SLOVENSKI STANDARD
SIST EN 13628-1:2003

01-julij-2003

Embalaža in Prožni embalažni materiali in Določevanje ostanka topil s statičnim vzorčevalnikom iz plinske faze (headspace) za plinsko kromatografijo in 1. del: Absolutna metoda

Packaging - Flexible packaging material - Determination of residual solvents by static headspace gas chromatography - Part 1: Absolute methods

Verpackung - Flexible Packstoffe - Bestimmung der Restlösemittel durch statische Dampfraumanalyse mittels Gaschromatographie - Teil 1: Absolute Verfahren

Emballage - Matériaux d'emballages souples - Détermination des solvants résiduels par chromatographie en phase gazeuse et espace de tete statique - Partie 1: Méthodes absolues

Ta slovenski standard je istoveten z: **EN 13628-1:2002**

ICS:

55.040	Materiali in pripomoji ki za pakiranje	Packaging materials and accessories

SIST EN 13628-1:2003 **en**

EUROPEAN STANDARD

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English version

Packaging - Flexible packaging material - Determination of residual solvents by static headspace gas chromatography - Part 1: Absolute methods

Emballage - Matériaux d'emballages souples - Détermination des solvants résiduels par chromatographie en phase gazeuse et espace de tête statique - Partie 1: Méthodes absolues

Verpackung - Flexible Packstoffe - Bestimmung der Restlösemittel durch statische Dampfraumanalyse mittels Gaschromatographie - Teil 1: Absolute Verfahren

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

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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.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This document EN 13628-1:2002 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of an identical text or by endorsement, at the latest by April 2003, and conflicting national standards shall be withdrawn at the latest by April 2003.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*

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.

1 Scope

This part of this European Standard specifies methods for the quantitative determination of residual solvents in flexible packaging by static headspace chromatography where the chemical identities of the residual solvents to be determined are known before commencing the analysis. 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 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. Quantification is achieved by the multiple headspace extraction (MHE) procedure using external or internal standards;

b) manually or automatically by using a heated gastight syringe or a loop without pressurization of the heated vials. Quantification is achieved by the standard addition method.

NOTE A prerequisite for a quantitative determination of residual solvents by headspace gas chromatography is that a partition equilibrium for the solvent between the gas phase and the solid phase has been reached before an aliquot of the headspace is withdrawn and transferred into the gas chromatograph.

During the analysis, there may be interferences from possible products of thermal decomposition. Additional peaks due to these products shall 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 are 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.

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;

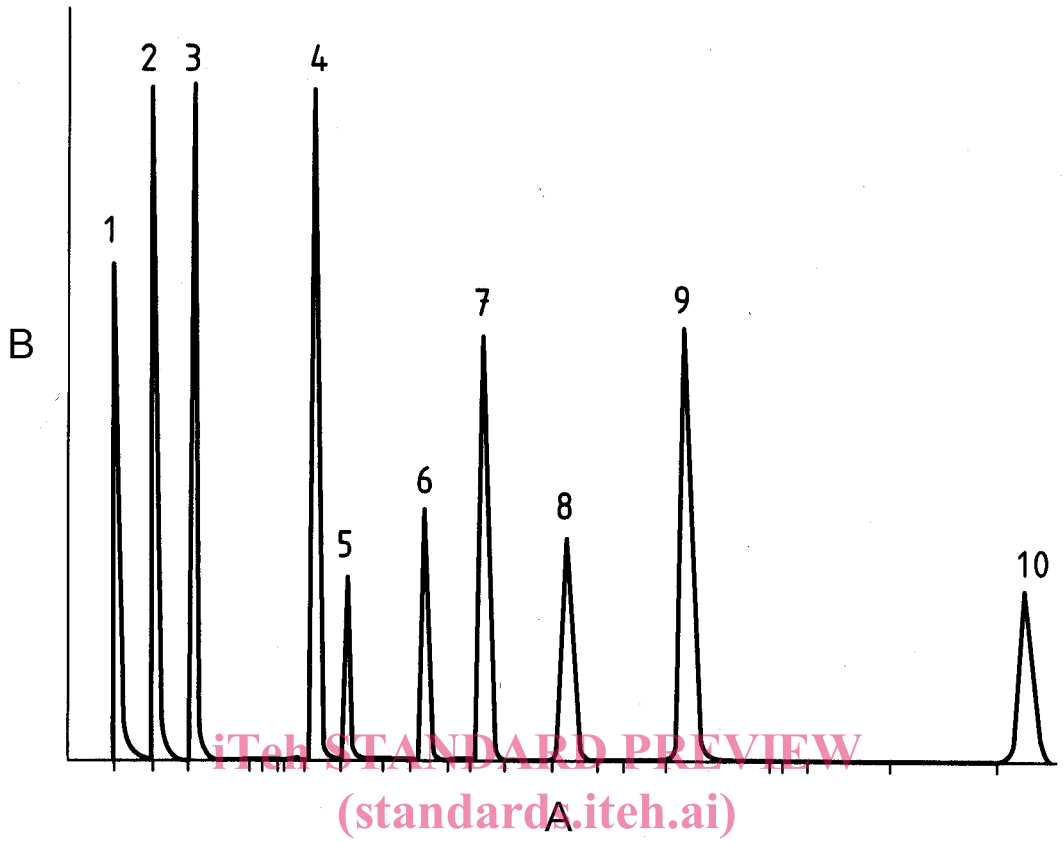
column filling: 80/120 mesh graphitised carbon, deactivated with polyethyleneglycol;

carrier gas: N_2, 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;

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



Key

1 4,23 methanol
2 6,67 ethanol
3 9,25 acetone
4 17, 06 ethyl acetate
5 19,1 butanol
6 23,34 trichloroethylene
7 28,4 isobutyl acetate
8 33,87 methyl isobutylketone
9 41,86 toluene
10 64.06 xylene

A Retention time (min)
B Peak height

Figure 1 — Example of chromatogram obtained with a packed column

or

b) Capillary column (fused silica):

length: 30 m;

internal diameter: 0,32 mm;

stationary phase: Poly(dimethylsiloxane), film thickness 3 µm;

carrier gas: He, 1,7 ml/min;

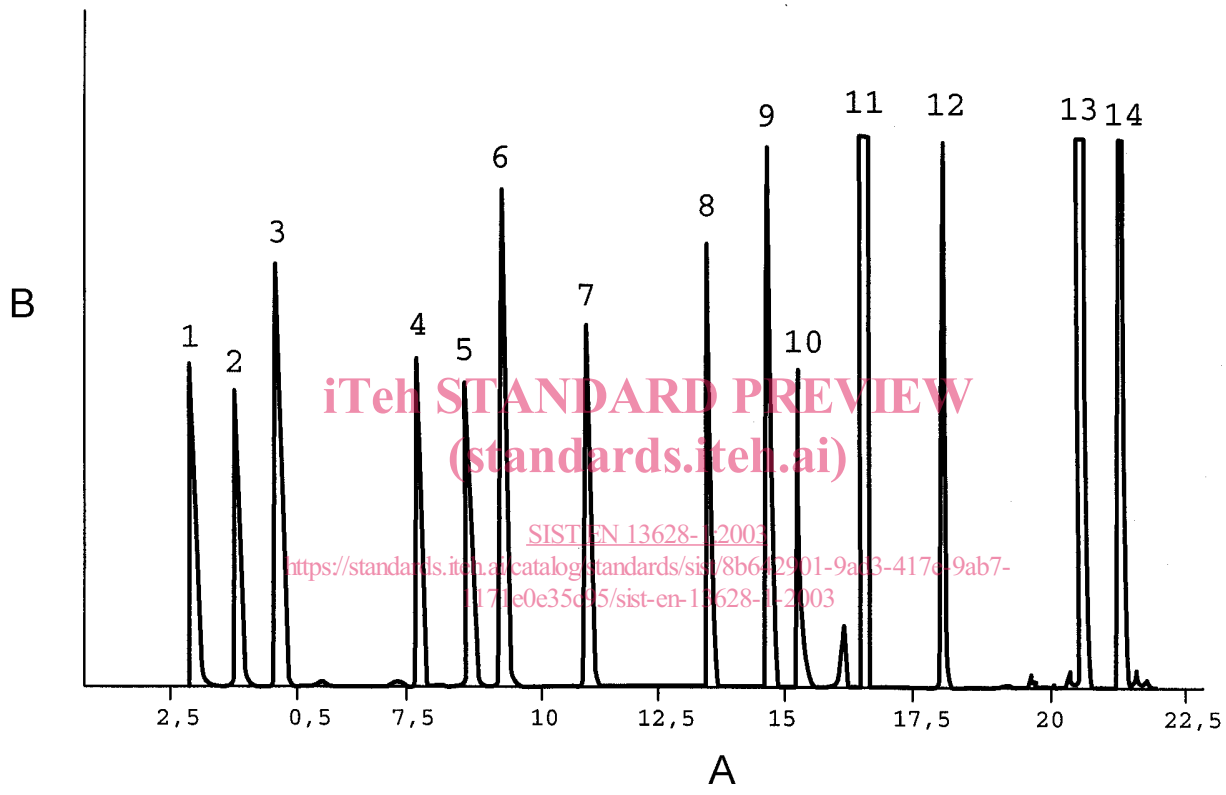
carrier gas split ratio: 1:20;

injector temperature: 230 °C;

detector (flame ionization) 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 for a mixture of solvents is shown in Figure 2.



Key

1 Methanol
2 Ethanol
3 Isopropanol
4 Methylethyl ketone
5 Ethyl acetate
6 Isobutanol
7 Isopropyl acetate
8 n-propyl acetate
9 Methylisobutyl ketone
10 Ethoxypropanol
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