SLOVENSKI STANDARD
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Materiali in predmeti v stiku z živili - Snovi v polimernih materialih, katerih koncentracija je omejena - 17. del: Določevanje karbonilklorida (fosgena) v polimernih materialih

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 17: Determination of carbonyl chloride in plastics

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 17: Bestimmung von Carbonylchlorid in Kunststoffen

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matieres plastiques soumises a des limitations - Partie 17: Détermination du chlorure de carbonyle dans les matieres plastiques

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English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 17: Determination of carbonyl chloride in plastics

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This Technical Specification (CEN/TS) was approved by CEN on 16 December 2004 for provisional application.

The period of validity of this CEN/TS 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 CEN/TS can be converted into a European Standard.

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

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Foreword

This document (CEN/TS 13130-17:2005) 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 EN 13130 has been prepared within the Standards, Measurement and Testing project, MAT1-CT92-0006, “*Development of Methods of Analysis for Monomers*” and has been prepared by Subcommittee (SC 1) of TC 194 "Utensils in contact with food" as one of a series of test methods for plastics materials and articles in contact with foodstuffs.

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.

This part of EN 13130 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 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 6: *Determination of vinylidene chloride in plastics*

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*

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-trimethylolpropane in food simulants*

Parts 1 to 8 are European Standards. Parts 9 to 28 are Technical Specifications.

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this CEN Technical Specification: 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

Carbonyl chloride or phosgene, CCl_2O, PM/Ref. No 14380, is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture, residual carbonyl chloride can remain in the polymer and may migrate into foodstuffs coming into contact with the plastics material or article.

The method has been pre-validated by a collaborative trial with two laboratories.

1 Scope

This document, part of EN 13130, specifies an analytical procedure for the determination of carbonyl chloride monomer in polymers. The method is applicable to polycarbonate as well as to other polymers and copolymers where these are soluble in methylene chloride. The level of carbonyl chloride monomer determined is expressed as milligrams of carbonyl chloride per kilogram of polymer. The method is appropriate for the quantitative determination of carbonyl chloride at a minimum level of 0,3 mg per kilogram of polymer.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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.*

3 Principle

The level of carbonyl chloride in the polymer is determined by dissolution of the polymer and concurrent derivatization with 2-aminophenol. The resulting 2-benzoxazolinone is analyzed by high performance liquid chromatography (HPLC) with ultra violet (UV) detection. Quantification is achieved by standard addition of carbonyl chloride to the test polymer. Confirmation of the identity of carbonyl chloride is carried out by diode array detection.

4 Reagents

NOTE 1 Carbonyl chloride reacts rapidly with moisture. Suitable precautions should be taken to protect carbonyl chloride solutions from moisture. The carbonyl chloride derivative is not sensitive to moisture.

NOTE 2 All reagents should be of recognized analytical quality unless otherwise stated.

4.1 Analytes

4.1.1 Carbonyl chloride, CCl_2O, molecular weight, 98,9, commercially available as a 20 % (w/w) solution in toluene, density at 20 °C 0,935 kg per litre, corresponding to a concentration of 1,93 mole per litre or 191 g per litre.

Store the commercial carbonyl chloride solution in a closed container for up to 6 months at + 1 °C in the dark.

4.1.2 2-benzoxazolinone, $C_7H_5NO_2$, molecular weight: 135,1, purity 98 %.

4.2 Chemicals

4.2.1 2-aminophenol (2-hydroxyanilin), C_6H_7NO, molecular weight: 109,1, purity 98 %.

4.2.2 Dichloromethane (DCM), dried over a bed of molecular sieve (5Å), H_2O < 0,005 %, purity > 99,5 %.

4.2.3 Toluene, dried over a bed of molecular sieve (5Å), H_2O < 0,005 %, purity > 99,5 %.

NOTE The water content of the toluene should be as low as possible. Toluene dried over a molecular sieve can be obtained from chemical suppliers. Alternatively, toluene can be dried before use, in which case the molecular sieve should be activated for maximum water absorption.

4.2.4 Methanol

4.2.5 Acetone

4.2.6 Acetonitrile, purity > 99,5 %, HPLC grade

4.2.7 Distilled water, HPLC grade

4.2.8 Sodium dihydrogen orthophosphate, $NaH_2PO_4 \cdot 2H_2O$

4.2.9 Glacial acetic acid

4.2.10 Hydrochloric acid, 1 N

4.2.11 Sodium sulfate, anhydrous

4.2.12 Mobile phase for high performance liquid chromatography, prepared as follows.

Dissolve 7,5 g of sodium dihydrogen orthophosphate (4.2.8) in 700 ml of water (4.2.7), add 250 ml acetonitrile (4.2.6) using a measuring cylinder and adjust to pH (3,6 ± 0,1) with glacial acetic acid (4.2.9).

NOTE 1 Approximately 5 ml should be sufficient.

Make up to 1 l with water (4.2.7).

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

4.3 Solutions

4.3.1 Standard solution of carbonyl chloride in toluene, 95,5 mg/l

Pipette into a 20 ml vial (5.3) 20,0 ± 0,1ml of toluene (4.2.3) and close with the septum and cap. Using a 10 µl syringe (5.5), add 10 µl of the 20 % carbonyl chloride solution (4.1.1) by injection through the septum and dispensing after the needle has immersed into the toluene. Remove the syringe and mix.

NOTE 1 The exact concentration of this standard solution is 95,5 mg/l.

Repeat the procedure to provide a second standard solution.