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Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Single-point procedures

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de la surface spécifique par méthodes par adsorption d'azote — Modes opératoires à un point de mesure

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Foreword

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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).

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

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document 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 third edition cancels and replaces the second edition (ISO 4652:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

— the former method A has been updated as an automatic volumetric method (method B);

— the former method B has been removed;

— the former methods C and D have been updated as an automatic gas chromatography method (method A);

— 4.4 has been clarified;

— Annex B on precision data has been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The specific surface area of carbon black is an important element which categorizes the type of carbon black. In this document, nitrogen adsorption at the relative pressure of 0,3 is measured to determine the specific surface area (the single-point method). The single-point method can be a reasonable alternative for process management and shipping management after testing with the multi-point method in ISO 18852, because the measurement time is shorter and relative magnitude of the specific surface area among the samples is kept.

The multi-point method determines specific surface area on the basis of monolayer amount of nitrogen adsorbed (V_m) which is derived by Formula (1):

$$V_m = 1/(M+B) \tag{1}$$

where

M is the slope of the BET plot (see ISO 9277);

B is the intercept of the BET plot (see ISO 9277),

The single-point method simplifies this technique by assuming the intercept (B) as zero and calculating the slope (M) with the straight line joining the origin and the point at the relative pressure of 0,3. Therefore, the monolayer amount and specific surface area determined by the single-point method is always lower than those determined by the multi-point method.

Although most of the operation is done automatically, the operator should be familiar with the operation and follow the instruction manual.

Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Single-point procedures

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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.

1 Scope

This document specifies two methods for the determination of the specific surface area of types and grades of carbon black for use in the rubber industry:

— method A: automatic gas chromatography method (carrier gas method);

— method B: automatic volumetric method.

Somewhat different results might be obtained from the two methods. The degassing procedure differs between method A and method B, and it is important to investigate the possibility of correcting the results by using standard reference blacks.

The results might also differ from those obtained using the multipoint method specified in ISO 18852, which is the preferred method.

These methods are not applicable to porous carbon blacks.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Method A: Automatic gas chromatography (carrier gas method)

4.1 Principle

The mixed gas of helium and nitrogen (volume fraction 70 % and 30 %, respectively) is dosed to the degassed sample at the temperature of liquid nitrogen. The nitrogen in the mixed gas is adsorbed on the surface of a test portion of carbon black, so that the composition of the gas changes. The nitrogen is then desorbed by warming the test portion and the ratio of the mixed gas changes again. Since the thermal conductivity varies depending on the mixed gas concentration, it is possible to determine the amount of absorbed nitrogen gas using the thermal conductivity detector (TCD) of the system. Almost all the operations are automatically performed.