
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)



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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 $\geq 99\%$;
- **L-glutamic acid**, purity $\geq 99\%$;
- **EDTA**, purity $\geq 99\%$.

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

5.3 Carrier gases:

- **helium gas**, purity $\geq 99,995\%$ or in accordance with the analyser manufacturer's instructions;
- **carbon dioxide gas**, purity $\geq 99,995\%$ or in accordance with the analyser manufacturer's instructions;
- **argon gas**, purity $\geq 99,995\%$ or in accordance with the analyser manufacturer's instructions.

5.4 Ethanol, purity $\geq 95\%$ by volume.

5.5 Methanol, purity $\geq 99,8\%$ 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 $\pm 0,2$ 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 \(1\)](#):

$$(r) = \frac{s}{w_N} \times 100 \quad (1)$$

where

(r) is the relative standard deviation, in %;

s is the standard deviation;

w_N is the mean nitrogen content, in mass %.

6.2 Balance, weighing to the nearest 0,1 mg.

6.3 Extraction apparatus, as specified for method C in ISO 1407:2011.

6.4 Beaker, capacity 300 cm³.

6.5 Stirrer.

6.6 Sieve, aperture 150 µm.

6.7 Absorbent tissue.

6.8 Drying oven, capable of maintaining a temperature of 100 °C ± 2 °C.

6.9 Roll mill, the temperature of whose rolls can be maintained at 100 °C ± 5 °C.

6.10 Combustion container, in accordance with the analyser manufacturer's instructions.

7 Sampling and preparation of the sample for the determination

7.1 Raw NBR, XNBR or NBIR rubber

Take a sample of 10 g to 50 g in accordance with the method specified in ISO 1795, and pass the sample between the surfaces of the mill rolls with the nip set at 0,2 mm ± 0,05 mm and with the surface temperature of the rolls maintained at 100 °C ± 5 °C.

Take a portion of about 3 g to 5 g from the sample prepared as specified above.

Extract this portion with ethanol by method C in ISO 1407:2011 and rinse the extracted material twice with a small amount of fresh ethanol. Pour the contents of the flask onto a clean 150 µm sieve to recover the extracted material. Gently blot the extracted material with absorbent tissue to remove excess solvent, and dry the pieces of extracted rubber, separated from each other, in the oven at 100 °C ± 2 °C until the mass does not change by more than 2,0 mg over a period of 10 min.

7.2 NBR latex

Take about 500 g of NBR latex sample by the appropriate method in ISO 123.

In order to coagulate the latex, add dropwise a small portion of the NBR latex sample, containing approximately 5 g of solid NBR, to 150 cm³ of ethanol or methanol in the 300 cm³ beaker, stirring the ethanol or methanol at the same time. After all the latex has been added, continue stirring for another 5 min. Then pour the contents of the beaker onto a clean 150 µm sieve to recover the coagulated polymer. Place the recovered polymer and 100 cm³ of fresh solvent into the beaker and stir for 5 min. Again pour the contents of the beaker onto the 150 µm sieve to recover the polymer. Finally, dry the

recovered polymer between the surfaces of the mill rolls with the nip set at $0,2 \text{ mm} \pm 0,05 \text{ mm}$ and with the surface temperature of the rolls maintained at $100 \text{ }^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$ until the mass does not change by more than 2,0 mg over a period of 10 min.

8 Procedure

8.1 Operate the apparatus in accordance with the manufacturer's instructions. A general procedure is as described in [8.2](#) to [8.11](#).

8.2 Take as the test portion 50 mg to 500 mg of the extracted material prepared by the appropriate method in [Clause 7](#) and weigh it to the nearest 0,1 mg in the combustion container. The size of the test portion should preferably be chosen so that its total nitrogen content is between 5 mg and 15 mg. Input the mass of the test portion into the microprocessor.

8.3 Place the combustion container in the analyser and switch on the apparatus to start the determination.

8.4 The combustion container is moved into the combustion unit where combustion of the test portion in an atmosphere of high-purity oxygen takes place.

8.5 The combustion gases are then transferred to the reduction unit by a carrier gas. (If necessary, a portion of the combustion gases is measured by means of a measuring tube before being transferred to the reduction unit.)

8.6 In the reduction unit, nitrogenous compounds in the combustion gases are completely converted into nitrogen gas, and residual oxygen gas is removed by absorption.

8.7 The water and carbon dioxide formed are removed by an absorber or another type of separator.

8.8 Finally, the reduced combustion gases are passed into a TCD which determines the amount of nitrogen gas produced.

8.9 Residual combustion gases in the system are swept out with carrier gas or oxygen gas.

8.10 The microprocessor converts the detector response into the nitrogen content of the test portion, in mass %.

8.11 Calibrate the apparatus with a reference material before carrying out each determination or series of determinations.

9 Calculation of bound acrylonitrile content

Calculate the bound acrylonitrile content, w_A , of the sample from [Formula \(2\)](#):

$$w_A = 3,79 \times w_N \quad (2)$$

where

w_A is the bound acrylonitrile content of the sample, in mass %;

w_N is the nitrogen content determined, in mass %.

The test result is the value from a single determination of bound acrylonitrile content.

10 Precision

An interlaboratory test programme to determine the precision of this combustion method using automatic analysers was conducted on NBR in 2006 (see [Annex B](#)).

11 Test report

The test report shall include the following particulars:

- a) a reference to this document, i.e. ISO 24698-1:2018;
- b) full identification of the rubber tested;
- c) full identification of the apparatus used;
- d) the reference material used;
- e) the result of the test;
- f) the date of the test.

Annex A **(informative)**

Examples of automatic analysers based on the Dumas combustion method

A.1 General

There are differences between automatic analysers with regard to the combustion conditions (including the combustion temperature) and the catalyst. There are also differences with regard to the method of reduction, the method of absorption of the by-products and the method of conversion of the detector response into the mass % of nitrogen in the sample. However, all automatic analysers based on the Dumas combustion method which meet the performance requirements specified in [6.1.2](#) are suitable for the determination of the bound acrylonitrile content of NBR by the method specified in this document.

A.2 Examples of suitable automatic analysers¹⁾

- Vario MAX CN, Rapid N III: Elementar Analysensysteme GmbH;
- Sumigraph NC-220F: Sumika Chemical Analysis Service, Ltd.;
- Macro Corder JM1000CN, Macro Corder JM3000N: J Science Lab Co., Ltd.;
- Turbo Nitrogen 4040 Protein Analyzer: Costech International S.p.A.;
- Tru Spec N, CNS-2000: LECO Corporation.

1) These are examples of suitable instruments available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these instruments.

Annex B (informative)

Precision

B.1 General

An interlaboratory test programme (ITP) to determine the precision of this test method was conducted, using automatic analysers based on the combustion method, on NBR in 2006. The precision evaluated was a type 1 precision in accordance with ISO/TR 9272.

Ten laboratories participated in the ITP and two of these analysed with two different analysers. For the laboratories that submitted data from two types of analyser, the data from these separate analysers were included in the ITP as data from individual laboratories. This gave a database for twelve laboratories or pseudo-laboratories.

Two different materials (NBR rubbers) were used in the ITP. A test result was taken to be the value from a single determination of bound acrylonitrile. Test results were obtained on three different days, at intervals of four days between each of the three tests.

The precision results as determined by this ITP should not be applied to acceptance or rejection testing of any group of materials or products without documentation that the results of this precision evaluation actually apply to the products or materials tested.

B.2 Precision results

The precision results are based on test results (as defined above) and given in [Table B.1](#). These results were obtained using outlier deletion procedures as described in ISO/TR 9272.

Repeatability: The repeatability r of the test method has been established as the appropriate value tabulated in [Table B.1](#) for each material. Two single test results that differ by more than this value should be considered suspect and suggest that some appropriate investigative action be taken.

Reproducibility: The reproducibility R of the test method has been established as the appropriate value tabulated in [Table B.1](#) for each material. Two single test results that differ by more than this value should be considered suspect and suggest that some appropriate investigative action be taken.