DRAFT FOR DEVELOPMENT

DD CEN/TS 13130-9:2005

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

Part 9: Determination of acetic acid, vinyl ester in food simulants

ICS 67.250

British Standards

National foreword

This Draft for Development is the English language version of CEN/TS 13130-9:2005.

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 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.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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.

The BSI copyright date displayed in this document indicates when the document was last issued.

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TECHNICAL SPECIFICATION

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English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 9: Determination of acetic acid, vinyl ester in food simulants

Matériaux et objets en contact avec des denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 9 : Détermination du vinyl ester d'acide acétique dans les simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 9: Bestimmung von Essigsäurevinylester 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 13130-9:2005) 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

Acetic acid, vinyl ester (vinyl acetate) $CH_3CO_2CH=CH_2$, PM/Ref. No 10120, is a monomer used in the manufacture of certain materials and articles intended to come into contact with foodstuffs. After manufacture, residual vinyl acetate can remain in the polymer and may migrate into foodstuffs coming into contact with the plastic material or product.

NOTE The following should be taken into account when carrying out a migration test. Vinyl acetate is known to hydrolyze in aqueous media. In acetic acid solutions the hydrolysis is almost complete after 10 d at 40 °C. Vinyl acetate is stable in olive oil.

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

1 Scope

This document, part of EN 13130, specifies an analytical procedure for the determination of vinyl acetate in the four conventional EU food simulants, water, 10 % (v/v) ethanol aqueous solution; 3 % (w/v) acetic acid aqueous solution and olive oil or an approved substitute. The level of vinyl acetate monomer determined is expressed as milligrams of vinyl acetate per kilogram of food simulant. The method is appropriate for the quantitative determination of vinyl acetate in approximate analyte concentration range of 1,2 mg/kg to 24 mg/kg food simulant.

NOTE 1 The method should also be applicable to other aqueous food simulants as well as to other fatty food simulants such as sunflower oil and a mixture of synthetic triglycerides.

NOTE 2 The suitability of the fat simulant should be assessed prior to setting up migration tests - it may be found necessary to use sunflower oil or a mixture of synthetic triglycerides if unacceptable interferences are found with olive oil.

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 vinyl acetate in food simulants is determined by headspace capillary gas chromatography, using flame ionization detection. Quantification is achieved using propionic acid, methyl ester (methyl propionate), as an internal standard with calibration against relevant food simulants samples, fortified with known amounts of vinyl acetate.

Confirmation of vinyl acetate levels is carried out by combined headspace gas chromatography/mass spectrometry, or by repeating the analytical procedure using a gas chromatograph capillary column of different polarity.

If automated headspace sampling cannot be performed, manual injection may be used ensuring that the gas syringe and needle are preheated to the same temperature as the headspace vials before the headspace is sampled.

4 Reagents

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

4.1 Analytes

4.1.1 Vinyl acetate, $CH_3CO_2CH=CH_2$, purity greater than 99 %.

4.1.2 Methyl propionate, $CH_3CH_2CO_2CH_3$, purity greater than 99 %, which is free of interfering substances which elute at the same retention time as vinyl acetate.

4.2 Chemicals

4.2.1 Water, HPLC grade.

4.2.2 N,N-dimethylacetamide (99,9 %)

4.3 Solutions

4.3.1 Stock solution of vinyl acetate in dimethylacetamide (6 mg/ml)

Weigh accurately approximately 0,15 g of the vinyl acetate into a tared stoppered 25 ml volumetric flask containing about 10 ml of dimethylacetamide, and dilute to the mark with dimethylacetamide. Calculate the exact concentration of vinyl acetate in milligrams per litre.

Repeat the procedure to provide a second standard stock solution.

NOTE The stock solutions can be stored in a well closed containers in the dark for a maximum period of three months at any temperature between 5 °C and 20 °C.

4.3.2 Internal standard stock solution of propionic acid, methyl ester (methyl propionate) in dimethylacetamide (6 mg/ml)

Weigh approximately 0,6 g of propionic acid methyl ester (methyl propionate), into a tared stoppered 100 ml volumetric flask containing about 20 ml of dimethylacetamide, and dilute to the mark with dimethylacetamide.

NOTE The stock solutions can be stored in well closed containers in the dark for a maximum period of three months at any temperature between 5 °C and 20 °C.

4.3.3 Standard solutions of vinyl acetate in dimethylacetamide

Into six 25 ml volumetric flasks, each containing approximately 5 ml of dimethylacetamide, add by pipette 0 ml, 0,5 ml, 1,0 ml, 2,0 ml, 3,0 ml and 5,0 ml of the stock solution (3.3.1). Pipette in 5,0 ml of internal standard solution (4.3.2). Dilute to the mark with dimethylacetamide to give standard solutions containing a nominal concentration of 0 mg/l, 120 mg/l, 240 mg/l, 480 mg/l, 720 mg/l and 1200 mg/l vinyl acetate. Shake thoroughly to mix.

Calculate the exact concentrations of the standard solutions in milligrams per litre.

Repeat the procedure using the second stock solution prepared in 4.3.1 to give a second set of standard solutions.

NOTE The standard solutions can be stored in well closed containers in the dark for a maximum period of three months at any temperature between 5 °C and 20 °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, fitted with a capillary column capable of resolving vinyl acetate and the internal standard from each other and other compounds present in the food simulants.

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

NOTE The following chromatographic column and conditions have been found to be satisfactory.

System 1

GC analytical column and temperature program for ethanol, water and olive oil food simulants.

Column:	25 m x 0,32 I.D dimethylsiloxane 5 µm film thickness
Temperature program:	50 °C held for 1 min, raised to 90 °C at 5 °C/min raised at 30 °C/min to 120 °C, held for 5 min.
Carrier gas:	Helium 2 ml/min.
Carrier gas split ratio	1 : 10
Run time:	20 min
Flame ionization detector	200 °C
Injector:	125 °C

System 2

GC analytical column and temperature program for 3 % w/v aqueous acetic acid food simulant.

Column:	25 m x 0,32 I.D polyethylene glycol 1 µm film thickness.
Temperature program:	54 °C held for 2 min, raised to 56 °C at 0,3 °C/min, raised to 200 °C at 30 °C/min, held for 5 min.
Carrier gas:	Helium 2 ml/min
Carrier gas split ratio:	1: 10
Run time:	30 min
Detector:	FID 200 °C
Injector:	125 °C

Headspace sampling

If an automatic sampling machine is not available to heat the samples and inject the resulting headspace into the chromatograph, a water bath and stopwatch may be used to perform these operations manually. The following GC headspace sampling conditions have been found to be satisfactory.

Headspace auto sampler conditions

	Aqueous simulants	Fat simulants
Sample vial temperature	80 °C	90 °C
Needle temperature	80 °C	90 °C
Transfer temperature	125 °C	125 °C
Thermostat time	15 min	30 min
Pressurization time	3 min	3 min
Injection time	0,1 min	0,1 min
Needle withdrawal time	0,1 min	0,1 min

Manual headspace sampling conditions

The head space should be sampled using a preheated glass gas syringe (10 ml) fitted with a hypodermic needle.

	Aqueous simulants	Fat simulants
Sample vial temperatures	80 °C	90 °C
Thermostat time	15 min	30 min
Injection volume	2 ml	2 ml
Typical retention times		
	System 1	System 2
vinyl acetate	4 min	4,0 min
methyl propionate	6 min	4,2 min
dimethylacetamide	14 min	12,6 min

5.2 20 ml glass headspace vials

5.3 Seals, polytetrafluoroethylene coated silicone or butyl rubber, and crimp on caps.

5.4 Syringe, 100 µl fitted with a hypodermic needle and a constant volume, dispensing adapter, calibrated to deliver 100 µl or a 100 µl micro syringe.

5.5 Crimping pliers, for sealing the headspace vials.

6 Samples

6.1 Preparation of test samples

6.1.1 General

The laboratory samples of food simulant to be analyzed shall be obtained as described in EN 13130-1.

NOTE Aqueous samples should ideally be analyzed as soon as possible after completion of the migration experiment (see EN 13130-1). Fat simulants may be stored for up to 10 d in a refrigerator at + 5 °C in completely filled and sealed glass containers.

From each sample, blank or calibration solution, at least duplicate vials shall be prepared, e.g. as a pair of measurements. For vinyl acetate, free samples of simulant of the same type as those to be analyzed are also required for calibration purposes.

Migration testing shall take account of possible loss of analyte in aqueous food simulants (see NOTE in the Introduction).

6.1.2 Aqueous food simulants

Transfer 5 ml of the aqueous food simulant obtained from the migration experiment (see EN 13130-1), into an empty, sealed headspace vial, using a 5 ml syringe fitted with a hypodermic syringe, taking care to avoid an air gap or vacuum in the syringe barrel. Inject 100 µl of the standard solution containing no vinyl acetate (4.3.3) using the 100 µl syringe (5.4).

6.1.3 Analysis of olive oil and other fat simulant samples

Transfer 5,00 g of the fat simulant obtained from the migration experiment (see EN 13130-1) into a headspace vial using a 10 ml syringe. Crimp seal the vial and inject 100 µl of the standard solution containing no vinyl acetate (4.3.3) using a 100 µl syringe (5.4). Mix thoroughly.

6.2 Preparation of blank samples

Proceed as described in 6.1.2 and 6.1.3, using vinyl acetate-free food simulant.

6.3 Preparation of calibration samples

6.3.1 Aqueous food simulants

NOTE 1 These solutions should be prepared immediately prior to analysis.

Pipette into six headspace vials 5,0 ml of the blank simulant. Crimp seal the vials. Inject into the vials 100 µl of the standard solutions (4.3.3) using the 100 µl syringe (5.4), to give nominal concentrations of 0 mg/l, 2,4 mg/l, 4,8 mg/l, 9,6 mg/l, 14,4 mg/l and 24,0 mg vinyl acetate per litre of food simulant. Shake the flasks to mix the solutions thoroughly.

Calculate the exact concentrations of vinyl acetate in the calibration samples in milligrams per litre.

NOTE 2 Assuming a density of 1,0 this gives a calibration graph in milligrams per kilogram.

NOTE 3 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.

Repeat the procedure using the second set of standard solutions prepared in 4.3.3 to give a second set of calibration samples.

6.3.2 Fat simulants

Weigh accurately 5,00 g of the fat simulant into separate headspace vials. Crimp seal the vials. Inject into each vial 100 µl of the intermediate standards (4.3.3) using the 100 µl syringe (5.4), to give nominal concentrations of 0 mg/kg, 2,4 mg/kg, 4,8 mg/kg, 9,6 mg/kg, 14,4 mg/kg and 24,0 mg vinyl acetate per kilogram of fat simulant. Shake the vials well to thoroughly mix the solutions.

Calculate the exact concentrations of vinyl acetate in the calibration samples in milligrams per kilogram.

Repeat the procedure using the second set of standard solutions prepared in 4.3.3 to give a second set of calibration samples.

7 Procedure

7.1 Headspace capillary GC analysis

Examine the baseline stability and response linearity of the detector when starting GC headspace measurements.

Maintain the same headspace GC operating conditions throughout the measurement of all samples prepared as described in 6.1 to 6.3.

7.2 Sample treatment

Heat the vials containing the test samples (6.1) and blanks (6.2) for the specified time period, and then inject a suitable portion of the headspace into the gas chromatograph.

7.3 Calibration

Heat the vials containing the first set of calibration samples (6.3) for the specified time period, and then inject a suitable portion of the headspace into the gas chromatograph. Obtain the integrated peak areas of the vinyl acetate and the internal standard. Calculate the peak area ratio for each of the calibration standards, by dividing the vinyl acetate peak area by the internal standard peak area. Construct the calibration line by plotting the mean of the duplicate peak area ratios (PAR) against the concentration of vinyl acetate in the calibration samples in milligrams per kilogram.

Repeat for the second set of calibration samples (6.3) to obtain a second calibration line.

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 agree to ± 5 % of one another.

If the correlation coefficients are less than 0,996, fresh intermediate standard solutions and calibration solutions shall be prepared from the original stock solutions.

If the levels of the two independently prepared stock solutions do not correspond to within ± 5 %, both stock solutions and standard solutions shall be discarded, fresh stock solutions of vinyl acetate shall be prepared and the entire procedure repeated from the beginning.

7.4 Evaluation of data

NOTE The following calculations assume that for all measurements exactly the same weight or volume of food simulant has been used, and that the same volume of internal standard, has been added.

If the GC chromatogram of the blank sample (6.2) shows an interfering peak in the vinyl acetate region, use different chromatographic conditions and/or use alternative food simulants, e.g. sunflower oil or a mixture of synthetic triglycerides, instead of olive oil.

NOTE No interferences arising from either of the four conventional EU food simulants, have so far been observed using the method and the headspace GC conditions specified in (4.1).

8 Expression of results

8.1 Calculation of vinyl acetate concentration in the test samples

8.1.1 Calculation from the calibration graph:

Calculate the mean of the peak area ratio values obtained from the two duplicate test samples and read the corresponding vinyl acetate concentration from the calibration graph (7.3).

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 peak area ratio of vinyl acetate/internal standard;

a is the slope of the regression line in kilograms per milligram;

x is the concentration of vinyl acetate in the food simulant in milligrams per kilogram;

b is the intercept of the regression line,

then the concentration of vinyl acetate in the food simulant, is given by:

$$C_{Aafs} = \frac{v - b}{a}$$

where

C_{Vafs} is the concentration of vinyl acetate in milligrams per kilogram.

Both procedures directly yield the concentration of vinyl acetate in the food simulant in milligrams per kilogram.

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

8.1.3 Calculation of the specific vinyl acetate migration

Depending on the fill volume of the test material and on the surface area/food simulant ratio, the vinyl acetate concentration in the test sample as determined according to 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

The method was validated by a within-laboratory precision experiment using the four official EU food simulants for establishment of precision data at the restriction criterion as well as by carrying out within-laboratory migration tests at 40 °C for 10 d with a vinyl acetate-based polymer coating in contact with 15 % ethanol and with olive oil.

8.2.2 Repeatability

Evaluation of the three within-laboratory precision experiment results, in accordance ISO 5725, at a vinyl acetate concentration of 12 mg/kg, yielded the following performance characteristics at the 95 % probability level (lab 1/lab 2/lab3):

Repeatability r = 1,3/1,4/0,7 mg vinyl acetate per kilogram in water;

r = 1,1/0,9/0,3 mg vinyl acetate per kilogram in 3 % w/v aqueous acetic acid;

r = 0,5/0,5/0,7 mg vinyl acetate per kilogram in 15 % v/v aqueous ethanol;

r = 1,0/0,3/0,4 mg vinyl acetate per kilogram in olive oil.

8.2.3 Detection limit

The within-laboratory detection limit (WDL), calculated from the calibration line method in accordance with DIN 32645 was found to be in the range of 0,3 mg to 1,1 mg vinyl acetate per kilogram of food simulant, depending on the type of food simulant. Thus the method is capable of quantitative determination of vinyl acetate at a minimum level of 1,1 mg/kg of food simulant.

9 Confirmation

9.1 Requirements

If the specific migration of vinyl acetate into the food simulant, determined as described in 8.1.3, exceeds a restriction criterion, e.g. a specific migration limit of 12 mg/kg, the determination shall be confirmed by either of the methods described in 9.2 and 9.3.

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 8.1.3 shall be taken as the true value.

9.2 Confirmation of vinyl acetate using gas chromatography/mass spectrometry

In the selective ion monitoring mode, re-analyze all the calibration, blanks and sample solutions, by transferring fresh aliquots into headspace vials, and injecting the headspace into the gas chromatograph. The ions monitored should be the molecular ion peaks of the vinyl acetate and internal standard (86/88 m/z) and the base ion peak for vinyl acetate and internal standard (43/57 m/z). Abundance ratios should be calculated for the most prominent ions in the spectra of standards and samples. Abundance ratios for samples should agree to within ± 5 % of ratios of standards. No ions with a relative abundance of > 5 % should be present in the samples which are not present in the standards.

NOTE 1 If this equipment is available then this procedure should be performed in preference to the procedure described in 8.3.

The peaks attributable to vinyl acetate and internal standard shall maximize within one half peak width (measured at half the peak height) or within 2 % of the absolute retention time of the standards, whichever is the smaller.

The peak area ratio 43/86 m/z for vinyl acetate in the test samples shall be the same as the peak area ratio 43/86 of vinyl acetate in the calibration samples.

NOTE 2 A peak area ratio 43/86 m/z for vinyl acetate of 8,3 ± 0,4 was obtained from calibration samples in all four simulants.

9.3 Confirmation of vinyl acetate using a GC analytical column of different polarity stationary phase

If the equipment as described in 8.2 is not available, the following confirmation procedure shall be carried out.

Select the analytical GC column of different polarity. Re-analyze all the calibration and sample solutions by transferring fresh aliquots into headspace vials and injecting the headspace into the gas chromatograph. For each GC column selected, the peaks attributable to vinyl acetate and the internal standard, should maximize within one half peak width (measured at half the peak height), or within 2 % of the absolute retention time of the standards, whichever is the smaller.

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 vinyl acetate 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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