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Rubber — Determination of ash —

Part 2: Thermogravimetric analysis (TGA)

Caoutchouc — Détermination du taux de cendres —

Partie 2: Analyse thermogravimétrique (TGA)

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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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For an explanation on 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 the following URL: 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*.

A list of all parts in the ISO 247 series can be found on the ISO website.

Rubber — Determination of ash —

Part 2: Thermogravimetric analysis (TGA)

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 two methods for the determination of ash from raw rubbers, compounded rubbers and vulcanizates using a thermogravimetric analyser (TGA).

The methods are applicable to raw, compounded or vulcanized rubbers of the M, O, R and U families described in ISO 1629:

— Method A is applicable for the determination of the ash from raw rubbers.

— Method B is applicable for the determination of the ash from compounded or vulcanized rubbers.

The methods are not applicable for the determination of the ash from raw rubbers, compounded or vulcanized rubbers containing chlorine, bromine or iodine.

This document does not cover the interpretation of the ash results from the inorganic chemical contents of compounded or vulcanized rubbers.

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 248-1, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

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:

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

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

4 Principle

A weighed test portion is heated in an atmosphere of nitrogen. After complete decomposition of the polymer, the atmosphere is switched from nitrogen to oxygen or air and the test portion is further heated until all the carbonaceous matter has been burnt off and a constant mass is reached. The mass of the residue represents the ash.

5 Reagents

5.1 **Dry nitrogen**, purity ≥ 99,99 %.

5.2 **Dry oxygen**, purity ≥ 99,99 %, or **air**.

6 Apparatus

6.1 **Thermogravimetric analyser**, comprising the following elements:

a) Thermo-balance;

b) Heating furnace;

c) Temperature programmer;

d) Gas flow controller, for controlling the purge gas to the balance and furnace, and keeping a constant flow rate.

6.2 **Sample pan**, platinum pan or alumina ceramic pan.

6.3 **Analytical balance**, capable of weighing to the nearest 0,1 mg.

7 Preparation of the test sample

7.1 For raw natural rubber, test samples shall be cut from the homogenized piece prepared in accordance with ISO 1795. For raw synthetic rubber, test samples shall be cut from the dried rubber obtained after carrying out the determination of volatile-matter content in accordance with the hot-mill method of ISO 248-1.

Take a test portion of about 2 g to 5 g from the homogenized sample and cut into pieces by hand.

7.2 Test samples of rubber compounds shall be sheeted on a mill and cut into pieces by hand.

7.3 Test samples of vulcanizates shall be sheeted or crumbed on a mill or comminuted by hand.

7.4 Care shall be taken to ensure that test samples of rubber compounds and vulcanizates are representative of the sample.

8 Calibration

Calibrate the thermogravimetric analyser (see 6.1) according to the manufacturer's instructions.

Temperature and mass calibration should be performed.

To ensure consistent results, the thermogravimetric analyser shall be calibrated periodically. It is recommended to perform mass calibration once a month.