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Packaging - Flexible packaging material - Determination of residual solvents by static headspace gas chromatography - Part 2: Industrial methods

Verpackung - Flexible Packstoffe - Bestimmung der Restlösemittel durch statische Dampfdruckanalyse mittels Gaschromatographie - Teil 2: Industrielle Verfahren

Emballage - Matériaux d'emballages souples - Détermination des solvants résiduels par chromatographie en phase gazeuse et espace de tête statique - Partie 2: Méthodes industrielles

Ta slovenski standard je istoveten z: EN 13628-2:2002

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55.040 T æ \ ä æ Á \ ä æ Packaging materials and accessories

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EUROPEAN STANDARD
NORME EUROPÉENNE
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EN 13628-2

October 2002

ICS 55.040

English version

**Packaging - Flexible packaging material - Determination of
residual solvents by static headspace gas chromatography -
Part 2: Industrial methods**

Emballage - Matériaux d'emballages souples -
Détermination des solvants résiduels par chromatographie
en phase gazeuse et espace de tête statique - Partie 2:
Méthodes industrielles

Verpackung - Flexible Packstoffe - Bestimmung der
Restlösemittel durch statische Dampfdruckanalyse mittels
Gaschromatographie - Teil 2: Industrielle Verfahren

This European Standard was approved by CEN on 26 August 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.



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Foreword

This document EN 13628-2:2002 has been prepared by Technical Committee CEN/TC 261 "Packaging", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2003, and conflicting national standards shall be withdrawn at the latest by April 2003.

This standard is part of a standard for determination of residual solvents by static headspace gas chromatography, which is published in two parts:

- *Part 1: Absolute methods*

- *Part 2: Industrial methods*

This part of EN 13628 should be read in conjunction with EN 13628-1.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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EN 13628-2:2002 (E)

1 Scope

This part of this European Standard specifies rapid methods as commonly used in quality control for monitoring the level of residual solvents used in the production of flexible packaging by static headspace chromatography. The procedures described in this part involve one single injection of the headspace which implies an incomplete extraction of the solvent. The values obtained may be lower than the absolute content which should be determined according to Part 1. Residues from thermal decomposition products are not within the scope of this standard.

The method is applicable to flexible packaging materials that may consist of mono- or multilayer plastic films, paper or board, foil or combinations thereof.

This method does not apply to residual solvents with amounts lower than 0,5 mg/m².

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

ISO 2859-1, *Sampling procedures for inspection by attributes - Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*.

ISO 2859-2, *Sampling procedures for inspection by attributes - Part 2: Sampling plans indexed by limiting quality (LQ) for isolated lot inspection*.

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*.

3 Principle

Specimens of the flexible packaging material are placed in a hermetically closed vial and heated under closely controlled conditions of time and temperature to vaporize solvents into the headspace. The amount of solvent released into the headspace is determined by transferring an aliquot of the headspace into a gas chromatograph for analysis. The transfer may be performed:

- a) by specific semi-automatic or automatic systems which allow pressurization of the heated vials;
- b) manually or automatically by using a heated gastight syringe or a loop without pressurization of the heated vials.

The relative amount of residual solvent is determined by single headspace extraction using external or internal standards.

NOTE For reproducibility the same incubation conditions should be used for each single analysis. During the analysis, there could be interferences from possible products of thermal decomposition. Additional peaks due to these products should not be considered for evaluation of residual solvents.

4 Reagents

4.1 General

All reagents shall be of a recognized analytical reagent grade.

NOTE Grades stated as being suitable for chromatography can be commercially available and are recommended for use as reference for standard calibration solutions. Appropriate safety precautions should be used when handling toxic and/or flammable solvents.

4.2 Reference solvents, for the preparation of standard calibration solutions.

4.3 Dilution solvent, with a retention time different from those of residual solvents in the sample.

NOTE Solvents such as hexane, cyclohexanone, acid amides and glycerol triacetate (triacetin) are appropriate.

5 Apparatus

5.1 Glass vials, of capacity 6 ml, 8 ml, 20 ml, 50 ml or 100 ml depending upon the specific requirements of accessory equipment, for example, the headspace sampler, fitted with an inert septum seal and aluminium crimp tops. The septum seal shall neither absorb nor release volatile components, shall be gas tight during incubation and shall permit samples of the headspace gas to be withdrawn by syringe for subsequent analysis.

NOTE Elastomers lined with polytetrafluoroethylene (PTFE) are suitable materials for septum seals.

5.2 Crimping tool, for sealing the vials with the aluminium crimp tops.

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5.3 Seal removing tool.

5.4 Analytical balance, capable of weighing to the nearest 0,1 mg.

5.5 Template, for cutting samples. The dimensions of this template shall be matched to the vial volume used.

5.6 Scalpel, or sharp knife.

5.7 Syringes (e.g. 1 µl, 10 µl)

5.8 Gas chromatograph, having a flame ionization detector or equivalent for the solvents to be determined.

5.9 Gas chromatographic column, either packed or capillary, that will give good resolution of the solvents to be determined from any other components that might be injected with the specimen of the headspace.

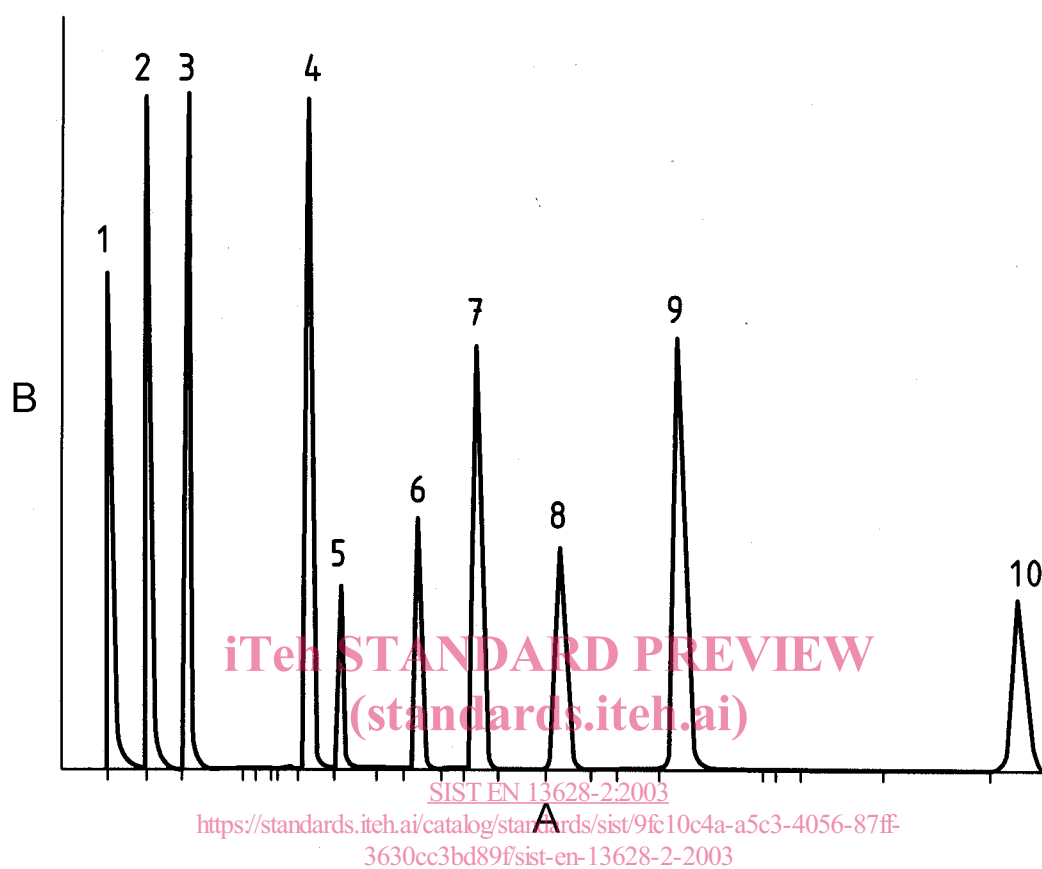
Examples for suitable columns and operation conditions are:

a) Packed column

length: 3 m;
internal diameter: 3,2 mm;
column filling: 80/120 mesh graphitised carbon, deactivated with polyethyleneglycol;
carrier gas: N₂, 20 ml/min;
injector temperature: 220 °C;
temperature programme: 80 °C; raised to 160 °C at 6 °C/min; raised to 225 °C at 1,5 °C/min; held for 16 min

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NOTE 1 A corresponding chromatogram obtained for a mixture of solvents is shown in Figure 1.



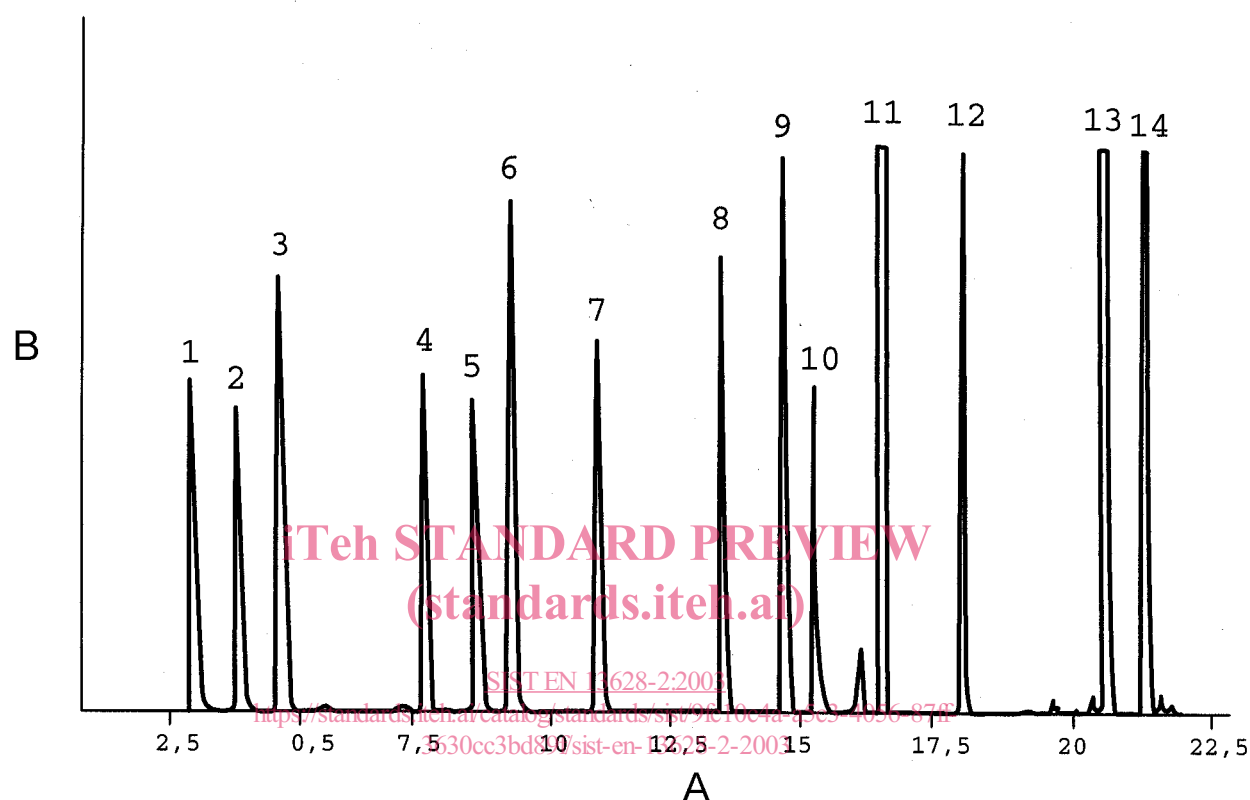
or

b) Capillary column: (fused silica):

length : 30 m;
 internal diameter : 0,32 mm;
 stationary phase : Poly(dimethylsiloxane) film thickness 3µm;
 carrier gas : He 1,7ml/min;
 carrier gas split ratio : 1:20;
 injector temperature : 230 °C;
 detector (flame ionisation) temperature : 280 °C;

temperature programme: 50 °C held for 5 min; raised to 100 °C at 5 °C/min; raised to 250 °C at 10 °C/min.

NOTE 2 A corresponding chromatogram is shown in Figure 2.



Key

- 1 Methanol
- 2 Ethanol
- 3 Isopropanol
- 4 Methyl ethyl ketone
- 5 Ethyl acetate
- 6 Isobutanol
- 7 Isopropyl acetate
- 8 n-propyl acetate
- 9 Methyl isobutyl ketone
- 10 Etoxypropanol
- 11 Toluene
- 12 n-butyl acetate
- 13 Xylene
- 14 Butyl cellosolve

- A Retention time (min)
- B Peak height

Figure 2 — Example of chromatogram obtained with a capillary column

NOTE Other apparatus which is more specific to the alternative methods is identified under the relevant clauses.