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MATERIALS AND ARTICLES IN CONTACT WITH FOODSTUFFS - PLASTICS SUBSTANCES SUBJECT TO LIMITATION - PART 8: DETERMINATION OF ISOCYANATES IN PLASTICS

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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 od 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 13130-5	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

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.

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WARNING: Isocyanates are hazardous substances, and some (e.g. 2,4 and 2,6-toluene diisocyanate) are carcinogenic. They are also suspected of causing sensitization. Handling and preparation of standard solutions should be undertaken in a fume hood. Skin and eye contact with isocyanates and inhalation of vapour, should be strictly avoided.

0 Introduction

Isocyanates, characterised by the -NCO group, are monomers used for the manufacture of materials and articles intended to come in contact with food. During manufacture residual isocyanates can remain in the polymer and may migrate into food coming into contact with the polymer

1 Scope

This Part of this European Prestandard describes a method for the determination of individual and total levels of residual isocyanates in plastics materials and articles.

This method is applicable to the analysis of polyurethane polymers. The total level of isocyanate monomers in materials and articles determined according to the procedure described in this prestandard is given in milligrams of NCO per kilogram of material or article. The method is capable of quantitative determination of individual isocyanates measured as NCO at 0,04 mg/kg and total isocyanates at 1,0 mg/kg.

> NOTE: The method has been applied to the analysis of 9 isocyanate monomers listed in 3.1. It has not been applied to the analysis of octadecyl isocyanate, diphenylether-4,4'-diisocyanate or 3,3'-dimethyl-4,4'-diisocyanatobiphenyl as samples of these monomers have not been obtained. There is no reason to anticipate that the method may not be suitable for the analysis of these monomers also.

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 procedure consists of two parts: screening and, if necessary, quantitative determination. Quantitative determination is applied only if isocyanates are detected by the screening procedure.

Materials and articles are initially screened for residual isocyanates by solvent extraction with dichloromethane and concurrent derivatisation with 9-(methylaminomethyl)anthracene. 1-Naphthyl isocyanate is used during the screening procedure to check that the derivatization procedure has been successful. The resultant fluorescent derivatives are analysed by high performance liquid chromatography with fluorescence detection.

Materials found to contain residual isocyanates are quantified by standard addition to the material or article under test, using 1-naphthyl isocyanate as internal standard.

If interferences are experienced with the internal standard then calibration is carried out by standard addition omitting the internal standard, as described in annex A.

Confirmation of isocyanate levels is carried out by re-analysing the sample extracts on an HPLC column with different elution characteristics.

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4 Reagents

NOTE: Isocyanates react extremely rapidly with moisture. Suitable precautions should be taken to ensure all glassware is dry. All laboratory glassware should be rinsed with diethyl ether (4.2.2) and baked at 105°C overnight before use. After baking, vials should be placed in a desiccator and stored until required. Isocyanate standards should be protected from moisture and stored under refrigeration at -20 °C.

All reagents should be of recognised analytical quality, unless otherwise specified.

4.1 Analytes

4.1.1 2,6-toluene diisocyanate $CH_3C_6H_3(NCO)_2$

4.1.2 diphenylmethane-4,4'-diisocyanate $OCNC_6H_4CH_2C_6H_4NCO$

4.1.3 2,4-toluene diisocyanate $CH_3C_6H_3(NCO)_2$

4.1.4 hexamethylene diisocyanate $OCNC_6H_{12}NCO$

4.1.5 cyclohexyl isocyanate $C_6H_{11}NCO$

4.1.6 1,5-naphthalene diisocyanate $C_{10}H_6(NCO)_2$

4.1.7 diphenylmethane-2,4'-diisocyanate $OCNC_6H_4CH_2C_6H_4NCO$

4.1.8 2,4-toluene diisocyanate dimer

4.1.9 phenyl isocyanate C_6H_5NCO

4.1.10 1-naphthyl isocyanate (internal standard, $C_{10}H_7NCO$), which contains no impurity at > 1 % by area which will elute at the same retention time as any of the nine individual isocyanate derivatives.

All standards should be of > 99 % purity.

4.2 Reagents

4.2.1 Dichloromethane (DCM, CH_2C_{l2}), <30 ppm H_2O, containing no impurity at > 1 %, by area, which elutes at the same HPLC retention time as the isocyanate derivatives or internal standard derivative peaks. DCM should be dried over a bed of molecular sieve (5 Å) for 24 h prior to use.

4.2.2 Diethylether ($(C_2H_5)_2O$), at least 99 % purity.

4.2.3 9-(Methylaminomethyl)anthracene (MAMA, $CH_3NHCH_2C_{14}H_9$), containing no impurity at > 1 %, by area, which elutes at the same HPLC retention time as the isocyanate derivatives or internal standard derivative peaks.

4.2.4 N,N'-Dimethylformamide ($HCON(CH_3)_2$), containing no impurity at > 1 %, by area, which elutes at the same HPLC retention time as the isocyanate derivatives or internal standard derivative peaks.

4.2.5 Individual stock standard solutions (1000 µg/ml)

Weigh 0.01 g of isocyanate standard (4.1), to an accuracy of 0,1 mg, in a 10 ml volumetric flask. Rapidly make-up to the mark with DCM (4.2.1) and shake thoroughly. Ultrasonification may be used as an aid to dissolution. Repeat the procedure to provide a second stock solution.

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