

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

Part 7: Determination of monoethylene glycol and diethylene glycol in food simulants

The European Standard EN 13130-7:2004 has the status of a
British Standard

ICS 67.250

National foreword

This British Standard is the official English language version of EN 13130-7:2004. It supersedes DD ENV 13130-7:1996 which is withdrawn.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.

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English version

Materials and articles in contact with foodstuffs - Plastics substances subject to limitation - Part 7: Determination of monoethylene glycol and diethylene glycol in food simulants

Matériaux et objets en contact avec les denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 7 : Détermination du monoéthylène glycol et du diéthylène glycol dans les simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 7: Bestimmung von Monoethylenglykol und Diethylenglykol in Lebensmittel-Simulantien

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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Foreword

This document (EN 13130-7:2004) 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 13130-7 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 6: *Determination of vinylidene chloride in plastics*
- 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-trimethylpropane 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

Monoethylene glycol (MEG) and diethylene glycol (DEG) are monomers used in the manufacture of plastics materials and articles intended to come into contact with foodstuffs. Residues of monoethylene glycol and diethylene glycol can remain in the plastic after processing to form materials and articles for food contact use, and can migrate into foodstuffs.

NOTE Commission Directive 2002/72/EC lists a combined specific migration limit of 30 mg/kg (T) of monoethylene glycol and diethylene glycol in foods or food simulants.

1 Scope

This part of this European Standard specifies methods for the determination of monoethylene glycol and diethylene glycol in the food simulants; water, 3 % (w/v) acetic acid, 15 % (v/v) ethanol and olive oil and other fatty food simulants, simulants D, e.g. a mixture of synthetic triglycerides or sunflower oil or corn oil. The methods are capable of determining monoethylene glycol and diethylene glycol in food simulants separately, or combined, at the specific migration limit SML (T) of 30 mg/kg.

NOTE This method was developed for the determination of monoethylene glycol and diethylene glycol in 15 % (v/v) aqueous ethanol, as required by the regulations in force at the time the development work was carried out. However, this method, developed for 15 (v/v) aqueous ethanol, should be applicable to the determination in 10 (v/v) aqueous ethanol.

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.*

EN ISO 1042; *Laboratory glassware – One-mark volumetric flasks (ISO 1042:1998).*

ISO 385 (all parts), *Laboratory glassware – Burettes.*

ISO 835 (all parts); *Laboratory glassware – Graduated pipettes.*

ISO 4788, *Laboratory glassware – Graduated measuring cylinders.*

3 Principle

After addition of an internal standard the aqueous food simulants or the olive oil water extract is directly injected for gas chromatographic analysis using a cold on-column injector. Detection limits are approximately 1 mg/kg in the food simulants. The concentrations of monoethylene glycol and diethylene glycol are measured by comparison of peak height or area ratios against standards.

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 Methanol

4.2 Monoethylene glycol

- 4.3 Diethylene glycol
- 4.4 Butan-1,4-diol, internal standard
- 4.5 Water (HPLC or deionized)
- 4.6 Heptane
- 4.7 Prepare a monoethylene glycol and diethylene glycol standard stock solution as follows:

Weigh accurately about 0,75 g each of monoethylene glycol and diethylene glycol into a beaker, dissolve in methanol and transfer with washings to a 100 ml volumetric flask. Dilute to the mark with methanol. This solution is stable for 1 month if stored in the dark at 5 °C in a stoppered flask. Prepare a second stock solution for validation purposes, see 7.2.2.

- 4.8 Prepare a butan-1,4-diol internal standard stock solution as follows:

Weigh about 1 g of butan-1,4-diol into a beaker, dissolve in methanol and make up to 100 ml mark with methanol in a volumetric flask. This solution is stable for 1 month if stored in the dark at 5 °C in a stoppered flask.

5 Apparatus

- 5.1 Analytical balance capable of weighing to 0,1 mg.
- 5.2 Graduated pipettes, conforming to the minimum requirements of ISO 835 and of 1 ml and 2 ml capacity.
- 5.3 Burette, conforming to the minimum requirements of ISO 385 and of 25 ml capacity.
- 5.4 Volumetric flasks, conforming to the minimum requirements of EN ISO 1042 and of 25 ml, 50 ml and 100 ml capacity.
- 5.5 Separating funnels with polytetrafluoroethylene stopcock, of 250 ml capacity.
- 5.6 Glass syringe, of 50 ml capacity.
- 5.7 0,2 µm disposable HPLC filters, or disposable C18 solid phase extraction cartridges 400 mg size.
- 5.8 Measuring cylinders, conforming to the minimum requirements of ISO 4788 and of 25 ml and 50 ml capacity.
- 5.9 Gas chromatograph (GC) fitted with a flame ionization detector (FID) and a cold on-column injector.

NOTE 1 This method has been developed for use with cold on-column injectors, which are available from all major GC suppliers. Previous studies at other laboratories have shown that split/splitless injection gives unreliable results. Higher than optimum carrier gas flow rates have been found to give less peak tailing and increased column lifetime for polyethylene glycol stationary phases. The GC column should be capable of resolving monoethylene glycol, diethylene glycol and butan-1,4-diol from each other, and from ethanol and acetic acid present in the simulants. GC capillary columns using a polyethylene glycol stationary phase have been found to be most suitable for example:

- a) 15 m X 0,53 mm internal diameter, film thickness 1 µm;
- Temperature 100 °C hold 2 min ramped to 150 °C at 10 °C/min, hold 4 min;
- Carrier gas Helium 50 KPa, 18 ml/min;

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Detector	Flame ionization detector 250 °C;
Injector	Cold on-column.

The retention times are monoethylene glycol, 4,0 min, butan-1,4-diol , 7,5 min and diethylene glycol, 8,1 min.

b) 12 m X 0,32 mm internal diameter, film thickness 1 µm;

Temperature	100 °C hold 1 min ramped to 200 °C hold 1 min at 10 °C/min;
Carrier gas	Helium 70 KPa, 7 ml/min;
Detector	Flame ionization detector 250 °C;
Injector	Cold on-column.

The retention times are monoethylene glycol, 3,8 min, butan-1,4-diol, 7,0 min and diethylene glycol, 7,6 min, and a typical GC chromatogram is given in annex A.

NOTE 2 As noted above this method has been developed for use with cold on-column injectors. However, it has been reported that successful results can be obtained using a polyethylene glycol, modified with nitroterephthalic acid, split-splitless injector as follows:

30 m x 0,32 mm internal diameter;

Temperature	100 °C for 1 min, then 10 °C /min to 150 °C for 8 min;
Detection temperature	Flame ionization detector 220 °C;
Injector	Split-splitless, 220 °C.

The precision data detailed in 8.2 is only relevant to cold on column injectors.

6 Samples

The food simulant samples for testing shall have been prepared as described in EN 13130-1. Blank samples of the food simulants are also required.

For each simulant, 50 ml samples are required for each test.

7 Procedure

7.1 Preparation of standards

7.1.1 Intermediate standards

Into each of five 25 ml volumetric flasks pipette 2 ml of the stock butan-1,4-diol solution. Add, using a burette or graduated pipettes, 0,5 ml, 1,0 ml, 2,5 ml, 5 ml and 10 ml of the stock monoethylene glycol/diethylene glycol solution and dilute to the mark with methanol to give 150 mg/l, 300 mg/l, 750 mg/l, 1500 mg/l and 3000 mg/l monoethylene glycol and diethylene glycol respectively. These standards are stable for 1 month if stored in the dark at 5 °C in stoppered flasks.

7.1.2 Internal standard, 800 mg/l butan-1,4-diol

Pipette 2 ml of the stock butan-1,4-diol solution into a 25 ml volumetric flask and dilute to the mark with methanol. This solution is stable for 1 month if stored in the dark at 5 °C in a stoppered flask.

7.1.3 Working standards for aqueous simulants

Fill each of five 50 ml volumetric flasks to the graduation mark with water and add, by pipette, 1 ml of each intermediate standard. Mix thoroughly to give, nominally, working standards containing 3 mg/l, 6 mg/l, 15

mg/l, 30 mg/l and 60 mg/l monoethylene glycol and diethylene glycol respectively. To a further 50 ml volumetric flask filled to the mark with water add, by pipette, 1 ml of the internal standard 800 mg/l butan-1,4-diol and mix well to act as a blank.

7.1.4 Working standards for olive oil

Weigh $50 \text{ g} \pm 1 \text{ g}$ of the olive oil blank simulant into a 250 ml separating funnel. Add, by pipette, 1 ml of the first intermediate standard, mix well and add $50 \text{ ml} \pm 2 \text{ ml}$ of heptane from the measuring cylinder (this transfers any remaining olive oil). Mix and add $20 \text{ ml} \pm 1 \text{ ml}$ of water using a measuring cylinder, shake vigorously for 1 min and allow 5 min to 10 min for the layers to separate.

NOTE 1 This can be facilitated by holding the separating funnel at an angle of about 45°C and slowly rotating it.

Run off the lower aqueous layer into a 100 ml beaker and re-extract the olive oil with a further $20 \text{ ml} \pm 1 \text{ ml}$ of water. Allow the phases to separate and run off the lower layer into the beaker to combine the extracts. Pass about 2 ml of the aqueous solution through a disposable filter, using a syringe, and collect the filtrate.

Repeat this process for each intermediate standard and the internal standard to act as a blank. These standards correspond to 0 mg/kg, 3 mg/kg, 6 mg/kg, 15 mg/kg, 30 mg/kg and 60 mg monoethylene glycol and diethylene glycol per kilogram olive oil.

NOTE 2 A C18 solid phase extraction cartridge can also be effectively used for removal of oil droplets.

7.2 Preparation of calibration graphs

7.2.1 Injection of standards

Inject a suitable quantity, $0,5 \mu\text{l}$ to $1 \mu\text{l}$, of each standard and measure the peak heights or areas. Divide the monoethylene glycol and diethylene glycol peak height/areas by the butan-1,4-diol peak height/area and plot this ratio against monoethylene glycol and diethylene glycol concentration. The calibration graph shall be linear with a correlation coefficient better than 0,998. Calculate the slope and intercept on the y axis from the line of best fit.

7.2.2 Validation of stock monoethylene glycol/diethylene glycol solution

Dilute the second stock standard monoethylene glycol/diethylene glycol prepared in 4.7 to give a 750 mg/l intermediate standard as described in 7.1.1, and dilute again to obtain a 15 mg/l working standard described in 7.1.3. Inject this working standard, in duplicate, for GC analysis and calculate the monoethylene glycol/butan-1,4-diol and diethylene glycol/butan-1,4-diol peak area ratios. Calculate the mean concentrations of monoethylene glycol and diethylene glycol found in the validation working standard using the slope and intercept values obtained in 7.2.1, correcting for the masses of monoethylene glycol and diethylene glycol used to prepare the first stock solution. The mean concentrations shall be within $\pm 1 \text{ mg/l}$ of the actual monoethylene glycol and diethylene glycol concentrations present in the validation working standard calculated from the masses of monoethylene glycol and diethylene glycol used to prepare the second stock standard. If the concentrations found are not within $\pm 1 \text{ mg/l}$, reject all solutions and start again.

7.3 Extraction of olive oil simulant migration test samples

Weigh $50 \text{ g} \pm 1 \text{ g}$ of the olive oil samples directly from the migration cell or containers into a 250 ml separating funnel. Add by pipette 1 ml of the internal standard solution (800 mg/l, butan-1,4-diol) and mix thoroughly. Rinse the measuring cylinder with $50 \text{ ml} \pm 2 \text{ ml}$ heptane and transfer to the separating funnel. Extract twice with water as described in 7.1.4.

7.4 Preparation of aqueous simulant migration test samples

Fill a series of dry 50 ml volumetric flasks with each aqueous sample to the mark and add, by pipette, 1 ml of the internal standard, mix well. Prepare a blank for each aqueous simulant in a similar fashion but fill to the mark with the blank simulant.

7.5 Injection of samples

Inject a suitable quantity, 0,5 µl to 1µl, in duplicate, for each sample and measure the peak areas or heights. After correction for the blank, if necessary, calculate the monoethylene glycol/butan-1,4-diol and diethylene glycol/butan-1,4-diol peak ratios and, from the slope and intercept values found in 7.2.1, the concentrations of diethylene glycol and monoethylene glycol in milligrams per litre. Alternatively, read directly from the calibration graph the concentrations of monoethylene glycol and diethylene glycol using the peak area ratios monoethylene glycol/butan-1,4-diol and diethylene glycol/butan-1,4-diol. Correct the concentrations for the actual masses of monoethylene glycol and diethylene glycol used to prepare the stock solution.

8 Expression of results

8.1 Method of calculation

8.1.1 As milligrams of monoethylene glycol and diethylene glycol per kilogram of food simulant

The procedure yields the concentration in milligrams per litre for aqueous food simulants and milligrams per kilogram for olive oil and simulants D.

NOTE Commission Directive 2002/72/EC [1] states that the specific gravity of all simulants should conventionally be assumed to be '1'. Milligrams of substance released per litre of simulant will thus correspond numerically to milligrams of substance released per kilogram of simulant and, taking into account of the provisions laid down in Directive 82/711/EEC, to milligrams of substance released per kilogram of foodstuff.

The migration results obtained above as milligrams per litre equate numerically as milligrams per kilogram.

8.1.2 As milligrams of monoethylene glycol and diethylene glycol per square decimetre of surface area of plastic material or article

From the total volume or mass of simulant and surface area of plastic employed in the migration test, calculate the migrated quantity of monoethylene glycol and/or diethylene glycol in milligrams per square decimetre.

8.1.3 Calculation of the specific migration

Depending on the fill volume of the test material and on the surface area/food simulant ratio, monoethylene glycol and/or diethylene glycol concentration in the sample as determined according to clause 7 may need mathematical transformation in order to calculate the specific migration value to be compared with SML. For guidance see EN 13130-1:2004, clause 13.

8.2 Precision

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

8.2.1 Validation

For validation of this method a precision experiment was conducted in 1997, involving twelve laboratories. Each participant in this experiment obtained six samples of 10 % (v/v) aqueous ethanol fortified with monoethylene glycol and diethylene glycol at levels as follows in Table 1.

Table 1 — Levels of monoethylene glycol and diethylene glycol in 10 % (v/v) aqueous ethanol

solution	level of monoethylene glycol	level of diethylene glycol
	mg/kg	mg/kg
C1 solution level I	37,52	26,20
C2 solution level I	37,52	26,20
C3 solution level II	31,76	39,24
C4 solution level II	31,76	39,24
C5 solution level III	26,62	29,54
C6 solution level III	26,62	29,54

For validation of this method a precision experiment was conducted in 1997, involving ten laboratories. Each participant in this experiment obtained six samples of olive oil fortified with monoethylene glycol and diethylene glycol at levels as follows in Table 2.

Table 2 — Levels of monoethylene glycol and diethylene glycol in olive oil

solution	level of monoethylene glycol	level of diethylene glycol
	mg/kg	mg/kg
D1 solution level I	31,59	29,47
D2 solution level I	31,59	29,47
D3 solution level II	26,65	39,28
D4 solution level II	26,65	39,28
D5 solution level III	37,58	25,33
D6 solution level III	37,58	25,33

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 monoethylene glycol for the 95% probability level yielded the following performance characteristics in Table 3:

Table 3 — Repeatability and reproducibility for the determination of monoethylene glycol

range	<i>r</i>	<i>R</i>
27 mg/kg to 38 mg/kg in 10 % (v/v) aqueous ethanol	1,87	5,92
27 mg/kg to 38 mg/kg in olive oil	2,08	6,72

Evaluation of the results of the precision experiment for diethylene glycol for the 95 % probability level yielded the following performance characteristics in Table 4:

Table 4 — Repeatability and reproducibility for the determination of diethylene glycol

range	<i>r</i>	<i>R</i>
27 mg/kg to 38 mg/kg in 10 % (v/v) aqueous ethanol	2,00	6,58
27 mg/kg to 38 mg/kg in olive oil	2,13	6,18

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 (DL) was found to be between 0,6 mg/l and 1,8 mg/l for monoethylene glycol and diethylene glycol in the aqueous and fat simulants.

9 Confirmation of monoethylene glycol and diethylene glycol

9.1 Requirement for confirmation

In cases where the combined levels of monoethylene glycol and diethylene glycol migration from materials and articles into foods, or food simulants, as determined according to 8.1, exceeds a restriction criterion, e.g. SML(T) = 30 mg/kg, the determination shall be confirmed by one of either of the methods described in 9.2.

9.2 Confirmation by re-analysis using confirmed by gas chromatography - mass spectrometry

In the selected ion mode, re-analyse the test samples(s), the working standards and the appropriate blank simulant(s) by gas chromatography - mass spectrometry, using the cold on-column injection technique. The ions monitored shall be:

monoethylene glycol	m/z 61
diethylene glycol	m/z 75
butan-1,4-diol	m/z 71.

The mass spectra for monoethylene glycol and diethylene glycol are shown in annex B. The peaks attributed to monoethylene glycol, diethylene glycol and butan-1,4-diol shall 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. Use the peak ratios so derived to calculate the levels of monoethylene glycol and diethylene glycol in food simulants. The level thus found shall be considered the true value.

10 Test report

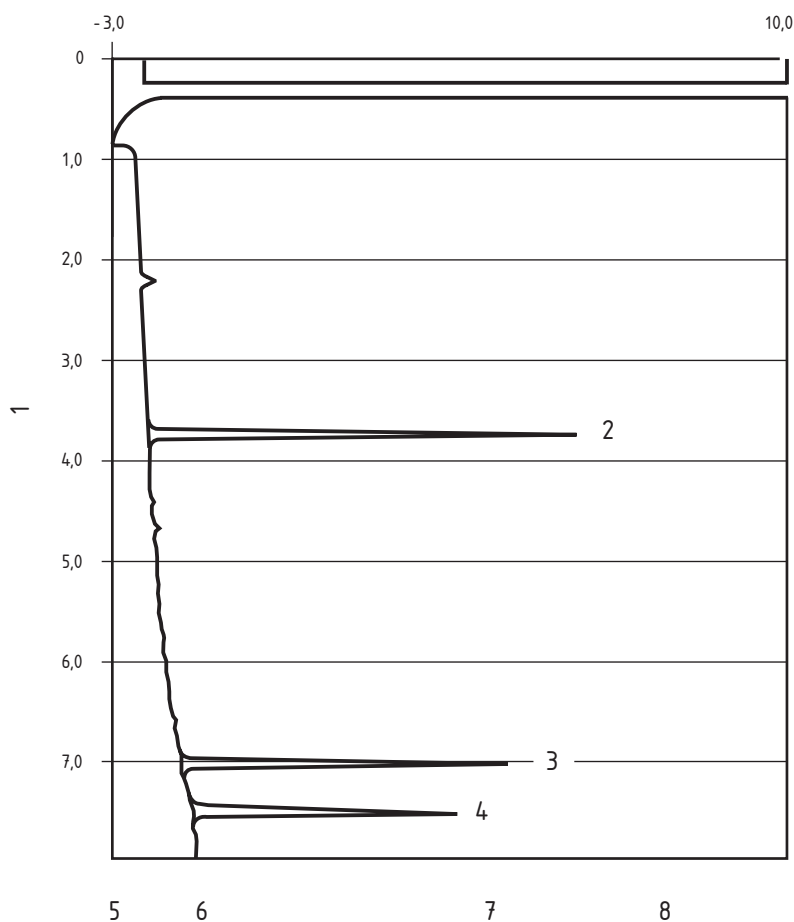
The test report shall contain the following, where known:

- a reference to this part of this standard;
- all information necessary for complete identification of the sample;
- 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) conditions of the test;
 - 1) foodstuffs or food simulants used;
 - 2) duration and temperature, and relation with "Conditions of contact in worst foreseeable use" see Table 3 of EN 13130-1:2004;
 - 3) area and geometry of the test specimen;
 - 4) volume of foodstuff or food simulant used where appropriate;
- g) any departures from the standard method, reasons for the departures;
- h) any particular requirements of the parts of this standard;
- i) any relevant comments on the test results;
- j) details of any confirmation procedure(s);
- k) limitation on the substance, that is 30 mg of monoethylene glycol alone or together with diethylene glycol /kilogram of food or foods simulant;
- l) identity of the laboratory conducting the test and the name of the analyst;
- m) date of sample arrival or sampling, the method of sampling, the date of the analysis, together with note on any intervening storage conditions;
- n) individual test results, and the mean of two or more determinations satisfying the repeatability criterion of 8.2.2, expressed in milligrams of monoethylene glycol and diethylene glycol per kilogram of food simulant.

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Annex A (informative)

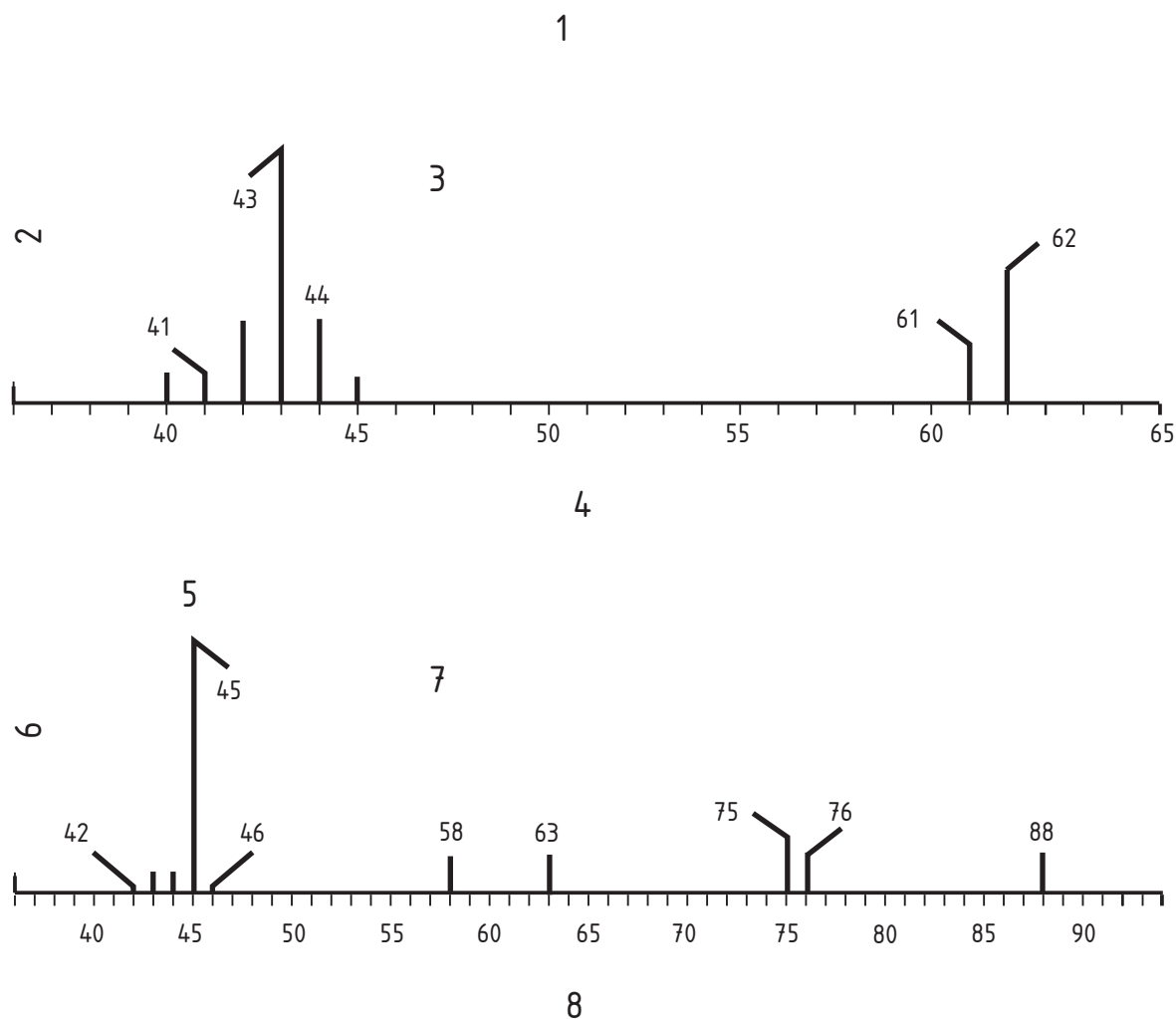


Key

- 1 Minutes
- 2 MEG 20 mg/L
- 3 BUG
- 4 DEG 20 mg/l
- 5 GC column temperature
- 6 12 m × 0,32 mm BP 20 1 μm
100 °C hold 1 min
- 200°C at 10 °C/min
- 7 Carrier gas
- 8 Helium 70 kPa

Figure A.1 — Typical gas chromatogram of glycols in water using cold on-column injection

Annex B (informative)



Keys

- 1 (154) Scan 4.865 min. of DATA:0303MS01.D
- 2 Abundance
- 3 MEG
- 4 Mass/Charge
- 5 (290) Scan 7.359 min. of DATA :0303MS01.D
- 6 Abundance
- 7 DEG
- 8 Mass/Charge

Figure B.1 — Mass spectra for monoethylene glycol and diethylene glycol

Bibliography

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic methods for the determination of repeatability and reproducibility of a standard measurement method*

[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.

[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.

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