.7.5 Proceed as specified in 9.1.9.

9.1.8 Method 4 — Manual titration of CTAB filtrate with sodium dodecyl sulfate (SDS) solution (4.6) to a specified colour end-point.

9.1.8.1 Transfer, by means of a pipette (5.15), a 10,00 cm^3 portion of the CTAB filtrate (see 9.1.4) into a 100 cm^3 conical flask (5.16) containing a 32 mm magnetic stirring bar (5.4). Add approximately 0,30 cm^3 (6 drops) of dichlorofluorescein indicator solution (4.7) and place the flask on a magnetic stirrer (5.5) positioned beneath the delivery tip of a burette (5.13) containing the SDS solution (4.6). Set the magnetic stirrer at a speed which will give rapid swirling of the titration mixture with minimum formation of foam.

Titrate with SDS solution until the pink colour is discharged and the mixture reverts to a clear yellow colour.

At the beginning of the titration, the colour is mostly yellow but will have a pink undertone. As the titration proceeds, the yellow disappears and the colour becomes a strong, clear pink.

This pink colour is the first of three distinct indications of the approach of the end-point and titrant may be added at the maximum flow rate of the burette until the pink colour develops. After the mixture has turned pink, the next stage is development of turbidity without much change in colour. The pink then begins to fade towards a salmon-orange and this is the final indication to proceed with the titration drop by drop. Continue until the salmon tinge is discharged and the mixture has turned to clear yellow.

Record the volume of the SDS solution used to the nearest 0,05 cm^3.

9.1.8.2 Repeat the operations described in 9.1.8.1 for the other four test portions (9.1.2).

9.1.8.3 Proceed as specified in 9.1.9.

9.1.9 Calculation of standardization factors

Plot the titration volumes V_s against the corresponding masses of the test portions m_s (figure 2 gives an example). Draw the best possible straight line through the points or use the method of least squares, and determine the slope a (in cubic centimetres per gram) and the volume V_0 (in cubic centimetres) at which the line intercepts the volume axis. Using the data corresponding to each test portion, calculate by means of the formula given in clause 10 the surface area of the standard reference black. The calculated surface areas shall not differ from the agreed surface area by more than $0,75 \times 10^3$ m^2/kg.

NOTE 6 Examples of the calculation are given in annex A. Standardization to determine new values of V_0 and a will be necessary whenever any new solutions are prepared. If the solutions are stored for long periods, standardization every month is recommended.

9.2 Determination

9.2.1 Weigh, to the nearest 0,1 mg, a mass of carbon black, dried as indicated in clause 6, according to its grade and its expected surface area as given in table 1.

Table 1

Grade	Expected CTAB surface area range 10^3 m^2/kg	Mass of test portion g
N 100	125 to 150	0,30
N 200	100 to 130	0,35
N 300	75 to 105	0,40
N 351 to N 440	50 to 75	0,60
N 500 to N 600	35 to 50	0,90
N 700	25 to 30	1,35

If the type of carbon black is totally unknown, so that its grade cannot be established, determination of the nitrogen adsorption specific surface area (in accordance with ISO 4652) or of the iodine adsorption number (in accordance with ISO 1304) will categorize it.

9.2.2 Equilibrate with CTAB solution as described in 9.1.3.

9.2.3 Filter as described in 9.1.4.

9.2.4 Titrate a 10,00 cm^3 portion of the CTAB filtrate by the same method as used for standardization of reagents.

Results may not be valid for any test in which the titration volume V_1 is less than 19 cm^3. In such cases, reduce the mass of the test portion m to the quantity

$$\frac{23\, m_1}{V_0 - V_1}$$

(where m_1 is the mass of the first test portion) and repeat the determination.

10 Expression of results

Calculate the CTAB surface area S_{CTAB}, in square metres per kilogram, from the equation

$$S_{CTAB} = \frac{V_0 - V}{m} \times \frac{S'_{CTAB}}{-a}$$

where

V is the volume, in cubic centimetres, of titrant required for the titration of the 10,00 cm^3 portion of CTAB filtrate;

V_0 and a are the standardization factors calculated in 9.1.9;

m is the mass, in grams, of the test portion;

S'_{CTAB} is the agreed value of the surface area accessible to CTAB, in square metres per kilogram, of the standard reference black used.

Express the results to the nearest 10^3 m^2/kg.

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the identification of the sample;

c) the test conditions;

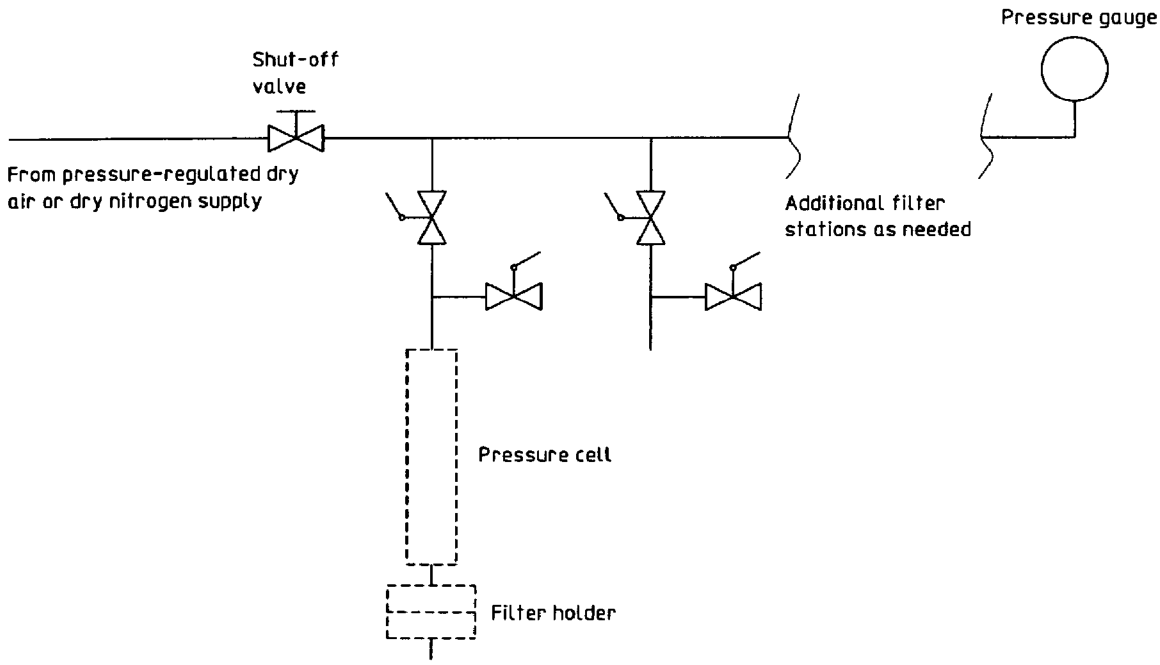
d) the mass of the test portion used;

e) the titration method used;

f) the results obtained from the individual determinations and their average, to the nearest whole number;

g) identification of the standard reference black and its agreed surface area accessible to CTAB;

h) the temperature used for drying the carbon blacks.



Suggested material: 3,15 mm or 6,3 mm standard brass pipe and fittings, brass valves. Toggle-type valves are convenient. If 3,15 mm pipe is used, the outlets to the pressure cells shall have 3,15 mm × 6,3 mm bushings. Polypropylene bushings are convenient.

Figure 1 — Pressure filtration manifold

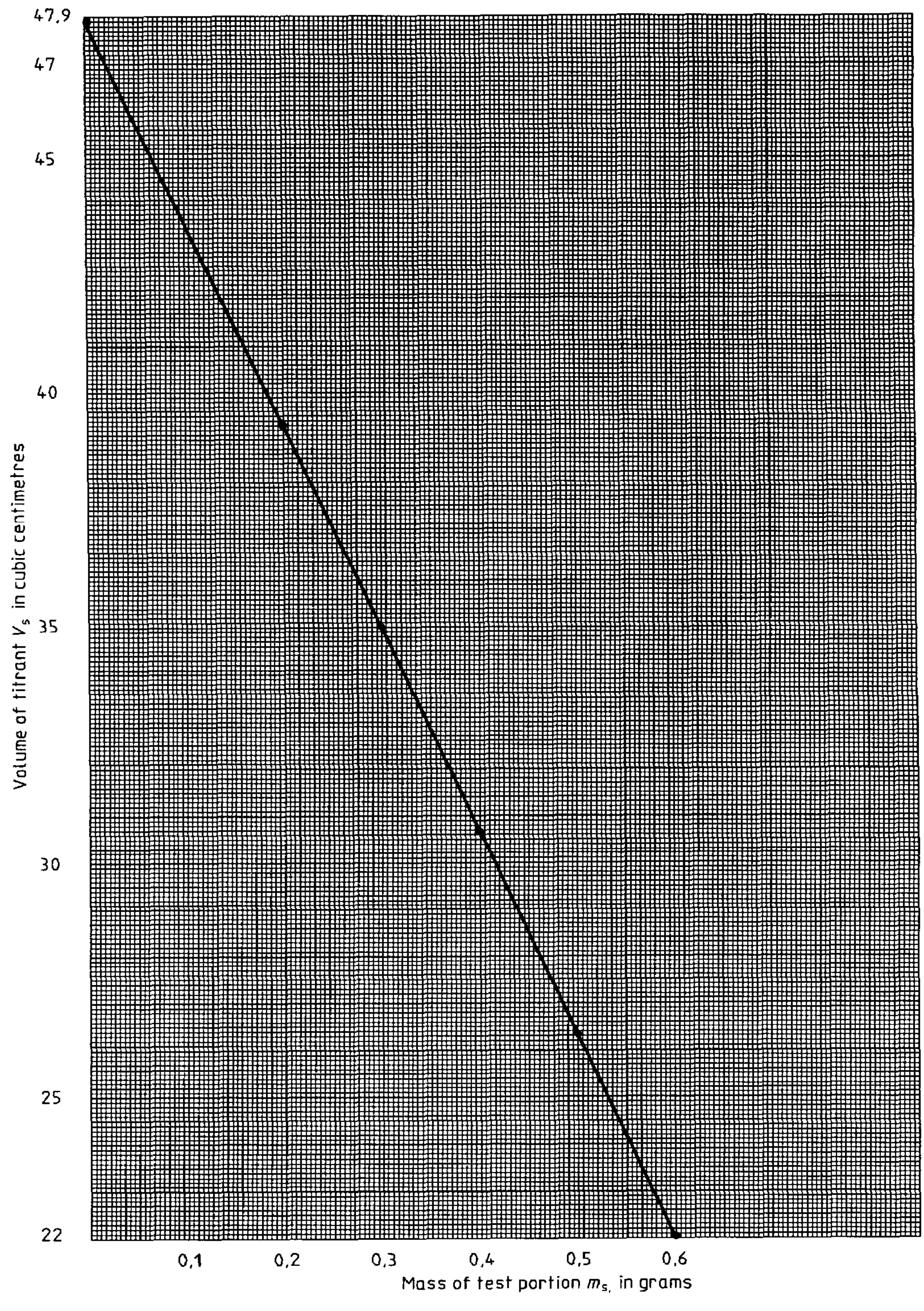


Figure 2 — Derivation of standardization factors

Annex A
(informative)

Examples of calculations of standardization factors

A.1 General

The purpose of this annex is to explain, by worked examples, the methods of calculation of standardization factors (as described in 9.1.9).

Both methods are based on the general equation of a straight line:

$$y = ax + b$$

where

a is the slope of the line;

b is the value on the y-axis, where the line intersects this axis.

In these examples, m_s stands for x, V_s for y, and V_0 for b.

For the construction of the graph (see figure 2) and calculations of standardization factors, the data for m_s and V_s corresponding to five test portions of industry reference black ITRB are used (see table A.1). The agreed surface area, accessible to CTAB, of ITRB is $83,0 \times 10^3$ m²/kg.

A.2 Calculation of V_0 and a

A.2.1 Calculation from graph

The values of V_0 and a can be derived from a graph (see figure 2) as follows:

V_0 is the intercept on the vertical (volume) axis: 47,9 in figure 2;

a is the slope of the line, which can be calculated from any two points on the line as follows:

$$a = \frac{V_2 - V_1}{m_2 - m_1}$$

Using, for example, the points in figure 2 with coordinates 0,30 and 35,0, and 0,45 and 28,5, the slope of the line is calculated as follows:

$$a = \frac{28,5 - 35,0}{0,45 - 0,30} = -43,33$$

Using the data for m_s and V_s and these standardization factors, the surface area for each test portion of ITRB is calculated (as described in clause 10). The values obtained and their differences from the agreed value are listed in table A.2.

This method is only practical if a suitably large scale is used.

A.2.2 Calculation by method of least squares

The values of V_0 and a can be calculated by regression using the method of least squares, as follows:

$$D = n\Sigma m_s^2 - (\Sigma m_s)^2$$

$$V_0 = \frac{\Sigma V_s \, \Sigma m_s^2 - \Sigma m_s V_s \, \Sigma m_s}{D}$$

$$a = \frac{n\Sigma m_s V_s - \Sigma m_s \, \Sigma V_s}{D}$$

Using the data from table A.1, the standardization factors are calculated as follows:

$$D = 5 \times 0,9 - 2^2 = 0,5$$

$$V_0 = \frac{153,55 \times 0,9 - 57,115 \times 2}{0,5} = 47,93$$

$$a = \frac{5 \times 57,115 - 2 \times 153,55}{0,5} = -43,05$$

Using the data for m_s and V_s and these standardization factors, the surface area for each test portion of ITRB is calculated (as described in clause 10). The values obtained and their differences from the agreed value are listed in table A.2.

Table A.1

Test portion No.	Mass of test portion, m_s g	m_s^2	Volume of titrant used, V_s cm^3	$m_s V_s$
1	0,200 0	0,040 0	39,25	7,850 0
2	0,300 0	0,090 0	35,00	10,500 0
3	0,400 0	0,160 0	30,85	12,340 0
4	0,500 0	0,250 0	26,45	13,225 0
5	0,600 0	0,360 0	22,00	13,200 0
$n = 5$	$\Sigma m_s = 2{,}000\ 0$	$\Sigma m_s^2 = 0{,}900\ 0$	$\Sigma V_s = 153{,}55$	$\Sigma m_s V_s = 57{,}115\ 0$

Table A.2

ITRB test portion No.	Calculated S'_{CTAB} (back-calculated), 10^3 m^2/kg	
	Standardization factors used	
	from graph	from least-squares method
1	82,85 (−0,15)	83,67 (+0,67)
2	82,37 (−0,63)	83,10 (+0,10)
3	81,65 (−1,35)	82,33 (−0,67)
4	82,18 (−0,82)	82,83 (−0,17)
5	82,69 (−0,31)	83,32 (+0,32)

Annex B
(informative)

Alternative method of setting up and using the automatic titration apparatus

NOTE 7 This method concerns the commonly used "ATMAST" apparatus.

In the standard set-up, when the electric eye detects the end-point (by turbidity), the pump delivering the titrant is stopped.

More precise results may be obtained by modifying the apparatus so that, instead of the pump being stopped, a solenoid valve in the feed line is closed.

The following is a suggested arrangement:

a) a convenient 400 cm^3 reservoir of titrant feeding by gravity to a three-way 50 cm^3 burette;

b) polyethylene tubing leading from the bottom of the burette to the titration needle, whose point is immersed during titration in the CTAB solution;

c) a solenoid shut-off valve in the polyethylene-tubing line;

d) the solenoid valve is connected to the terminals formerly used to connect up the pump.

ICS 83.040.20

Descriptors: rubber industry, rubber, ingredients, carbon black, tests, determination, specific area.

Price based on 12 pages