INTERNATIONAL STANDARD

ISO 3900

Second edition
1995-12-15

Rubber — Nitrile latex — Determination of bound acrylonitrile content

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

Reference number
ISO 3900:1995(E)

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.

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.

International Standard ISO 3900 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 3900:1976), which has been technically revised.

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Rubber — Nitrile latex — Determination of bound acrylonitrile content

WARNING — Persons using this standard should be familiar with normal laboratory practice. This standard 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.

1 Scope

This International Standard specifies a method for the determination of the bound acrylonitrile content of emulsion-polymerized NBR latices having a bound acrylonitrile content, expressed as a percentage of the polymer content, of between 18 % and 45 %. The method is also applicable to, for example, carboxylic-nitrile-butadiene (XNBR) latices and nitrile-isoprene (NIR) latices.

NOTE 1 The determination includes any water-insoluble nitrogen-containing additives present in the latex.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 123:1985, *Rubber latex — Sampling.*

ISO 124:—[1], *Latex, rubber — Determination of total solids content.*

ISO 1407:1992, *Rubber — Determination of solvent extract.*

ISO 1656:—[2], *Rubber, raw natural, and rubber latex, natural — Determination of nitrogen content.*

3 Principle

A film is prepared from the latex and then extracted with water to remove water-soluble nitrogen-containing material, and finally dried to constant mass. The dry film is analysed by the method laid down in ISO 1656. A known mass of the film 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 distilled and 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.

NOTE 2 Some automatic analysers utilize the micro-Dumas method to analyse for nitrogen; at present this is not published as an ISO method. It is also noted that, while automatic analysers generally have a high repeatability, there is often poor reproducibility between instruments.

4 Reagents

The reagents specified in ISO 1656 for the semi-micro method shall be used, except that sulfuric acid

1) To be published. (Revision of ISO 124:1992)

2) To be published. (Revision of ISO 1656:1988)

(c = 0,05 mol/dm^3) shall be employed and sodium hydroxide (c = 0,02 mol/dm^3) is not required.

5 Apparatus

5.1 Mould, preferably of glass, 1 mm deep and square-shaped with approximately 50-mm sides.

5.2 Extraction apparatus, in accordance with ISO 1407.

5.3 Apparatus as specified in ISO 1656 for the semi-micro method.

5.4 Oven, capable of being maintained at 70 °C ± 2 °C.

5.5 Desiccator, capable of holding the mould (5.1).

6 Sampling

Sampling shall be carried out in accordance with one of the methods specified in ISO 123.

7 Procedure

If the total solids content of the latex is not known, determine it in accordance with ISO 124.

If the total solids content of the latex is greater than 41 % (*m/m*), dilute the latex with water to a solids content of 40 % (*m/m*) ± 1 % (*m/m*).

Pour the latex into the mould (5.1) placed on a perfectly horizontal surface, and scrape off the excess latex with a straightedge. Transfer the mould to the oven (5.4) and dry at 70 °C ± 2 °C for 16 h (i.e. overnight).

NOTE 3 At this stage, it is not essential that the film be totally dry, but it is necessary that it can be handled.

Transfer the mould to the desiccator (5.5) and allow to cool. Demould the film and roll it (or a portion of it) in filter paper so that no part of the film is anywhere in contact with any other part of the film, and place the roll in the extraction cup of the extraction apparatus (5.2). Pour into the extraction flask sufficient water to fill the extraction cup two or three times. Assemble the extraction apparatus and reflux continuously for 4 h such that each extraction takes 15 min to 30 min. Remove the extracted film and dry it to constant mass in accordance with ISO 124.

Proceed in accordance with the method in ISO 1656 for the semi-micro method, using 100 mg of the dried extracted film, weighed to the nearest 0,5 mg. Determine its nitrogen content, also carrying out the specified blank test, using boric acid solution to absorb the distilled ammonia, and sulfuric acid (c = 0,05 mol/dm^3) to titrate the distillate. Take the end-point of the titration as the colour change from bright green to grey.

8 Expression of results

Calculate the bound acrylonitrile content, expressed as a percentage by mass of the dried extracted film, using the formula

$$\frac{10{,}62 \times c(V_1 - V_2)}{m}$$

where

c is the actual concentration, expressed in moles per cubic decimetre, of the sulfuric acid solution used;

V_1 is the volume, in cubic centimetres, of the sulfuric acid solution used in the titration of the distillate from the test portion;

V_2 is the volume, in cubic centimetres, of the sulfuric acid solution used in the blank titration;

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

9 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary to identify the sample;

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

d) any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional;

e) the date of the test.

ICS 83.040.10

Descriptors: rubber, latex, nitrile latex, chemical analysis, determination of content, acrylonitriles, extraction methods.

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