DRAFT FOR DEVELOPMENT

DD CEN/TS 13130-20:2005

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

Part 20: Determination of epichlorohydrin in plastics

ICS 67.250

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British Standards

National foreword

This Draft for Development is the official English language version of CEN/TS 13130-10:2005.

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 18, an inside back cover and a back cover.

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TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

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CEN/TS 13130-20

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English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 20: Determination of epichlorohydrin in plastics

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 20 : Détermination de l'épichlorohydrine dans les matières plastiques

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 20: Bestimmung von Epichlorhydrin in Kunststoffen

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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Ref. No. CEN/TS 13130-20:2005: E

Contents

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Foreword

This document (CEN/TS 13130-20:2005) 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.

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Introduction

Epichlorohydrin, 1-chloro-2,3-epoxypropane, C_3H_5OCl, PM/Ref. No 16750, is a monomer used in the manufacture of certain plastics materials and articles, but mainly coatings intended to come into contact with foodstuffs.

After manufacture residual epichlorohydrin can remain in the finished product and may migrate into foodstuffs coming into contact with that product.

This analytical method allows the determination of the residual content of epichlorohydrin in coatings.

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

NOTE A limit on the residual quantity of 1 mg/kg polymer in final product has been set. However, epichlorohydrin is mainly used in coatings on non-plastics substrates. The amount of coating on a final article, e.g. coated cans, cannot be determined with an acceptable accuracy. Therefore, the residual content of epichlorohydrin in coatings cannot be determined. The area of a coating is easy to determine and so the amount of residual epichlorohydrin content can be determined in milligrams per square decimetre.

1 Scope

This document, part of EN 13130, specifies an analytical procedure for the determination of residual epichlorohydrin in coatings.

The method is appropriate for the quantitative determination of epichlorohydrin in the analyte concentration range of 5 ng/ml to 80 ng/ml of extract. In general this allows for the detection of epichlorohydrin at the level of 1 mg/kg of polymer or in the case of coatings, where the amount of polymer cannot be determined, detection of 1 µg epichlorohydrin per square decimetre of coating is feasible.

In order to obtain reliable and reproducible results, it is essential that the method described in this part of EN 13130 is followed as closely as possible.

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

To determine the residual amount of epichlorohydrin in a coating, the sample material is extracted with dioxane for 6 h at room temperature. Subsequently, the extract is distilled by means of a micro-distillation. The concentration of epichlorohydrin in the distilled fraction thus obtained is determined by derivatization of the epoxide with an aromatic sulfonic acid, i.e. 9,10-dimethoxyanthracene-2-sulfonic acid (DAS), followed by reversed phase high performance liquid chromatography (HPLC) with fluorescence detection. Depending on the quality and type of the HPLC column, it is possible to separate the two isomers that are formed in the derivatization reaction of epichlorohydrin with DAS. Quantification is achieved by means of external standard calibration using dioxane solutions fortified with known amounts of epichlorohydrin.

Confirmation of epichlorohydrin is carried out by normal phase HPLC with fluorescence detection.

4 Reagents

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

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4.1 Analyte

4.1.1 Epichlorohydrin (C_3H_5OCl), purity > 99 %.

4.2 Chemicals

4.2.1 Acetonitrile (HPLC grade).

4.2.2 9,10-Dimethoxyanthracene-2-sulfonic acid sodium salt, i.e. DAS-Na, $C_{16}H_{13}NaO_5S$.

4.2.3 Dioxane > 99,5 %, absolute; over molecular sieve (H_2O < 0,01 %).

4.2.4 Hexane

4.2.5 Cation exchange resin, strongly acid with sulfonic acids as functional groups, ion form H^+.

4.2.6 Isopropanol

4.2.7 Methanol

4.2.8 Water, deionized (HPLC quality).

4.3 Preparation of the DAS reagent

Dissolve approximately 100 mg DAS-Na (4.2.2) in 20 ml methanol/water (80/20). Heat the solution slightly to obtain a clear solution. In a glass beaker, wash an amount of the ion exchange resin (4.2.5) with methanol/water (80/20) and subsequently pour the slurry into a glass column with a diameter of 1 cm, until the resin has reached a height of ± 20 cm. Elute the DAS solution through the ion exchange resin and flush the column with a mixture of methanol/water (80/20). Start to collect the eluate at the moment the eluate has decreased to a pH of approximately pH 1 to pH 2. Continue collecting the eluate until the pH starts to increase. Evaporate the solution thus obtained to dryness by means of a nitrogen stream.

NOTE The dry residue should be protected from light and is stable for at least 1 year.

WARNING Do not deviate from the prescribed ratio of DAS-Na versus ion exchange resin.

4.4 Solutions

4.4.1 Stock solution of epichlorohydrin in dioxane (1 mg/ml)

Weigh to the nearest 0,1 mg approximately 50 mg of epichlorohydrin (4.1.1) in a 50 ml volumetric flask. Make up to the mark with dioxane (4.2.3) and mix thoroughly.

Calculate the exact concentration in milligrams of epichlorohydrin per millilitre of solution.

Repeat the procedure to obtain a second stock solution.

NOTE The solutions can be stored in closed containers in dark for a maximum period of 3 months at any temperature between - 20 °C and + 20 °C.

4.4.2 Diluted stock solutions of epichlorohydrin in dioxane (0,04 mg/ml)

Pipette 2 ml of the stock solution (4.4.1) into a 50 ml volumetric flask. Make up to the mark with dioxane and mix thoroughly.

Calculate the exact concentration in milligrams of epichlorohydrin per millilitre of solution.

Repeat the procedure using the second stock solution to obtain a second diluted stock solution.

4.4.3 Standard solutions of epichlorohydrin in dioxane

Pipette into a series of 25 ml volumetric flasks, 0 ml, 0,5 ml, 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of the diluted stock solution (4.4.2). Make up to the mark with dioxane and mix carefully.

The standard solutions thus obtained contain approximately 0 µg, 0,8 µg, 1,6 µg, 3,2 µg, 4,8 µg, 6,4 µg or 8,0 µg epichlorohydrin per millilitre.

Calculate the exact concentrations in micrograms of epichlorohydrin per millilitre of solution.

Repeat the procedure using the second diluted stock solution to obtain a second series of standard solutions.

4.4.4 DAS solution in acetonitrile (5 mg/ml)

Prepare from the DAS reagent (4.3) a solution in acetonitrile which contains approximately 5 mg DAS-reagent per millilitre of acetonitrile.

The DAS solution in acetonitrile shall be prepared freshly before use and protected against light. The solution is only stable for one day at room temperature.

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 High performance liquid chromatograph (HPLC)

NOTE A chromatograph with an automatic injector or 20 µl injection loop, and a fluorescence detector, set to $\lambda_{excitation}$ 262 nm and $\lambda_{emission}$ 490 nm, connected to a strip chart recorder or integrator is preferred.

5.2 HPLC column, capable of producing a symmetric peak for epichlorohydrin, and capable of separating epichlorohydrin from peaks originating from solvents and excess of DAS.

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

NOTE The column and parameters established for the column used in the development of the method are given below.

Column: Stainless steel 250 mm x 4,6 mm, filled with C_8 coated silica, particle size 5 µm (load of 10 % carbon and end capped)

Column temperature:	ambient
Eluent :	acetonitrile (4.2.1) water (4.2.8)

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Gradient	time in min	% acetonitrile	% water
	0	55	45
	5	55	45
	15	70	30
	18	70	30
	21	55	45
	31	55	45

Flow rate: 2 ml/min
Injection volume: 20 µl
Detection: fluorescence
Wavelength: $\lambda_{excitation}$ = 262 nm
$\lambda_{emission}$ = 490 nm

5.3 Crimper, for sealing sample vials.

5.4 22 ml sample vials with polytetrafluoroethylene coated butyl rubber septa and aluminium crimp caps.

5.5 De-capper, for de-capping sample vials closed with an aluminium crimp cap.

5.6 Injection syringe, 50 µl.

5.7 Injection syringe, 100 µl.

5.8 Equipment for micro-distillation, as shown in Figure A.1.

6 Samples

6.1 General

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

NOTE This method only describes the determination of epichlorohydrin from coatings, but is expected to be suitable for any other materials containing epichlorohydrin.

6.2 Preparation of test samples - extraction of epichlorohydrin from coated articles

NOTE 1 In general test samples are extracted with dioxane for 6 h at room temperature. In the case of very thick samples, the extraction time should be extended or carried out at elevated temperature using a closed system to avoid loss of epichlorohydrin.

For cans, fill a can with 50 ml dioxane, close the can with an epoxide-free coated end and extract for 6 h at room temperature on a roller bank.

NOTE 2 A typical surface to volume ratio is 2,5 dm^2/50 ml.

For coated packaging material, cut 2 dm^2 coated material into pieces and immerse in 50 ml dioxane for a period of 6 h at room temperature.

Transfer 10,0 ml of the solution obtained into a 22 ml headspace vial, add a magnetic stirring bar and seal the vial with a polytetrafluoroethylene lined septum. This vial is known as vial A.

NOTE 3 A few anti bumping granules can be added if a heating plate with a magnetic stirrer is not available.

6.3 Blank sample preparation

Transfer 10,0 ml of dioxane which has not been in contact with the coating to a 22 ml headspace vial, add a few anti-bumping granules, if added at 6.2, and close the vial.

6.4 Calibration solution preparation

Pipette into a series of 22 ml sample vials (5.4) 10,0 ml blank dioxane solvent. Add 100 µl of each standard solution of epichlorohydrin as prepared in 4.4.3. Add a few anti-bumping granules and close the vials with polytetrafluoroethylene lined septa.

The solutions thus obtained contain approximately 0 ng/ml, 8 ng/ml, 16 ng/ml, 32 ng/ml, 48 ng/ml, 60 ng/ml or 80 ng epichlorohydrin per millilitre of dioxane.

Calculate the exact concentration in nanograms of epichlorohydrin per millilitre of solution.

Repeat the procedure using the second set of standard solutions to obtain a second set of calibration solutions.

7 Procedure

NOTE The solution in the vials is distilled to remove any polymeric material and the distilled epichlorohydrin is derivatized. The derivative is submitted to HPLC using fluorescence detection.

7.1 Distillation of the dioxane extract

The equipment for the micro-distillation of epichlorohydrin from the dioxane extract is shown in Figure A.1.

Vial A is the vial as prepared in 6.2 and contains 10,0 ml of the test sample extract.

Close an empty 22 ml vial with a septum and aluminium cap and mark the vial at a volume of 3 ml. Record the mass of this empty, closed vial. This vial is known as vial B.

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Connect vial A with vial B, as shown in Figure A.1, by means of the stainless steel tube, provided with a polytetrafluoroethylene sleeve, in such way that the tube penetrates the septum into vial A for only 1 cm to 2 cm, whereas in vial B the end of the tube is positioned far into the vial. To avoid blocking of the tubing, pierce the septa of the sample vials by making a pinhole only, in order to do this ensure the ends of the stainless steel tubing are sharpened like injection needles. Pierce an injection needle just through the septum of vial B to avoid pressure build-up in the vial.

Place vial A on a hot plate and vial B in a beaker containing ice/water. Heat vial A, while stirring, until the extract is boiling. Continue boiling the dioxane extract in vial A until the distilled liquid in vial B has reached the 3 ml mark. Remove vial A from the heating plate and remove the venting needle and the stainless steel tubing from vial B. Dry carefully the outside of vial B with a tissue. Re-weigh vial B and determine the volume of the distilled fraction to the nearest 0,01 ml.

For calculation of the volume of the distilled fraction, assume that the density of dioxane is 1 g/ml, instead of 1,03 g/ml.

NOTE The distilled fraction can be stored in the refrigerator in a well closed container for several weeks.

7.2 Derivatization of epichlorohydrin with DAS

The derivative of epichlorohydrin with DAS is very sensitive to light. Therefore, handle under conditions of dimmed light, including the HPLC analysis. Exclude daylight and artificial light where possible.

De-cap the sample vial B containing the distilled fraction and add 50 µl of freshly prepared DAS solution in acetonitrile (4.4.4). Close the vial with a septum and aluminium cap. Place the vial in an oven for a period of 17 h ± 1 h at 75 °C. After this derivatization period, cool the solution to room temperature. Fill a HPLC vial with the derivatized solution.

NOTE The derivatized solutions are stable for a maximum of two days, when stored at room temperature with the exclusion of light. During these two days, the composition of the derivatized solution changes slightly. Therefore, in the case were derivatized solutions are stored, a series of derivatized standard solutions should be stored at the same time, under the same conditions. After two days, HPLC analysis is no longer possible because of too many interferences in the HPLC determination.

7.3 Treatment of blank sample

Treat the vials with the blank samples prepared in 6.3 in the same way as described in 7.1 and 7.2.

7.4 Treatment of calibration solutions

Treat the vial with the calibration solution prepared in 6.4 in the same way as described in 7.1 and 7.2.

7.5 HPLC analysis

Analyze the derivatized test samples, blanks and calibration solutions prepared in 7.1 to 7.4 by means of HPLC.

NOTE 1 The conditions described in 5.2 were found to be suitable.

Measure the peak area of the epichlorohydrin derivative in the chromatograms obtained.

Inject each solution at least in duplicate.

When using an autosampler, cover the vials with a light shield, e.g. a piece of aluminium foil.

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

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

The detector shall be capable of detecting at least 10 ng of epichlorohydrin per millilitre concentration of sample injected at a signal to noise ratio of 3:1.

Under the conditions given in 5.2 the two isomers formed during the derivatization of epichlorohydrin with DAS are separated, resulting in a double peak with peak maxima at retention times of approximately 8,6 min and 9,1 min.

NOTE 2 Chromatographic separation of the two isomers formed in the derivatization reaction strongly depends on the quality of the HPLC column used. Also deviation from the type of column mentioned in 5.2 could result in lesser separation. In those cases, only one peak is obtained for the epichlorohydrin-DAS derivative.

7.6 Calibration

Measure the area of the epichlorohydrin peak(s). If the two isomers of the epichlorohydrin-DAS derivative are separated, sum the peak areas. Correct for the volume (in ml) distilled by multiplying the measured peak area with the volume of the distilled fraction. Divide the corrected peak area by 10, the initial volume of the dioxane solution, and plot that value against the concentration of epichlorohydrin in the calibration solutions in micrograms per millilitre of dioxane.

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

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

7.7 Evaluation of data

7.7.1 High performance liquid chromatography interferences

Following the method described no interferences have been observed.

7.7.2 Calculation of analyte level in dioxane solution

7.7.2.1 Graphical determination

Calculate the average of peak area values obtained from the test samples according to 7.5 and correct for the volume distilled, by multiplying the measured area with the volume (in ml) distilled. Divide the corrected peak area by 10, the initial volume of the dioxane solution. Read the epichlorohydrin concentration of the test solution from the calibration graph (7.6) in micrograms per millilitre.

7.7.2.2 Calculation from the regression parameters

Calculate the corrected peak area as described above. Use the calculated peak area in the following formula.

Calculate the regression line of the calibration graph including the sample value:

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

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where

y is the peak area of the epichlorohydrin derivative;

a is the slope of the regression line;

x is the concentration of epichlorohydrin in the calibration solution in micrograms per millilitre;

b is the intercept of the regression line,

then the epichlorohydrin concentration in the dioxane extract, is given by:

$$\mathrm{C}_{\mathrm{ECH,extr}} = \frac{y-b}{a}$$

where

$C_{\mathrm{ECH,extr}}$ is the epichlorohydrin concentration in the dioxane extract in micrograms per millilitre of extract.

Both procedures yield directly the epichlorohydrin concentration in the extract in micrograms per millilitre of extract.

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

7.7.3 Calculation of the epichlorohydrin content in the test sample

7.7.3.1 Residual content in milligrams per kilogram

Calculate the residual content of epichlorohydrin in the food contact material as follows:

$$Q = \frac{X \times V}{W}$$

where

Q is the concentration of epichlorohyrin in the food contact material in milligrams per kilogram

X is concentration of epichlorohydrin in the dioxane solution as obtained in 7.7.2, in micrograms per millilitre;

V is the volume of dioxane used to extract the test sample, in millilitres;

W is the mass of the test sample, in grams.

7.7.3.2 Residual content in milligrams per square decimetre

Calculate the residual content (mg/dm²) of epichlorohydrin in the food contact material as follows:

$$Q = \frac{X \times V}{0{,}001S}$$

where

Q the residual content (mg/dm²) of epichlorohydrin in the food contact material in milligrams per square decimetre;

X is concentration of epichlorohydrin in the dioxane solution as obtained in 7.7.2, micrograms per millilitre;

V is the volume of dioxane used to extract the test sample, in millilitres;

S is the surface area of the test sample, in square decimetres.

NOTE The restriction for the residual content (QM) of epichlorohydrin given in Commission Directive 90/128/EEC is expressed as 1 mg/kg in food contact material (FCM). It should be noted that expression of QM in milligrams per kilogram in final product is difficult or even impossible to handle because data on thickness and weight of the coating on a FCM are often missing and it is difficult to determine the amount of coating on a FCM. Therefore, it is proposed to set a food contact area-related QM restriction of 20 µg/6 dm² in cases where the thickness of the coating cannot clearly be determined. This area-related QM limit is workable for thicknesses less than and equal to 0,25 mm. For thicknesses greater than 0,25 mm the proposed area-related QM value can come into conflict with the prescribed mass related QM. Therefore, above 0,25 mm thickness the currently legal QM value of 1 mg/kg of polymer should be applied. The above proposal would not only solve any difficulties concerning the amount of coating extracted in the determination of epichlorohydrin, but it would also exclude effects of thickness of the coating layer on a FCM.

8 Precision

8.1 Validation

This method was pre-validated by a collaborative trial with two laboratories. In each laboratory a within-laboratory precision experiment was performed by standard addition of epichlorohydrin to an extract of an epoxy coating at the level of 0,8 µg/dm^2.

8.2 Repeatability

Evaluation of the within-laboratory precision experiment results according to ISO 5725, at a concentration of 40 ng epichlorohydrin/ ml extract for the 95 % probability level, yielded the following performance characteristics (lab 1/lab 2):

Repeatability: r = 9,6/9,7 ng epichlorohydrin per millilitre of extract, equal to 1,2 µg/6 dm²

8.3 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 of 5,3 ng/ml to 6,9 ng/ml extract or 0,6 µg/6 dm^2 to 0,8 µg/6 dm^2. Thus the method is capable of quantitative detection of epichlorohydrin at a minimum level of 6,9 ng/ml extract, i.e. 0,8 µg/6 dm^2 applying a surface/volume ratio of 2,5 dm^2/50 ml extraction solvent.

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9 Confirmation

9.1 Requirement for confirmation

If the residual concentration of epichlorohyrin in the polymer, as determined in accordance with 7.7.3, exceeds the restriction, e.g. a compositional limit of 1,0 mg/kg of polymer or the optional 20 µg/6 dm² until a new limit is set, the determination shall be confirmed by the method described in 9.2.

The confirmation is qualitative in the sense that it demonstrates the correct identity of the measured analyte and the absence of interferences. For the purposes of quantification the result as calculated according to 7.7.3 shall be taken as the true value.

9.2 Confirmation by normal phase HPLC

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

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

Column:	Stainless steel 250 mm x 4,6 mm, filled with Lichrosorb Si60, particle size 5 µm
Column temperature:	ambient
Eluent :	hexane (4.2.4) isopropanol (4.2.6)

gradient

time in min	% hexane	% propanol
0	95	5
3	95	5
5	60	40
8	60	40
10	20	80
15	20	80
17	95	5
27	95	5

Flow rate:	1 ml/min
Injection volume:	20 µl
Detection:	Fluorescence
Wavelength:	$\lambda_{excitation}$ = 262 nm $\lambda_{emission}$ = 490 nm

9.3 HPLC analysis

The derivatized test samples, blanks as well as calibration samples prepared in 7.1 to 7.4 are analyzed as they are without any further sample treatment.

Each solution shall be injected at least in duplicate.

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

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

The detector has to be able to detect at least 10 ng of epichlorohydrin/ml concentration of sample injected at a signal to noise ratio of 3:1.

Under the conditions given in 9.2 the retention time obtained for the derivatized epichlorohydrin was approximately 14,3 minutes.

9.4 Calibration

See 7.6.

9.5 Evaluation of data

See 7.7.

10 Test report

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

a) Identification of the sample;

b) name of the laboratory;

c) name responsible analyst;

d) date of report;

e) date of analysis;

f) analyte;

g) a reference to this method;

h) sample details, such as:

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

  2) origin and denotation of the sample;

  3) date and method of obtaining the laboratory sample;

  4) storage conditions;

i) results expressed in milligrams of epichlorohydrin per kilogram of polymer or milligrams of epichlorohydrin per 6 dm^2. Results shall be reported as the average value from two or more determinations satisfying the repeatability criterion of 8.2;

j) details of confirmation procedure, if any;

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

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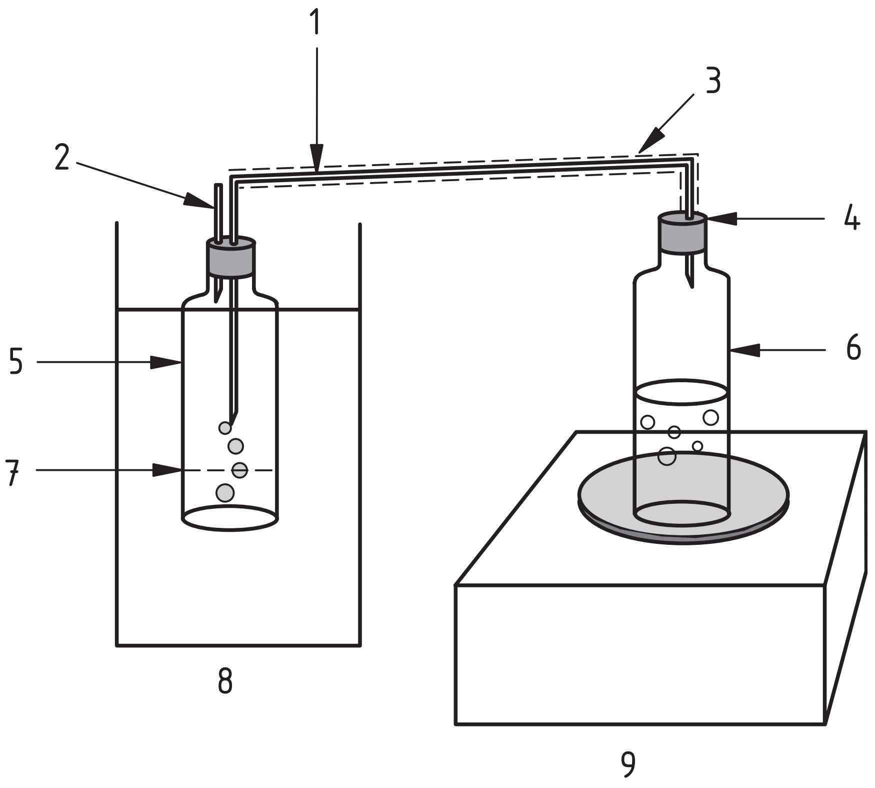
Annex A
(normative)

Equipment for the micro-distillation of epichlorohydrin from dioxane extract



Key

1 Stainless steel tubing, ends are injection needle type sharpened, internal diameter 1 mm, length approx. 200 mm
2 Injection needle for venting
3 Sleeves of PTFE tube
4 PTFE lined septum
5 Vial B
6 Vial A
7 3 ml mark
8 Cold water/ice bath
9 Hot plate and magnetic stirrer

Figure A.1 — Equipment for the micro-distillation of epichlorohydrin from dioxane extract

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[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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