

INTERNATIONAL STANDARD

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6810**

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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*



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Foreword

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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.

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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 colloiddally 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 dm³.

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 cm³ of purified water in a suitable container. Add 100 cm³ 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 cm³ of formaldehyde solution (4.3), using the magnetic stirrer (5.5). Dilute to 1 dm³ 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 dm³ 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 cm³ of formaldehyde solution (4.3) and dilute to 1 dm³ 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 cm³ 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 cm³ and add 40 cm³ of water.

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

5 Apparatus¹⁾²⁾

5.1 Analytical balance, accurate to 0,1 mg.

5.2 Oven, capable of being maintained at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ or $125\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

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.

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 Waukegan 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.

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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 $0,1\text{ cm}^3$ 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 $5,00\text{ cm}^3$ and $10,00\text{ cm}^3$, complying with the requirements of ISO 648, class A.