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

Part 6: Determination of vinylidene chloride in plastics

The European Standard EN 13130-6:2004 has the status of a British Standard

ICS 67.250

British Standards

National foreword

This British Standard is the official English language version of EN 13130-6:2004. It supersedes DD ENV 13130-6:1999 which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee CW/47, Materials and articles in contact with foodstuffs, to Subcommittee CW/47/1, Migration from plastics, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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

Cross-references

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Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 6: Determination of vinylidene chloride in plastics

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Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 6: Bestimmung von Vinylidenchlorid in Kunststoffen

This European Standard was approved by CEN on 24 March 2004.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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Contents

Foreword

This document (EN 13130-6:2004) has been prepared by Technical Committee CEN/TC 194 “Utensils in contact with food”, the secretariat of which is held by BSI.

This document was prepared by Subcommittee SC1 of TC 194 as one of a series of analytical test methods for plastics materials and articles in contact with foodstuffs.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2004, and conflicting national standards shall be withdrawn at the latest by November 2004.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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.

EN 13130-6 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 other 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 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-trimethylopropane in food simulants*

Parts 1 to 8 are European Standards.

Parts 9 to 28 are Technical Specifications, prepared within the Standards, Measurement and Testing project, MAT1-CT92-0006, "*Development of Methods of Analysis for Monomers*".

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: 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

Vinylidene chloride (1,1-dichloroethylene), $H_2C{=}CCl_2$, is a monomer used in the manufacture of certain plastics films and coatings intended to come into contact with foodstuffs. During the manufacture of vinylidene chloride polymers and copolymers, residual vinylidene chloride monomer can remain in the polymer and can migrate into food coming into contact with the polymer.

The method described in this European Standard should be used in conjunction with part 1 of this standard, which describes the procedures required prior to the determination of vinylidene chloride.

1 Scope

This part of this European Standard specifies a method for the determination of vinylidene chloride (VdC) in plastics materials and articles.

The method is applicable to poly(vinylidene chloride) (PVdC) films, PVdC coated films, and laminates and coextruded materials containing PVdC. The level of vinylidene chloride determined is expressed as milligrams of vinylidene chloride per kilogram of polymer. The method is appropriate for the quantitative determination of vinylidene chloride at a level of 5 mg/kg in plastics materials and articles.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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 vinylidene chloride in a polymer is determined by headspace gas chromatography with automated sample injection, using either electron capture detection (ECD) or flame ionization detection (FID). As it is not possible to obtain test material free of vinylidene chloride, the method of standard addition is employed. Quantification is achieved using 1-chloropropane (1-CP) as an internal standard.

Plastics samples are dissolved in N,N-dimethylacetamide (DMA) prior to headspace analysis. For samples not soluble in this solvent, e.g. laminates and co-extrusions, determination can be carried out by the 'hot jar' method in which the polymer alone is heated to release the vinylidene chloride.

If interferences are experienced with the internal standard then calibration is carried out by external standardization.

If automated headspace sampling cannot be performed then manual injection as described in annex A shall be applied.

Confirmation of vinylidene chloride levels is carried out either by combined gas chromatography/mass spectrometry (GC/MS) or by re-analysis on a second GC column of different polarity.

4 Reagents

WARNING: All chemicals are hazardous to health to a greater or lesser extent. It is beyond the scope of this standard to give instructions for the safe handling of all chemicals, that meet, in full, the legal obligations in all countries in which this standard may be followed. Therefore, specific warnings are not given and users of this standard shall ensure that they meet all the necessary safety requirements in their own country.

4.1 Vinylidene chloride, $H_2C=CCl_2$, of purity greater than 99 % (w/w).

4.2 1-Chloropropane, $CH_3(CH_2)_2Cl$, containing no impurity > 1 % by area which will elute at the same GC retention time as vinylidene chloride.

4.3 N,N-Dimethylacetamide $CH_3CON(CH_3)_2$ free of any interferences (< 1 % area) with the vinylidene chloride and 1-chloropropane peaks.

4.4 Standard solutions of vinylidene chloride in N,N-dimethylacetamide with defined concentrations in the range 25 mg/kg to 500 mg/kg, prepared as described in 4.4.1 and 4.4.2.

4.4.1 Prepare concentrated standard vinylidene chloride solutions at approximately 2,5 g/kg and 1,2 g/kg as follows:

a) To a 50 ml flask or vial tared with cap, add approximately 50 ml (46,5 g) N,N-dimethylacetamide (4.3), close and weigh to an accuracy of 0,1 mg. Add to the N,N-dimethylacetamide a quantity of approximately 0,1 ml (0,125 g) vinylidene chloride (4.1) and shake the closed flask. Determine the exact mass of vinylidene chloride added, in grams per kilogram, by re-weighing to an accuracy of 0,1 mg.

b) Repeat item a) using 0,05 ml (0,06 g) vinylidene chloride.

c) Repeat items a) and b) to provide a second set of concentrated standard solutions.

4.4.2 Prepare dilute standard vinylidene chloride solutions as follows;

a) To two 20 ml flasks or vials tared with cap, add 18 ml and 16 ml N,N-dimethylacetamide and weigh to an accuracy of 0,1 mg. Add 2 ml of the 2,5 g/kg concentrated standard solution to the first flask or vial and 4 ml to the second. Cap, re-weigh to an accuracy of 0,1 mg and shake thoroughly.

b) Repeat item a) using 19,6 ml and 18 ml dimethylacetamide and 0,4 ml and 2 ml of the 1,25 g/kg standard. The dilute standard solutions shall be stored in suitable glass septum-capped vials with minimal headspace volume.

c) Repeat items a) and b) using the second solutions prepared in 4.4.1 to provide a second set of four dilute standard vinylidene chloride solutions.

NOTE The standard solutions with known vinylidene chloride concentrations of approximately 25 mg/kg, 125 mg/kg, 250 mg/kg and 500 mg/kg, respectively can be stored at - 20 °C for up to four weeks, protected from light.

4.5 Solution of 1-chloropropane (4.2) in N,N-dimethylacetamide at approximately 0,4 g/kg

4.5.1 Prepare a concentrated standard 1-chloropropane solution at approximately 2,0 g/kg as follows:

To a 50 ml flask or vial tared with cap, add 50 ml of dimethylacetamide. Close and weigh to an accuracy of 0,1 mg. Add to the dimethylacetamide, approximately 0,1 ml (0,090 g) of 1-chloropropane and shake the closed flask or vial. Determine the exact mass of 1-chloropropane added by re-weighing to an accuracy of 0,1 mg.

4.5.2 Prepare a dilute standard 1-chloropropane solution at approximately 0,4 g/kg as follows:

To a 20 ml flask or vial tared with cap, add 16 ml N,N-dimethylacetamide and weigh to an accuracy of 0,1 mg. Add 4 ml of 1-chloropropane solution, as prepared in 4.5.1. Close, re-weigh to an accuracy of 0,1 mg and shake thoroughly to mix.

5 Apparatus

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

5.1 Gas-chromatograph, equipped with either an electron capture detector or a flame ionization detector and fitted with an automated headspace sampler.

5.2 Gas-chromatographic column, capable of the separation of N,N-dimethylacetamide from vinylidene chloride and the internal standard such that the peaks of vinylidene chloride and the internal standard do not overlap by more than 1 % peak area with each other and with other compounds.

NOTE The following are examples of GC columns found to be suitable for vinylidene chloride analysis:

a) 2 m x 2 mm internal diameter stainless steel column packed with n-octane chemically bonded to 100 mesh to 120 mesh uncoated silica beads;

b) 2,8 m x 2 mm internal diameter glass column packed with 80 mesh to 100 mesh cross linked porous polymer;

c) 50 m x 0,32 mm internal diameter fused silica capillary column with 1,2 µm film thickness of 100 % dimethylsiloxane;

d) 50 m x 0,32 mm internal diameter fused silica capillary column with 1,2 µm film thickness of 85 % dimethyl-, 7 % cyanopropyl- 7 % phenyl-, 1 % vinyl-siloxane;

e) 50 m x 0,53 mm internal diameter fused silica capillary column with 5,0 µm film thickness of 100 % dimethylsiloxane.

5.3 Glass sample vials, of size suitable for the particular autosampler employed, with polytetrafluoroethylene faced silicone rubber septa and crimp-closures.

5.4 Microsyringe, of 50 µl capacity.

6 Samples

6.1 Sample storage

Keep the samples refrigerated at - 20 °C enclosed in several layers of non-lacquered aluminium foil.

6.2 Test sample preparation

6.2.1 General

The following precautions are advisable:

a) To avoid cross-contamination by volatilization, carry out preparation of the polymer samples in an area remote to that used for handling vinylidene chloride and the 1-chloropropane solutions;

b) To avoid loss of standard solutions to the septum when making additions, add solutions directly to the polymer dissolved in N,N-dimethylacetamide contained within the vial, rather than injecting them through the septum;

c) To reduce volatilization, add internal standard and vinylidene chloride to dissolved polymer samples as quickly as possible, injecting solutions into the solvent and sealing vials immediately after addition.

Polymer samples that dissolve in N,N-dimethylacetamide are treated as in 6.2.2. Samples not soluble in this solvent are treated as in 6.2.4.

6.2.2 Preparation of dissolved test sample solutions and standard addition solutions

6.2.2.1 General

Use a representative sample of the test material or article cut into small pieces. To ten vials (5.3) tared with caps, add 0,2 g ± 0,005 g of sample and weigh to an accuracy of 0,1 mg.

6.2.2.2 Prepare the test sample solutions as follows:

a) To one of the vials prepared in 6.2.2, add 4 ml of N,N-dimethylacetamide and weigh. Add 20 µl of dilute 1-chloropropane internal standard solution (4.5.2) and reweigh. Seal the vial.

b) Repeat item a) to provide a duplicate test sample solution.

6.2.2.3 Prepare the sample addition solutions as follows:

a) To one of the vials prepared in 6.2.2, add 4 ml of N,N-dimethylacetamide. Then, weighing after each addition, add 20 µl of dilute 1-chloropropane internal standard solution (4.5.2) and 20 µl of the 25 mg/kg (4.4.2) dilute standard vinylidene chloride solution. Seal the vial.

b) Repeat item a) to provide a duplicate sample.

c) Repeat item a) using the 125 mg/kg, 250 mg/kg and 500 mg/kg dilute vinylidene chloride standard solutions, preparing duplicate samples.

A second set of samples and standard additions shall be prepared using the second set of dilute vinylidene chloride standard solutions.

Calculate the concentration of vinylidene chloride and 1-chloropropane, in milligrams per kilogram of polymer sample. Place the vials in either an ultrasonic bath or on an orbital shaker for 2 h to dissolve vinylidene chloride polymer.

WARNING: It is important that the mass of sample does not exceed the limit specified as this is a standard addition method. If sample masses are not kept constant errors will be introduced in the individual points of the standard addition graph.

6.2.3 Preparation of blank samples

6.2.3.1 To a vial (5.3) add 4 ml N,N-dimethylacetamide and 20 µl of dilute 1-chloropropane internal standard solution. Seal vial with cap and septum.

6.2.3.2 To a vial (5.3) add 0,2 mg of sample and 4 ml of N,N-dimethylacetamide. Seal vial with cap and septum. Place the vial in an ultrasonic bath or on an orbital shaker for 2 h to dissolve the vinylidene chloride polymer.

6.2.4 Preparation of 'hot jar' test samples

Prepare test samples, standard additions and blank samples as in 6.2.2 and 6.2.3 with the following alterations:

Cut samples in strips and arrange in the vial so as to expose the maximum surface area for equilibration. Omit the addition of 4 ml of N,N-dimethylacetamide to the test samples, the standard additions and the blank samples.

7 Procedure

7.1 GC preparation

7.1.1 GC parameters

Establish the appropriate GC parameters.

NOTE The following range of parameters have been found suitable for column a):

Temperature:

Injector	175 °C
Column	65 °C (isothermal)

Detector	200 °C
Carrier gas and flow rate	nitrogen, 30 ml/min

Parameters found suitable for column e) are as follows:

Temperature:	
Injector	250 °C
Column	40 °C (isothermal)
Detector	200 °C
Carrier gas and flow rate	helium 8 ml/min

Split ratio 5:1.

7.1.2 Electron capture detector optimization

The electron capture detector shall be operated to give optimum sensitivity and linearity combined. Use make-up gas, carrier flow and gas filters as recommended by the manufacturer.

7.1.3 Performance of GC measurements

Equilibrate the sample solutions prepared according to 6.2.1 in the thermostat of the automated headspace sampler for 2 h at 70 °C ± 1 °C for the dissolved polymer solutions (6.2.2 and 6.2.3) and 24 h at 70 °C ± 1 °C for the 'hot jar' samples (6.2.4) before commencing the analysis programme. The long heating period for the hot jar is necessary for the vinylidene chloride in the polymer to equilibrate with the headspace.

It is possible that some plastics may generate very small quantities of vinylidene chloride by thermal degradation during the ‘hot jar’ sampling technique. If the test result obtained from using this technique exceeds any numerical restriction and if the hot jar conditions of 24 h at 70 °C are too severe in relation to the intended use of the plastics, then the plastics shall be retested using the dissolution procedure and this result shall be taken in preference.

Identify the vinylidene chloride and 1-chloropropane peaks on the basis of their retention times and measure the respective peak areas.

7.2 Calibration

Using the duplicate test sample solutions and the standard addition solutions, establish a standard addition graph (8.1.2).

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

8 Expression of results

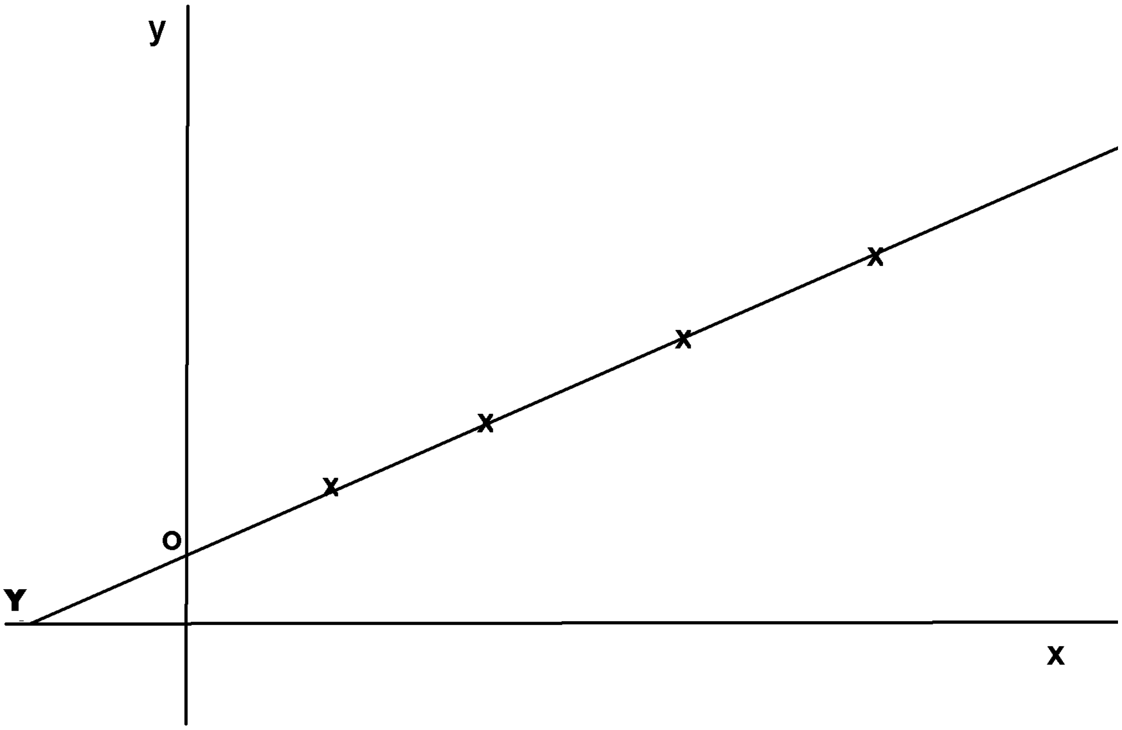
8.1 Method of calculation

8.1.1 GC interferences

If the blank samples with 1-chloropropane omitted show an interference in the 1-chloropropane region of the chromatogram exceeding 10 % of the area of 1-chloropropane in the calibration samples, and if the analysis of replicate control samples reveals that this interference varies by more than ± 20 % in absolute size, then the method of external calibration given in 8.1.3 shall be used.

8.1.2 Graphical determination using internal standard

Construct the calibration graph according to Figure 1, plotting 1-chloropropane concentration multiplied by vinylidene chloride peak area divided by 1-chloropropane peak area (y-axis) versus the vinylidene chloride concentration added to the test material in milligrams per kilogram of polymer sample (x-axis).



where

o is the corrected peak area ratio resulting from the sample solution;

X is the corrected peak area ratio resulting from the fortified sample solutions.

Figure 1 - Calibration graph

Read the vinylidene chloride concentration of the test sample solutions from the calibration graph by back extrapolation to the x-axis where the magnitude of the intercept (Y) is equal to the vinylidene chloride concentration. The intercept can be determined by means of a linear regression using a statistical programme for the method of least squares. The procedure yields the vinylidene chloride concentration in the polymer in milligrams per kilogram.

8.1.3 Graphical determination via external standardization

In cases where interferences are experienced with the internal standard (8.1.1), calibration is carried out using external standardization in which peak area values of vinylidene chloride are used to construct the calibration graph.

8.1.4 Calculation of the concentration using a response factor

Calculate the response factor using the concentrations in the standard solutions and the area of the peaks as follows:

$$f_r = \frac{C_{VDC} \times A_{1-CP}}{C_{1-CP} \times A_{VDC}} \qquad (1)$$

where:

f_r is the response factor;

C_{VDC} is the vinylidene chloride concentration of the standard solution, in milligrams per kilogram;

$C_{1\text{-}CP}$ is the 1-chloropropane concentration of the standard solution, in milligrams per kilogram;

A_{VDC} is the peak area corresponding to C_{VDC} corrected for the value of the blank sample;

$A_{1\text{-}CP}$ is the peak area corresponding to $C_{1\text{-}CP}$ corrected for the value of the blank sample.

f_r shall be calculated using the results from a minimum of four calibration samples.

Calculation of the concentration in the test samples as follows:

$$C_{VDCa} = f_r \times C_{1-CPa} \frac{A_{VDCa}}{A_{1-CPa}} \qquad (2)$$

where:

f_r is the response factor;

C_{VDCa} is the vinylidene chloride concentration of the test solution, in milligrams per kilogram;

$C_{1\text{-}CPa}$ is the 1-chloropropane concentration of the test solution, in milligrams per kilogram;

A_{VDCa} is the peak area corresponding to C_{VDCa} corrected for the value of the blank sample;

$A_{1\text{-}Cpa}$ is the peak area corresponding to $C_{1\text{-}CPa}$ corrected for the value of the blank sample.

8.2 Precision

8.2.1 Validation

Method performance has been evaluated by carrying out a precision experiment according to ISO 5725-1:1994 and ISO 5725-2:1994.

For validation of this method a precision experiment was conducted in 1997, involving eight laboratories. Each participant in this experiment obtained two samples of polymer granules fortified with vinylidene chloride as follows in Table 1.

Table 1 — Levels of vinylidene chloride in plastics

solution	level of vinylidene chloride mg/kg
Sample 1	2,6
Sample 2	29,00

Calibration solutions were prepared with comparable concentrations so that the calibration samples could be corrected.

8.2.2 Repeatability and reproducibility

Evaluation of the results of the precision experiment for vinylidene chloride for the 95% probability level yielded the following performance characteristics in Table 2:

Table 2 — Repeatability and reproducibility for the determination of vinylidene chloride

range	*r*	*R*
2,6 mg/kg to 29 mg/kg in plastics	1,26	2,67

The difference between two single results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value, *r*, on average not more than once in 20 cases in the normal and correct operation of the method.

Single results on identical test material reported by two laboratories will differ by more than the reproducibility value, *R,* on average not more than once in 20 cases in the normal operation of the method.

8.2.3 Detection limit

The detection limit was 1 mg/kg for GC-ECD and GC-FID.

9 Confirmation

9.1 Requirement for confirmation

In cases where vinylidene chloride exceeds a restriction criterion, e.g. QM = 5 mg/kg in the final material or article, the determination shall be confirmed by one of either of the methods described in 9.2 or 9.3. As the method of determination is by headspace GC, a new set of test samples and standard addition samples will need to be prepared for the confirmation. The same reagents shall be used for the confirmation analysis as were used for the primary analysis.

9.2 Confirmation by combined gas chromatography-mass spectrometry

In the selected ion mode, analyse the test sample(s), the standard addition samples and the blank samples. The ions monitored shall be m/z 96 and m/z 98 for vinylidene chloride and m/z 78 and m/z 80 for 1-chloropropane. The peaks attributed to vinylidene chloride and 1-chloropropane 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. If necessary, calculate the true concentration as indicated in 8.1. The ratios of 96/98 and 78/80 for the test samples shall be within ± 10 % of the values for vinylidene chloride and 1-chloropropane respectively in the calibration standard.

NOTE The confirmation is qualitative in that it should demonstrate the correct identity of the measured peak and the absence of interferences. If the GC/MS analysis clearly indicates the absences of interferences then the result calculated according to 8.1 should be the true value. In the case of interferences, the peak areas obtained from single ion monitoring should be used to calculate the vinylidene chloride level in the simulant, assuming ion ratios are within the tolerances given in 9.2.

9.3 Confirmation by analysis on a GC column of different polarity

Analyse the test sample(s), standard addition samples and control samples using an alternative GC phase of different polarity e.g. a more polar polysiloxane phase such as 85 % dimethyl-,7 % cyanopropyl- and 1 % vinyl- siloxane or similar, instead of a less polar phase such as 100 % dimethylsiloxane or similar, or vice versa. For each column, the peaks attributed to vinylidene chloride and to 1-chloropropane, if employed, should maximize within one-half peak width (H/2) or within 2 % of the absolute retention time of the standard, whichever is the smaller. If the levels of putative vinylidene chloride found using the two columns agree to within 10 %, then the average of the two values shall be considered to be the true value. If not, procedure 9.2 shall be followed.

10 Test report

The test report shall contain the following, where known:

a) a reference to this part of this standard;

b) all information necessary for complete identification of the sample;

c) form of the plastics;

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

e) intended conditions of use, where known;

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

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

h) any relevant comments on the test results;

i) details of any confirmation procedure(s);

j) limitation on the substance, that is 5 mg per kilogram final product;

k) identity of the laboratory conducting the test and the name of the analyst;

l) date of sample arrival or sampling, the method of sampling, the date of the analysis, together with note on any intervening storage conditions;

m) individual test results, and the mean of two or more determinations satisfying the repeatability criterion of 8.2.2, expressed in milligrams of vinylidene chloride per kilogram of plastics.

Annex A
(normative)

Manual sample injection

In the case of non-availability of an automated headspace sampler, manual sample injection may be applied provided that the repeatability criterion stated in 8.2 is satisfied.

Performance of sample injection:

Equilibrate the samples prepared according to 6.2.1 in a water bath for two hours at a constant temperature of 70 °C ± 1 °C for dissolution samples or 24 h at 70 °C for 'hot jar' samples. Take an aliquot of 1 ml or 2 ml from the sample headspace with the aid of a prewarmed (100 °C) or heatable syringe, pressing the plunger of the syringe when penetrating the septum and moving the plunger up and down several times before withdrawing the sample. Inject the aliquot into the GC. Keep the sample vial thermostated throughout this procedure.

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[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. L 220, p18.

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