

Sampling and testing carbon black for use in the rubber industry —

**Part 11: Method for determination of
specific surface area by nitrogen
adsorption using single-point
procedures**

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee PRI/53, Carbon black, upon which the following body was represented:

British Rubber Manufacturers' Association

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National foreword

This Part of BS 5293 has been prepared by Technical Committee PRI/53 and is identical with ISO 4652-1:1994 *Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Part 1: Single-point procedures*, published by the International Organization for Standardization (ISO).

Cross-reference

International Standard	Corresponding British Standard
ISO 1126:1992	BS 5293 <i>Methods for sampling and testing carbon black for use in the rubber industry</i> Part 5:1992 <i>Method for determination of loss of mass on heating</i> (Identical)

The Technical Committee has reviewed the provisions of ISO 6809:1989, to which normative reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 18, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

WARNING — Persons using this part of ISO 4652 should be familiar with normal laboratory practice. This part of ISO 4652 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 ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 4652 specifies four methods for the determination of the specific surface area of types and grades of carbon black for use in the rubber industry.

The four methods are the following:

- method A using the Ni-Count-1 apparatus (clause 3);
- method B using the Areameter apparatus (clause 4);
- method C using gas chromatography (clause 5);
- method D using the Monosorb surface-area analyser (clause 6).

NOTE 1 Somewhat different results may be obtained from the four methods. The degassing procedure differs from method to method, and it is important to investigate the possibility of correcting the results by using standard reference blacks.

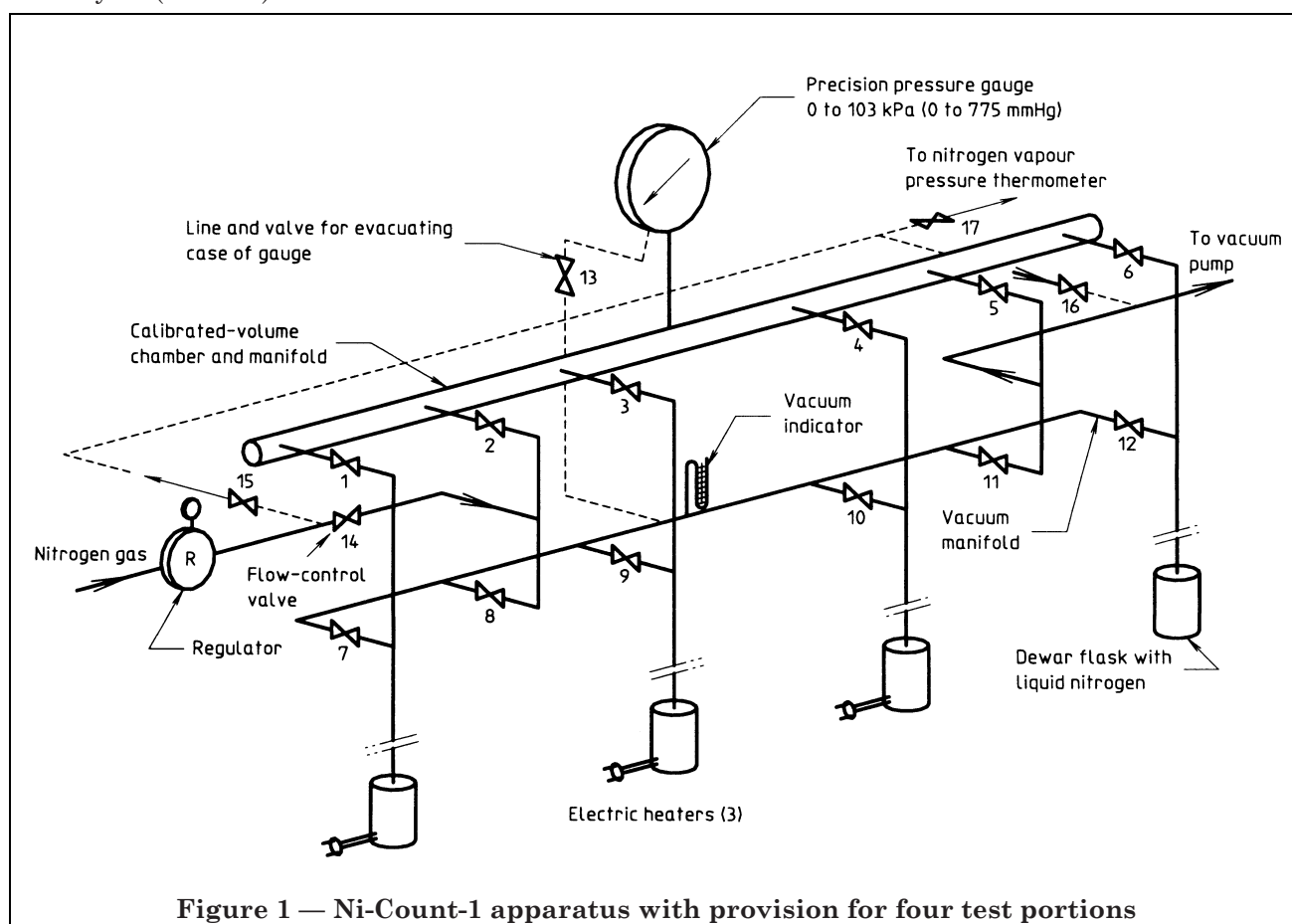
NOTE 2 The nomenclature of carbon blacks is given in ISO/TR 12245:1994, *Carbon blacks used in rubber products — Classification system*.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 4652. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 4652 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 1126:1992, *Rubber compounding ingredients — Carbon black — Determination of loss on heating*.

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



3 Method A, using Ni-Count-1 apparatus

3.1 Principle

A test portion is degassed and weighed, then exposed to nitrogen at the temperature of liquid nitrogen. The amount of nitrogen adsorbed on to the carbon black surface at equilibrium is determined. From this value and the mass of the degassed test portion, the specific surface area is calculated.

3.2 Reagents

3.2.1 Nitrogen, in a cylinder, or another source of prepurified nitrogen of recognized analytical quality.

The nitrogen supply to the Ni-Count-1 apparatus (see 3.3.1) shall be controlled at a pressure of 70 kPa to 140 kPa. If nitrogen from a cylinder is used, the cylinder shall be fitted with a two-stage regulator capable of maintaining the outlet pressure within the specified range.

3.2.2 Liquid nitrogen (approximately 300 cm³ are required for the determination).

WARNING — Use caution. Gloves and safety glasses should be worn as the temperature of liquid nitrogen is – 196 °C.

3.3 Apparatus

3.3.1 Ni-Count-1 specific surface area apparatus¹⁾ (see Figure 1 and Figure 2), or an equivalent one-point adsorption apparatus.

3.3.2 Heater and voltage-control device, capable of maintaining a temperature of 300 °C ± 10 °C, for degassing the test portion.

NOTE 3 The heater (see Figure 1) is furnished with the Ni-Count-1 apparatus.

3.3.3 Vacuum pump, capable of producing an ultimate pressure of $(1,3 \times 10^{-2})$ Pa [(1×10^{-4}) mmHg].

3.3.4 Dewar flask, of capacity approximately 265 cm³ and height 145 mm.

NOTE 4 This is supplied with the Ni-Count-1 apparatus.

3.3.5 Nitrogen vapour pressure thermometer (see Figure 2).

NOTE 5 This is supplied with the Ni-Count-1 apparatus.

3.3.6 Sample tubes (see Figure 3).

The recommended volumes are given in Table 1.

3.3.7 Stopcock grease or poly(chlorotrifluoroethylene) lubricant.

NOTE 6 This is supplied with the Ni-Count-1 apparatus.

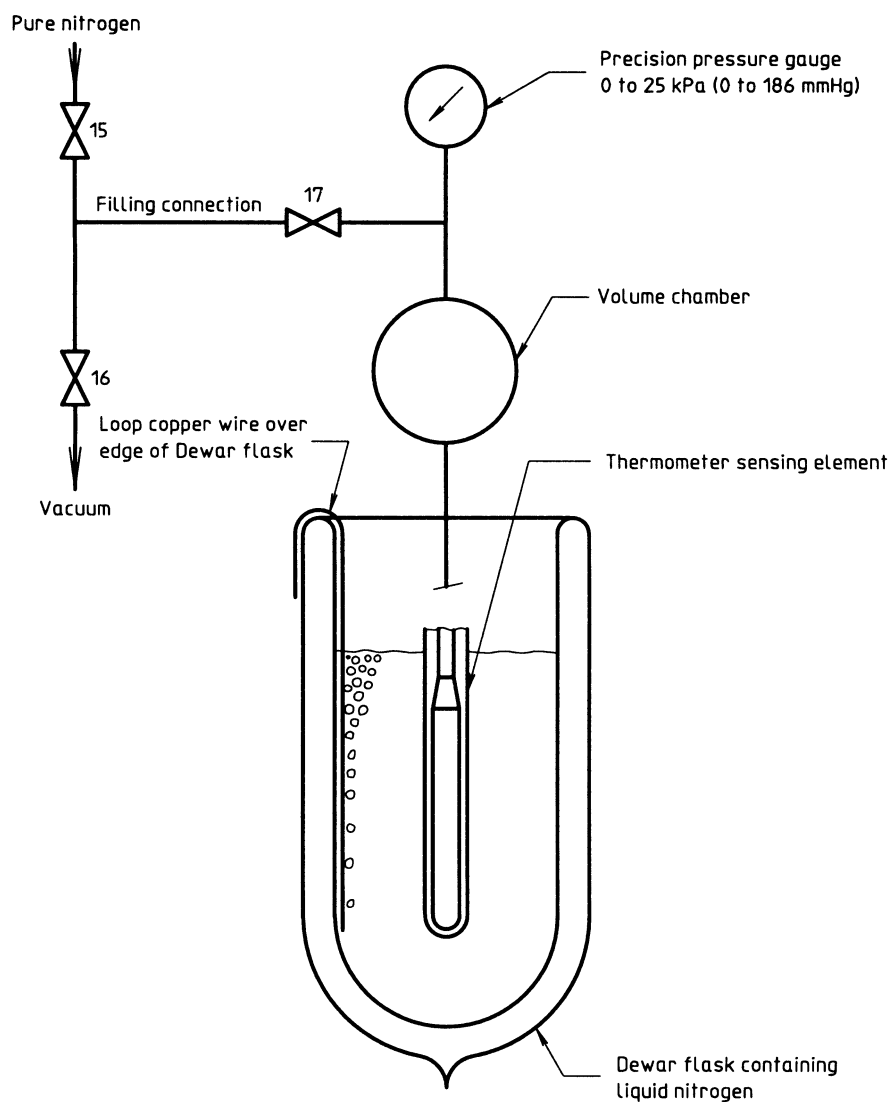
3.3.8 Fine glass wool

3.3.9 Analytical balance, accurate to 0,1 mg.

3.4 Preparation of the sample

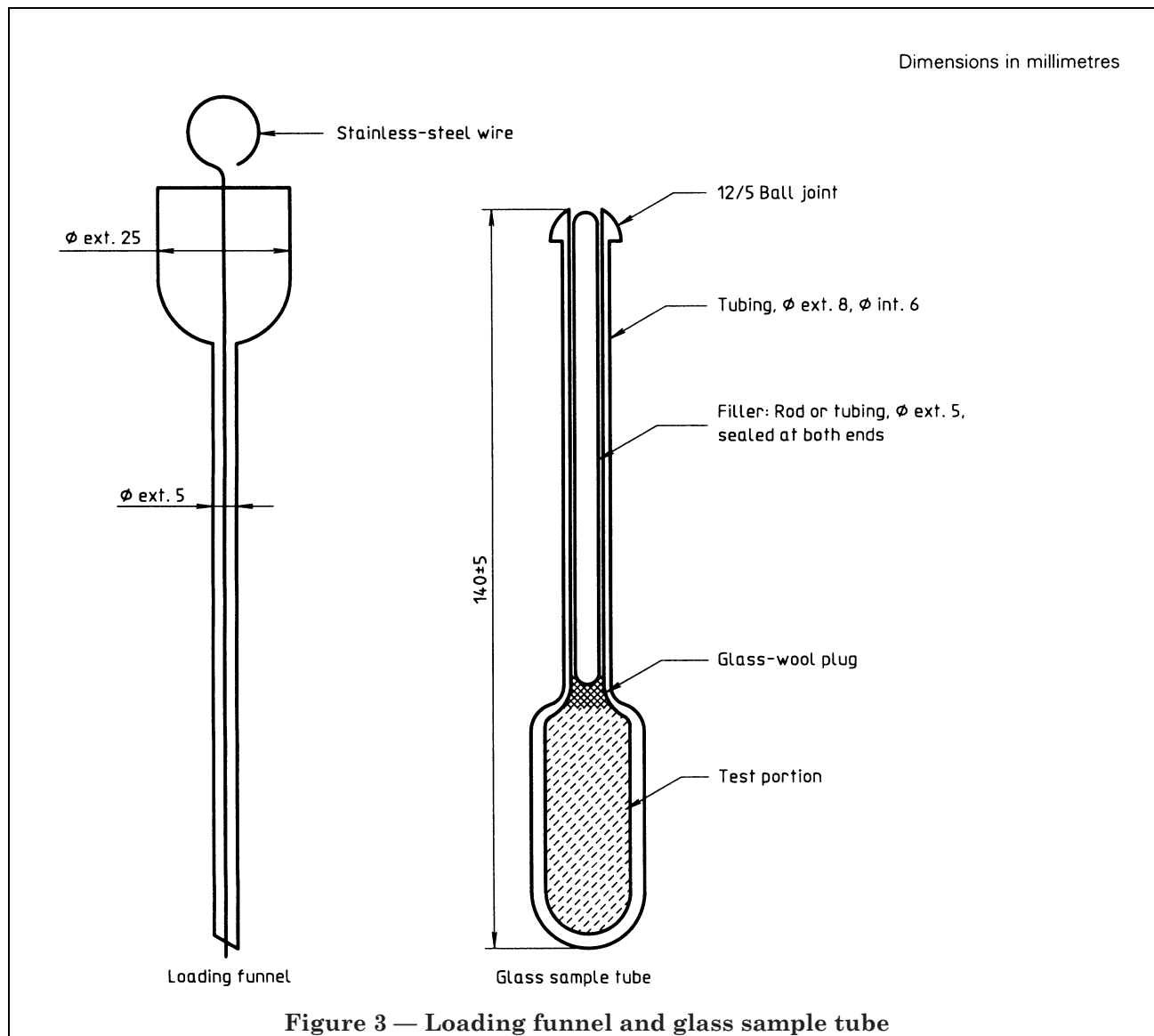
Pellets of carbon black need not be crushed. Unagitated, unpelletized carbon black may be densified if desired.

¹⁾ Available from E.G. & G. Chandler Engineering, 2001 N. Indianwood Avenue, Broken Arrow, Oklahoma, USA.
Postal address: P.O. Box 470710, Tulsa, Oklahoma 74147-0710, USA.



NOTE The system is filled with pure nitrogen to a pressure of 13 kPa to 20 kPa (100 mmHg to 150 mmHg).

Figure 2 — Nitrogen vapour pressure thermometer



3.5 Test conditions

The test should preferably be carried out in ambient conditions of either $23\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ and $(50 \pm 5)\%$ relative humidity or $27\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ and $(65 \pm 5)\%$ relative humidity.

The reagents and the apparatus shall be maintained at temperature equilibrium in the same room for at least 24 h before being used.

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

3.6 Procedure

3.6.1 Preparation and calibration of apparatus

3.6.1.1 The all-metal Ni-Count-1 apparatus has an internal volume adjusted to $139,5\text{ cm}^3$. This internal volume includes all lines to the sample valve, and the volume in the bellows of the pressure gauge is adjusted so that the gauge indicates $66,7\text{ kPa}$ (500 mmHg) at a room temperature of $27\text{ }^{\circ}\text{C}$. The tables of surface area versus pressure (furnished with the Ni-Count-1 apparatus) will yield accurate specific surface areas if the internal volume of the instrument has been accurately adjusted at the factory to $139,5\text{ cm}^3$. To confirm the volume, it is recommended that tests be made on a standard reference black having an agreed nitrogen surface area independently determined by a multipoint method.