SLOVENSKI STANDARD
SIST ENV 13130-2:2000

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Materiali in predmeti v stiku z ģivili - Snovi v plastiļ nih masah, katerih koncentracija je omejena - 2. del: Doloļ evanje tereftalne kisline v modelnih raztopinah ģivil

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 2: Determination of terephthalic acid in food simulants

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Grenzwerten unterliegen - Teil 2: Bestimmung von Terephthalsäure in Prüflebensmitteln

Matériaux et objets en contact avec les denrées alimentaires - Matieres plastiques soumises a des limitations - Partie 2: Détermination de l'acide téréphtalique dans les simulants

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English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 2: Determination of terephthalic acid in food simulants

Matériaux et objets en contact avec les denrées alimentaires - Matières plastiques soumises à des limitations - Partie 2: Détermination de l'acide téréphtalique dans 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
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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 13130-3: Determination of acrylonitrile in food and 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).

Annex A to this prestandard is 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

Terephthalic acid (TA) is a comonomer used in the manufacture of polyester plastics. Residues of terephthalic acid can be present in the plastics after processing to form materials and articles intended to come into contact with foodstuffs. When these plastics are in contact with foodstuffs, the residual terephthalic acid monomer can migrate into the foodstuff. European Commission Directive 90/128/EEC lists a specific migration limit of 7,5 mg/kg of terephthalic acid in foods or food simulants.

1 Scope

This Part of this European Prestandard specifies methods for the determination of the monomer terephthalic acid in food simulants; distilled water, 3 % w/v acetic acid aqueous solution, 15 % v/v ethanol aqueous solution and olive oil or its approved substitutes, sunflower oil or a mixture of synthetic triglycerides. The methods are capable of determining terephthalic acid in the food simulants at the level of the specific migration limit in Commission Directive 90/128/EEC.

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.

ISO 385-1	Laboratory glassware - Burettes - Part 1 - General requirements
ISO 385-2	Laboratory glassware - Burettes - Part 2 - Burettes for which no waiting time is specified
ISO 385-3	Laboratory glassware - Burettes - Part 3 - Burettes for which a waiting time of 30s is specified
ISO 648:1977	Laboratory glassware - One mark pipettes
ISO 1042:1983	Laboratory glassware - One neck volumetric flasks
ISO 4788:1980	Laboratory glassware - Graduated measuring cylinders
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 aqueous simulant test samples are directly analysed by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection. The olive oil test samples are extracted with dilute sodium hydrogen carbonate and the resultant aqueous solution is acidified and analysed by HPLC.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water of equivalent purity.

4.1 Methanol (HPLC grade).

4.2 Sodium acetate, trihydrate.

4.3 Orthophthalic acid.

4.4 Terephthalic acid.

4.5 Orthophosphoric acid 85 % w/v.

4.6 Water (HPLC grade).

4.7 Sodium hydrogen carbonate solution 0,1 % w/v.

4.8 Acetic acid, 50 % v/v in water.

4.9 Propan-2-ol.

4.10 Heptane.

4.11 pH 3,6 buffer solution.

Dissolve 25,0 g of sodium acetate, trihydrate in 350 ml of water, add 5,0 ml ± 0,1 ml of orthophosphoric acid and adjust to pH 3,6 ± pH 0,2 with glacial acetic acid (approximately 50 ml). Make up to 500 ml with water.

4.12 Terephthalic acid standard stock solution.

Weigh accurately about 0,05 g terephthalic acid and, by continuous stirring, dissolve in about 90 ml of methanol. Make up to 100 ml with methanol in a volumetric flask. Prepare a second terephthalic acid standard stock solution for validation purposes, see 7.3.

NOTE 1: The solution can be warmed to 50 °C to facilitate dissolution of the terephthalic acid, which will take at least 1 h.

4.13 Orthophthalic acid internal standard stock solution.

Weigh about 0,1 g of orthophthalic acid and dissolve in propan-2-ol. Make up to 100 ml with propan-2-ol in a volumetric flask.

4.14 Mobile phase for high performance liquid chromatography.

Using a measuring cylinder add 150 ml of methanol to 150 ml of pH 3,6 buffer and dilute to 1 l with water.

NOTE 2: Degassing the mobile phase can be necessary with some HPLC equipment.

5 Apparatus

5.1 Analytical balance capable of weighing accurately to 0,1 mg.

5.2 pH meter with an accuracy of ± pH 0,1.

5.3 Volumetric flasks, of 25 ml, 50 ml and 100 ml capacity, conforming to the minimum requirements of ISO 1042.

5.4 Measuring cylinders, of 5 ml, 25 ml, 50 ml, 100 ml and 250ml capacity, conforming to the minimum requirements of ISO 4788.

5.5 Pipettes, of 5 ml and 50 ml capacity, conforming to the minimum requirements of ISO 648.

5.6 Graduated pipette, of 2 ml capacity, conforming to the minimum requirements of

ISO 835.

5.7 Separating funnels, of 250 ml capacity.

5.8 Glass syringe, of 50 ml capacity.

5.9 Octadecylsilyl silica (ODS) 400 mg, C18 reverse phase disposable cartridges.

5.10 HPLC 0,2 µm membrane filters.

5.11 Burette, of 25 ml capacity, conforming to the minimum requirements of ISO 385.

5.12 High performance liquid chromatograph with a 10 µl injection loop, and a variable wavelength UV detector, set to 242 nm, connected to a strip chart recorder or integrator.

The column shall be capable of fully resolving terephthalic acid from orthophthalic acid, isophthalic acid and peaks arising from injection media.

NOTE: The following column has been found to be suitable: ODS 5 µm 250 mm x 4,6 mm column, with a pre-column. With a flow rate of 1,5 ml/min, orthophthalic acid and terephthalic acid have retention times of 4,7 min and 6,9 min respectively.

With other reverse phase columns, adjustments can be made to the methanol concentration of the mobile phase to give acceptable retention times.

6 Samples

The food simulant samples for testing shall have been prepared as described in Part 1 of this prestandard. Blank samples of the food simulants are also required. For each simulant, 50 ml samples are required for each test.

7 Procedure

7.1 Preparation of standards

7.1.1 Intermediate standards

Prepare freshly on the day of use. Into five 25 ml volumetric flasks add, by burette, 1,0 ml, 2,0 ml, 5,0 ml, 10,0 ml, and 20,0 ml of the terephthalic acid stock solution (4.12). Into each add, by pipette, 5 ml of the orthophthalic acid internal standard stock solution (4.13) and dilute to the mark with methanol. These standards contain 20 mg/l, 40 mg/l, 100 mg/l, 200 mg/l, and 400 mg/l terephthalic acid.

7.1.2 Internal standard

Pipette 5 ml of the orthophthalic acid internal standard stock solution (4.13) into a 25 ml volumetric flask and dilute to the mark with methanol.

7.1.3 Working standards for aqueous simulants

Pipette 2 ml of each intermediate standard into five 100 ml conical flasks, and add, by pipette, 50 ml of water to give working standards containing approximately 0,8 mg/l, 1,6 mg/l, 4,0 mg/l, 8,0 mg/l and 16,0 mg/l terephthalic acid. Pipette 2 ml of the internal standard (7.1.2) into a further 100 ml conical flask and add, by pipette, 50 ml of water to give a blank standard 0,0 mg/l terephthalic acid.

7.1.4 Working standards for olive oil simulant

Using a measuring cylinder, pour 50 ml ± 1 ml of the olive oil blank from the migration test into a 250 ml separating funnel. Add, by pipette, 2 ml of the first intermediate standard, mix well and add 50 ml ±

2 ml of heptane rinsing the measuring cylinder in the process. Mix, and add 20 ml ± 1 ml of dilute sodium hydrogen carbonate solution using a measuring cylinder.

Shake gently for 1 min, then allow 15 min for the phases to separate.

NOTE: This is facilitated by holding the separating funnel at an angle of about 45° and slowly rotating it.

Run off the lower aqueous layer into a 100 ml beaker and re-extract the oil with a further 20 ml ± 1 ml of sodium hydrogen carbonate solution. Allow the phases to separate and run off the lower aqueous layer into the beaker to combine the extracts. Pass the aqueous solution through a C18 cartridge using a syringe or suction with a flow rate of about 10 ml/min to 20 ml/min. Collect the eluent into a 50 ml volumetric flask, add 1 ml ± 0,1 ml of 50 % acetic acid using a graduated pipette and dilute to the mark with water, mixing thoroughly. Filter 1 ml to 2 ml through a 0,2 µm filter for subsequent analysis in 7.2. Repeat this procedure for each intermediate standard, and the internal standard (7.1.2)., to give working standards containing 0 mg/l, 0,8 mg/l, 1,6 mg/l, 4,0 mg/l, 8,0 mg/l, and 16,0 mg/l terephthalic acid.

7.2 Preparation of calibration graphs

Inject the two sets of working standards into the high performance liquid chromatograph using a 10 µl loop. Divide the terephthalic acid peak height or area by the orthophthalic acid peak height or area and plot these values against the concentration of terephthalic acid in the standards in milligrams per litre, for both the aqueous and the olive oil simulant.

The calibration graphs should have a correlation coefficient of better than 0,997.

7.3 Validation of preparation of standard solutions

Dilute 10 ml of the second stock terephthalic acid standard solution (4.12) and 5 ml of the orthophthalic acid internal standard stock solution (4.13) to 25 ml as described in 7.1.1 to obtain a 200 mg/l intermediate standard. Dilute the intermediate standard as described in 7.1.3 to prepare a second 8,0 mg/l working standard and inject in duplicate for HPLC analysis.

Calculate the terephthalic acid/orthophthalic acid peak ratio and the mean concentration of terephthalic acid found in the second working standard by interpolation using the calibration graph obtained in 7.2, correcting for the mass of terephthalic acid used to prepare the first stock solution. The mean concentration should be within ± 0,5 mg/l of the actual terephthalic acid concentration in the second working standard, calculated from the mass of terephthalic acid used to prepare the second stock standard and the dilution factor. If the concentration is not within ± 0,5 mg/l, reject all solutions and start again.

7.4 Suitability of orthophthalic acid as an internal standard

Carry out the instructions described in 7.5 and 7.6, for each simulant, but omitting addition of the internal standard in each case. Inject each sample and the blank simulant prepared in this fashion and measure the peak area of any peak with a retention time equal to that of orthophthalic acid, measured from the standard chromatograms. After correction for the blank, if the interfering peak area exceeds 5 % of the orthophthalic acid peak area measured from injection of the standards, seek an alternative internal standard.

NOTE: Isophthalic acid has been found to be a suitable alternative, and has a longer retention time than terephthalic acid.

7.5 Extraction of olive oil simulant migration test samples

Pour the olive oil samples directly from the migration cell or container into a dry 50 ml measuring cylinder until the 50 ml ± 1 ml mark is reached. Pour the oil into a 250 ml separating funnel. Add, by