
**Rubber compounding ingredients —
Determination of multipoint nitrogen
surface area (NSA) and statistical
thickness surface area (STSA)**

*Ingrédients de mélange du caoutchouc — Détermination de la
surface par adsorption d'azote (NSA) et de la surface par épaisseur
statistique (STSA) par méthode multipoints*

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This third edition cancels and replaces the second edition (ISO 18852:2012), which has been technically revised with the following changes:

- [Clause 6](#): deletion of the description of what operators do not have to do; specification of what operators have to do. The verification of measured values was moved to [Clause 8](#) for measurement procedure;
- [Clauses 7](#) and [10](#): addition of explanations for operators to carry out the procedure easily;
- [Clause 8](#): the description for selecting data points has been modified;
- the specification of sample mass has been moved to [Clause 7](#) for sample preparation, and the previous [Clause 9](#) was deleted.

Rubber compounding ingredients — Determination of multipoint nitrogen surface area (NSA) and statistical thickness surface area (STSA)

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International 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 a method for the determination of the nitrogen surface area (NSA) of carbon blacks and other rubber compounding ingredients, like silicas and zinc oxides, based on the Brunauer, Emmett and Teller (BET) theory of gas adsorption using a multipoint determination as well as the determination of the statistical thickness surface area (STSA), otherwise known as the external surface area. STSA, however, is not applicable to silica and zinc oxide.

The method can also be used for verifying “single-point” procedures described in ISO 4652 and other standards.

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2 Normative references (standards.iteh.ai)

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4652:2012, *Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Single-point procedures*

ISO 5794-1:2010, *Rubber compounding ingredients — Silica, precipitated, hydrated — Part 1: Non-rubber tests*

3 Principle

A sample of carbon black, silica, zinc oxide, etc., is placed in a cell of known volume and degassed. Known volume of nitrogen gas is dosed into the cell that is kept at the temperature of liquid nitrogen. The pressure in the cell decreases to the equilibrium due to the adsorption by the sample. The adsorbed amount at the equilibrium is derived from the difference between the amount of the dosed gas and that of remainder in the cell. The pressure is measured together with the temperature in the system, and the volume of the system is measured with a gas that does not adsorb such as helium before the test. NSA is determined by the BET analysis on the adsorption amount obtained, and STSA is determined by t-plot analysis.

4 Apparatus

4.1 Automatic volumetric adsorption measurement apparatus, consisting of a pressure gauge or transducer, a calibration volume, a Dewar flask and all other accessories required for the analysis.

The pressure gauge or transducer shall have the $\pm 0,25$ % accuracy of full scale at the range from 0 kPa to 133 kPa.

Calibration volume is where the volume of the nitrogen to be adsorbed is measured and shall have a known internal volume with a valve or stopcock. It shall have been calibrated by its manufacturer prior to testing.

Sample port volume is where the adsorption to samples takes place, and the pressure at adsorption equilibrium described in [Clause 3](#) is the pressure of this part. A connector connects the apparatus with an adsorption cell.

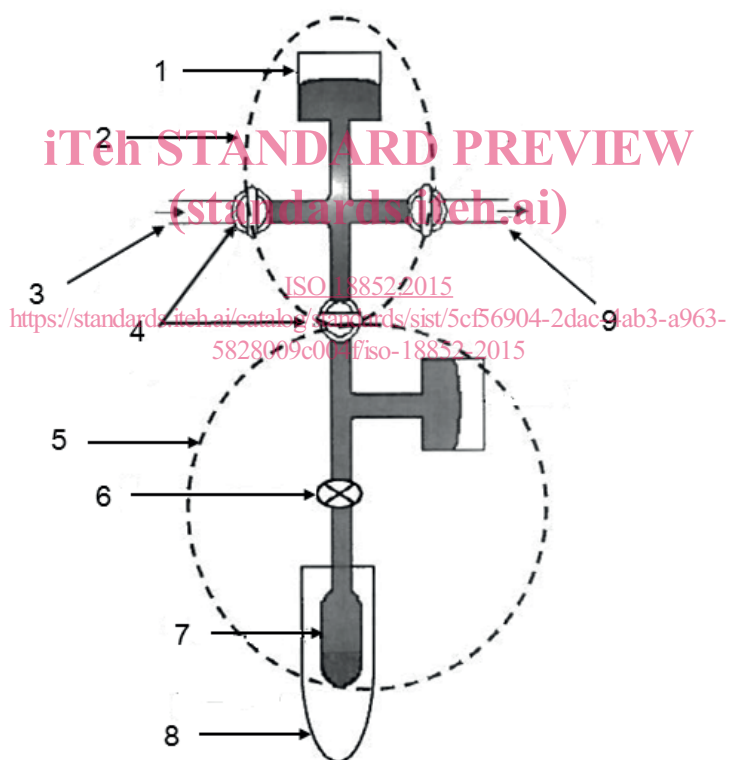
NOTE Commercially, there are two types of the apparatus with different calibration method for the volume of the adsorption cell; those which a calibration factor is applied to the volume of the blank cell determined prior to testing, and those which the volume of the cell with a sample is directly measured during testing (with or without parallel measurement of the volume of another blank cell). Either type is suitable.

4.2 Adsorption cell (hereinafter called “cell”), made of glass and capable of maintaining a vacuum at a pressure 2,7 Pa or below when connected to the adsorption apparatus.

A stopper or valve needs to be attached in order to avoid adsorption after drying.

4.3 Analytical balance, with 0,1 mg sensitivity.

4.4 Degassing station and heating mantle, capable of maintaining the temperatures in [Table 1](#) together with vacuum suction or purge gas flow in order to perform degassing procedure specified in [Clause 7](#).



Key

- | | | | |
|---|------------------------------|---|-----------------|
| 1 | pressure gauge or transducer | 6 | connector |
| 2 | calibration volume | 7 | adsorption cell |
| 3 | gas doser | 8 | Dewar flask |
| 4 | valve | 9 | to vacuum pump |
| 5 | sample port volume | | |

Figure 1 — Diagram of adsorption measurement apparatus

5 Reagents

- 5.1 Liquid nitrogen**, 99,9 % or higher purity.
- 5.2 Ultra-high-purity nitrogen gas**, 99,995 % or higher purity.
- 5.3 Ultra-high-purity helium gas**, 99,99 % or higher purity.

NOTE Helium is used for the calibration of the volume of the automatic volumetric adsorption measurement apparatus since it does not adsorb to the cell or sample.

6 Preparation of automatic volumetric adsorption measurement apparatus

- 6.1** Turn on the automatic volumetric adsorption measurement apparatus and computer.
- 6.2** Connect the cylinders of helium and nitrogen to the automatic volumetric adsorption measurement apparatus with gas tubes, and check if the secondary pressure of the regulator is as specified by the manufacturer of the apparatus.
- 6.3** Check if there is no leakage between the connected tubes.
- 6.4** Carry out gas purging for the tubes to the apparatus when the cylinders are replaced or the apparatus is not used for a month or longer.
- 6.5** Verify periodically that the error sources in the apparatus have been appropriately minimised in accordance with the user's manual.

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7 Sample preparation

7.1 General

Prior to the determination of NSA or STSA, adsorbed contaminants such as atmospheric molecules shall be removed from the sample surface (= degassing). The degassing conditions vary by the material measured and shall be as given in [Table 1](#).

Table 1 — Degassing condition

Material	Temperature °C	Time, minimum h	Reference
Carbon black	300 ± 10	0,5	ISO 4652-1:2012, 3.6.1.2, 4.4, 5.5.6, 6.7.4, and 6.7.5
Silica	155 ± 5	1,0	ISO 5794-1:2010, D.4.6 and E.3.2
Zinc oxide (Type A or B) ^a	300 ± 10	0,5	
Zinc oxide (Type C) ^a	155 ± 5	1,0	ISO 5794-1:2010, D.4.6

^a The various zinc oxide grades are listed in Table D.1 of ISO 9298:1995.

7.2 Vacuum suction method

7.2.1 After degassing a blank cell by vacuum suction, fill the cell with nitrogen gas ([5.2](#)) or helium gas ([5.3](#)) of atmospheric pressure. Insert a glass rod into the adsorption cell stem, if recommended by the

manufacturer of the apparatus, to reduce the dead volume. Disconnect the cell from the adapter and promptly seal it with a stopper or valve. Weigh the cell to the nearest 0,1 mg and record the mass m_1 .

7.2.2 Weigh a sample dried in advance so that the NSA of the sample is equivalent to 20 m² to 50 m², and put it in the cell. Drop the sample clinging on the cell stem into the cell with a pipe cleaner.

7.2.3 Connect the cell containing a sample to the degassing station and degas under vacuum suction with heating in accordance with the operating instructions.

7.2.4 After degassing, fill the cell with the gas, of atmospheric pressure, used in [7.2.1](#). Disconnect the cell from the adapter and promptly seal it with a stopper or valve. Weigh the cell to the nearest 0,1 mg and record the mass m_2 .

7.2.5 Calculate the sample mass as follows:

$$m_0 = m_2 - m_1 \quad (1)$$

where

m_0 is the sample mass, expressed in grams;

m_2 is the mass of the cell, glass rod, gas in the cell, sample and stopper or valve, expressed in grams;

m_1 is the mass of the cell, glass rod, gas in the cell and stopper or valve, expressed in grams.

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7.3 Purge gas flow method

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7.3.1 Connect a blank cell to the adapter and open the valve connected to the cell. Insert a gas tube for nitrogen gas ([5.2](#)) or helium gas ([5.3](#)) to the bottom of the cell and fill the cell with the gas. Disconnect the cell from the adapter and promptly seal it with a stopper or valve. Weigh the cell to the nearest 0,1 mg and record the mass m_1 .

7.3.2 Weigh a sample dried in advance so that the NSA of the sample is equivalent to 20 m² to 50 m², and put it in the cell. Drop the sample clinging on the cell stem into the cell with a pipe cleaner.

7.3.3 Connect the cell containing a sample to the adapter and open the valve connected to the cell. Insert the gas tube used in [7.3.1](#) again to the bottom of the cell and apply an appropriate amount of gas flow so that the sample is not stirred up. While applying the gas, heat the sample in accordance with a condition in [Table 1](#) until the mass becomes constant.

The degassing time might vary significantly from sample to sample, and a reasonable margin of excess time is recommended.

7.3.4 After degassing, disconnect the cell from the adapter and promptly seal it with a stopper or valve. Weigh the cell to the nearest 0,1 mg and record the mass m_2 .

7.3.5 Calculate the sample mass m_0 as given in [7.2.5](#).

8 Measurement procedure

8.1 Measurement

8.1.1 Since most of the operations are conducted automatically, it is important to become thoroughly familiar with the procedures and to follow carefully the operating instructions.

8.1.2 Determine the saturation vapour pressure of the liquid nitrogen (5.1) in a Dewar flask by measuring its temperature. This pressure is affected by the purity of the liquid nitrogen and the ambient pressure. Impurities dissolved in the liquid nitrogen such as oxygen usually cause the temperature of the liquid nitrogen to increase, thus the saturation vapour pressure increases by some kilopascals.

NOTE Some apparatuses are capable of measuring the saturation vapour pressure automatically.

8.1.3 Input relative-pressure range, number of data points (see 8.2), saturation vapour pressure, sample mass and other information to the computer in accordance with the operating instructions. This relative pressure is expressed as a fraction of saturation vapour pressure P_0 and measured pressure P (P/P_0).

8.1.4 Check if there is no defect on the O-ring before measurement.

NOTE If this procedure is not done appropriately, a leakage which leads to an underestimation of the value might occur during the measurement.

8.1.5 Connect the cell containing the degassed sample (Clause 7) to the adapter. A glass rod may be inserted into the cell stem in order to decrease the volume of the cell and eventually to improve precision.

NOTE While the cell stem is in contact with the liquid nitrogen, the level of the liquid nitrogen might affect the cell stem. Hence, some error to the measurement might occur.

8.1.6 Start the measurement. Adsorbed amount is calculated at the input relative pressures (see 8.1.3) by the apparatus.

8.2 Selection of data points and determination of nitrogen surface area (NSA)

8.2.1 General

NSA is usually determined by making the BET plot and selecting a certain range of data points from the plot. The selection of data points shall be done in accordance with Procedure A (8.2.2) or B (8.2.3). Relative pressure range and the number of data points to perform adsorption measurement (8.1.3) shall also be determined as specified in the procedures.

8.2.2 Procedure A

Procedure A is the standard method that generates high precision and shall be applied when the relative pressure range of data points to be used for determining NSA or STSA of a sample is unknown.

Measure the adsorbed amount at relative pressures equally distributed in the range from 0 to 0,5. A minimum of 10, and preferably 20 or more data points shall be obtained. The type of adsorption isotherm may be determined at this point.

For calculating NSA, prepare a BET plot based on the measured amount of gas adsorbed by using an appropriate tool, for example, the function in the software of an automatic volumetric adsorption measurement apparatus.

Select a range with the best linearity on the BET plot and calculate NSA. The usual relative-pressure range for determining NSA is from 0,05 to 0,3. It, however, is recommended to adjust the range depending on the adsorbed amount measured since the end point tends to be shifted downward as NSA increases.