SLOVENSKI STANDARD
SIST EN 14479:2004

01-september-2004

Embalaģa ï Proģni embalaģni materiali ï Določ evanje ostanka topil z dinamič nim vzorč evalnikom iz plinske faze (headspace) za plinsko kromatografijo - Absolutna metoda

Packaging - Flexible packaging material - Determination of residual solvents by dynamic headspace gas chromatography-Absolute method

Verpackung, Flexible Packstoffe - Bestimmung der Restlösemittel durch dynamische Dampfraumanalyse mittels Gaschromatographie - Absolutes Verfahren

Emballage - Matériaux d'emballage souples - Détermination des solvants résiduels par chromatographie en phase gazeuse et espace de tete dynamique - Méthode absolue

Ta slovenski standard je istoveten z: **EN 14479:2004**

ICS:

55.040	Materiali in pripomoč ki za pakiranje	Packaging materials and accessories

SIST EN 14479:2004 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14479

April 2004

ICS 55.040

English version

Packaging - Flexible packaging material - Determination of residual solvents by dynamic headspace gas chromatography-Absolute method

Emballage - Matériaux d'emballage souples - Détermination des solvants résiduels par chromatographie en phase gazeuse et espace de tête dynamique - Méthode absolue

Verpackung, Flexible Packstoffe - Bestimmung der Restlösemittel durch dynamische Dampfraumanalyse mittels Gaschromatographie - Absolutes Verfahren

This European Standard was approved by CEN on 2 February 2004.

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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 Central Secretariat has the same status as the official versions.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 14479:2004: E

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Foreword

This document (EN 14479:2004) 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of an identical text or by endorsement, at the latest by October 2004, and conflicting national standards shall be withdrawn at the latest by October 2004.

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

1 Scope

This standard describes methods for the quantitative determination of residual solvents in flexible packaging by dynamic 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 multiplayer 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 Principle

Dynamic extraction of the vapour phase of a sample is carried out using an inert gas for continuous stripping of the solvent. In this way thermodynamic equilibrium between the sample and its vapour phase is never reached.

The gas is fed in directly over the thermostatically controlled sample for a period of time sufficient to allow extraction of most of the solvent present.

Concentration methods using adsorbant solids or cryogenic liquids are required before the GC analysis because of the large volume of gas extracted. The procedure can thus be divided into two phases:

a) extraction and entrapment

b) desorption and transfer to the GC column.

3 Reagents

3.1 General

All reagents shall be of 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.

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

3.3 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.

4 Apparatus

4.1 Dynamic headspace device

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

4.3 Template for cutting samples.

4.4 Scalpel or sharp knife

4.5 Syringes (e.g. 1 µl, 10 µl).

4.6 Gas chromatograph, having a flame ionization detector or a mass-spectrometer or an FTIR spectrometer or another detector suitable for the solvents to be determined.

4.7 Gas chromatographic column, either packed or capillary, that will give good resolution of the solvents to be determined from any other components that might eventually be present in the headspace. Examples for suitable columns and operation conditions are:

a) Packed column:

length : 3m;

internal diameter : 3,2 mm;

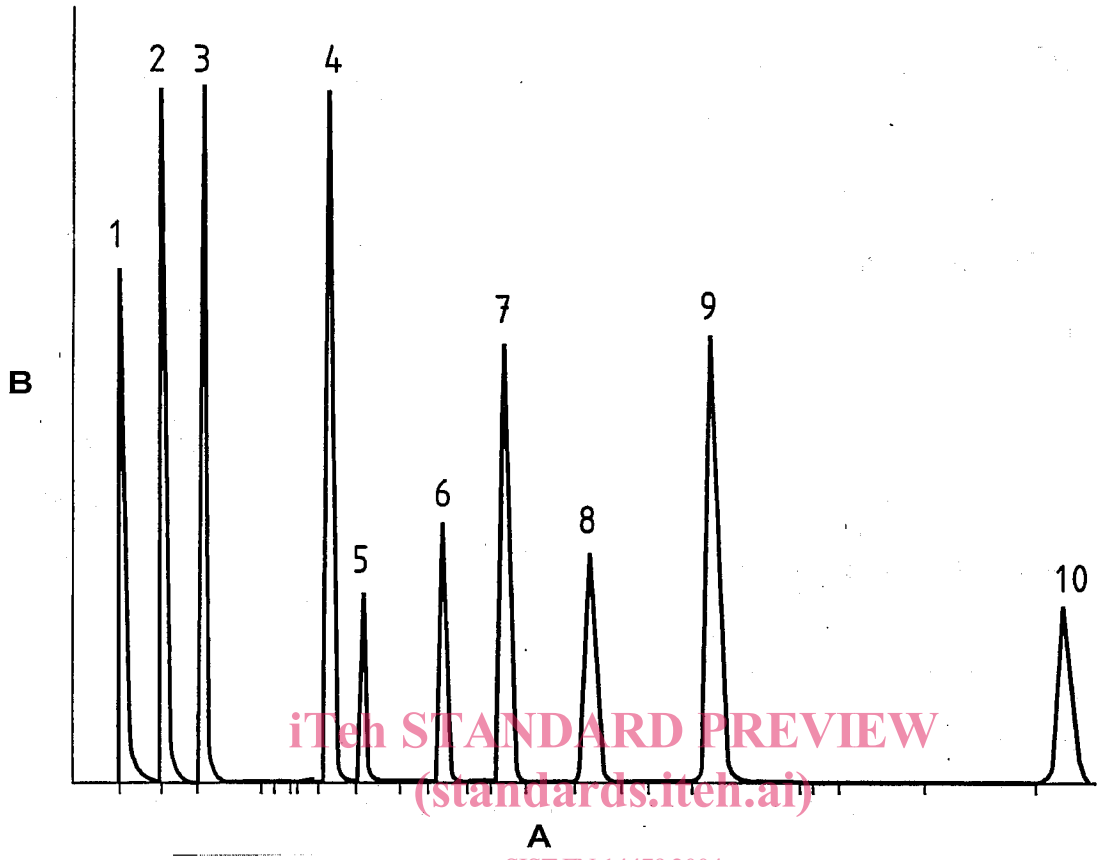
column filling : 80/120 mesh graphitised carbon, deactivated with polyethylene glycol;

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 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 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 A corresponding chromatogram for a mixture of solvents is shown in Figure 2.