INTERNATIONAL STANDARD

ISO 24698-1

Second edition
2018-09

Rubber, raw — Determination of bound acrylonitrile content in acrylonitrile-butadiene rubber (NBR) —

Part 1: Combustion (Dumas) method

Caoutchouc brut — Détermination du contenu en acrylonitrile lié dans le caoutchouc acrylonitrile-butadiène (NBR) —

Partie 1: Méthode par combustion (Dumas)

Reference number
ISO 24698-1:2018(E)

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Published in Switzerland

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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 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 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 24698-1:2008), which has been technically revised.

The main changes compared to the previous edition are as follows:

— addition in the scope of two types of raw rubbers, XNBR and NBIR, that contain acrylonitrile;

— addition of specified argon gas as carrier gas in 5.3;

— amendment of finishing condition of sample weight in 7.1 and 7.2.

A list of all the parts in the ISO 24698 series can be found on the ISO website.

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.

Rubber, raw — Determination of bound acrylonitrile content in acrylonitrile-butadiene rubber (NBR) —

Part 1: Combustion (Dumas) method

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.

CAUTION — 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 a method for the determination of the bound acrylonitrile content in NBR by an automatic analyser which uses a combustion process. The method is also applicable to XNBR (carboxylic acrylonitrile-butadiene rubber) and NBIR (acrylonitrile- butadiene-isoprene rubber) as well as NBR latex.

NOTE This document and ISO 24698-2 can give different results on the same rubber sample.

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 123, *Rubber latex — Sampling*

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

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:

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

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

4 Principle

The nitrogen in a sample of raw rubber is converted into oxides of nitrogen in an atmosphere of high-purity oxygen in the combustion unit of the analyser. The oxides of nitrogen are then converted into elemental nitrogen by a catalyst in the reduction unit. The carbon dioxide and water vapour produced are removed by absorption or another means of separation. Finally, the resultant gas is passed, with a carrier gas, into a thermal conductivity detector (TCD) to determine the nitrogen content.

5 Reagents and materials

5.1 Reference materials:

— **L-aspartic acid**, purity ≥99 %;

— **L-glutamic acid**, purity ≥99 %;

— **EDTA**, purity ≥99 %.

5.2 Oxygen gas, purity ≥99,99 % or in accordance with the analyser manufacturer's instructions.

5.3 Carrier gases:

— **helium gas, purity** ≥99,995 % or in accordance with the analyser manufacturer's instructions;

— **carbon dioxide gas, purity** ≥99,995 % or in accordance with the analyser manufacturer's instructions;

— **argon gas, purity** ≥99,995 % or in accordance with the analyser manufacturer's instructions.

5.4 Ethanol, purity ≥95 % by volume.

5.5 Methanol, purity ≥99,8 % by volume.

6 Apparatus

6.1 Automatic analyser.

6.1.1 General

The automatic analyser consists of the following components:

a) a combustion unit, capable of maintaining a minimum operating temperature in accordance with the manufacturer's instructions for combustion of the sample in an atmosphere of high-purity oxygen;

b) a high-purity oxygen feeder, capable of feeding enough high-purity oxygen for complete combustion;

c) a reduction unit, capable of fully converting liberated nitrogenous compounds to nitrogen gas;

d) an absorber (or another type of separator) of by-products, capable of removing the water and carbon dioxide formed;

e) a TCD, capable of detecting the nitrogen gas formed;

f) a microprocessor, capable of calibrating the apparatus with a standard reference material and of converting the detector response into mass % of nitrogen in the sample.

6.1.2 Performance requirements

The accuracy of the system shall be demonstrated by performing ten successive determinations using a reference material such as L-aspartic acid, L-glutamic acid or EDTA. The mean of the ten determinations with the reference material shall be within ±0,2 percentage points of the theoretical value. The relative standard deviation shall be within 0,5 % by mass of nitrogen for the reference material.

NOTE The relative standard deviation, (r), is given by Formula (1):

