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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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*

Reference number
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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.

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 4581 was prepared by Technical Committee ISO/TC 61, *Plastics*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

1 Scope and field of application

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 Reference

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

3 Principle

Dissolution of a test portion in dimethylformamide and injection of a small volume of the solution into a gas chromatograph equipped with flame ionization detector to obtain separation and detection of 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. For obtaining a lower detection limit of the order of 1 ppm, an alternative method is specified in the annex. In this method, the test portion is dissolved in *N,N*-dimethylacetamide, propylene carbonate or ethylmethylketone, and the solution is injected into a gas chromatograph equipped with nitrogen-phosphorus detector. The solvent 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 and

5.1 Gas chromatograph, with flame ionization detector and recorder.

Gas chromatographic operating conditions

Column : Stainless steel or glass tubing, 1 to 2 m length, 3 to 4 mm internal diameter is recommended. The column is packed with Porapak Q¹⁾ of particle size from 50 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 to 180 °C isothermal.

Temperature of injection port : 200 to 230 °C.

Temperature of detector compartment : 230 °C.

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

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

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.