DRAFT FOR DEVELOPMENT

DD CEN/TS 13130-15:2005

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

Part 15: Determination of 1,3-butadiene in food simulants

ICS 67.250

British Standards

National foreword

This Draft for Development is the official English language version of CEN/TS 13130-15:2005.

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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TECHNICAL SPECIFICATION

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CEN/TS 13130-15

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English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 15: Determination of 1,3-butadiene in food simulants

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 15: Détermination du 1,3-butadiène dans les simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 15: Bestimmung von 1,3-Butadien 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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Contents

Foreword

This document (CEN/TS 13130-15:2005) 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

1,3-butadiene, C_4H_6 or $CH_2=CH-CH=CH_2$, PM/Ref. No 13630, is a monomer used in the manufacture of certain plastics materials and articles intended to come into contact with foodstuffs. After manufacture, residual butadiene monomer can remain in the polymer and may migrate into foodstuffs coming into contact with that plastics article.

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 a considerable non-reproducible loss of up to 90 %, due to volatilization of 1,3-butadiene can be obtained when using aqueous food simulants.

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

1 Scope

This document, part of EN 13130, specifies an analytical procedure for the determination of butadiene monomer in the food simulants water, 3 % w/v aqueous acetic acid, 15 % v/v aqueous ethanol and olive oil. The level of butadiene monomer determined is expressed as mg butadiene/kg of food. The method is appropriate for the quantitative determination of butadiene at a range of 0,01 mg/kg to 0,1 mg/kg in food simulants.

NOTE The method should also be applicable to other aqueous food simulants and to the other fatty food simulants such as 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 butadiene in a food or a food simulant is determined by headspace gas chromatography (HSGC) with automated sample injection and using flame ionization detection (FID). Quantification is achieved using an internal standard (n-pentane) with calibration against relevant food simulants samples fortified with known amounts of butadiene. Confirmation of butadiene levels 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 1,3-butadiene, $CH_2=CH-CH=CH_2$, purity greater than 99,5 % (GC).

4.1.2 n-pentane, $CH_3-(CH_2)_3-CH_3$, purity greater than 99,5 % (GC).

4.2 Chemicals

4.2.1 N,N-dimethylacetamide (DMAA), $CH_3-CO-N(CH_3)_2$, purity greater than 99,5 % (GC); density $d^T = 0{,}960\,0 - (0{,}000\,94 \times T)$, where T is temperature in °C.

4.2.2 Purified nitrogen (purity 99,999 9 %).

4.3 Solutions

4.3.1 Stock solutions of butadiene in DMAA with defined concentrations of approximately 5mg/g

Weigh a 50 ml sample vial (5.4), including septum and cap, to an accuracy of 1,0 mg. Pipette 50 ml DMAA (4.2.1) into the vial (5.4), close and weigh again to 1,0 mg. Insert hollow needles equipped with miniature two-way valves for introducing butadiene and for venting air and weigh again to an accuracy of 1,0 mg. Introduce, under a fume hood, approximately 0,25 g butadiene (4.1.1) by bubbling through the DMAA with the valves in the open position. Re-weigh the vial plus needles to an accuracy of 1,0 mg with the valves in the closed position and then remove the needles.

NOTE 1 For safety reasons, the use of hollow needles with miniature two-way valves is recommended but not necessary. Alternatively, two separate needles can be used.

Calculate the concentration of butadiene in milligrams per gram of solution.

Repeat the procedure to provide a second stock solution.

NOTE 2 The stock solutions can be stored at – 20 °C up to 3 months protected from light in septum-capped glass vials with minimum headspace; storage at + 4 °C with the exclusion of light should not exceed one week.

4.3.2 Diluted stock solutions of butadiene in DMAA with a defined concentration of approximately 50 µg butadiene per gram DMAA

Weigh a 20 ml sample vial (5.4), including septum and cap, to an accuracy of 1,0 mg. Pipette 18,0 ml of DMAA into the sample vial (5.4) and close the vial with the septum and cap. Weigh again to an accuracy of 1,0 mg. Insert 2,0 ml of the stock solution (4.3.1) through the septum using a syringe (5.5), mix thoroughly and re-weigh to an accuracy of 1,0 mg. Repeat this procedure taking the 1:10 diluted stock solution instead of the stock solution (4.3.1) to obtain then the required 50 µg per gram diluted stock solution.

Calculate the concentration of butadiene in the diluted stock solution in micrograms per gram of solution.

Repeat the procedure to provide a second diluted stock solution.

4.3.3 Standard solutions with defined concentrations in the range 1 µg/ml to 10 µg/ml (see NOTE in 6.3)

Weigh a series of five sample vials (volume 20 ml (5.4)), including septum and cap, to an accuracy of 1,0 mg. Pipette 19,6 ml, 19,2 ml, 18,4 ml, 17,6 ml and 16,0 ml DMAA into the vials, close and weigh again to 1,0 mg. Add 0,4 ml, 0,8 ml, 1,6 ml, 2,4 ml and 4 ml of the diluted stock solution (4.3.2) through the septum using a syringe, mix thoroughly and re-weigh to an accuracy of 1,0 mg.

Calculate the concentrations of butadiene in the standard solutions in micrograms per millilitre of solution and in micrograms per 20 µl solution, using the density according to 4.2.1.

Repeat the procedure using the second diluted stock solution prepared in 4.3.2 to provide a second set of standard solutions.

NOTE The standard solutions can be stored at – 20 °C for up to 3 months protected from light in septum capped glass vials with minimum headspace; storage at + 4 °C with the exclusion of light should not exceed one week.

4.3.4 Internal standard stock solution of n-pentane in DMAA with a defined concentration of approximately 5 mg/g

Weigh a 50 ml sample vial (5.4) including the septum and cap to an accuracy of 1,0 mg. Pipette 50 ml DMAA (4.2.1) into the vial (5.4), close and weigh again to 1,0 mg. Insert 0,4 ml n-pentane (4.1.2) through the septum using a syringe, mix and re-weigh to an accuracy of 1,0 mg.

Calculate the concentration of pentane in the internal standard stock solution in milligrams per gram of solution.

4.3.5 Diluted internal standard stock solution of n-pentane in DMAA with a defined concentration of approximately 50 µg n-pentane per gram DMAA

Weigh a 20 ml sample vial (5.4), including the septum and cap, to an accuracy of 1,0 mg. Pipette 18,0 ml of DMAA into the sample vial (5.4) and close the vial with septum and cap. Weigh again to an accuracy of 1,0 mg. Insert 2,0 ml of the internal standard stock solution (4.3.4) through the septum using a syringe (5.5), mix thoroughly and re-weigh to an accuracy of 1,0 mg. Repeat this procedure taking the 1:10 diluted stock solution instead of the stock solution (4.3.4).

Calculate the concentration of pentane in the diluted stock solution in micrograms per gram of solution.

4.3.6 Internal standard solution of n-pentane in DMAA with a defined concentration of approximately 12,5 µg n-pentane/g DMAA corresponding to 0,25 µg/20 µl

Weigh a 20 ml sample vial (5.4), including septum and cap, to an accuracy of 1,0 mg. Pipette 15,0 ml of DMAA into the sample vial (5.4) and close the vial with the septum and cap. Weigh again to an accuracy of 1,0 mg. Insert 5,0 ml of the diluted internal standard stock solution (4.3.5) through the septum using a syringe (5.5), mix thoroughly and re-weigh to an accuracy of 1,0 mg.

Calculate the concentration of pentane in the internal standard solutions in micrograms per millilitre of solution and in micrograms per 20 µl solution, using density according to 4.2.1.

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 Gas chromatograph, equipped with a flame ionization detector (FID), and fitted with an automatic headspace sampler.

5.2 Gas-chromatographic column, capable of the separation of DMAA from butadiene and n-pentane such that the peaks of butadiene and n-pentane do not overlap by more than 1% peak area with other compounds.

NOTE Depending on the type of gas chromatograph, automatic headspace sampler and column used for the determination, the appropriate operating conditions have to be established.

The following are examples of GC columns known to be suitable for butadiene analysis:

10 m x 0,32 mm ID PLOT (Porous Layer Open Tubular) fused silica capillary column with a 10 µm thick layer of porous styrene-divinylbenzene polymer.

Pre-column 1 m x 2 mm packed with porous styrene-divinylbenzene polymer 100/150 mesh, coupled with two packed columns in series where each column of the dimensions 2 m x 2 mm is packed with graphitized carbon black 80/100 mesh containing with 0,19 % picric acid.

30 m x 0,53 mm ID megabore gas-solid capillary column with ethylvinylbenzene-divinylbenzene polymer.

For guidance, the operating conditions established for column a) were the following:

Headspace sampler:	
Equilibration time:	30 min
Sample temperature:	70 °C
Transfer line:	120 °C
Sampling time:	12 s

In case of insufficient detection sensitivity the sample temperature during equilibration may be raised to 80 °C or even to 90 °C.

Gas chromatograph:	
Injector:	220 °C
Oven program:	70 °C (5 min), 8 °C/min to 220 °C (15 min)
Detector (FID):	250 °C
Carrier gas:	Helium at 25 kPa inlet pressure and 33 cm/s linear velocity
Injection mode:	split (ratio = 1:10)
FID gases:	to be optimized according to the manufacturer's specifications

5.3 Glass sample vials, 20 ml, or of another size suitable for the particular autosampler employed, with PTFE-coated butyl or silicone 20 ml rubber septa and crimp-closures.

5.4 Glass sample vials, 50 ml, 20 ml and 10 ml with closures as in 5.3.

5.5 Microsyringes, 50 µl and syringes, 2 ml.

6 Samples

6.1 Preparation of test samples

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. Butadiene-free samples of the same type as those to be analyzed shall also be prepared for use for calibration purposes.

NOTE Since the determination of butadiene in food simulants is performed only slightly above the detection limit of the method, extreme care should be taken with respect to possible adventitious contamination during preparation of the test samples.

The following precautions are advisable:

a) purge the empty sample vials (5.3) with purified nitrogen (4.2.2) before filling with food simulant;

b) carry out the migration test procedure and the preparation of the food simulant sub-samples in a different laboratory to that used for handling butadiene and n-pentane solutions, to avoid cross-contamination by volatilization.

Migration testing (see EN 13130-1) has to take account of possible loss of analyte in aqueous food simulants (see NOTE in the Introduction).

Place 2,0 ml of the food simulant obtained from the migration experiment into a sample vial (5.3) using a 2 ml syringe (5.5). Add 20 µl DMAA (4.2.1) and close the vial with septum and cap. Add 20 µl n-pentane internal standard solution (4.3.6) to the food simulant by injection through the septum using the 50 µl syringe (5.4).

Prepare each test sample at least in duplicate.

6.2 Preparation of blank samples

Place 2,0 ml of butadiene-free food simulant into a sample vial (5.3) using a 2 ml syringe (5.5). Add 40 µl DMAA (4.2.1) and close the vial with the septum and cap.

Prepare blank samples at least in duplicate.

6.3 Preparation of calibration samples

Place 2,0 ml of butadiene-free food simulant into a series of 5 sample vials (5.3) using a 2 ml syringe (5.5) and close the vials with the septum and cap. Add into each vial 20 µl of one of the five standard solutions (4.3.3) through the septum using a 50 µl syringe (5.5). Then add into each vial 20 µl n-pentane internal standard solution (4.3.6) in the same way.

Prepare each calibration sample at least in duplicate.

To ensure that the calibration range has a reasonable order of magnitude compared to the sample concentrations, the calibration solutions shall be prepared at five levels spanning approximately 0,5 to 5 times (0,5, 1, 2, 3 and 5) the restriction criteria, e.g. the specific migration limit (SML-value) is 0,020 mg/kg. This means that 20 µl of the lowest (highest) concentrated standard solution shall contain not less (more) than 0,02 (0,2) µg butadiene.

7 Procedure

7.1 GC analysis

7.1.1 General

When starting measurements, baseline stability and response linearity of the detector shall be examined.

The same operating conditions of the HSGC system shall be maintained throughout the measurement of all samples prepared in 6.1 to 6.3.

Each sample shall be determined at least in duplicate, i.e. as a pair of measurements.

NOTE Under the conditions given in 6.2 the retention times of butadiene and n-pentane were 15 min and 24 min, respectively.

7.1.2 Sample treatment

The test samples, blanks and calibration samples prepared in 6.1 to 6.3, shall be analyzed as they are without any further sample treatment.

7.1.3 Execution of determination

Equilibrate the sample solutions in the thermostat of the automated headspace sampler before sampling and commencing the analysis programme (see NOTE in 6.1).

Identify the butadiene and n-pentane peaks on the basis of their retention times and measure the respective peak areas.

Divide the butadiene peak area by the n-pentane peak area to obtain the peak area ratio (PAR).

7.2 Calibration

Calibration samples (6.3) shall be measured as described in 7.1. Construct or calculate the calibration curve plotting PAR values against the concentration of butadiene in the food simulant in milligrams per kilogram.

NOTE Commission Directive 90/128/EEC [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 area measurement, shall agree to within ± 5 % of one another.

7.3 GC interferences

Following the method described above, no interferences have been observed.

8 Expression of results

8.1 Determination of butadiene concentration in the test samples

NOTE The following calculations assume that for all measurements exactly the same volume or mass of sample solution, 2 ml of food simulant, has been used and, for the internal standard, that the same volume of standard solution, i.e. same mass of internal standard, has been added.

8.1.1 Graphical determination

Calculate the average of PAR values obtained from the test samples according to 7.1 and read the butadiene concentration of the test sample from the calibration graph (7.2).

8.1.2 Calculation from the regression parameters

If the regression line equation is:

$$y[PAR] = (a \times X) + b$$

where

$y[PAR]$ is the butadiene/n-pentane peak area ratio;

a is the slope of the regression line in milligrams per kilogram;

X is the concentration of butadiene in the food simulant in milligrams per kilogram;

b is the intercept of the regression line,

the butadiene concentration in the food simulant $C_{\text{But,fs}}$ is given by:

$$\mathrm{C}_{\text{But,fs}} = \frac{y-b}{a}$$

where

$C_{But,fs}$ is the concentration in of butadiene in the food simulant in milligrams per kilogram.

Both procedures yield directly the butadiene concentration in the food simulant in milligrams per kilogram.

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

8.1.3 Calculation of the specific butadiene migration

Depending on the fill volume of the test material and on the surface area/food simulant ratio, the butadiene concentration in the test sample, as determined according to 8.1.1 or 8.1.2, 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-validated by a collaborative trial with three laboratories; in each laboratory a within-laboratory precision experiment using the four official EC food simulants for establishment of precision data at the restriction criterion was carried out as well as migration testing using an ABS copolymer sample being in contact with 15 % ethanol and olive oil, respectively.

8.2.2 Repeatability

Evaluation of the within-laboratory precision experiment results according to ISO 5725, at a concentration of 23 µg/l, yielded the following performance characteristics at the 95 % probability level (lab 1/lab 2/lab 3):

Repeatability: r = 4,0/5,1/3,4 µg butadiene per kilogram of water;

r = 5,9/4,1/1,0 µg butadiene per kilogram of 3 % w/v aqueous acetic acid;

r = 4,5/6,0/1,7 µg butadiene per kilogram of 15 % v/v aqueous ethanol;

r = 3,5/6,4/0,9 µg butadiene per kilogram of olive oil.

8.2.3 Detection limit

The within-laboratory detection limits (WDL), based on the calibration curve method according to DIN 32645, were found to be in the range 4 µg/kg to 9 µg butadiene per kilogram of food simulant, regardless to the type of food simulant. Thus the method is capable of quantitative detection at a minimum value of 9 µg butadiene per kilogram of food simulant.

9 Confirmation

9.1 Requirement for confirmation

If the specific migration of butadiene into the food simulant, determined in accordance with 8.1.1 or 8.1.2, exceeds the restriction criterion, e.g. the specific migration limit is not detectable at a detection limit of 0,02 mg/kg, the result of the determination shall be confirmed by the method described in 9.2.

9.2 Confirmation by combined gas chromatography/mass spectrometry

In the selected ion mode, re-analyze the test sample(s) (6.1), the calibration samples (6.2) and the blank sample (6.3). The ions monitored should be m/z 39, 53 and 54 for butadiene and m/z 72 and 57 for n-pentane. The peak ratio 53/54 of the butadiene in the test sample has to be the same as the ratio of the butadiene peaks in the calibration sample. Measured ratios at the SML-concentration were found to be 0,70/1 to 0,90/1. The peaks attributed to butadiene and pentane 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.

The confirmation is qualitative in the sense that it demonstrates the correct identity of the measured peak and the absence of interferences. If the GC/MS analysis clearly indicates the absence of interferences, the result calculated according to Clause 8.1.1or 8.1.2 shall be the true value. If there are interferences, the peak area ratios of the specified ions should be used to calculate the relevant butadiene level in the food simulant.

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 1,3-butadiene per kilogram of food simulant.

Bibliography

[1] Commission of the European Communities, Commission Directive of 23 February 1990 relating to plastics materials and articles intended to come into contact with foodstuffs (90/128/EEC), Official Journal of the European Communities, 13 December 1990, no. L349, p26. Corrigendum of the previous publication, Official Journal of the European Communities, 21 March 1990, no. 75. p19.

[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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