INTERNATIONAL STANDARD

ISO 24698-2

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

Part 2: Kjeldahl method

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

Partie 2: Méthode Kjeldahl

Reference number
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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 24698-2:2008), 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 NBR latex to the scope and in sample preparation (7.2);

— amendment of of finishing condition of sample weight in 7.1;

— addition of ITP results of NBR latex in a new Annex C.

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 2: Kjeldahl 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 the Kjeldahl method. 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 24698-1 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 https://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 an ammonium salt by digestion with potassium sulfate, sulfuric acid and a copper sulfate catalyst. Ammonia is then released by the addition of strong alkali and steam-distilled into boric acid solution and titrated with standard volumetric acid solution.

5 Reagents and materials

5.1 Reference material: ammonium sulfate, purity ≥99,5 %.

5.2 Standard volumetric acid solutions:

— sulfuric acid solution, 0,05 mol/dm^3;

— hydrochloric acid solution, 0,05 mol/dm^3.

5.3 Sulfuric acid, ρ = 1,84 g/cm^3.

5.4 Sodium hydroxide solution, 10 mol/dm^3.

5.5 Potassium sulfate (K_2SO_4).

5.6 Copper sulfate ($CuSO_4 \cdot 5H_2O$).

5.7 Indicators, in accordance with the analyser manufacturer's instructions:

— methyl red;

— methylene blue;

— bromophenol blue.

5.8 Boric acid solution, in accordance with the analyser manufacturer's instructions (commonly, 3 % or 4 % solution).

5.9 Ethanol, purity ≥95 % by volume.

5.10 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 digestion unit, capable of maintaining a minimum operating temperature in accordance with the manufacturer's instructions for digestion of the sample;

b) a digestion tube, capable of being used for both digestion and distillation;

c) a distillation unit, capable of introducing a fixed volume of sodium hydroxide into the digestion tube when it is placed in the unit and of steam-distilling, for a fixed time, the liberated NH_3 and condensing it into a fixed volume of ammonia-absorbing boric acid solution in a titration vessel;

d) a titration unit, capable of introducing the boric acid solution into the titration vessel before distillation and of titrating the distillate with standard volumetric acid solution photometrically using a photo-cell or potentiometrically;

e) a microprocessor, capable of calibrating the instrument with a reference material and of converting the titration result into mass % of nitrogen in the sample.