



# SLOVENSKI STANDARD

## SIST EN 13130-6:2004

01-september-2004

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Materials and articles in contact with foodstuffs - Plastics substances subject to limitation  
- Part 6: Determination of vinylidene chloride in plastics

**iTeh STANDARD PREVIEW**

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen,  
die Beschränkungen unterliegen - Teil 6: Bestimmung von Vinylidenchlorid in  
Kunststoffen

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Matériaux et objets en contact avec les denrées alimentaires - Substances dans les  
matieres plastiques soumises a des limitations - Partie 6 : Détermination du chlorure de  
vinylidene dans les matieres plastiques

**Ta slovenski standard je istoveten z: EN 13130-6:2004**

### ICS:

67.250      Materiali in predmeti v stiku z      Materials and articles in  
živili      contact with foodstuffs

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN 13130-6**

May 2004

ICS 67.250

English version

**Materials and articles in contact with foodstuffs - Plastics  
substances subject to limitation - Part 6: Determination of  
vinylidene chloride in plastics**

Matériaux et objets en contact avec les denrées  
alimentaires - Substances dans les matières plastiques  
sujettes à des restrictions - Partie 6 : Détermination du  
chlorure de vinylidène dans les matières plastiques

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln  
- Substanzen in Kunststoffen, die Beschränkungen  
unterliegen - Teil 6: Bestimmung von Vinylidenchlorid in  
Kunststoffen

This European Standard was approved by CEN on 24 March 2004.

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of 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.



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COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

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## Foreword

This document (EN 13130-6:2004) has been prepared by Technical Committee CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.

This document was prepared by Subcommittee SC1 of TC 194 as one of a series of analytical test methods for plastics materials and articles in contact with foodstuffs.

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 November 2004, and conflicting national standards shall be withdrawn at the latest by November 2004.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This standard is intended to support Directives 2002/72/EC [1], 89/109/EEC [2], 82/711/EEC [3] and its amendments 93/8/EEC [4] and 97/48/EC [5], and 85/572/EEC [6].

At the time of preparation and publication of this part of EN 13130 the European Union legislation relating to plastics materials and articles intended to come into contact with foodstuffs is incomplete. Further Directives and amendments to existing Directives are expected which could change the legislative requirements which this standard supports. It is therefore strongly recommended that users of this standard refer to the latest relevant published Directive(s) before commencement of a test or tests described in this standard.

EN 13130-6 should be read in conjunction with EN 13130-1.

Further parts of EN 13130, under the general title *Materials and articles in contact with foodstuffs - Plastics substances subject to limitation*, have been prepared, and others are in preparation, concerned with the determination of specific migration from plastics materials into foodstuffs and food simulants and the determination of specific monomers and additives in plastics. The other parts of EN 13130 are as follows.

- Part 1: *Guide to test methods for the specific migration of substances from plastics to foods and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants*
- Part 2: *Determination of terephthalic acid in food simulants*
- Part 3: *Determination of acrylonitrile in food and food simulants*
- Part 4: *Determination of 1,3-butadiene in plastics*
- Part 5: *Determination of vinylidene chloride in food simulants*
- Part 7: *Determination of monoethylene glycol and diethylene glycol in food simulants*
- Part 8: *Determination of isocyanates in plastics*
- Part 9: *Determination of acetic acid, vinyl ester in food simulants*
- Part 10: *Determination of acrylamide in food simulants*
- Part 11: *Determination of 11-aminoundecanoic acid in food simulants*
- Part 12: *Determination of 1,3-benzenedimethanamine in food simulants*
- Part 13: *Determination of 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A) in food simulants*

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- Part 14: *Determination of 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indoline in food simulants*
- Part 15: *Determination of 1,3-butadiene in food simulants*
- Part 16: *Determination of caprolactam and caprolactam salt in food simulants*
- Part 17: *Determination of carbonyl chloride in plastics*
- Part 18: *Determination of 1,2-dihydroxybenzene, 1,3- dihydroxybenzene, 1,4- dihydroxybenzene, 4,4'-dihydroxybenzophenone and 4,4'dihydroxybiphenyl in food simulants*
- Part 19: *Determination of dimethylaminoethanol in food simulants*
- Part 20: *Determination of epichlorohydrin in plastics*
- Part 21: *Determination of ethylenediamine and hexamethylenediamine in food simulants*
- Part 22: *Determination of ethylene oxide and propylene oxide in plastics*
- Part 23: *Determination of formaldehyde and hexamethylenetetramine in food simulants*
- Part 24: *Determination of maleic acid and maleic anhydride in food simulants*
- Part 25: *Determination of 4-methyl-pentene in food simulants*
- Part 26: *Determination of 1-octene and tetrahydrofuran in food simulants*
- Part 27: *Determination of 2,4,6-triamino-1,3,5-triazine in food simulants*
- Part 28: *Determination of 1,1,1-trimethylpropane in food simulants*

Parts 1 to 8 are European Standards.

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Parts 9 to 28 are Technical Specifications, prepared within the Standards, Measurement and Testing project, MAT1-CT92-0006, "Development of Methods of Analysis for Monomers".

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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.

## Introduction

Vinylidene chloride (1,1-dichloroethylene),  $\text{H}_2\text{C}=\text{CCl}_2$ , is a monomer used in the manufacture of certain plastics films and coatings intended to come into contact with foodstuffs. During the manufacture of vinylidene chloride polymers and copolymers, residual vinylidene chloride monomer can remain in the polymer and can migrate into food coming into contact with the polymer.

The method described in this European Standard should be used in conjunction with part 1 of this standard, which describes the procedures required prior to the determination of vinylidene chloride.

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**EN 13130-6:2004 (E)****1 Scope**

This part of this European Standard specifies a method for the determination of vinylidene chloride (VdC) in plastics materials and articles.

The method is applicable to poly(vinylidene chloride) (PVdC) films, PVdC coated films, and laminates and coextruded materials containing PVdC. The level of vinylidene chloride determined is expressed as milligrams of vinylidene chloride per kilogram of polymer. The method is appropriate for the quantitative determination of vinylidene chloride at a level of 5 mg/kg in plastics materials and articles.

**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).

EN 13130-1:2004, *Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 1: Guide to test methods for the specific migration of substances from plastics to foods and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants.*

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**3 Principle**

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The level of vinylidene chloride in a polymer is determined by headspace gas chromatography with automated sample injection, using either electron capture detection (ECD) or flame ionization detection (FID). As it is not possible to obtain test material free of vinylidene chloride, the method of standard addition is employed. Quantification is achieved using 1-chloropropane (1-CP) as an internal standard.

Plastics samples are dissolved in N,N-dimethylacetamide (DMA) prior to headspace analysis. For samples not soluble in this solvent, e.g. laminates and co-extrusions, determination can be carried out by the 'hot jar' method in which the polymer alone is heated to release the vinylidene chloride.

If interferences are experienced with the internal standard then calibration is carried out by external standardization.

If automated headspace sampling cannot be performed then manual injection as described in annex A shall be applied.

Confirmation of vinylidene chloride levels is carried out either by combined gas chromatography/mass spectrometry (GC/MS) or by re-analysis on a second GC column of different polarity.

**4 Reagents**

**WARNING: All chemicals are hazardous to health to a greater or lesser extent. It is beyond the scope of this standard to give instructions for the safe handling of all chemicals, that meet, in full, the legal obligations in all countries in which this standard may be followed. Therefore, specific warnings are not given and users of this standard shall ensure that they meet all the necessary safety requirements in their own country.**

**4.1** Vinylidene chloride,  $\text{H}_2\text{C}=\text{CCl}_2$ , of purity greater than 99 % (w/w).



**4.2** 1-Chloropropane,  $\text{CH}_3(\text{CH}_2)_2\text{Cl}$ , containing no impurity > 1 % by area which will elute at the same GC retention time as vinylidene chloride.

**4.3** N,N-Dimethylacetamide  $\text{CH}_3\text{CON}(\text{CH}_3)_2$  free of any interferences (< 1 % area) with the vinylidene chloride and 1-chloropropane peaks.

**4.4** Standard solutions of vinylidene chloride in N,N-dimethylacetamide with defined concentrations in the range 25 mg/kg to 500 mg/kg, prepared as described in 4.4.1 and 4.4.2.

**4.4.1** Prepare concentrated standard vinylidene chloride solutions at approximately 2,5 g/kg and 1,2 g/kg as follows:

- a) To a 50 ml flask or vial tared with cap, add approximately 50 ml (46,5 g) N,N-dimethylacetamide (4.3), close and weigh to an accuracy of 0,1 mg. Add to the N,N-dimethylacetamide a quantity of approximately 0,1 ml (0,125 g) vinylidene chloride (4.1) and shake the closed flask. Determine the exact mass of vinylidene chloride added, in grams per kilogram, by re-weighing to an accuracy of 0,1 mg.
- b) Repeat item a) using 0,05 ml (0,06 g) vinylidene chloride.
- c) Repeat items a) and b) to provide a second set of concentrated standard solutions.

**4.4.2** Prepare dilute standard vinylidene chloride solutions as follows;

- a) To two 20 ml flasks or vials tared with cap, add 18 ml and 16 ml N,N-dimethylacetamide and weigh to an accuracy of 0,1 mg. Add 2 ml of the 2,5 g/kg concentrated standard solution to the first flask or vial and 4 ml to the second. Cap, re-weigh to an accuracy of 0,1 mg and shake thoroughly.
- b) Repeat item a) using 19,6 ml and 18 ml dimethylacetamide and 0,4 ml and 2 ml of the 1,25 g/kg standard. The dilute standard solutions shall be stored in suitable glass septum-capped vials with minimal headspace volume.
- c) Repeat items a) and b) using the second solutions prepared in 4.4.1 to provide a second set of four dilute standard vinylidene chloride solutions.

**NOTE** The standard solutions with known vinylidene chloride concentrations of approximately 25 mg/kg, 125 mg/kg, 250 mg/kg and 500 mg/kg, respectively can be stored at - 20 °C for up to four weeks, protected from light.

**4.5** Solution of 1-chloropropane (4.2) in N,N-dimethylacetamide at approximately 0,4 g/kg

**4.5.1** Prepare a concentrated standard 1-chloropropane solution at approximately 2,0 g/kg as follows:

To a 50 ml flask or vial tared with cap, add 50 ml of dimethylacetamide. Close and weigh to an accuracy of 0,1 mg. Add to the dimethylacetamide, approximately 0,1 ml (0,090 g) of 1-chloropropane and shake the closed flask or vial. Determine the exact mass of 1-chloropropane added by re-weighing to an accuracy of 0,1 mg.

**4.5.2** Prepare a dilute standard 1-chloropropane solution at approximately 0,4 g/kg as follows:

To a 20 ml flask or vial tared with cap, add 16 ml N,N-dimethylacetamide and weigh to an accuracy of 0,1 mg. Add 4 ml of 1-chloropropane solution, as prepared in 4.5.1. Close, re-weigh to an accuracy of 0,1 mg and shake thoroughly to mix.

## 5 Apparatus

**NOTE** An instrument or item of apparatus is listed only where it is special, or made to a particular specification, usual laboratory equipment being assumed to be available.

**5.1** Gas-chromatograph, equipped with either an electron capture detector or a flame ionization detector and fitted with an automated headspace sampler.