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Plastics/rubber — Polymer dispersions and rubber latices (natural and synthetic) — Determination of residual monomers and other organic components by capillary-column gas chromatography —

Part 1:
Direct liquid injection method

Plastiques/caoutchouc — Dispersions de polymères et latex de caoutchouc (naturel et synthétique) — Détermination des monomères résiduels et autres constituants organiques par chromatographie en phase gazeuse sur colonne capillaire —

Partie 1: Méthode d'injection liquide directe

ICS: 83.040.10; 83.080.01

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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. 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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 13741-1 was prepared by Technical Committee ISO/TC 61, Plastics, Subcommittee SC 9, Thermoplastic materials, in close collaboration with ISO/TC 45, Rubber and rubber products.

ISO 13741 consists of the following parts, under the general title Plastics/rubber — Polymer dispersions and rubber latices (natural and synthetic) — Determination of residual monomers and other organic components by capillary-column gas chromatography:

— Part 1: Direct liquid injection method

— Part 2: Headspace method

Introduction

The requirements imposed today by authorities include the assessment of the content of residual monomers and organic saturated volatiles, for health and environmental reasons sometimes down to minute traces. Former standards for measurement of residual volatiles based on distillation linked with titration cannot cope with such exigences.

This part of ISO 13741 presents an advanced method for the determination, by gas chromatography, of residual monomers and other organic components in polymer dispersions and latices. This standard provides a method that is in line with present-day requirements for analytical methods and is intended for use instead of ISO 2008:1987, Rubber latex, styrene-butadiene — Determination of volatile unsaturates, and ISO 3899:1988, Rubber — Nitrile latex — Determination of residual acrylonitrile content, where precise measurements of volatile-matter content are needed, and expands their scope to include other volatile organic components.

Plastics/rubber — Polymer dispersions and rubber latices (natural and synthetic) — Determination of residual monomers and other organic components by capillary-column gas chromatography —

Part 1:
Direct liquid injection method

WARNING — This part of ISO 13741 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this part of ISO 13741 to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use

1 Scope

1.1 This part of ISO 13741 specifies a method for the determination of residual monomers and other (saturated) organic components in aqueous polymer dispersions and latices as well as in related products. It makes use of capillary-column gas chromatography with direct injection of the liquid sample.

1.2 Residual monomers and saturated volatiles that have been successfully determined by this method include acrylic and methacrylic esters, acrylonitrile, butadiene, styrene, vinyl acetate, vinyl chloride as well as by-products such as acetaldehyde and ethylbenzene. Butadiene could be co-eluted with cis-2-butene.

NOTE N-butyl acrylate and dibutyl ether cannot be separated with DB1 column. In this case use DB5 column.

1.3 Since the chromatograms obtained normally contain a series of peaks, it is only possible to determine the content of those volatiles for which response factors have been determined. For the identification of unknown peaks including SVOC (semi volatile organic compounds), auxiliary methods like mass spectroscopy or the use of a second GC column with a different polarity are advisable. Toluene equivalent can also be used for the alternative quantification method of unknown peaks.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 13741. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 13741 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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

3 Principle

A test sample is diluted with water containing an internal standard and injected onto the liner of a gas chromatograph with a capillary column, a flame ionization detector and a linear temperature programming capability.

4 Reagents

Unless otherwise stated, use only reagents of recognized analytical grade and only grade 1 water as defined in ISO 3696.

4.1 Carrier gas: nitrogen, or helium of 99,995 % (or higher) purity.

NOTE hydrogen can also be used but with a safety care.

4.2 Propionitrile, 99 % purity, for use as internal standard.

Propionitrile has been found to be a suitable internal standard, but other at least partly water-soluble organic compounds not found in the sample could be used as the internal standard, e.g. iso-butyl acetate or methyl iso-butyl ketone. The internal standard shall yield a clear chromatographic separation and shall not interfere with any component originally present in the sample.

4.3 Monomers and other organic compounds of interest, 99 % purity, for comparison purposes.

4.4 Dimethylformamide (DMF)

4.5 Tetrahydrofuran (THF).

5 Apparatus

Ordinary laboratory equipment, plus the following:

5.1 Gas chromatograph, having an injection port designed for split operation, with a liner of at least 1 cm^3 volume, a flame-ionization detector (FID) and a linear temperature programming capability for the column.

5.2 Capillary column, of length 30 m and internal diameter 0,53 mm, 0,32 mm or 0,25 mm made of fused silica that is covered inside with a 1 µm to 5 µm thick film of a dimethylpolysiloxane.

5.3 Integrator or **suitable recorder.** Recording or integration is made using a chromatography data system(DTS) such as Chromeleon.

5.4 Microsyringe, capacity 10 µl to 50 µl.

NOTE 1 Automated sample injector is recommended for large number of test samples.

5.5 Analytical balance, accurate to 0,1 mg.

5.6 Volumetric flasks, capacity 50 ml and 1000 ml.

6 Preparation of apparatus

6.1 Partly fill the insert liner with glass wool to retain solids during injection.

6.2 Control the detector temperature so that it is constant to within 1 °C, without thermostat cycling which causes an uneven baseline.

Table 1 — Typical operating conditions[1)]

Detector	flame ionization
Air flow rate	300 or 450 ml/min
Hydrogen flow rate	30 ml/min
Make-up gas flow rate	30 ml/min
Column	
Length	30 m
Inside diameter	0,25 mm, 0,32 mm or 0,53 mm
Film thickness	1 µm to 5 µm (dimethylpolysiloxane)
Carrier gas	nitrogen or helium
Flow rate	0,8 to1,2 ml/min or 4 ml/min
Purge rate	1 to 2 ml/min
Temperatures	
Injection port	150 °C to 200 °C [3)]
Detector block	250 °C
Initial column temperature	50 °C
Hold time	5 min
Program rate	5 °C/min
Final column temperature	200 °C (or higher as needed)
Final hold time [2)]	7 min (or longer)
Injection volume	1 µl
Split ratio	10:1 to 100:1

1) It may be necessary to modify these conditions if separation problems are encountered or if other conditions are specified in the gas chromatograph manufacturer's instructions. For instance a column with an inside diameter < 0,53 mm may be more suitable: in this case, reduce the carrier gas flow rate to ca. 1 cm^3/min

2) After the final hold, heating to 300 °C or 320 °C is recommended to purge the column.

3) Methyl methacrylate is not thermally stable above 175 °C

7 Calibration

7.1 For reliable results, it is necessary to calibrate the instrument for each analysis with respect to sensitivity and retention time.

This is done by determining the response factors and retention times for each component expected to be present in the dispersion or the latex by injecting small amounts of the internal standard together with the individual components, or mixtures thereof, dissolved in a solvent (e.g. dimethylformamide or tetrahydrofuran).