INTERNATIONAL STANDARD ISO 3899

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Rubber — Nitrile latex — Determination of residual acrylonitrile content

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

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FOREWORD

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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.

International Standard ISO 3899 was drawn up by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the Member Bodies in July 1975.

It has been approved by the Member Bodies of the following countries :

Australia	India	Sweden
Belgium	Italy	Thailand
Brazil	Mexico	Turkey
Canada	Netherlands	United Kingdom
Czechoslovakia	Poland	U.S.A.
France	Portugal	U.S.S.R.
Germany	Romania	
Hungary	Spain	

No Member Body expressed disapproval of the document.

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Rubber — Nitrile latex — Determination of residual acrylonitrile content

1 SCOPE AND FIELD OF APPLICATION

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 REFERENCE

ISO 123, *Rubber latex — Sampling.*

3 PRINCIPLE

Distillation of the 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.

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, 0,012 5 M, freshly standardized with a standard volumetric sodium thiosulphate solution.

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 Volumetric flask, of capacity 100 cm³.

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

Weigh 25,0 ± 0,2 g of latex into the distillation flask (5.1) and add 100 cm³ of water and 1 cm³ of the silicone antifoaming agent (4.1). Place 25 cm³ 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 cm³) 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 cm³ 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 cm³) portions of methanol and add the washings to the volumetric flask. Dilute to the mark with methanol.

* The term millilitre (ml) is commonly used for the cubic centimetre (cm³), particularly to denote the capacity of laboratory glassware. Apparatus with either type of marking is satisfactory for use with this International Standard.

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 ± 2 °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.

Carry out a blank determination, 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

$$\frac{42{,}4 \times T \times (V_0 - V_1)}{V_2}$$

where

T is the molar concentration of the iodine solution;

V_0 is the volume, in cubic centimetres, of iodine solution used in the blank titration;

V_1 is the volume, in cubic centimetres, of iodine solution used in the sample titration;

V_2 is the volume, in cubic centimetres, of the aliquot of diluted distillate.

The results of duplicate determinations shall agree within 0,005 unit.

NOTE — The above formula is derived from the following expression :

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

where

M is the molar mass of acrylonitrile = 53,06 g;

m is the mass, in grams, of latex.

9 TEST REPORT

The test report shall include the following particulars :

a) a reference to this International Standard;

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

c) the date of testing;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standard to which reference is made, or regarded as optional.