

INTERNATIONAL STANDARD

ISO
3899

Second edition
1988-12-01



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Rubber — Nitrile latex — Determination of residual acrylonitrile content

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

ITeH STANDARD PREVIEW
(standards.iteh.ai)

ISO 3899:1988

<https://standards.iteh.ai/catalog/standards/sist/f90f1e11-2b8b-4614-acde-e9172c323f4e/iso-3899-1988>

Reference number
ISO 3899 : 1988 (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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 3899 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

[ISO 3899:1988](https://standards.iteh.ai/catalog/standards/sist/f90ffe11-2b8b-4614-aede-0112322574c4/iso-3899-1988)

[https://standards.iteh.ai/catalog/standards/sist/f90ffe11-2b8b-4614-aede-](https://standards.iteh.ai/catalog/standards/sist/f90ffe11-2b8b-4614-aede-0112322574c4/iso-3899-1988)

This second edition cancels and replaces the first edition (ISO 3899 : 1976) of which it constitutes a minor revision.

Rubber — Nitrile latex — Determination of residual acrylonitrile content

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 % (*m/m*).

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 1042 : 1983, *Laboratory glassware — One-mark volumetric flasks*.

3 Principle

Distillation of a test portion of latex and collection of the distillate in methanol. Addition of *n*-dodecyl mercaptan to the distillate and titration of the excess with a standard iodine solution.

4 Reagents

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

WARNING — All recognized health and safety precautions shall be taken when using the procedure specified in this International Standard.

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 % (*m/m*) *n*-dodecyl mercaptan in propan-2-ol.

4.5 Potassium hydroxide, 6 % (*m/m*) solution in 95 % (*V/V*) ethanol. The ethanol shall be free from aldehydes.

4.6 Acetic acid, glacial.

4.7 Iodine, standard volumetric solution, $c(I_2) = 0,0125 \text{ mol/dm}^3$, freshly standardized with a standard reference solution of sodium thiosulfate.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Distillation apparatus, consisting of distillation flask of capacity 500 cm³, stillhead, vertical water-cooled condenser and receiver of capacity 100 cm³ with glass stopper through which pass a glass tube connected to the condenser and a shorter exit glass tube leading into a beaker of capacity 50 cm³.

5.2 One-mark volumetric flask, of capacity 100 cm³, conforming with the requirements of ISO 1042.

5.3 Conical flask, of capacity at least 250 cm³.

6 Sampling

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

7 Procedure

7.1 Determination

Weigh $25 \text{ g} \pm 0,2 \text{ g}$ 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 the 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 dissolved 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 the propan-2-ol (4.3). Pipette 10 cm^3 of the mercaptan solution (4.4) into the flask. Add 1 cm^3 of the potassium hydroxide solution (4.5) and allow to react for exactly 2 min at $23 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$. Add 2 cm^3 of the glacial acetic acid (4.6) to stop the reaction. The resulting pH should be between 4 and 6. Titrate with the 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, using 50 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

$$\frac{M \times 2c(V_0 - V_1)}{1\,000} \times \frac{100}{V_2} \times \frac{100}{m}$$

$$= \frac{20 M c (V_0 - V_1)}{m V_2}$$

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 (= 50 cm^3);

m is the mass, in grams, of the test portion ($\approx 25 \text{ g}$).

The results of duplicate determinations shall agree to within 0,005 % (m/m).

9 Test report

The test report shall include the following particulars:

- a reference to this International Standard;
- the results, and the units in which they are expressed;
- the date of testing;
- any unusual features noted during the determination;
- any operation not included in this International Standard or in the International Standard to which reference is made, as well as any operation regarded as optional.

UDC 678.745.3.031 : 543.8

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

Price based on 2 pages