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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 24698-1:2008(E)

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Contents

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 24698-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

ISO 24698 consists of the following parts, under the general title *Rubber, raw — Determination of bound acrylonitrile content in acrylonitrile-butadiene rubber (NBR)*:

— *Part 1: Combustion (Dumas) method*

— *Part 2: Kjeldahl method*

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

Part 1:
Combustion (Dumas) method

WARNING — Persons using this part of ISO 24698 should be familiar with normal laboratory practice. This part of ISO 24698 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 ensure compliance with any national regulatory conditions.

CAUTION — Certain procedures specified in this part of ISO 24698 may 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 part of ISO 24698 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 NBR latex.

NOTE Parts 1 and 2 of this International Standard may not necessarily give the same result for any given rubber sample.

2 Normative references

The following referenced documents are indispensable for the application 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 1407:1992, *Rubber — Determination of solvent extract*

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

3 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.

4 Reagents and materials

4.1 Reference materials:

- L-aspartic acid, purity $\geqslant$ 99 %;
- L-glutamic acid, purity $\geqslant$ 99 %;
- EDTA, purity $\geqslant$ 99 %.

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

4.3 Carrier gases:

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

4.4 Ethanol, purity $\geqslant$ 95 % by volume.

4.5 Methanol, purity $\geqslant$ 99,8 % by volume.

5 Apparatus

5.1 Automatic analyser.

5.1.1 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.

5.1.2 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 Relative standard deviation (%) $= \frac{s}{w_\mathrm{N}} \times 100$

where

s is the standard deviation;

w_N is the mean nitrogen content, in mass %.

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

5.3 Extraction apparatus, as specified for method B in ISO 1407:1992.

5.4 Beaker, capacity 300 cm^3.

5.5 Stirrer.

5.6 Sieve, aperture 150 µm.

5.7 Absorbent tissue.

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

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

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

6 Sampling and preparation of the sample for the determination

6.1 Raw NBR 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 B in ISO 1407:1992 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 0,1 mg over a period of 10 min.

6.2 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 cm^3 of ethanol or methanol in the 300 cm^3 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 cm^3 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 mm ± 0,05 mm and with the surface temperature of the rolls maintained at 100 °C ± 5 °C until the mass does not change by more than 0,1 mg over a period of 10 min.

7 Procedure

7.1 Operate the apparatus in accordance with the manufacturer's instructions. A general procedure is as described in 7.2 to 7.11.

7.2 Take as the test portion 50 mg to 500 mg of the extracted material prepared by the appropriate method in Clause 6 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.

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

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

7.5 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.)

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

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

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

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

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

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

8 Calculation of bound acrylonitrile content

Calculate the bound acrylonitrile content w_A of the sample, in mass %, from the following equation:

$$w_\mathrm{A} = 3{,}79 \times w_\mathrm{N}$$

where w_N is the nitrogen content determined, in mass %.

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

9 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).

10 Test report

The test report shall include the following particulars:

a) a reference to this part of ISO 24698;

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 5.1.2 are suitable for the determination of the bound acrylonitrile content of NBR by the method specified in this part of ISO 24698.

A.2 Examples of suitable automatic analysers

— 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.

NOTE These are examples of suitable instruments available commercially. This information is given for the convenience of users of this part of ISO 24698 and does not constitute an endorsement by ISO of these instruments.