INTERNATIONAL STANDARD

ISO 3900

Third edition
2023-07

Rubber — Nitrile latex — Determination of bound acrylonitrile content

Caoutchouc — Latex de nitrile — Détermination de la teneur en acrylonitrile lié

Reference number
ISO 3900:2023(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 document 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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 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 third edition cancels and replaces the second edition (ISO 3900:1995), which has been technically revised.

The main changes are as follows:

— addition of an introduction to explain the purpose of this revision;

— deletion of the note in Clause 1 and in Clause 4;

— update of the apparatus in Clause 5;

— in Clause 7, deletion of the preparation for dried film of NBR latex and addition of the preparation dried NBR latex in accordance with ISO 24698-2;

— modification of the determination method of bound acrylonitrile content in accordance with the macro-method in ISO 1656;

— update of the procedure and addition of the suitable determination conditions, in Clause 8;

— addition of the precision data in Annex A.

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.

Introduction

Generally, the bound acrylonitrile content in nitrile latex is between 18 % and 45 %, expressed in the nitrogen content in the range from 4,7 % to 11,9 %. In the previous version of this document, the bound acrylonitrile content of emulsion-polymerized NBR lattices was determined in accordance with the semi-micro method in ISO 1656. However, the semi-micro method is applicable to natural rubber and natural rubber latex with lower nitrogen content; it is not applicable to determine nitrile latex. The purpose of this document is to use the macro-method in ISO 1656 to determine the bound acrylonitrile content in nitrile latex and to give the suitable determination conditions for nitrile latex.

Rubber — Nitrile latex — Determination of bound acrylonitrile content

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.

1 Scope

This document specifies a method for the determination of the bound acrylonitrile content of emulsion-polymerized NBR lattices.

The method is applicable to NBR lattices having a bound acrylonitrile content between 18 % and 45 %. It is also applicable to, for example, carboxylic-nitrile-butadiene (XNBR) lattices and nitrile-isoprene (NIR) lattices.

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 1656:2019, *Rubber, raw natural, and rubber latex, natural — Determination of nitrogen content*

ISO 24698-2:2018, *Rubber, raw — Determination of bound acrylonitrile content in acrylonitrile-butadiene rubber (NBR) — Part 2: Kjeldahl method*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology 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 NBR latex is coagulated with ethanol or methanol and dried to constant mass. A known mass of dried sample is digested with a mixture of sulfuric acid, potassium sulfate and catalyst to convert the nitrogen present into ammonium hydrogen sulfate. The mixture is made alkaline and the liberated ammonia is absorbed in a solution of boric acid. The bound acrylonitrile content is calculated from the volumes of standard volumetric solution required in the test and blank titrations.

5 Reagents

Use the reagents specified in ISO 1656 for the macro-method, except that sulfuric acid standard volumetric solution (c = 0,1 mol/dm^3) shall be used and sodium hydroxide standard volumetric solution (c = 0,02 mol/dm^3) is not required.

6 Apparatus

Use ordinary laboratory apparatus and Kjeldahl apparatus with a digestion flask of capacity 800 cm^3.

7 Sampling and preparation of test sample

Take about 500 g of NBR latex sample in accordance with the methods specified in ISO 123.

Prepare the test sample in accordance with ISO 24698-2:2018, 7.2. Dry the coagulated polymer in the oven maintained at 100 °C ± 2 °C until the mass does not change by more than 2,0 mg over a period of 10 min.

8 Procedure

Proceed in accordance with the method in ISO 1656 for the macro-method. Suitable determination conditions are given in Table 1.

Determine the nitrogen content, also carrying out the specified blank test, using boric acid solution to absorb the ammonia, and sulfuric acid standard volumetric solution (c = 0,1 mol/dm^3) to titrate the absorbed solution.

Take the end-point of the titration as the colour change from bright green to grey, then to light purple.

Table 1 — Suitable determination conditions

Procedure Subclause of ISO 1656:2019	Conditions	Amount
5.4.1	Size of test portion	0,5 g to 1,0 g (weigh to the nearest 0,1 mg). The nitrogen content is between 50 mg and 100 mg
	Titanium dioxide catalyst mixture or Selenium catalyst mixture	9,0 g or 6,5 g
	Sulfuric acid	20 cm^3
	Water	250 cm^3
	Sodium hydroxide solution	100 cm^3
5.4.2	Boric acid solution	100 cm^3
	Mixed indicator solution	Three drops
NOTE In order to prevent strongly boiling, explosion proof boiling particles can be added in the digestion flask.		

9 Expression of results

Calculate the bound acrylonitrile content, w_A, expressed as a percentage by mass of the dried sample, using Formula (1).

$$w_A = \frac{10{,}61 \times c(V_1 - V_2)}{m} \quad (1)$$

where

c is the exact concentration of the sulfuric acid standard solution, in moles per cubic decimetre;

V_1 is the volume of the sulfuric acid standard solution required for the titration of test sample, in cubic centimetres;

V_2 is the volume of the sulfuric acid standard solution required for the titration in the blank test, in cubic centimetres;

m is the mass of dried sample used for the determination, in grams.

Express the results to the nearest 0,01 %.

10 Precision

See Annex A.

11 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 3900:2023;

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

c) the results and the units in which they are expressed;

d) details of any operation not included in this document to which reference is made, as well as details of any operation regarded as optional;

e) the date of the test.

Annex A
(informative)

Precision

A.1 General

The interlaboratory test programme (ITP) to determine the precision of this test method was conducted in 2022. The precision evaluated was a type 1 precision in accordance with method A of ISO 19983:2022.

Six laboratories participated in the ITP and two different materials (NBRL-1 and NBRL-2) were used. Test results were obtained on two different days at interval of one week. Two measurements were determined in each day. Titanium dioxide catalyst mixture and selenium catalyst mixture were used in this ITP.

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.

A.2 Precision results

The precision results using TiO_2 catalyst are given in Table A.1. The precision results using selenium catalyst are given in Table A.2. These results were obtained using outlier deletion procedures as described in ISO 19983.

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

b) Day to day 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.

c) 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.