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

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

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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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This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

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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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