International Standard 6810

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

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

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

1 Scope and field of application

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 References

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

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

ISO 1126, *Carbon black for use in the rubber industry — Determination of loss on heating.*

ISO 1304, *Carbon black for use in the rubber industry — Determination of iodine adsorption number.*

ISO 4652, *Rubber compounding ingredients — Carbon black — Determination of specific surface area — Nitrogen adsorption methods.*

ISO 6809, *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 attacks.

4.1 Buffer solution, of pH 7, 0,05 mol/dm^3 solution.

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

NOTE — Further details concerning the preparation have been published (see clause 12). An equivalent buffer solution is available commercially.

4.2 Hexadecyltrimethyl ammonium bromide (CTAB), 0,01 mol/dm^3 solution.

Dissolve 3,64 g 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 to 37 °C to facilitate dissolution. Cool to a temperature between 22 and 25 °C before use.

NOTE — The temperature of this solution should 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, approximately 0,002 2 mol/dm^3 solution (for methods 1, 2 and 3).

Dissolve 1,00 g of 100 % solid sodium di(2-ethylhexyl) sulfosuccinate in purified water containing 2,5 cm^3 of the 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 trightly and store in a cool place.

NOTES

1 Once its container has been opened, the 100 % solid reagent should be stored in a desiccator.

2 The reagent solution may be subject to slow bio-degradation in the absence of formaldehyde. It should be used within 6 months.

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

Dissolve 1,5 g of 100 % liquid 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), 0,002 1 mol/dm^3 solution (for method 4).

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

NOTE — 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, 0,36 % (m/m) ethanolic solution (indicator 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, 0,20 % (m/m) aqueous ethanolic solution (indicator 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)]

5.1 Analytical balance, accurate to 0,1 mg.

5.2 Oven, capable of being controlled at 105 ± 2 °C or 125 ± 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 line or a cylinder with regulator).

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

A typical assembly of a 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.

NOTE — It is essential that this is thoroughly cleaned after use.

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

5.10 Filter holder.

NOTE — It is essential that this is thoroughly cleaned after used.

1) Details of suitable commercially available products may be obtained from the Secretariat of ISO/TC 45 (BSI).

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 the CTAB solution (4.2).

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

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 Jar, of capacity 100 to 200 cm^3, wide mouthed, with a screw cap.

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

5.20 Desiccators.

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

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

5.22 Automatic titration apparatus (for method 1).

5.23 Beakers, of capacity 100 cm^3, Berzelius tall form.

5.24 Variable resistance, for use with the light source (5.21).

6 Preparation of the sample

Dry an adequate amount of the sample for 1 h at a temperature of 105 ± 2 °C or 125 ± 2 °C as described in ISO 1126. Allow to cool to ambient temperature in a desiccator. 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 ± 2 °C and 50 ± 5 % relative humidity or 27 ± 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 — Storage of the CTAB solution at temperatures below 22 °C will result in slow crystallization.

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

8 Preparation of filters

WARNING NOTE — New batches of filters (5.9) should be evaluated to ensure normal filtration times. The maximum time taken to avoid errors should be 8 min.

The filter discs to be used for removing the colloidally dispersed carbon black from the depleted CTAB solutions shall be saturated with CTAB solution before use as follows:

Submerge 25 filters, one at a time, in a least 100 cm^3 of the CTAB solution (4.2) in the wide-mouthed jar (5.18). During this operation, remove and discard the packing papers separating the filter discs. Allow the filters to soak for at least 48 h before use. The CTAB solution used for soaking the discs shall be discarded after use. Soaked filter discs should be used within 1 month or discarded.

Some filter discs tend to curl during pre-soaking, making leak-free installation in the filter holder difficult. To prevent the filter discs from curling, a glass stopper, of nearly similar diameter and heavy enough to keep the discs flat during soaking, should be placed on top of them. This should be removed within the first 24 h.

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 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 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 3 cm in an ultrasonic cleaning bath modified to provide concurrent stirring, and agitate for 6 min. The water temperature in the bath should be kept between 22 and 27 °C throughout the equilibration procedure,

otherwise variations in adsorption equilibrium can occur. It is usual for the water temperature to rise during the operation. If 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 to the stainless steel pressure cell and hand tighten sufficiently to avoid leakage. (Polytetra-fluoroethylene sealing tape can be used if found necessary.) Remove excess liquid from a pre-soaked filter disc by blotting with paper towels and then install with the shiny surface facing the inlet, before it dries completely, according to the instructions furnished with the filter holder. Pour the equilibrated carbon black suspension (9.1.3) through a small funnel into the pressure cell. Connect the cell to the dry compressed air or dry nitrogen source regulated at 0,4 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 foaming. 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, 9.1.6, 9.1.7 and 9.1.8.

NOTES

1 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.

2 It is not generally practicable to titrate immediately after filtration; filtrate collection vials should, 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 according to the manufacturer's instructions. Ascertain that the titrant reservoir contains sufficient sodium di(2-ethylhexyl) sulfosuccinate solution (4.4) and that fluid lines and the pump head are free from air bubbles and have been sufficiently flushed with titrant. The power should be on and the titrant reservoir stopper should be loosened to admit air as liquid flows out.

9.1.5.2 Place 45 cm^3 of purified water in a Berzelius beaker (5.2.3) 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, a 10,00 cm^3 portion of the CTAB filtrate into the beaker, taking care to avoid formation of excessive foaming. Place the beaker into 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 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 clean tissue (do not use solvent). The apparatus is now ready for another sample.

9.1.5.3 Repeat the operations described 9.1.5.1 and 9.1.5.2 for the other four test portions (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 follows.

9.1.6.1.2 Place 55 cm^3 of water in a beaker (5.23) 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 foaming.

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

9.1.6.1.3 Connect a variable resistance (5.24) in series with a light source (5.21) 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 looked at 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 of turbidity is observed. Stop the titrant addition and keep on stirring for about 10 s. The filament is just visible when looked at 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 — 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 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 beaker (5.23) 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.

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

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

9.1.6.4 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, a 10,00 cm^3 portion of the CTAB filtrate into a 100 cm^3 beaker containing the 32 mm magnetic stirring bar (5.4). Add approximately 0,15 cm^3 (3 drops) of the 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 (5.21) directly behind and sligthly 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 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 a 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.

NOTE — 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, a 10,00 cm^3 portion of the CTAB filtrate into a 100 cm^3 conical flask containing the 32 mm magnetic stirring bar (5.4). Add approximately 0,30 cm^3 (6 drops) of the dichlorofluorescein indicator solution (4.7) and place the flask on a magnetic stirrer positioned beneath the delivery tip of the burette (5.13) containing the SDS solution. 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 plain yellow colour.

NOTE — 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 plain 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. 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_o (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 should not differ from the agreed surface area by more than 0,75 m^2/g.

NOTE — Examples of the calculation are given in annex A. Standardization to determine new values of V_o 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 Weight, 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 m^2/g	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-N 440	50 to 75	0,60
N 500-N 600	35 to 50	0,90
N 700	25 to 30	1,35

NOTE — 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.

NOTE — Results may not be valid for any test in which the titration volume, V, is less than 19 cm^3. In such cases, reduce the mass of the test portion, m, to the quantity $\frac{23\,m}{V_o - V}$ (where m 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 gram, from the formula

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

where

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

V_o 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 gram, of the standard reference black used.

Express the results to the nearest 1,0 m^2/g.

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) the proper identification of the sample;

c) the test conditions;

d) the mass of the test portion used;

e) the titration method used;

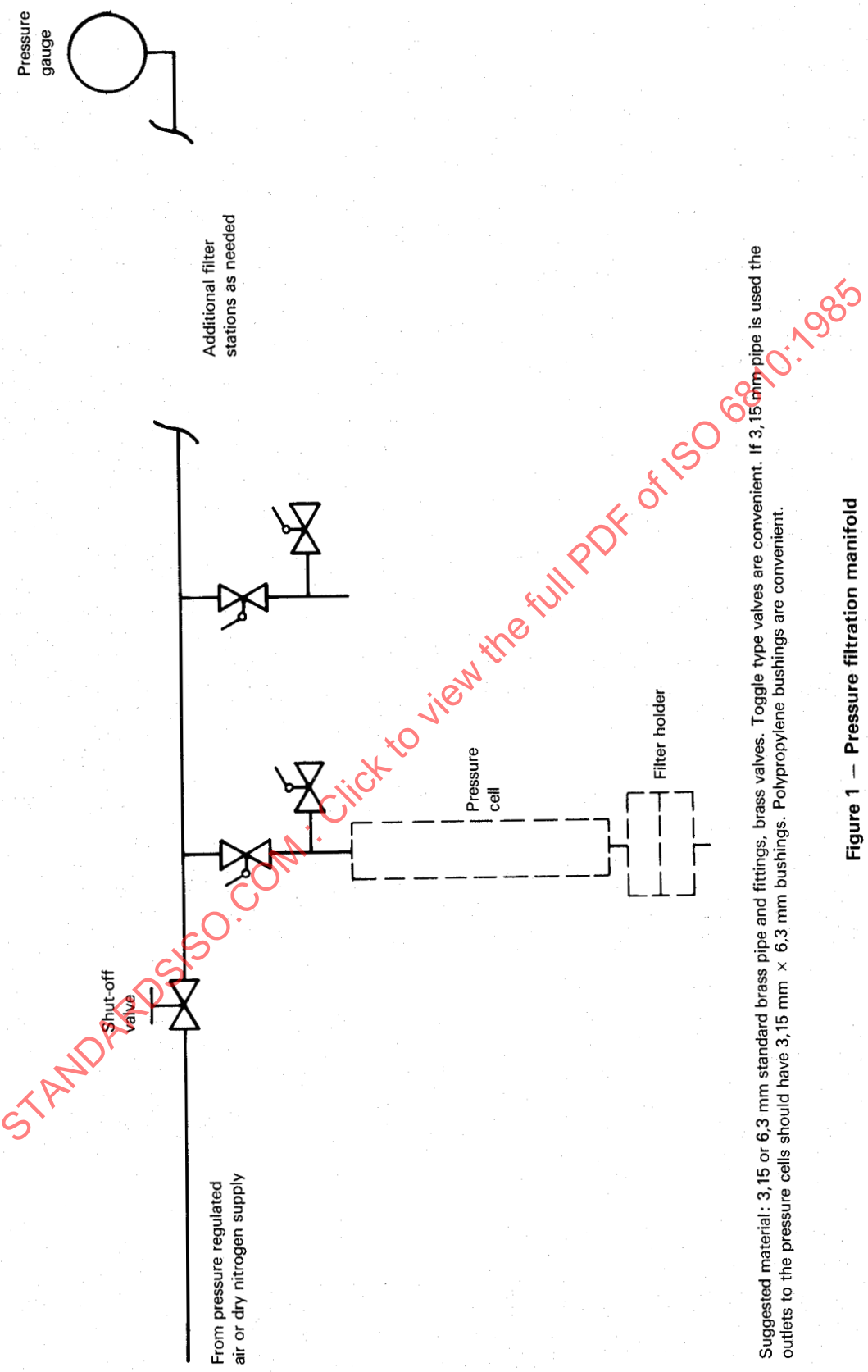
f) the results obtained from individual determinations and their average, to the nearest whole number;

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

h) the temperature used for drying the carbon blacks.

12 Bibliography

BATES *et al. J. Res. NBS*, **29**, (1942), p. 183.



Suggested material: 3,15 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 should have 3,15 mm × 6,3 mm bushings. Polypropylene bushings are convenient.

Figure 1 — Pressure filtration manifold

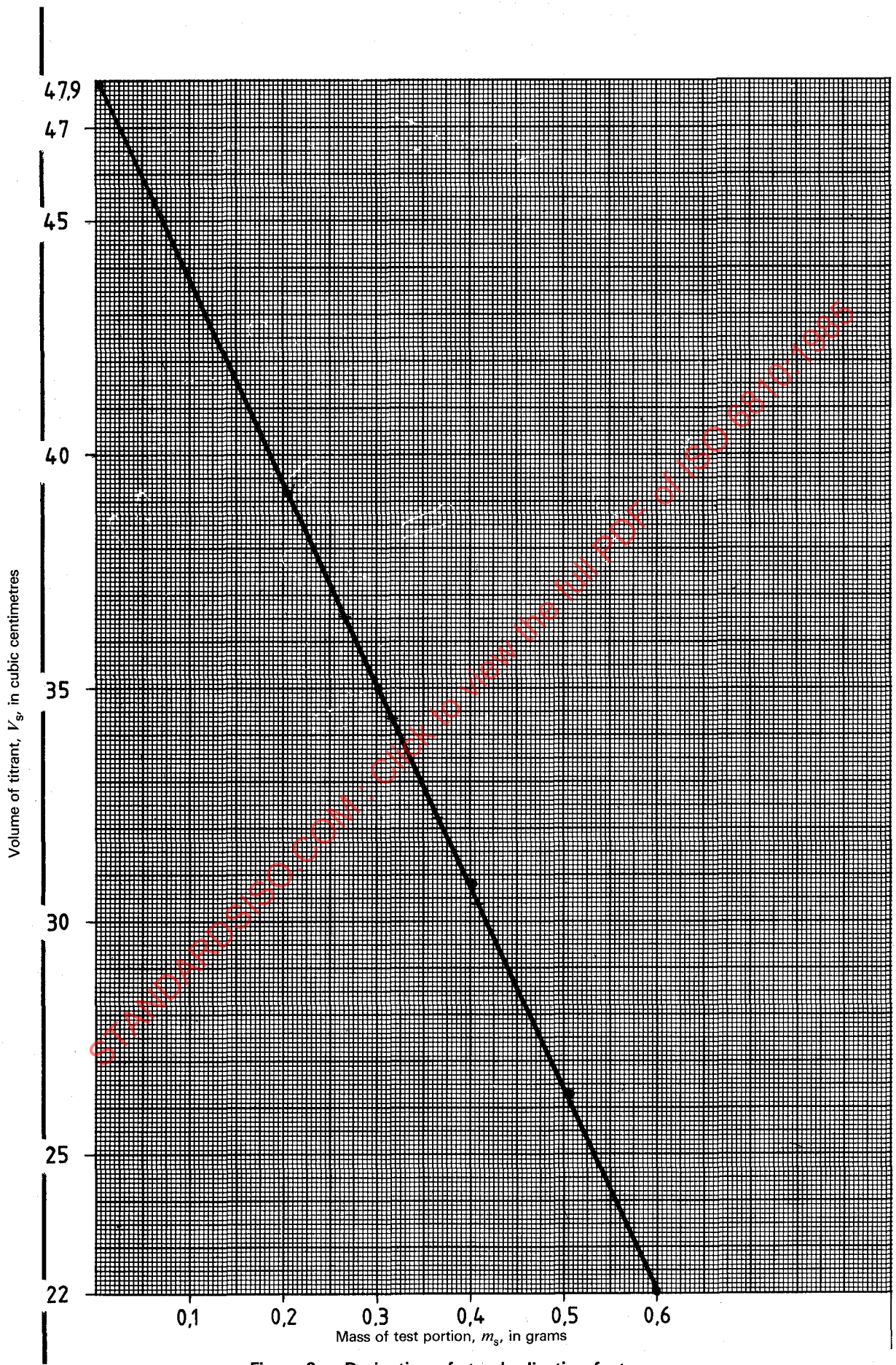


Figure 2 — Derivation of standardization factors