INTERNATIONAL STANDARD

ISO 3899

Third edition
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Rubber — Nitrile latex — Determination of residual acrylonitrile content

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

Reference number
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Foreword

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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 3899 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This third edition cancels and replaces the second edition (ISO 3899:1988), of which it constitutes a minor revision the main purpose of which was to update the normative references clause.

Rubber — Nitrile latex — Determination of residual acrylonitrile content

WARNING — Persons using this International 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.

CAUTION — Certain procedures specified in this International Standard 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 International Standard specifies a method for the determination of the residual acrylonitrile content of nitrile rubber latices which have a residual acrylonitrile content of less than 0,2 % (by mass).

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 1042, *Laboratory glassware — One-mark volumetric flasks*

3 Principle

A test portion of latex is distilled and the distillate collected in methanol. *n*-Dodecyl mercaptan is added to the distillate and the excess titrated with standard iodine solution.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Silicone antifoaming agent, which does not affect the result of the determination.

4.2 Methanol.

4.3 Propan-2-ol.

4.4 Mercaptan solution, 1,25 % (by mass) *n*-dodecyl mercaptan solution in propan-2-ol.

4.5 Potassium hydroxide, 6 % (by mass) solution in 95 % (by volume) ethanol. The ethanol shall be free from aldehydes.

4.6 Acetic acid, glacial.

4.7 Iodine, standard volumetric solution, $c(I_2)$ = 0,012 5 mol/dm^3, freshly standardized with a standard reference solution of sodium thiosulfate.

5 Apparatus

Ordinary laboratory apparatus plus the following:

5.1 Distillation apparatus, consisting of a distillation flask of capacity 500 cm^3, a still head, a vertical water-cooled condenser and a receiver of capacity 100 cm^3 with a glass stopper through which pass a glass tube connected to the condenser and a shorter glass exit tube leading into a beaker of capacity 50 cm^3.

5.2 One-mark volumetric flask, of capacity 100 cm^3, conforming with the requirements of ISO 1042.

5.3 Conical flask, of capacity at least 250 cm^3.

6 Sampling

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

7 Procedure

7.1 Determination

Weigh 25 g ± 0,2 g of latex into the distillation flask (see 5.1) and add 100 cm^3 of water and 1 cm^3 of the silicone antifoaming agent (4.1). Place 25 cm^3 of methanol (4.2) in the receiver and assemble the distillation apparatus so that the end of the tube connected to the condenser is immersed in the methanol.

Put sufficient methanol (for example 10 cm^3) in the beaker to cover the end of the exit tube. Immerse the receiver and beaker in ice.

NOTE The purpose of the methanol in the beaker is to collect any acrylonitrile which is not trapped by the liquid in the receiver.

Distil the mixture, adjusting the rate of boiling to control frothing, and collect 50 cm^3 of distillate in the receiver.

Empty the contents of the receiver and beaker into the volumetric flask (5.2). Rinse through the condenser into the receiver twice with small (for example 5 cm^3) portions of methanol and add the washings to the volumetric flask. Dilute to the mark with methanol.

Pipette a 50 cm^3 aliquot portion of the diluted distillate into the conical flask (5.3) containing 25 cm^3 of propan-2-ol (4.3).

Pipette 10 cm^3 of mercaptan solution (4.4) into the flask.

Add 1 cm^3 of potassium hydroxide solution (4.5) and allow to react for exactly 2 min at 23 °C ± 2 °C. Add 2 cm^3 of glacial acetic acid (4.6) to stop the reaction. The resulting pH should be between 4 and 6. Titrate with freshly standardized iodine solution (4.7) to a yellow colour which persists for at least 60 s. Discard the iodine solution remaining in the burette, unless it is required for immediate use.

7.2 Blank test

Carry out a blank determination, following the procedure described in 7.1 but omitting the distillation stage and using 500 cm^3 of a (1 + 1) methanol-water mixture.

8 Expression of results

The residual acrylonitrile content is given, as a percentage by mass of the latex, by the formula

$$\frac{M \times 2c(V_0 - V_1)}{1000} \times \frac{100}{V_2} \times \frac{100}{m}$$

$$= \frac{20\,Mc(V_0 - V_1)}{m\,V_2}$$

where

M is the molar mass, in grams per mole, of acrylonitrile (= 53,06 g/mol);

c is the actual concentration, in moles of I_2 per cubic decimetre, of the iodine solution (4.7);

V_0 is the volume, in cubic centimetres, of iodine solution used in the blank titration (see 7.2);

V_1 is the volume, in cubic centimetres, of iodine solution used in the determination (see 7.1);

V_2 is the volume, in cubic centimetres, of the aliquot of diluted distillate taken (= 50 cm^3);

m is the mass, in grams, of the test portion (ca 25 g).

If results of duplicate determinations do not agree to within 0,005 % (by mass), they shall be rejected and additional determinations carried out.

9 Precision

No precision data are currently available.

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

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

c) the date of testing;

d) any unusual features noted during the determination;

e) details of 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.

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