DRAFT FOR DEVELOPMENT

DD CEN/TS
13130-25:2005

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

Part 25: Determination of 4-methyl-1-pentene in food simulants

ICS 67.250

BSI
British Standards

National foreword

This Draft for Development is the official English language version of CEN/TS 13130-25:2005.

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 14, an inside back cover and a back cover.

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English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 25: Determination of 4-methyl-1-pentene in food simulants

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 25 : Détermination du 4-méthyl-1 pentène dans les simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 25: Bestimmung von 4-Methyl-1-Penten 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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Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (CEN/TS 13130-25:2005) 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

4-methyl-1-pentene, C_6H_{12}, PM/Ref. No 22150 is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture residual 4-methyl-1-pentene can remain in the finished product and may migrate into foodstuffs coming into contact with that product.

NOTE However, the following should be taken into account when carrying out a migration test. From migration experiments carried out at 10 d for 40 °C it was recognized that there was an irreproducible and considerable loss of 4-methyl-1-pentene, 35 % to 98 %, due to volatilization when using aqueous food simulants.

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

1 Scope

This document, part of EN 13130, specifies an analytical procedure for the determination of 4-methyl-1-pentene in food simulants distilled water, 3 % (w/v) aqueous acetic acid aqueous solution, 15 % (v/v) aqueous ethanol aqueous solution and rectified olive oil. The level of 4-methyl-1-pentene monomer determined is expressed as milligrams per kilogram of food simulant. In principle, the method is appropriate for the quantitative determination of 4-methyl-1-pentene at a minimum level of 0,005 mg/kg in all of the four food simulants. However, in the case of interferences, which have been observed for some olive oil batches, the detection limit can be compromised correspondingly.

NOTE The method should also be applicable to other aqueous food simulants as well as to other fatty food simulants such as corn oil, sunflower oil and a mixture of synthetic triglycerides.

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 4-methyl-1-pentene in food simulants is determined by headspace gas chromatography (HSGC). Quantification is achieved using an internal standard, cyclohexane. Calibration is realized by analysis of relevant food simulant samples containing known amounts of 4-methyl-1-pentene and cyclohexane.

NOTE If automatic headspace equipment is applied, the use of an internal standard is not required. Repeatability may even be improved without use of the internal standard.

Confirmation of 4-methyl-1-pentene is carried out by combined gas chromatography/mass spectrometry (GC/MS).

4 Reagents

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

4.1 Analytes

4.1.1 4-methyl-1-pentene, C_6H_{12}, molecular weight: 84,16, purity ≥ 98 %.

4.1.2 Cyclohexane, C_6H_{12}, molecular weight: 84,16, purity ≥ 99,5 %.

4.2 Chemical

N,N-dimethylacetamide - distilled (DMAA)

4.3 Solutions

4.3.1 Stock solution of 4-methyl-1-pentene (1 mg/ml)

Weigh to the nearest 0,1 mg approximately 25 mg of 4-methyl-1-pentene into a 25 ml volumetric flask, which contains approximately 20 ml of DMAA. Make up to the mark with DMAA and mix carefully.

Calculate the concentration in milligrams of 4-methyl-1-pentene per millilitre of solution.

Repeat the procedure to obtain a second stock solution.

NOTE The stock solution can be stored in a well closed container, with the exclusion of light, for a maximum of 3 weeks at any temperature between - 20 °C and + 10 °C.

4.3.2 Internal standard stock solution of cyclohexane in DMAA (1 mg/ml)

Weigh to the nearest 0,1 mg, approximately 25 mg of cyclohexane into a 25 ml volumetric flask. Make up to the mark with DMAA.

Calculate the concentration in milligrams of cyclohexane per millilitre of solution.

NOTE The solution can be stored in a well closed container, with the exclusion of light, for a maximum of 2 weeks at any temperature between - 20 °C and + 10 °C.

4.3.3 Diluted internal standard solution of cyclohexane in DMAA (50 µg/ml)

Transfer with a microsyringe 500 µl of the internal standard stock solution (4.3.2) into a 10 ml volumetric flask and make up to the mark with DMAA.

Calculate the concentration in micrograms of cyclohexane per millilitre of solution.

NOTE The solution can be stored in a well closed container, with the exclusion of light, for a maximum of 2 weeks at any temperature between - 20 °C and + 10 °C.

4.3.4 Diluted standard solution (10 µg/ml)

Transfer into a 10 ml volumetric flasks approximately 7 ml DMAA. Add with the aid of a microsyringe 100 µl of the standard stock solution of 4-methyl-1-pentene (4.3.1). The tip of the syringe shall be submerged into DMAA at the moment the syringe is emptied. Make up to the mark with DMAA.

The standard solution thus obtained contains 10 µg of 4-methyl-1-pentene/ml DMAA.

Repeat the procedure using the second standard stock solution.

NOTE The solution can be stored in a closed container, with the exclusion of light, for a maximum of 2 weeks at any temperature between - 20 °C and + 10 °C.

5 Apparatus

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

5.1 Gas chromatograph, equipped with an automatic headspace sampler and a flame ionization detector.

Appropriate operating conditions shall be established for the specific equipment used for the determination.

The GC column used should be capable of separating 4-methyl-1-pentene fully from cyclohexane and also from peaks originating from the simulants and/or solvents used.

NOTE The following column and parameters have been found to be suitable:

Column: 27,5 m x 0,32 mm fused silica PLOT (Porous Layer Open Tubular) provided with a 10 µm thick layer of porous styrene-divinylbenzene polymer

GC parameters:

column oven	isothermal 2 min at 70 °C, then at 7,5 °C/min to 220 °C
detector(FID)	250 °C
carrier gas	helium
inlet pressure	180 kPa
flow rate 5,7 ml/min	
FID gasses	optimized according to manufacturer's specifications

Headspace conditions for aqueous food simulants:

Sample temperature	80 °C
Transfer temperature	110 °C
Thermostatting time	30 min
Pressurization time	0,5 min
Injection time	0,10 min
Cycle time	30 min

Headspace conditions for olive oil and other fatty simulants:

Sample temperature	90 °C
Transfer temperature	120°C
Thermostatting time	30 min
Pressurization time	0,5 min
Injection time	0,25 min
Cycle time	30 min

GC and headspace apparatus should be optimized according to manufacturer's instruction.

5.2 Microsyringes, 10 µl, 50 µl, 100 µl and 500 µl.

6 Samples

6.1 Test sample preparation

6.1.1 General

Laboratory samples of the food simulant to be analyzed shall be obtained as described in EN 13130-1. Samples shall be kept refrigerated at + 4 °C in closed containers with the exclusion of light up to four weeks. Analyte-free samples of food simulants of the same type as those to be analyzed shall also be prepared for calibration purposes.

NOTE In the case of interferences, which were observed for some olive oil batches, the detection limit of the method can be compromised correspondingly and may be insufficient. In such a case it is recommended either to search for a fatty food simulant free of interferences or to deviate from the food simulant volume to contact surface ratio as given in EN 13130-1 by application of a considerably lower fatty food simulant volume.

WARNING Take into account possible loss of analyte in aqueous food simulants (see NOTE in the Introduction).

If an automatic headspace sampler is used, the addition of the internal standard is not required to obtain reliable results. In that case all references to the internal standard may be omitted. If manual injection is used, the internal standard technique shall be used.

From each sample, blank or calibration solution, at least duplicate vials shall be prepared, e.g. as a pair of measurements.

6.1.2 Aqueous solutions

Transfer into a 22 ml headspace vial 10,0 ml of food simulant obtained from a migration experiment (see EN 13130-1). Add with an injection syringe 10 µl of the diluted internal standard solution (4.3.3). Submerge the tip of the syringe needle into the simulant at the moment the syringe is emptied and the swirl the syringe before retraction. Immediately close the vial with a septum and aluminium cap.

6.1.3 Olive oil

Transfer into a 22 ml headspace vial 10 g ± 0,1 g of olive oil obtained from a migration experiment (see EN 13130-1). Add with an injection syringe 10 µl of the diluted internal standard solution (4.3.3). Submerge the tip of the syringe needle into the simulant at the moment the syringe is emptied and swirl the syringe before retraction. Immediately close the vial with a septum and aluminium cap.

6.2 Blank sample preparation

Treat simulants which have not been in contact with packaging material in the same way as described in 6.1.2 and 6.1.3.

6.3 Calibration sample preparation

6.3.1 Calibration curves for aqueous food simulants

Transfer into a series of six headspace vials 10,0 ml of the appropriate food simulant. Transfer with a microsyringe 0 µl, 5 µl, 10 µl, 20 µl, 30 µl, and 40 µl of the diluted standard solution (4.3.4) and 10 µl of the diluted internal standard (4.3.3) into the headspace vials containing the food simulant. The solutions thus obtained contain approximately 0 ng/ml, 5 ng/ml, 10 ng/ml, 20 ng/ml, 30 ng/ml and 40 ng/ml of 4-methyl-1-pentene and 50 ng of cyclohexane per millilitre food simulant.

Repeat the procedure with the second set of standard solutions (4.3.4).

6.3.2 Calibration curve for olive oil

Weigh 10 g ± 0,1 g of olive oil into a series of six headspace vials; proceed further as described in 6.3.1. The solutions thus obtained contain approximately 0 ng/g, 5 ng/g, 10 ng/g, 20 ng/g, 30 ng/g and 40 ng/g of 4-methyl-1-pentene and 50 ng of cyclohexane per gram of olive oil.

Repeat the procedure with the second set of standard solutions (4.3.4).

7 Procedure

7.1 Headspace gas chromatographic analysis

7.1.1 General

Examine the baseline stability and response linearity of the detector before starting measurements.

Maintain the same operating conditions throughout the measurement of all samples and calibration solutions.

Inject each solution at least in duplicate, i.e. fill two headspace vials with the same solution for analysis.

NOTE 1 The detector should be able to detect at least 5 ng of 4-methyl-1-pentene per millilitre of simulant at a signal to noise ratio of 3:1.

NOTE 2 Under the conditions given in 5.2 the retention times were 15,2 min and 17,1 min for 4-methyl-1-pentene and cyclohexane, respectively.

7.1.2 Sample treatment and execution of the determination

Place the headspace vials with the samples prepared in 6.1 and 6.2 in the autosampler and analyze the samples using the conditions given in 5.1.

Identify the analyte and internal standard peaks on the basis of their retention times and measure the respective peak heights/areas. Divide the 4-methyl-1-pentene peak height/area by the cyclohexane peak height/area to obtain the peak ratio.

7.2 Calibration

Inject each of the calibration solutions, prepared as described in 6.3.1 and 6.3.2, in duplicate, into the GC column. Measure the peak height or area of 4-methyl-1-pentene and cyclohexane. Divide the 4-methyl-1-pentene peak height/area by the cyclohexane peak/area and plot these peak ratio values against the concentration of 4-methyl-1-pentene in the calibration solution in milligrams per kilogram.

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.

The calibration curves shall be rectilinear and the correlation coefficient shall be 0,996 or better.

The two sets of calibrant solutions made from independently prepared stock solutions shall be cross-checked by generating two calibration curves which on the basis of peak ratio measurement shall agree to within ± 5 % of one another.

7.3 Evaluation of data

NOTE The following calculations assume that for all measurements exactly the same mass or volume of water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol or olive oil has been used and, for the internal standard, that invariably the same volume of internal standard solution has been added.

Following the method described no interferences have been detected.

8 Expression of results

8.1 Calculation of analyte level

8.1.1 Graphical determination

Calculate the average of the peak area/height ratios obtained from the test samples in accordance with 7.1 and read the 4-methyl-1-pentene concentration of the test samples from the calibration graph (7.2).

8.1.2 Calculation from the regression parameters

If the regression line equation is:

$$y = (a \times x) + b$$

where

y is the peak area ratio of 4-methyl-1-pentene/cyclohexane;

a is the slope of the regression line;

x is the concentration of 4-methyl-1-pentene in the food simulant in milligrams per kilogram;

b is the intercept of the regression line,

then the concentration of 4-methyl-1-pentene in the food simulant is given by:

$$C_{MeP,fsd} = \frac{y - b}{a}$$

where

$C_{MeP,fs}$ is the concentration of 4-methyl-1-pentene in milligrams per kilogram.

Both procedures yield directly the 4-methyl-1-pentene concentration in the food simulant in milligrams per kilogram.

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

8.2 Calculation of the specific 4-methyl-1-pentene migration

Depending on the fill volume of the test material and on the surface area/food simulant ratio, the concentration in the laboratory sample as determined in accordance with 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.

8.3 Precision

8.3.1 Validation

This method was pre-validated by a collaborative trial with two laboratories. In each laboratory within-laboratory precision experiments were performed using the four official EC food simulants for establishment of precision data at the restriction criterion as well as migration testing using a

4-methyl-1-pentene containing polymer film sample having been in contact for 10 d at 40 °C with 15 % v/v aqueous ethanol and olive oil.

8.3.2 Repeatability

Evaluation of the within-laboratory precision experiment results according to ISO 5725, at a concentration of 20 µg 4-methyl-1-pentene/kg food simulants, yielded the following performance characteristics on the 95 % probability level (lab 1/lab 2):

Repeatability:

r = 0,5 µg 4-methyl-1-pentene per kilogram water (laboratory 1 only);

r = 1,5/0,92µg 4-methyl-1-pentene per kilogram 3 % v/v aqueous acetic acid;

r = 1,5 µg 4-methyl-1-pentene per kilogram 15 % v/v aqueous ethanol (laboratory 1 only);

r = 1,5/10,8µg 4-methyl-1-pentene per kilogram olive oil.

NOTE The high *r*-value in case of olive oil was due to an occurring GC interference.

8.3.3 Detection limit

For interference-free simulants, the within-laboratory detection limits (WDL), based on the calibration curve method according to DIN 32645, were found to be in the range of 3 µg/kg food simulant. Thus the method is capable of quantitative determination of 4-methyl-1-pentene at a minimum level of 5 µg/kg food simulant.

NOTE Possible interferences with olive oil may influence the WDL value correspondingly.

9 Confirmation

9.1 Requirement for confirmation

If the specific migration of 4-methyl-1-pentene, calculated according to the procedure given in EN 13130-1, from the analyte level calculated according to 8.2 exceeds a restriction, i.e. a specific migration limit of 0,02 mg/kg, the result of the determination shall be confirmed by the method described in 9.2.

The confirmation is qualitative in the sense that it demonstrates the correct identity of the measured analyte and the absence of interferences. For the purposes of quantification the result as calculated according to 8.2 shall be taken as the true value.

9.2 Confirmation by GC/MS

In the selected ion mode, re-analyze the test samples, the blank samples and the calibration samples (6.1 to 6.3). The ions monitored should be m/z = 41,42, 43, 55, 56, 69 and 84 for 4-methyl-1-pentene and m/z = 41, 42, 43, 55, 56, 69, and 84 for cyclohexane. The peak ratio 41/42/43/55/56/69/84 of 4-methyl-1-pentene in the test sample shall be the same as the ratio of the 4-methyl-1-pentene peak in the calibration sample.

NOTE Measured ratios at the specific migration limit were found to be 1,0/0,5/1,5/0,1/0,7/0,2/0,1.

The peaks attributed to 4-methyl-1-pentene and cyclohexane should maximize within one-half peak width (measured at half-height, H/2) or within 2 % of the absolute retention time of standards, whichever is the smaller.

10 Test report

The test report shall include at least the following, where applicable:

a) identification of the sample;

b) name of the laboratory;

c) name of responsible analyst;

d) date of report;

e) date of analysis;

f) analyte;

g) a reference to this method;

h) sample details, such as:

   1) type of food/food simulant/material/article;

   2) origin and denotation of the sample;

   3) date and method of obtaining the laboratory sample;

   4) storage conditions;

i) results expressed in milligrams of 4-methyl-1-pentene per kilogram of food simulant. Results shall be reported as the average value from two or more determinations satisfying the repeatability criterion of 8.3.2;

j) details of confirmation procedure, if any;

k) reasons for modifications introduced into the test method, if any.

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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