SIST

SLOVENSKI STANDARD
SIST ENV 13130-5:2000

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Materiali in predmeti v stiku z ġivili - Snovi v plastiį nih masah, katerih koncentracija je omejena - 5. del: Doloį evanje vinilidenklorida v modelnih raztopinah ġivil

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 5: Determination of vinylidene chloride in food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Grenzwerten unterliegen - Teil 5: Bestimmung von Vinylidenchlorid in Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Matieres plastiques soumises a des limitations - Partie 5: Détermination du chlorure de vinylidene dans les simulants

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Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 5: Determination of vinylidene chloride in food simulants

Matériaux et objets en contact avec les denrées alimentaires - Matières plastiques soumises à des limitations - Partie 5: Détermination du chlorure de vinylidène dans les simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Grenzwerten unterliegen - Teil 5: Bestimmung von Vinylidenchlorid in Prüflebensmitteln

This European Prestandard (ENV) was approved by CEN on 18 February 1999 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

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

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Contents

Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.

This Part of this European Prestandard has been prepared by a Subcommittee (SC1) of TC194 'Utensils in contact with food' as one of a series of analytical test methods for plastics materials and articles in contact with foodstuffs.

Further Parts of this prestandard 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 substances in plastics.

Their titles are as follows:

ENV 13130-1	Guide to the test methods for the specific migration of substances from plastics into food and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants
ENV 13130-2	Determination of terephthalic acid in food simulants
ENV 13130-3	Determination of acrylonitrile in food and food simulants
ENV 13130-4	Determination of 1,3-butadiene in plastics
ENV 13130-6	Determination of vinylidene chloride in plastics
ENV 13130-7	Determination of monoethylene glycol and diethylene glycol in food simulants
ENV 13130-8	Determination of isocyanates in plastics

Method development for other monomers subject to limitation is being coordinated by the Measurement and Testing Programme of DG XII (formerly BCR).

Annexes A and B to this standard are normative where applicable.

This Part of this prestandard should be read in conjunction with Part 1 of this prestandard.

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

WARNING: Vinylidene chloride is a highly flammable, photosensitive, hazardous substance which is volatile at room temperature. The handling and preparation of solutions of vinylidene chloride should be carried out in a fume cupboard and skin and eye contact with vinylidene chloride solutions or the inhalation of vinylidene chloride vapour should be avoided. Vinylidene chloride should be protected from prolonged exposure to light and stored at 5 °C.

0 Introduction

Vinylidene chloride (VdC) (1,1-dichloroethylene), $H_2C=CCl_2$, 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 Part of the prestandard is to be used in conjunction with Part 1 of this prestandard which describes the procedures required prior to the determination of vinylidene chloride.

1 Scope

This Part of this European Prestandard specifies a method for the determination of vinylidene chloride monomer in food simulants. The method is applicable to aqueous food simulants, to the fatty food simulant olive oil and to the approved alternatives, sunflower oil and a mixture of synthetic triglycerides. The level of vinylidene chloride determined is expressed as milligrams of vinylidene chloride per kilogram of food simulant. The method is appropriate for the quantitative determination of vinylidene chloride at a level of 0,05 mg/kg.

2 Normative references

This European Prestandard incorporates by dated and 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 and revisions of any of these publications apply to this European Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ENV 13130-1 Guide to the test methods for the specific migration of substances from plastics into food and food simulants and the determination of substances in plastics and the selection of conditions of exposure to food simulants

3 Principle

The level of vinylidene chloride in a food simulant is determined by headspace gas chromatography with automated sample injection and using either electron capture or flame ionization detection. Quantification is achieved using 1-chloropropane (1-CP) as internal standard with calibration against blank samples of simulant fortified with vinylidene chloride. If blank samples of simulant cannot be obtained then the method of standard addition described in annex A is employed.

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

If automated headspace sampling cannot be performed, manual injection as described in annex B can 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

4.1 Analyte and internal standard

4.1.1 Analyte

Vinylidene chloride (VdC), $H_2C=CCl_2$, of purity greater than 99 %.

4.1.2 Internal standard

1-Chloropropane (1-CP), $CH_3(CH_2)_2Cl$, of purity greater than 98 %.

4.2 Chemicals

N,N-dimethylacetamide (DMA), $CH_3CON(CH_3)_2$, of purity greater than 99 %.

4.3 Solutions of standards and internal standard

4.3.1 Prepare stock solutions of vinylidene chloride as follows:

a) Stock solution of vinylidene chloride with a defined concentration of approximately 2,5 g/kg. To a 20 ml flask or vial with cap, add 20 ml N,N-dimethylacetamide (4.2), close and weigh to an accuracy of 0,1 mg. Add to the N,N-dimethylacetamide a quantity of approximately 40 µl (0,048g) vinylidene chloride (4.1.1) and shake the closed flask. Weigh to an accuracy of 0,1 mg. Determine the exact mass of vinylidene chloride added in grams per kilogram.

b) Repeat item a) to provide a second stock solution.

4.3.2 Intermediate solutions of vinylidene chloride

Intermediate solutions of vinylidene chloride in N,N-dimethylacetamide with defined concentrations in the range 5 mg/kg to 50 mg/kg are prepared as follows:

a) To five 10 ml flasks or vials tared with cap, add 10 ml of N,N-dimethylacetamide and weigh to an accuracy of 0,1 mg. Add respectively 20 µl, 50 µl, 100 µl, 150 µl and 200 µl of the 2.5 g/kg stock solution of vinylidene chloride (4.3.1a). Cap, re-weigh to an accuracy of 0,1 mg and shake thoroughly.

b) Repeat item a) using the second stock solution prepared in 4.3.1b to provide a second set of five intermediate solutions of vinylidene chloride.

NOTE: The stock and intermediate solutions of vinylidene chloride, may be stored at - 20 °C for up to four weeks, protected from light. Solutions should be stored in suitable glass, septum-capped vials, with minimal headspace volume.

4.3.3 Internal standard stock solution

Prepare a stock solution of 1-chloropropane with a defined concentration of approximately 20 g/kg as follows:

To a 10 ml flask or vial tared with cap, add 10 ml N,N-dimethylacetamide. Close and weigh to an accuracy of 0,1 mg. Add to the N,N-dimethylacetamide, approximately 0,20 ml 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.

4.3.4 Intermediate internal standard solution

Prepare an intermediate solution of 1-chloropropane with a defined concentration of approximately 200 mg/kg as follows:

To a 10 ml flask or vial tared with cap, add 10 ml N,N-dimethylacetamide and weigh to an accuracy of 0,1 mg. Add 100 µl of 1-chloropropane stock solution (4.3.3). 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 an electron capture detector (ECD) or flame ionization detector (FID) and fitted with an automated headspace sampler.

5.2 Gas-chromatographic column, capable of the separation of N,N-dimethylacetamide from vinylidene chloride and 1-chloropropane such that the peaks of vinylidene chloride and 1-chloropropane do not overlap by more than 1 % peak area with each other and with other compounds.

NOTE 1: The following are examples of GC columns known to be suitable for vinylidene chloride analysis:

a) 50 m x 0,32 mm internal diameter 100 % dimethyl siloxane fused silica capillary column with 1,2 µm film thickness;

b) 50 m x 0,32 mm internal diameter 85 % dimethyl-, 7 % cyanopropyl-, 7 % phenyl-, 1 % vinyl- siloxane fused silica capillary column with 1,2 µm film thickness;

c) 50 m x 0,53 mm internal diameter polyethylene glycol capillary column with 2,0 µm film thickness.

NOTE 2: Depending on the type of gas chromatograph, automated headspace sampler and GC column, the appropriate operating conditions should be established. For guidance, the following range of parameters have been found suitable for column a):

Injector temperature:	120 °C
Oven programme:	70 °C (isothermal)
Detector (ECD):	280 °C
Carrier gas and flow rate:	helium, 1 ml/min
Injection mode:	split (ratio = 20:1).

Parameters found suitable for column c) are as follows:

Injector:	200 °C
Oven programme:	75 °C for 10 min, 20 °C/min to 200 °C, hold 5 min.
Detector (FID):	250 °C
Carrier gas and flow rate:	helium, 5 ml/min
Injection mode:	split (ratio= 4:1).

Electron capture detector optimization. If a capillary column is employed, ensure the detector has a make-up gas. Nitrogen or argon/methane (95 % : 5 %) is generally recommended and the flow should be optimized as recommended in the GC manufacturer's instructions. Gases should be purified of moisture, oxygen and electron capturing species by passage through suitable filters, to the level recommended by the detector manufacturer.

5.3 Glass sample vials, of size suitable for the particular autosampler employed, with polytetrafluoroethylen faced silicone rubber septa and crimp-closures.

5.4 Microsyringes, of 50 µl, 100 µl and 200 µl capacity.

6 Samples

6.1 Laboratory samples

The laboratory samples of food simulant to be analysed are obtained as described in Part 1 of this prestandard. Samples shall be kept refrigerated at 5 °C with the exclusion of light, in glass, gas-tight containers with minimal headspace volume. Vinylidene chloride-free samples of simulant are also required for calibration purposes. The simulant shall be the same as that used for the exposure.

NOTE: Since the determination of vinylidene chloride in food simulant is performed close to the limit of detection of the method, extreme care should be taken with respect to possible adventitious contamination during preparation of the test samples and standards and to loss by volatilization. The following precautions are advisable.

a) To avoid cross-contamination by volatilization, carry out the migration test procedure and the preparation of the simulant test samples in a remote area to that used for handling vinylidene chloride and 1-chloropropane solutions.

b) To avoid loss of standard solutions to the septum when making additions and subsequent loss during headspace equilibration, it is preferable to add solutions directly to the food simulant contained within the vial, rather than injecting them through the septum. Cool the simulant to 5 °C before addition.

c) To reduce volatilization, add vinylidene chloride and internal standard to simulant samples as quickly as possible, injecting solutions down the side of vials and sealing vials immediately after addition.

6.2 Preparation of test samples

Conduct all weighings to an accuracy of 0,1 mg.

6.2.1 Aqueous food simulants

To a sample vial (5.3), add 10 ml of food simulant and weigh. Then, weighing after each addition, add 20 µl N,N-dimethylacetamide and 20 µl of intermediate 1-chloropropane solution (4.3.4) and reweigh. Seal vial. Repeat this procedure to provide a duplicate test sample.

6.2.2 Fatty food simulant

Follow the procedure outlined in 6.2.1 but use 10 g ± 0,01 g olive oil.

6.3 Preparation of blank samples

To a sample vial, add 10 ml or 10 g, as appropriate, of food simulant and 20 µl of N,N-dimethylacetamide. Seal vial. Prepare a duplicate blank sample.

6.4 Preparation of calibration samples

NOTE: If vinylidene chloride-free food simulant is not available, the method of standard addition described in annex A shall be employed.

To a sample vial (5.3), add 10 ml of aqueous food simulant or 10 g ± 0,01 g of fatty food simulant and weigh. Then, weighing after each addition, add 20 µl of intermediate 1-chloropropane solution and 20 µl