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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

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 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.

9 Procedure

9.1 Method A

9.1.1 Switch on and stabilize the instrument, set the temperature of furnace to 30 °C.

9.1.2 Place an empty sample pan on the platform and tare the sample pan.

9.1.3 Take about 10 mg to 20 mg from the test portion of raw rubber (see 7.1) and weigh to the nearest 0,1 mg. Place it in the sample pan. Position the sample pan on the sample platform and load the sample pan onto the thermo-balance.

9.1.4 Set the flow rate according to the manufacturer's instructions. Raise the temperature to 500 °C at a rate of 20 °C/min or 30 °C/min under an atmosphere of nitrogen. Maintain at this temperature for 1 min.

9.1.5 Switch from the stream of nitrogen to a stream of oxygen or air. Raise the temperature to 550 °C. Maintain the temperature at 550 °C for 5 min or until the mass is constant.

9.1.6 Calculate the ash using the apparatus micro-processor.

9.2 Method B

9.2.1 Switch on and stabilize the instrument, set the temperature of furnace to 30 °C.

9.2.2 Place an empty sample pan on the platform and tare the sample pan.

9.2.3 Take a test portion of about 10 mg to 20 mg from the test sample of compounded or vulcanized rubbers (see 7.2 or 7.3) and weigh to the nearest 0,1 mg. Place it in the sample pan. Position the sample pan on the sample platform and load the sample pan onto the thermo-balance.

9.2.4 Set the flow rate according to the manufacturer's instructions. Raise the temperature to 550 °C at a rate of 20 °C/min or 30 °C/min under an atmosphere of nitrogen. Maintain at this temperature for 1 min.

9.2.5 Switch from the stream of nitrogen to a stream of oxygen or air. Raise the temperature to 650 °C. Maintain the temperature at 650 °C for 5 min or until the mass is constant.

For compounded or vulcanized rubbers containing inorganic fillers such as calcium carbonate, a temperature of 850 °C should be used.

9.2.6 Calculate the ash using the apparatus micro-processor.

10 Expression of results

The ash content, w, is given, as a percentage mass fraction, by Formula (1):

$$w = \frac{m_1}{m_0} \times 100 \tag{1}$$

where

m_0 is the mass of the test portion, in milligrams;

m_1 is the mass of the ash, in milligrams.

11 Precision

See Annex A.

12 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 247-2:2018;

b) all details necessary for identification of the sample tested;

c) the method employed — method A or method B;

d) the heating rate and the final temperature reached;

e) the type of apparatus used;

f) the result of the test;

g) details of any procedures not specified in this document as well as details of any incidents which could have influenced the results;

h) the date of the test.

Annex A
(informative)

Precision

A.1 General

An interlaboratory test programme (ITP) to determine the precision of the two test methods specified in this document was conducted using thermogravimetric analysers in April 2017. The precision evaluated was a type 1 precision in accordance with Method B in ISO 19983. Precision concepts and nomenclature are also given in ISO 19983.

Nine laboratories participated in the ITP and four different materials (Raw rubber: NR and NBR, compounded SBR, vulcanized NR) were used. A test result was taken to be the average value of two measurements. Test results were obtained on two different days at intervals of one week. Two measurements were repeated for each day.

A.2 Precision results

The precision results are given in Table A.1 for method A and Table A.2 for method B. These results were obtained using outlier deletion procedures as described in ISO 19983.

Repeatability: The difference between two day test (value) averages, found on nominally identical material samples under correct operation of this test method, exceed the tabulated day-to-day repeatability on average not more than once in 20 cases.

Reproducibility: The difference between two independently measured test (value) averages, found in two laboratories using correct operation of this test method on nominally identical material samples, exceed the tabulated reproducibility not more than once in 20 cases.

Table A.1 — Precision data for Method A

Material	Mean content	Within lab			Between labs			No. of labs[a]
		s_{rD}	r_D	(r_D)	s_R	R	(R)	
NR	0,29	0,039	0,110	37,9	0,100	0,283	97,6	8
NBR	0,55	0,067	0,190	34,5	0,096	0,272	49,5	9

The symbols used in this table are defined as follows:

s_{rD} is the day-to-day repeatability standard deviation;

r_D is the day-to-day repeatability, in measurement units;

(r_D) is the relative day-to-day repeatability, in percent;

s_R is the reproducibility standard deviation;

R is the reproducibility, in measurement units;

(R) is the relative reproducibility, in percent.

[a] The final number of laboratories in the ITP after deletion of outliers.