SLOVENSKI STANDARD
SIST ENV 13130-3:2000

01-april-2000

Materiali in predmeti v stiku z ģivili - Snovi v plastiļ nih masah, katerih koncentracija je omejena - 3. del: Doloļ evanje akrilonitrila v ģivilih in modelnih raztopinah ģivil

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 3: Determination of acrylonitrile in food and food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Grenzwerten unterliegen - Teil 3: Bestimmung von Acrylnitril in Lebensmitteln und Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Matieres plastiques soumises a des limitations - Partie 3: Détermination de l'acrylonitile dans les denrées alimentaires et les simulants

Ta slovenski standard je istoveten z: **ENV 13130-3:1999**

ICS:

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

SIST ENV 13130-3:2000 **en**

EUROPEAN PRESTANDARD
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ENV 13130-3

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ICS 67.250

English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 3: Determination of acrylonitrile in food and food simulants

Matériaux et objets en contact avec les denrées alimentaires - Matières plastiques soumises à des limitations - Partie 3: Détermination de l'acrylonitile dans les denrées alimentaires et les simulants

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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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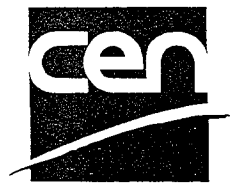

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

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 13130-4	Determination of 1,3-butadiene in plastics
ENV 13130-5	Determination of vinylidene chloride in food simulants
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, B and C to this prestandard 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.

0 Introduction

Acrylonitrile, CH_2=CH-CN, is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. During the manufacture of acrylonitrile copolymers, residual acrylonitrile 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 acrylonitrile.

The method has been validated by collaborative trial using fruit juice, wine and sunflower oil.

1 Scope

This Part of this European Prestandard specifies a method for the determination of acrylonitrile monomer in foods and 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, as well as to liquid and solid foodstuffs such as beverages and soft margarine. The level of acrylonitrile monomer determined is expressed as milligrammes of acrylonitrile per kilogram of food or food simulant. The method is appropriate for the quantitative determination of acrylonitrile at a minimum level of 0,02 mg/kg in food or food simulants.

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 acrylonitrile (AN) in a food, or a food simulant, is determined by headspace gas chromatography with automated sample injection, using nitrogen specific detection. Quantification is achieved using propionitrile (PN) as an internal standard with calibration against blank samples fortified with acrylonitrile. If blank samples 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 as described in annex B.

If automated headspace sampling cannot be performed, manual injection as described in annex C may be applied.

Confirmation of acrylonitrile 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 Acrylonitrile, CH_2=CH-CN, purity greater than 99% (w/w).

4.2 Propionitrile, CH_3-CH_2-CN, containing no impurity at > 1 % by area which will elute at the same GC retention time as acrylonitrile.

4.3 Propylene carbonate, CH_3-CH-OCOO-CH_2, boiling point 240 °C to 243 °C at normal pressure, free of any interferences (< 1 % area) with the acrylonitrile and propionitrile peaks.

4.4 Nitrogen, purified to 99,9999%.

4.5 Standard solutions of acrylonitrile in propylene carbonate with defined concentrations in the range 5 µg/ml to 25 µg/ml, prepared as described in 4.5.1 and 4.5.2.

4.5.1 Prepare concentrated standard acrylonitrile solutions at approximately 12,5 mg/ml as follows:

a) Fill a 100 ml volumetric flask with 50 ml propylene carbonate (4.3), close and weigh to an accuracy of 0,2 mg. Add to the propylene carbonate a quantity of approximately 1,5 ml (1,25 g) acrylonitrile (4.1) and shake the closed flask. Determine the exact mass of acrylonitrile added by re-weighing to an accuracy of 0,2 mg. Fill the flask to the 100 ml mark.

b) Repeat item a) to provide a second concentrated standard solution.

4.5.2 Prepare dilute standard acrylonitrile solutions as follows:

a) With an accuracy of 0,1 ml throughout, dilute one of the solutions prepared in 4.5.1 by a factor of 100 in two steps, taking for each step 10 ml acrylonitrile solution and 90 ml propylene carbonate, to give an intermediate standard solution of approximately 125 µg acrylonitrile per millilitre. Place 48 ml or 45 ml or 40 ml propylene carbonate into three 55 ml glass vials and add 2 ml or 5 ml or 10 ml of the intermediate standard solution, respectively. Close the vials with a polytetrafluoroethylene (PTFE) seal and cap and shake thoroughly.

b) Repeat item a) using the second solution prepared in 4.5.1 to provide a second set of three dilute standard acrylonitrile solutions.

NOTE: The standard solutions with known acrylonitrile concentrations of approximately 5 µg/ml, 12.5 µg/ml and 25 µg/ml, respectively, may be stored at 4 °C for up to four weeks.

4.6 Dilute standard propionitrile solution in propylene carbonate, with a known concentration of approximately 25 µg/ml of propionitrile (4.2) prepared by following an analogous procedure to that described in 4.5.

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 a nitrogen specific detector and fitted with an automatic headspace sampler.

5.2 Gas-chromatographic column, capable of the separation of propylene carbonate from acrylonitrile and propionitrile such that the peaks of acrylonitrile and propionitrile do not overlap by more than 1 % peak area with other compounds.

NOTE: The following are examples of GC columns known to be suitable for acrylonitrile analysis:

a) 2 m x 3 mm internal diameter stainless steel column packed with 15 % polyethylene glycol 1500 on 60 mesh to 100 mesh diatomite support;

b) 1.8 m x 2 mm internal diameter stainless steel column packed with 0,2 % polyethyleneglycol 1500 on 80 mesh to 100 mesh graphitized carbon black USP (S7) solid support;

c) 3 m x 2 mm internal diameter glass column packed with 20 % polyethylene glycol 20 on 60 mesh to 80 mesh flux-calcined diatomite support;

d) 25 m x 0,32 mm internal diameter, fused silica capillary column with 1.2 μm film thickness of 100 % dimethylpolysiloxane;

e) 12 m x 0,20 mm internal diameter, fused silica capillary column with 0,33 μm film thickness of free fatty acid phase (modified polyethylene glycol).

5.3 Sample vials, 25 ml, or of another size suitable for the particular autosampler employed, with butyl rubber septa and crimp-closures.

NOTE: The butyl rubber septa should not give rise to acrylonitrile or interference peaks and in some circumstances PTFE-faced septa are preferred.

5.4 Microsyringes, of 50 μl capacity and syringes, of 5 ml capacity.

6 Samples

6.1 Laboratory samples

The laboratory samples of food, or food simulant, to be analysed are obtained as described in Part 1 of this prestandard. Acrylonitrile-free samples of the same type as those to be analysed are also required for use for calibration purposes. Keep the samples refrigerated at 4 °C in closed containers with the exclusion of light.

NOTE: Acrylonitrile losses are unlikely during sampling, losses during transport and short-term storage for up to 4 weeks are unlikely.

6.2 Test sample preparation

NOTE: Since the determination of acrylonitrile in food or food simulant is performed close to the detection limit of the method, extreme care should be taken with respect to possible adventitious contamination during preparation of the test samples.

The following precautions are advisable:

a) purge the empty sample vials (5.3) with purified nitrogen before filling with food or food simulant;

b) to avoid cross-contamination by volatilization, carry out the migration test procedure and the preparation of the food or food simulant subsamples in a different laboratory to that used for handling acrylonitrile and propionitrile solutions;

c) to avoid loss of standard solutions to the septum when making additions, it is preferable, particularly with PTFE-faced septa, to add these directly to the food, or food simulant, contained within the vial, rather than injecting them through the septum.

6.2.1 Preparation of test sample solutions.

For liquid foods, place 5,0 ml ± 0,1 ml of the food or food simulant, in a sample vial (5.3) using a 5 ml syringe (5.4). For solid foods, such as soft margarine, weigh 5,0 g ± 0,1 g of food into the sample vial. Add 20 µl propylene carbonate (4.3) and 20 µl propionitrile standard solution (4.6) to the food, or food simulant, using the 50 µl syringe (5.4) and close the vial with septum and cap.

6.2.2 Preparation of food, or food simulant calibration samples.

NOTE: If the food or food simulant is not available free of acrylonitrile, use the method of standard addition described in annex A.

Follow the procedure described in 6.2.1 adding 20 µl of one of the dilute standard acrylonitrile solutions (4.5) in place of the propylene carbonate.

6.2.3 Preparation of blank samples

Follow the procedure described in 6.2.1 employing acrylonitrile-free food or food simulant, adding further propylene carbonate (20 µl) in place of the propionitrile.

7 Procedure

7.1 GC preparation

7.1.1 GC parameters

Depending on the type of gas chromatograph and column used for the determination, establish the appropriate GC parameters.

NOTE: The range of parameters which may be employed for packed columns are as follows:

Temperature:

Injector	140 °C to 200 °C
Column	80 °C to 90 °C (isothermal)
Detector	140 °C to 200 °C

Carrier gas and flow rate:

Helium or nitrogen	20 ml/min to 40 ml/min.

7.1.2 Nitrogen specific detector optimization

Optimize the air and hydrogen flow rates according to the manufacturer's instructions.

NOTE: As the influence of the carrier gas flow rate (see 7.1.1) on the detector sensitivity is low, hydrogen and air flow rates may in most cases be left unchanged after installation of a new rubidium bead. Any necessary change of detector sensitivity may be achieved by adjustment of detector voltage. The rubidium bead should be renewed if an acrylonitrile concentration of 20 µg/l in the sample solution yields a signal/noise ratio smaller than 3 and if the fault does not lie elsewhere.

7.1.3 Calibration

Each sample has to be determined at least in duplicate.

With the aid of the three dilute standard acrylonitrile solutions, establish a calibration curve based on fortification of acrylonitrile-free food or food simulant. For this calibration use aliquots of the same type of food or food simulant, as that to be analysed.