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Plastics — Styrene/acrylonitrile copolymers — Determination of residual acrylonitrile monomer content — Gas chromatography method

*Plastiques — Copolymères styrène/acrylonitrile — Dosage de
l'acrylonitrile monomère résiduel — Méthode par chromatographie en
phase gazeuse*



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Foreword

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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 4581 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 4581:1987), the annex of which has been technically revised.

Annex A forms an integral part of this International Standard.

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Plastics — Styrene/acrylonitrile copolymers — Determination of residual acrylonitrile monomer content — Gas chromatography method

1 Scope

This International Standard specifies a method for determining the content of residual acrylonitrile monomer in styrene/acrylonitrile copolymers and blends by gas chromatography. Bearing in mind that gas chromatography offers a wide range of possible conditions, the method specified in this International Standard is that shown to have been suitable in practice.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2561:1974, *Plastics — Determination of residual styrene monomer in polystyrene by gas chromatography*.

3 Principle

A test portion is dissolved in dimethylformamide and a small volume of the solution injected into a gas chromatograph equipped with flame ionization detector to separate and detect the volatile components. The solvent contains a known amount of propionitrile or acetonitrile as an internal standard for quantitative evaluation. With this method, a lower detection limit of the order of 3 parts per million (ppm) of acrylonitrile in the copolymer may be expected. To obtain a lower

detection limit of the order of 0,3 ppm to 0,4 ppm, an alternative method is specified in annex A. In this method, the test portion is dissolved in propanediol-(1,2)-carbonate, and the solution is injected into a gas chromatograph equipped with a thermionic nitrogen detector. The solution also contains propionitrile as an internal standard.

4 Reagents

During the analysis, use only reagents of recognized analytical grade. Special safety precautions shall be observed when handling the following reagents, especially acrylonitrile.

4.1 Dimethylformamide, of purity such that no impurity peaks occur within the range of retention times of the substances to be determined.

4.2 Propionitrile.

Acetonitrile may be used as internal standard instead of propionitrile, if it has been shown that the same results are obtained.

4.3 Acrylonitrile.

5 Apparatus

Ordinary laboratory apparatus, plus the following:

5.1 Gas chromatograph, with flame ionization detector and recorder.

Gas-chromatographic operating conditions

Column: Stainless-steel or glass tubing, 1 m to 2 m length, 3 mm to 4 mm internal diameter is recommended. The column shall be packed with

Porapak Q¹⁾ of particle size from 50 mesh to 100 mesh. To prevent non-volatile material contained in the test solution from entering the column, suitable means shall be provided, such as a glass liner in the injection port or a pre-column of 5 cm length with the same packing as the column and mounted in such a way as to allow frequent renewal.

The method of packing is not specified, but shall be such as to obtain a satisfactory separation efficiency of the column. The column shall be aged for 24 h at 230 °C with gas flow. Change in column dimensions is permissible only if this has proved to give the same results.

Column and, if applicable, pre-column temperature: 160 °C to 180 °C isothermal.

Temperature of injection port: 200 °C to 230 °C.

Temperature of detector compartment: 230 °C.

Carrier gas: helium (or nitrogen) as specified in ISO 2561.

Flowrate of carrier gas: to be adjusted such that propionitrile is eluted in 5 min to 10 min.

Flame ionization detector in which the hydrogen and air flowrates are adjusted such as to give

- a) high sensitivity of response;
- b) a linear relationship between response and concentration in the measured range;
- c) an only insignificant effect of small changes in flowrates on response and sensitivity.

Recommended recorder speed: 1,0 cm/min or 1,27 cm/min.

The silicone-rubber septum for injection shall have a layer of polytetrafluoroethylene on each side in order to delay attack by dimethylformamide. If the septa available have a protective layer on one side only, two shall be used. Even so, it will be necessary to renew them frequently.

5.2 Microsyringes, of capacity of 1 µl to 50 µl.

5.3 Analytical balance, accurate to within 0,5 mg.

1) Porapak Q is the trade-name of a product of Millipore Corporation. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement of this product by ISO. By agreement between the interested parties, alternative products of equal performance may be used.

6 Preparation of test sample

The sample may be taken from material in the form of powder, pellets or a moulding. Large pieces shall be reduced to fragments sufficiently small to permit a sample amount as close as possible to 1,0 g to be weighed out. This size-reduction operation shall not cause the sample to become heated.

7 Procedure

7.1 Preparation of the internal-standard solution

Transfer exactly 1 ml of propionitrile (4.2) by means of a pipette into a 100 ml one-mark volumetric flask and dilute to the mark with dimethylformamide (4.1), both reagents having been kept at 20 °C ± 1,0 °C. Transfer with a pipette exactly 5 ml of the solution thus obtained into another 100 ml one-mark volumetric flask and dilute to the mark with dimethylformamide. If necessary, further dilute this diluted solution with dimethylformamide, depending on the acrylonitrile concentration in the polymer sample, in order to be able to use the same detector sensitivity. During the dilution process, maintain the liquids at a temperature of 20 °C ± 1,0 °C.

7.2 Preparation of the sample solution

Weigh, to the nearest 1 mg, approximately 1 g of the polymer sample and transfer to a 20 ml one-mark volumetric flask fitted with a ground-glass stopper. Add approximately 15 ml of dimethylformamide (4.1). After closing the flask, allow the polymer to dissolve, with shaking if necessary. After complete dissolution, add exactly 1 ml of the internal-standard solution, prepared in accordance with 7.1 and kept at 20 °C ± 1,0 °C, from a pipette and dilute to the mark with dimethylformamide. Keep the solution for injection into the gas chromatograph.

7.3 Preparation of acrylonitrile solutions for calibration

7.3.1 Preparation of a stock standard solution

Weigh, to the nearest 1 mg, 0,1 ml of acrylonitrile (4.3) together with an appropriate amount of dimethylformamide (4.1) into a 100 ml one-mark volumetric flask and dilute to the mark with dimethylformamide which has been kept at