DRAFT FOR DEVELOPMENT

DD CEN/TS
13130-11:2005

Materials and articles in contact with foodstuffs — Plastics substances subject to limitation —

Part 11: Determination of 11-aminoundecanoic acid in food simulants

ICS 67.250

BSI
British Standards

National foreword

This Draft for Development is the official English language version of CEN/TS 13130-11:2005.

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature because the method was not evaluated using recognized ring trial procedures. As a consequence there are no reproducibility data for the method. It should be applied on this provisional basis, so that information and experience of its practical application may be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the European organization responsible for its conversion to a European standard. A review of this publication will be initiated 2 years after its publication by the European organization so that a decision can be taken on its status at the end of its 3-year life. Notification of the start of the review period will be made in an announcement in the appropriate issue of *Update Standards*.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into a European Standard, to extend the life of the Technical Specification or to withdraw it. Comments should be sent in writing to the Secretary of BSI Subcommittee CW/47/1, Migration from plastics, at British Standards House, 389 Chiswick High Road, London W4 4AL, giving the document reference and clause number and proposing, where possible, an appropriate revision of the text.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the *BSI Catalogue* under the section entitled "International Standards Correspondence Index", or by using the "Search" facility of the *BSI Electronic Catalogue* or of British Standards Online.

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Summary of pages

This document comprises a front cover, an inside front cover, the CEN/TS title page, pages 2 to 15 and a back cover.

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English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 11: Determination of 11-aminoundecanoic acid in food simulants

Matériaux et objets en contact avec les denrées alimentaires - Matières plastiques et substances soumises à des limitations - Partie 11 : Détermination de l'acide 11-aminoundecanoïque dans les simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 11: Bestimmung von 11-Aminoundecansäure in Prüflebensmitteln

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 13130-11:2005) 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

11-aminoundecanoic acid, $C_{11}H_{23}O_2N$, PM/Ref. No 12788 is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture, residual acrylamide can remain in the polymer and may migrate into foodstuffs coming into contact with that product.

This analytical method should be used in conjunction with EN 13130-1, which describes the procedures to be applied prior to the determination of 11-aminoundecanoic acid in food simulants.

NOTE Although the method is applicable to olive oil, it should be taken into account that 11-aminoundecanoic acid is unstable in olive oil during storage. Therefore the method should only be applied in case of short exposure periods with olive oil. Alternatively iso-octane could be used as a substitute fatty food simulant.

1 Scope

This document, part of EN 13130, specifies a method for the determination of 11-aminoundecanoic acid in the food simulants water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol, olive oil and iso-octane. The level of 11-aminoundecanoic acid determined is expressed as milligrams per kilogram of food simulant.

The method is appropriate for the quantitative determination of 11-amino-undecanoic acid in approximate analyte concentration range of 0,5 mg/kg to 10 mg/kg of food simulants.

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 11-aminoundecanoic acid in food simulants is determined by preparing a derivative of 11-aminoundecanoic acid with a fluorescent component. The derivative is subsequently determined by high performance liquid chromatography (HPLC) and fluorescence detection. Quantification is achieved using the calibration curve method.

NOTE A method for confirmation of the presence of 11-aminoundecanoic acid has not yet been established.

4 Reagents

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

4.1 Analyte

11-aminoundecanoic acid, $C_{11}H_{23}O_2N$, molecular weight 201, purity greater than 99 %.

NOTE The monomer is stable for a long period at room temperature if protected from light.

4.2 Chemicals

4.2.1 Acetic acid, glacial

4.2.2 Acetic acid, 96 %

4.2.3 Acetone

4.2.4 di-Sodium tetraborate-10-hydrate, $Na_2 B_4O_7$ 10. H_2O

4.2.5 Boric acid

4.2.6 Fluorescamine, 4-phenylspiro-[furan 2-(3), 1-phthalan]-3,3-dione

4.2.7 Iso-octane

4.2.8 Methanol

4.2.9 Nitrogen

4.2.10 Phosphoric acid 85 %

4.2.11 Sodium dihydrogen phosphate monohydrate

4.2.12 Sodium hydroxide

4.2.13 Water deionized (HPLC quality)

4.3 Solutions

4.3.1 Stock solution of 11-aminoundecanoic acid in water (1,25 mg/ml)

Weigh to the nearest 0,1 mg approximately 60 mg of 11-aminoundecanoic acid in a 50 ml volumetric flask. Dissolve the 11-aminoundecanoic acid in the smallest possible volume, approximately 1 ml, of 96 % acetic acid (4.2.2) and fill the conical flask up to the mark with water (4.2.13).

Calculate the actual concentration in milligrams 11-aminoundecanoic acid per millilitre of solution.

Repeat the procedure to obtain a second stock solution.

The two primary standard solutions of analyte shall be checked against one another. The response factor, i.e. detector response divided by concentration of analyte solution, of the two primary standard solutions shall not differ more than 5 %. If there is agreement within 5 %, subsequent diluted standard solutions shall be made from only one of the primary standard solutions.

If the levels of the two independently prepared stock solutions do not correspond to within ± 5 %, both stock solutions shall be discarded, and new solutions shall be prepared.

4.3.2 Stock solution of 11-aminoundecanoic acid in iso-octane (0,5 mg/ml)

Weigh to the nearest 0,1 mg approximately 25 mg 11-aminoundecanoic acid in a 50 ml volumetric flask. Dissolve the 11-aminoundecanoic acid in approximately 5 ml 100% acetic acid (4.2.1) and fill up to the mark with iso-octane (4.2.7).

Calculate the actual concentration of 11-aminoundecanoic acid in milligrams per millilitre solution.

Repeat the procedure to obtain a second stock solution, see 4.3.1.

4.3.3 Diluted standard solution of 11-aminoundecanoic acid in aqueous food simulants (25 μg/ml)

Pipette 2 ml of the stock solution (4.3.1) into a series of three 100 ml volumetric flasks. Make up to the mark with the appropriate aqueous food simulant, water, 3 % w/v aqueous acetic acid and 15 % v/v aqueous ethanol.

Calculate the actual concentration 11-aminoundecanoic acid in micrograms per millilitre of aqueous food simulant.

4.3.4 Phosphoric acid, 8,5 %

Pipette 10 ml of the 85 % phosphoric acid (4.2.10) into a measuring flask of 100 ml and make up to the mark with water (4.2.13).

4.3.5 Phosphate buffer, 5 mM, pH 3

Weigh 690 mg of sodium dihydrogen phosphate monohydrate (4.2.11) and dissolve in approximately 900 ml water (4.2.13). Adjust to pH (3 ± 0,2) by adding small portions of 8,5 % phosphoric acid (4.3.4). Make up to 1 litre with water (4.2.13).

4.3.6 Mobile phase for HPLC

Prepare a mixture of 300 ml of 5 mM phosphate buffer, pH 3 (4.3.5) with 700 ml of methanol (4.2.8).

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

4.3.7 Borate buffer, 400 mM, pH 9

Weigh 15,3 g of di-sodium tetraborate (4.2.4) and dissolve in approximately 90 ml water (4.2.13). Adjust to pH (9 ± 0,2) by adding small portions of 5 % boric acid. Make up to 100 ml with water (4.2.13).

4.3.8 Sodium hydroxide solution, 18 %

Weigh 18 g sodium hydroxide (4.2.12), and dissolve in 100 ml water (4.2.13).

4.3.9 Fluorescamine solution

Weigh 20 mg fluorescamine (4.2.6) and dissolve in 50 ml acetone (4.2.3).

NOTE The solution is stable for several months at room temperature when stored in the dark.

5 Apparatus

NOTE An instrument or item of apparatus is listed only where it is special or made to a particular specification, usual laboratory glassware and equipment being assumed to be available.

5.1 High performance liquid chromatograph, preferably with an automatic injector with 20 µl injection loop, and a variable fluorimetric detector, set to an emission of 390 nm and an excitation of 480 nm, connected to a strip chart recorder or integrator.

5.2 HPLC column, capable of producing a symmetric peak of 11-aminoundecanoic acid derivative, and capable to separate 11-aminoundecanoic acid from peaks originating from simulants and/or solvents used.

NOTE For guidance, the parameters established for the column selected are given below:

Column	Stainless steel 200 mm x 4,6 mm, filled with octadecyl coated silica, particle size 5 µm
Column temperature:	Ambient
Mobile phase:	30 % aqueous phosphate buffer, pH 3 (4.3.5)
	70 % methanol (4.2.8)
Flow rate	1 ml/min
Injection volume:	20 µl
Detection	fluorometric
Wavelength:	emission 390 nm excitation 480 nm

A broad range of different reversed phase columns are suitable for this analysis.

Depending on the type of equipment used for the determination, the appropriate operating conditions have to be established.

5.3 Shaking machine

5.4 Vortex mixer

5.5 Heating block, capable of maintaining a temperature of 40 °C, and provided with holes to accept suitable reaction tubes.

6 Samples

6.1 General

The laboratory samples of food simulants to be analyzed shall be obtained as described in EN 13130-1. Analyte-free samples of food simulants of the same type as those to be analyzed shall also be prepared for use in calibration and as blanks.

The migration experiments can be performed without special precautions, while the laboratory samples of water, 3 % w/v aqueous acetic acid and 15 % v/v aqueous ethanol may be stored for several weeks in a refrigerator. The extracts obtained with olive oil or iso-octane shall be analyzed immediately after the exposure time.

NOTE It is recommended that the containers in which the migration experiments (see EN 13130-1) with iso-octane are carried out are washed with 10 % of the initial volume of iso-octane, using 100 % acetic acid, to dissolve any 11-aminoundecanoic acid that is absorbed on the wall of the container. The acetic acid should be added to the iso-octane. The final volume should be corrected for this addition.

Samples of recovery tests with 11-aminoundecanoic acid in the relevant food simulants and which have been stored under the same conditions as used in the migration experiments, shall be submitted to the same procedure as the laboratory food simulant samples.

6.2 Test sample preparation

6.2.1 Food simulants, water and 15 % v/v aqueous ethanol

Pipette 1,0 ml of the food simulant obtained from the migration experiment (see EN 13130-1) into a 10 ml reaction tube and continue as described in Clause 7.

6.2.2 Food simulant, 3 % w/v aqueous acetic acid

Pipette 1,0 ml of the food simulant obtained from the migration experiment (see EN 13130-1) into a 10 ml reaction tube, add 100 µl of 18 % sodium hydroxide (4.3.8) and mix on the vortex mixer. Continue as described in Clause 7.

Make sure that the pH, of the 3 % w/v aqueous acetic acid simulant, after addition of the 0,4 M borate buffer (4.3.7), is between 8,0 and 9,5. If the pH is less than 8,0, add additional amounts of 18 % sodium hydroxide solution.

6.2.3 Iso-octane

Pipette 5,0 ml of the iso-octane obtained from the migration experiment (see EN 13130-1) into a conical flask. Add 5 ml of iso-octane (4.2.7) and mix. Add, by pipette, 5,0 ml of 96 % acetic acid (4.2.2). Place the conical flask for 30 min on a shaking machine (5.3). Allow the phases to separate and retract, by means of a pipette, 1,0 ml of the, lower, acetic acid layer and transfer into a reaction tube. Place the tube in the heating block at 40 °C and evaporate the acetic acid extract to dryness under a gentle stream of nitrogen (4.2.9). Add, by pipette, 1,0 ml water (4.2.13) to the residue and mix thoroughly for 10 s on a vortex mixer (5.4). Continue as described in Clause 7.

6.2.4 Olive oil

Weigh 5 g ± 0,1 g of the olive oil obtained from the migration experiment (see EN 13130-1) into a conical flask. Add 5 ml of iso-octane (4.2.7) and mix. Add, by pipette, 5,0 ml of 96 % acetic acid (4.2.2). Place the conical flask for 30 min on a shaking machine (5.3). Allow the phases to separate and retract, by means of a pipette, 1,0 ml of the, lower, acetic acid layer and transfer into a reaction tube. Place the tube in the heating block at 40 °C and evaporate the acetic acid extract to dryness under a gentle stream of nitrogen (4.2.9). Add, by pipette, 1,0 ml water (4.2.13) to the residue and mix thoroughly for 10 seconds on a vortex mixer (5.4). Continue as described in Clause 7.

6.2.5 Blank sample preparation

Follow the procedures described in 6.2.1 to 6.2.4 using 11-aminoundecanoic acid free food simulants.

6.3 Calibration sample preparation

6.3.1 Calibration curves in aqueous food simulant

Pipette, into a series of 50 ml volumetric flasks, 0 ml, 1 ml, 2 ml, 4 ml, 10 ml and 20 ml of each of the diluted standard solutions (4.3.3). Fill the volumetric flasks up to the mark with the appropriate, analyte free, food simulant and mix thoroughly.

Calculate the actual concentration of 11-aminoundecanoic acid in milligrams per millilitre aqueous food simulant. The solutions thus obtained contain approximately 0 μg/ml, 0,5 μg/ml, 1,0 μg/ml, 2,0 μg/ml, 5,0 μg/ml and 10 μg of 11-aminoundecanoic acid per millilitre of simulant. Transfer 1 ml of each standard solutions into a reaction tube and follow the procedures described in 6.2.1 and 6.2.2.

6.3.2 Calibration curve in olive oil and iso-octane

Weigh, into a series of 25 ml conical flasks, 5,0 g ± 0,01 g of analyte free olive oil. Add, by means of a suitable injection syringe 0 μl, 5 μl, 10 μl, 20 μl, 50 μl and 100 μl of the stock solution (4.3.2), and mix thoroughly.

Transfer into a series of 25 ml conical flasks, 5,0 ml of analyte free iso-octane. Add, by means of a suitable injection syringe, 0 μl, 5 μl, 10 μl, 20 μl, 50 μl and 100 μl of the stock solution (4.3.2), and mix thoroughly.

Calculate the actual concentration of 11-aminoundecanoic acid in milligrams per millilitre of food simulant. The solutions thus obtained contain approximately 0 μg/ml, 0,5 μg/ml, 1,0 μg/ml, 2,0 μg/ml, 5,0 μg/ml and 10 μg of 11-aminoundecanoic acid per millilitre of simulant. Follow the procedures described in sections 6.2.3 and 6.2.4.

7 Procedure

7.1 General

Prior to the HPLC analysis, the test samples, blanks and calibration samples as prepared in 6.2 to 6.3 are subjected to a reaction to convert the 11-aminoundecanoic acid into a fluorescent component.

7.2 Sample treatment

Add, to the solutions prepared in 6.2.1 to 6.2.4, by pipette, 1,0 ml of the 0,4 M borate buffer pH 9,0 (4.3.7) and mix for 10 seconds on a vortex mixer (5.4). Add, while mixing on the vortex mixer (5.4), using a pipette, 1,0 ml of the fluorescamine solution (4.3.9). Transfer this solution into a vial suitable for HPLC injections.

7.3 Execution of determination

When starting HPLC analyses, examine baseline stability and response linearity of the instrument. The same operating conditions of the HPLC system shall be maintained throughout the analysis of all test samples and solutions described in Clause 6. Each test sample and solution shall be analyzed at least in duplicate.

Insert the solutions as prepared in 7.2 into the automatic sampler and start the HPLC analysis using suitable conditions recommended in 5.2 or other suitable conditions.

Identify the 11-aminoundecanoic acid peak in the chromatogram on the basis of the retention time and measure the peak area.

7.4 Quantification

Calculate the amount of 11-aminoundecanoic acid using the calibration curve method, as follows.

Plot the peak area of the 11-aminoundecanoic acid derivative versus the concentration of 11-aminoundecanoic acid in the calibration samples. Calculate the regression parameters and the correlation coefficient.

The calibration graph shall be rectilinear and the correlation coefficient shall be 0,996 or better. If either of the two requirements is not met, fresh standard solutions shall be prepared from the original stock solutions. Analysis of the solutions and construction of the calibration graph shall be repeated.

7.5 HPLC interferences

Following the method described above, no interferences should be observed in the chromatograms.

8 Expression of results

8.1 Calculation of 11-aminoundecanoic acid level in test samples

NOTE the following calculations assume that for all measurements exactly the same mass or volume of food simulant has been used.

8.1.1 Graphical determination

Calculate the average peak areas obtained of the duplicate injections of the test samples obtained in 6.2 and read the 11-aminoundecanoic acid concentration of the test samples from the calibration graph in micrograms per gram.

NOTE Commission Directive 2002/72/EC [1] states that the specific gravity of all simulants should conventionally be assumed to be ‘1’. Milligrams of substance released per litre of simulant will thus correspond numerically to milligrams of substance released per kilogram of simulant and, taking into account of the provisions laid down in Directive 82/711//EEC [3], to milligrams of substance released per kilogram of foodstuff.

8.1.2 Calculation from regression parameters

If the regression line equation is:

$$Y = (A \times X) + B$$

where

Y is the peak area of 11-aminoundecanoic acid;

A is the slope of the regression line in micrograms per gram;

X is the concentration of 11-aminoundecanoic acid in the food simulant in micrograms per gram;

B is the intercept of the regression line,

then the 11-aminoundecanoic acid concentration in the food simulant is given by:

$$C_{\text{11-aminoundecanoic acid}} = \frac{Y - B}{A}$$

where

$C_{\text{11-aminoundecanoic acid}}$ is the concentration of 11-aminoundecanoic acid in micrograms per gram.

NOTE The method applying a calculation from the regression parameters is the preferred method.

8.1.3 Calculation of the specific migration

Depending on the fill volume of the test material and on the surface area/food simulant ratio, the 11-aminoundecanoic acid concentration in the laboratory sample as determined according to Clause 7 may need mathematical transformation to calculate the specific migration value to be compared with the specific migration limit (SML). For guidance see EN 13130-1:2004, Clause 13.

8.2 Precision

8.2.1 Validation

This method was pre-evaluated in 1995 by a within-laboratory precision experiment using the four official EU food simulant and iso-octane for establishment of precision data at the restriction criterion, as well as by carrying out recovery experiments including storage for 10 d at 40 °C with the official food simulants and 2 d at 20 °C with iso-octane.

8.2.2 Repeatability

Evaluation of the results of the precision experiment, in accordance with ISO 5725, at a concentration of 2 mg 11-aminoundecanoic acid per kilogram of food simulant for the 95 % probability level yielded the following performance characteristics:

Repeatability (r) = 0,18 mg 11-aminoundecanoic acid per kilogram water;

0,01 mg 11-aminoundecanoic acid per kilogram 3 % w/v aqueous acetic acid;

0,02 mg 11-aminoundecanoic acid per kilogram 15 % v/v aqueous ethanol;

0,17 mg 11-aminoundecanoic acid per kilogram olive oil;

0,12 mg 11-aminoundecanoic acid per kilogram iso-octane;

8.2.3 Detection limit

The within-laboratory detection limit (WDL), based on the calibration curve method in accordance with DIN 32645, were found to be in the range of 0,02 mg/kg to 0,8 mg 11-aminoundecanoic acid per kilogram of food simulant.

Thus the method is capable of quantitative determination of 11-aminoundecanoic acid at a minimum level of 0,8 mg/kg of food simulant.

9 Confirmation

9.1 Requirements

If the specific migration of 11-aminoundecanoic acid into the food simulant, when determined as described in 8.1.3, exceeds the restriction criterion, e.g. SML = 5 mg/kg, the determination shall be confirmed by the method described in 9.2.

9.2 Confirmation

A procedure for confirmation of the identity has not yet been established.

10 Test report

The test report shall include the following, where applicable:

a) all information necessary for complete identification of the sample, e.g. chemical type, trade mark, grade, batch number, thickness, etc.;

b) form of the plastics, e.g. film, bottle, pot, etc.;

c) use/class of food for which the sample is intended to contact, where known, and where possible food classification reference number; see Table 2 of EN 13130-1:2004;

d) intended conditions of use, where known, e.g. time/temperature;

e) conditions of the test;

   1) part(s) of EN 13130 used;

   2) foodstuffs or food simulants used;

   3) duration and temperature, and relation with "Conditions of contact in worst foreseeable use", as given in Table 3 of EN 13130-1:2004;

   4) area and geometry of the test specimen;

   5) volume of foodstuff or food simulant used where appropriate;

f) any departures from the standard method, reasons for the departures;

g) any particular requirements of the parts of this document;

h) any relevant comments on the test results;

i) details of any confirmation procedure(s);

j) individual triplicate or quadruplicate test results, and the mean of these results expressed in milligrams of 11-aminoundecanoic acid per kilogram of food simulant.

Bibliography

[1] Commission of the European Communities, Commission Directive 2002/72/EC of 6 August 2002 relating to plastics materials and articles intended to come into contact with foodstuffs, Official Journal of the European Communities, 15 August 2002, no. L220, p18.

[2] Commission of the European Communities, Council Directive of 21 December 1988 on the approximation of the laws of the Member States relating to materials and articles intended to come into contact with foodstuff (89/109/EEC), Official Journal of the European Communities, 11 February 1989, no. L 40, p 38.

[3] Commission of the European Communities, Council Directive of 18 October 1982 laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs (82/711/EEC), Official Journal of the European Communities, 23 October 1982, no. L 297, p 26.

[4] Commission of the European Communities, Commission Directive of 15 March 1993 amending Council Directive 82/711/EEC laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs (93/8/EEC), Official Journal of the European Communities, 14 April 1993, no. L 90, p 22.

[5] Commission of the European Communities, Commission Directive of 97/48/EC of 29 July 1997 amending Council Directive 82/711/EEC laying down the basic rules necessary for testing migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs, Official Journal of the European Communities, 12 August 1997, no. L 222, p 10.

[6] Commission of the European Communities, Council Directive of 19 December 1985 laying down the list of simulants to be used for testing migration of constituents of plastics materials and articles intended to come into contact with foodstuffs (85/572/EEC), Official Journal of the European Communities, 31 December 1985, no. L372, p14.

[7] ISO 5725, *Accuracy (trueness and precision) of measurement methods and result.*

[8] DIN 32645, *Chemical analysis; decision limit; detection limit and determination limit; estimation in case of repeatability; terms, methods, evaluation.*

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