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BSI Standards Publication

Vulcanized rubber — Determination of free sulfur by gas chromatography (GC) and high performance liquid chromatography (HPLC)

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National foreword

This British Standard is the UK implementation of ISO 20163:2019.

The UK participation in its preparation was entrusted to Technical Committee PRI/22, Testing and analysis of rubber.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Caoutchoucs vulcanisés — Dosage du soufre libre par chromatographie en phase gazeuse (CG) et chromatographie en phase liquide haute performance (HPLC)

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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).

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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 2, *Testing and analysis*.

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.

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Vulcanized rubber — Determination of free sulfur by gas chromatography (GC) and high performance liquid chromatography (HPLC)

WARNING 1 — 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 and to determine the applicability of any other restrictions.

WARNING 2 — Certain procedures specified in this document might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies chromatographic methods for the determination of the inorganic free sulfur (Sx) not consumed during the vulcanization of the rubber. This method is used primarily to compare the free sulfur content in different samples.

NOTE 1 In this document, "free sulfur" is used instead of "inorganic free sulfur".

NOTE 2 The residual curing agent containing sulfur is not concerned by this measure.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1407:2011, *Rubber — Determination of solvent extract*

3 Terms and definitions

There are no term and definition 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 Principle

The free sulfur is quantified following two techniques: gas chromatography/mass spectrometric detector (GC-MS) or gas chromatography/flame ionization detector (GC/FID) (method A) and high performance liquid chromatography/ultraviolet detector (HPLC/UV) (method B).

A sample of vulcanized rubber is first extracted using an appropriate solvent according to ISO 1407:2011 (Method B). A known quantity of triphenylphosphine (TPP) is added to the obtained solution. TPP reacts with free sulfur to form a stable compound, triphenylphosphine sulfide (TPPS). See Figure 1.

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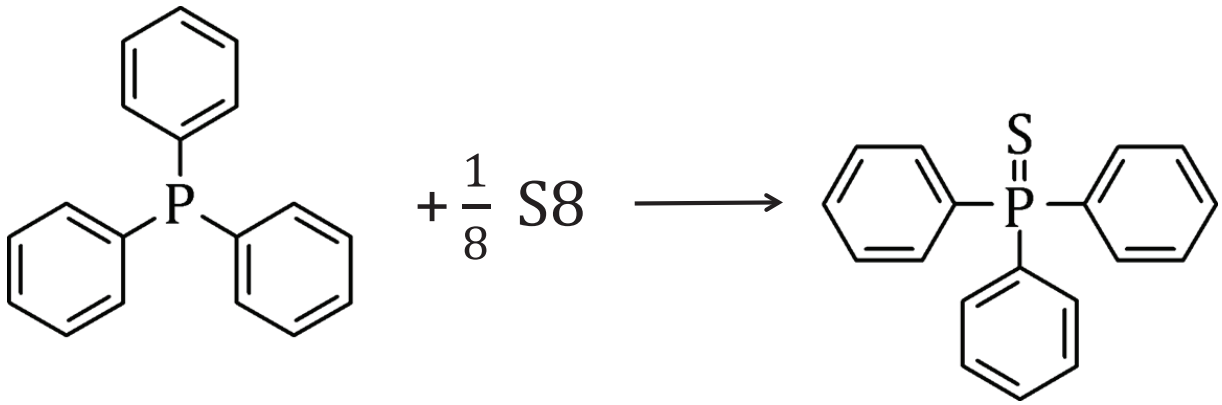

TPP, Triphenylphosphine
(CAS 603-35-0)

TPPS, Triphenylphosphine sulfide
(CAS 3878-45-3)

Figure 1 — TPP reaction with free sulfur for derivatization

TPPS, which contains aromatic rings, offers the following advantages:

— firstly, to have a more active chromophore in UV than sulfur, therefore being more easily detectable in HPLC/UV;

— secondly, to have sufficient thermal stability to be injected in GC.

The following calibration methods are possible for both types of chromatography (GC and HPLC). The preparation of samples is the same for GC and HPLC.

— Standard addition method is the preferred calibration method, used to correct matrix effects and when samples contain unknown contents of free sulfur.

— External calibration is used in case of absence of matrix effect and to check known contents of free sulfur.

5 Apparatus

5.1 Balance, precision 0,01 mg.

5.2 Soxhlet extractor.

5.3 Material for gas chromatography (Method A).

5.3.1 Injector for either split or splitless injection.

5.3.2 Mass spectrometric (MS) detector or **flame ionization detector (FID)**.

5.3.3 Mass spectrometer with the following specifications:

— transfer line temperature: 300 °C;

— ionizing energy: 70 eV;

— scan range: 33 m/z to 600 m/z;

— analysis mode: full scan.

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