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Plastics — Determination of residual styrene monomer in polystyrene (PS) and impact-resistant polystyrene (PS-I) by gas chromatography

Plastiques — Détermination du styrène monomère résiduel dans le polystyrène (PS) et le polystyrène résistant au choc (PS-I) par chromatographie en phase gazeuse

FDIS stage

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Foreword

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Field Code Changed

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This fourth edition cancels and replaces the third edition (ISO 2561:2012), which has been technically revised.

The main changes are as follows:

— ~~Adding~~adding headspace injection as another sample introducing option for gas chromatography.

~~A list of all parts in the ISO 2561 series can be found on the ISO website.~~

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at ~~www.iso.org/members.html.~~www.iso.org/members.html.

Plastics— — Determination of residual styrene monomer in polystyrene (PS) and impact-resistant polystyrene (PS-I) by gas chromatography

1 Scope

This document specifies a method for the determination of the residual styrene monomer in polystyrene (PS) and impact-resistant polystyrene (PS-I) by gas chromatography. It can also be used for the simultaneous determination of other volatile aromatic hydrocarbons in PS and PS-I.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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- 472, *Plastics — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ——ISO Online browsing platform: available at ~~https://www.iso.org/obp~~https://www.iso.org/obp

— ——IEC Electropedia: available at ~~http://www.electropedia.org/~~https://www.electropedia.org/

4 Principle

PS or PS-I sample is dissolved in solvent containing an internal standard. To obtain separation of styrene and other volatile materials, gas chromatography method is employed, in which three sample introducing options are available:

— Option A: a small volume of the polymer solution is injected directly into a gas chromatograph.

— Option B: a small volume of the supernatant solution remaining after precipitation of polymer by addition of a precipitator is injected into a gas chromatograph.

— Option C: a small volume of vapor of the polymer solution under thermal equilibrium is injected into a gas chromatograph.

5 Reagents and materials

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 -Internal standard

The internal standard shall be selected based on consideration of the retention times of the materials contained in the polymer sample and solvent. Recommended candidates are n-butylbenzene, cyclopentanol, 1,2,4-trimetylbenzene and 1,4-diethylbenzene of sufficient purity for analytical use.

5.2 Solvent

Use dimethylformamide, butanone, dichloromethane, or tetrahydrofuran. Tetrahydrofuran is used only for method A. Only dimethylformamide is used in method C.

5.3 Precipitator

Use 2,2,4-trimethylpentane or ethanol.

5.4 Aromatic hydrocarbons

Use styrene and other aromatic hydrocarbons such as ethylbenzene, cumene or α-methylstyrene, if required. Styrene shall be checked for self-polymerizaiton before use. The criterion for acceptance is that the mixture of styrene and ethanol of the same volume shall be clear. When determining the content of other aromatic hydrocarbons in the sample, other aromatic hydrocarbons such as ethylbenzene, cumene or α-methylstyrene shall be used.

5.5 Carrier gases and fuel gases for gas chromatograph

Hydrogen, helium or nitrogen, according to the type of detector used, shall be used as carrier gas. Use hydrogen and air as fuel gases. If detectors are used which require carrier gases and fuel gases other than those mentioned, the carrier gases and fuel gases shall be specified.

~~WARNNING~~ WARNING — **Strict observance of safety regulations is essential when using hydrogen.**

6 Apparatus

6.1 General

Normal laboratory equipment and the following apparatus are required. Typical operating conditions are described in ~~Annex A.~~Annex A.

6.2 Gas chromatograph

6.2.1 Injection port~~:~~. Use an injection port for liquid samples or gas samples. When using a capillary column, an injection port with splitter may be applicable.

6.2.2 Headspace ~~Sampler:~~**sampler.** only used in method C, including backflush capability, thermostated sample tray, and associated accessories ~~fulfill~~fulfil these requirements while providing for automatic sequential sampling of headspace vapors.

6.2.3 Column~~:~~. The column diameter and length, as well as the packing material and stationary phase shall be selected based on consideration of column resolution and calibration curve linearity. Both packed columns and capillary columns are acceptable. Capillary columns are recommended in the light of accuracy.

6.2.4 Detector~~:~~. Use a suitable detector.

NOTE The most commonly used detector is a hydrogen flame ionization detector (FID).

6.3 Data processor

Use a recorder or microcomputer to record the signals from the detector.

6.4 Sample injection syringe

Use a micro-syringe of the 1-µl to 50-µl type. A micro-syringe integrated with the auto-injector may also be used.