BRITISH STANDARD

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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

The European Standard EN 13130-1:2004 has the status of a British Standard

ICS 67.250

BSI
British Standards

National foreword

This British Standard is the official English language version of EN 13130-1:2004. It supersedes DD ENV 13130-1:1999 which is withdrawn.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

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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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

Matériaux et objets en contact avec des denrées alimentaires - Substances dans les matières plastiques soumises à des limitations - Partie 1 : Guide des méthodes d'essai pour la migration spécifique dans les denrées alimentaires et les simulants d'aliments de substances contenues dans les matières plastiques, détermination des substances dans les matières plastiques et choix des conditions d'exposition aux simulants d'aliments

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln - Substanzen in Kunststoffen, die Beschränkungen unterliegen - Teil 1: Anleitung für Testmethoden für die spezifische Migration von Substanzen aus Kunststoffen in Lebensmitteln und Lebensmitteln-Simulantien, Bestimmung der Substanzen in Kunststoffen und Auswahl von Expositions bedingungen für Lebensmitteln-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.

CEN members are the national standards bodies of 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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Contents

Foreword

This document (EN 13130-1:2004) 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 to provide guidance in the preparation of samples for testing in a series of test methods contained in other parts of this standard.

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.

Informative annex E details the relationship of this standard with the European Union Directives.

At the time of preparation and publication of this standard 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.

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 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-trimethylopropane in food simulants*

Parts 2 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*" [1)] .

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.

[1)] Franz R. and Rijk.R; Development of methods of analysis for monomers and other starting substances with SML and/or QM limits in Directives 2002/72/EC and 92/39/EEC. European Commission, BCR information: Chemical analysis, EU report 17610 EN, ECSC-EC-EAEC. Brussels - Luxembourg 1997.

Introduction

EN 13130-1 is intended to give guidance on the selection of the most appropriate type of test, test conditions and test method for a given application of a plastics material or article and is intended to be read in its entirety before testing protocols are finalized.

The general criteria for the operation and assessment of testing laboratories as well as the general criteria for laboratory accreditation bodies are set out in EN ISO/IEC 17025, EN 45002 and EN 45003. It is recommended that laboratories using this standard validate their procedures by taking part in a proficiency scheme. Suitable proficiency schemes are operated in Germany and in the United Kingdom, for example the German Assessment Scheme for Food Testing (GAFT) and the Food Analysis Performance Assessment Scheme (FAPAS) conducted by the Central Science Laboratory of the Ministry of Agriculture, Fisheries and Food.

1 Scope

This part of this European Standard provides a guide to the selection of the appropriate conditions of contact of food simulants with the test article before the determination of specific migration of those substances subject to a migration limit.

NOTE According to Directive 2002/72/EC[2] the determination of the migration of specified components in foodstuffs instead of the use of simulants is permitted. However, in that situation there is no need to give guidance on the test conditions of time and temperature as contact conditions shall be equal to conditions applied in real.

Also general guidance is given for the determination of the amount of the substance in the final plastics material or article.

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 10088-1; *Stainless steels – Part 1: List of stainless steels.*

EN ISO 8442-2:1997; *Materials and articles in contact with foodstuffs – Cutlery and table holloware – Part 2: Requirements for stainless steel and silver-plated cutlery (ISO 8442-2:1997).*

ISO 648; *Laboratory glassware -— One-mark pipettes.*

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

ISO 5725 (all parts); *Accuracy (trueness and precision) of measurement methods and results.*

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1
plastics
organic macromolecular compounds obtained by polymerization, polycondensation, polyaddition or any similar process from molecules with a lower molecular weight or by chemical alteration of natural molecules.
Silicones and other macromolecular compounds should also be regarded as plastics. Other substances or matter can be added to such compounds.

3.2
final material/article
material or article in its ready-for-use state or as sold

3.3
sample
material or article under investigation

3.4
test specimen
portion of the sample on which a test is performed

3.5
test piece
portion of the test specimen

3.6
conventional oven
oven where the air within the oven is heated and this heat is then transferred to the food through the plastics as opposed to a microwave oven where the food itself is heated directly by microwave irradiation

3.7
food simulant
medium intended to simulate a foodstuff (see clauses 4 to 7)

3.8
specific migration
mass of the substance transferred to the simulant as determined in the test method

3.9
residual content
mass of the substance present in the final material or article

3.10
specific migration limit (SML)
maximum permitted level of a named substance migrating from the final material or article into food or food simulants

3.11
SML(T)
maximum permitted level of a named substance migrating from the final material or article into food or food simulants expressed as total of moiety or substance(s) indicated

3.12
compositional limit (Qm)
maximum permitted amount of the "residual" monomer, additive or substance in the material or article

3.13
Qm(T)
maximum permitted amount of the "residual" monomer, additive or substance in the material or article expressed as total of moiety or substance(s) indicated

3.14
quantity per surface area (QMA)
maximum permitted amount of residual monomer, additive or substance in the material or article expressed as $mg/6dm^2$

3.15
reduction factor
numbers, 2 to 5, which can be applied to the result of the migration tests relevant to certain types of fatty foodstuffs and which is conventionally used to take account of the greater extractive capacity of the simulant for such foodstuffs

3.16
migration test
test for the determination of specific migration of substance, using food simulant under conventional test conditions

3.17
substitute fat test
test carried out which uses test media under conventional substitute test conditions when the use of a migration test into fatty food simulant(s) is not feasible

3.18
test media
substances used in "substitute tests", iso-octane, 95 % ethanol in aqueous solution and modified polyphenylene oxide (MPPO)

3.19
alternative fat test
tests, with suitable, usually, volatile media, that can be used instead of migration tests with fatty food simulants

3.20
'volatile' test media
volatile substances used in alternative fat tests

3.21
extraction tests
tests in which media having strong extraction properties under very severe test conditions, are used

3.22
dissolution test
tests in which the sample is dissolved to liberate the substance from the plastics test specimen

3.23
pouch
receptacle of known dimensions manufactured from plastics film/sheet to be tested, which when filled with food simulant or test medium exposes the food contact side of the film/sheet to the food simulant or test medium

3.24
reverse pouch
pouch which is fabricated such that the plastics surface intended to come into contact with foodstuff is the outer surface. All of its edges are sealed to prevent the inner surfaces coming into contact with the food simulant or test medium during the test period. The reverse pouch is intended to be totally immersed in the food simulant or test medium

3.25
cell
device in which a plastics film to be tested can be mounted which when assembled and filled with food simulant or test medium, exposes the food contact side of the film to the food simulant or test medium

3.26
repeatability value 'r'
value below which the absolute difference between two single test results obtained under repeatability conditions can be expected to lie with a probability of 95 %, as described in ISO 5725

3.27
reproducibility value 'R'
value below which the absolute difference between two single test results obtained under reproducibility conditions can be expected to lie with a probability of 95%, as described in ISO 5725

3.28
repeatability conditions
conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time

3.29
reproducibility conditions
conditions where test results are obtained with the same method on identical material in different laboratories with different operators using different equipment

4 General

4.1 Summary

When determining the specific migration of substances from plastics materials and articles into foodstuffs, food simulants or test media, the test procedure is carried out in two stages. The first stage is the exposure of the plastics material or article to the foodstuff, food simulant or test medium under conditions of use or simulated conditions of use. The second stage is the determination of the migrant in the foodstuff, food simulant or test medium. This part of this standard comprises advice and instructions on the procedures to be followed, where appropriate, in preparing the plastics sample for exposure, the selection of conditions of exposure to food simulants or test media and the calculation of migration levels when the analysis of the migrating substance is complete.

In addition, guidance is given in the preparation of plastics test specimens for the determination of the residual content of a substance or group of substances.

Procedures for sampling plastics materials and articles and foodstuffs for analysis are described in clause 11.

Methods for the chemical analysis of the individual substances or groups of substances in foodstuffs, food simulants and test media, and as residues in plastics materials and articles, are given in other parts of this standard.

4.2 Precautions in handling and testing

Many substances which are the subject of testing are volatile substances which migrate spontaneously from plastics. When testing a plastics material or article containing a volatile substance, careful consideration needs to be given to possible loss of the substance by volatilization after sampling and during testing. Loss of volatiles after sampling and before testing, can be minimized by low temperature storage or hermetic sealing with limited void volume (see clause 11).

In many applications of plastics materials and articles under actual conditions of use, it is possible that volatile substances will not migrate exclusively into the foodstuff but be lost to the surrounding atmosphere.

Considerations are given to the classification of substances on the basis of volatility in annex A.

Cutting or any mechanical treatment of the sample to prepare test specimens or test pieces, for testing with foodstuffs, food simulants or test media, can have an irreversible effect on the composition and/or morphology of the edges of the sample. As a result, with tests performed with test pieces totally immersed in the foodstuff or food simulant the obtained migration value might not be a true reflection of the real migration under actual conditions of use. Plastics sensitive to this phenomenon are acrylonitrile/butadiene/styrene terpolymers (ABS), polystyrene and other styrene co-polymers. With these plastics types, cut edges shall preferably not be in contact with the foodstuff, food simulant or test medium. Care shall also be taken to avoid mechanical damage to surfaces of these types of plastics.

4.3 Analysis of a substance in a food simulant - migration test

Where a plastics material or article is intended to be used in contact with a wide variety of foodstuffs it could be impracticable to test with all possible foodstuffs. Frequently, the presence of interfering substances in the foodstuff precludes the use of simple analytical methods. For these reasons migration testing with conventional food simulants is permissible.

In general, the methods described in other parts of this standard have been devised for use with the conventional food simulants. When the analysis is in the liquids chosen by convention to simulate foodstuffs, this part of this standard is intended to give advice on the selection of the most appropriate test conditions and test method for a given application of a plastics material or article and shall be read in its entirety before testing protocols are started.

4.4 Analysis of substance in a foodstuff

In some cases it could be necessary to carry out the analysis for a migrant in an actual foodstuff. This is particularly so for enforcement authorities where a sample of the plastics material or article which has not been in contact with the foodstuff is not available. Testing in actual foodstuff could also be appropriate when the testing in food simulants under the conventional conditions, taking into account reduction factors is known to produce invalid results.

For some analytical procedures, for example headspace gas chromatography analyses of volatile substances, analyses in a wide variety of foodstuffs can be possible. When the analysis is carried out in an actual foodstuff particular care needs be taken to ensure the validity of the test result, since the performance characteristics of the method are unlikely to have been established for the foodstuff. Where a particular procedure has also been found to be suitable for determinations in foodstuffs, this will be indicated in the part of this standard relevant to that particular substance.

4.5 Analysis of a substance in a test medium - substitute fat tests

Where the determination of a specified substance in a fatty food simulant is not feasible, for technical reasons connected with the method of analysis, then a substitute fat test using a test medium (iso-octane, 95% ethanol or modified polyphenylene oxide) can be used. Validity of the test result needs to be verified, since the performance characteristics of the test method might not have been established for the particular test medium.

4.6 Analysis of a substance in a ‘volatile’ test medium - alternative fat tests

Alternative fat tests using ‘volatile’ test media can be used to demonstrate compliance with the relevant specific migration limit. The alternative fat test conditions and the ‘volatile’ test medium shall be selected with great care as the migration into the ‘volatile’ test medium shall be equivalent to or higher then the migration into the fatty food simulant. Validity of the test result needs to be verified, since the performance characteristics of the method are unlikely to have been established for the particular ‘volatile’ test medium used.

4.7 Analysis of a substance in a plastics material or article

For those plastics substances which are subject to compositional limits, expressed as maximum quantity of substance, in milligrams, present per kilogram of plastics (mg/kg) - QM, or as maximum quantity of substance, in milligrams, present per 6 square decimetres of surface area of plastics (mg/6 dm^2), an analysis is carried out on the plastics material and article prior to contact with any foodstuff.

4.8 Multi-analyte analyses

Some plastics materials and articles contain several substances subject to specific migration limitations and/or compositional limitations. For the determination of the migration of more than one substance, one test simulant, test medium or one sample of foodstuff derived from a single exposure of the plastics to the food simulant, test medium or a single sample of foodstuff, can be prepared for the analyses. The test simulant, test medium or sample of foodstuff shall be divided to allow each substance to be individually determined, using the appropriate individual analytical test methods. If one of the substances is designated a ‘volatile’ substance, then the procedures for exposure to simulants, test media and sampling, have to be those applicable to volatile substances. Where the analyses are for substances in the plastics material or article, a suitable sample shall be appropriately divided for the analyses of the individual substances.

4.9 Multilayer materials and articles

There are many plastics constructions in food contact applications where the food contact surface is chemically different from the other layers. For a substance with a compositional limit (QM, mg/kg), the limit can apply only to the layer containing the substance. The concentration in the particular layer is calculated from the analytical measurement of the substance in the multilayer material, if the thickness and density of the layer is known or can be measured.

Where a specific migration limit (SML) applies, it is essential that the test is carried out with the food simulant in contact only with the food contact surface, to replicate the intended conditions of use with foodstuffs. A substance which originates in the non-food contact layer(s) can permeate through to the food contact layer and migrate into the simulant during the exposure period. An analysis on the food simulant for substance will determine if such permeation/migration has occurred.

5 Types of test

5.1 Substitute tests

"Migration" tests for the determination of the specific migration of plastics substances are carried out using the "food simulants" and "conventional migration test conditions", see 6.1, 6.2 and Table 1.

5.2 Substitute tests

If the migration test using fatty food simulants is not feasible, for technical reasons connected with the method of analysis, "substitute tests" which use test media under conventional substitute test conditions can be appropriate. The substitute tests involve the use of all of the substitute test media, 95 % ethanol in aqueous solution, iso-octane and modified polyphenylene oxide under the test conditions corresponding to the test conditions for simulant D, see Table 4. A new test specimen is used for each test. The reduction factors, 2 to 5, are applicable to these substitute tests, see clause 6. To ascertain compliance with the migration limit the highest value obtained from the tests with the three test media, is selected.

The use of substitute tests is justified, when the migration test carried out with each of the four 'D' fatty food simulants (rectified olive oil, synthetic mixture of tryglycerides, sunflower oil, corn oil - 6.2) are found to be inapplicable due to technical reasons connected with the method of analysis, e.g. interferences, inadequate detection limit, reaction with fat simulant etc.

5.3 Alternative fat tests

5.3.1 General

An alternative fat test can be carried out either with a volatile medium or as an extraction test.

5.3.2 Alternative tests with volatile media

Alternative tests are carried out using volatile test media such as iso-octane and 95 % ethanol in aqueous solution or other volatile solvents or mixtures of solvents. An alternative test can be used to demonstrate compliance with a specific migration limit (SML), provided that:

a) the 'alternative test' result obtained is equal to or greater than that obtained in a corresponding migration test with a fatty food simulant (simulant D);

b) the 'alternative test' result does not exceed the specific migration limit, after application of appropriate reduction factors.

If either or both conditions are not fulfilled, the migration tests (5.1) shall be performed.

5.3.3 Extraction tests

Extraction tests are carried out with 'other test media' having strong extractive power under severe test conditions. An extraction test can be used for testing for compliance with a specific migration limit (SML), if it is generally recognized, on the basis of scientific evidence, that the result obtained is equal to or higher than that obtained in with a fatty food simulant (simulant D).

5.4 Residual content determination

5.4.1 "QM" test

The "QM" test measures the total quantity of the substance under test, in the plastics material or article sample. This can usually be achieved by dissolution of test specimens of the plastics in a suitable solvent. After precipitation of the polymer the substance is quantitatively determined in the solvent using a suitable analytical method. An alternative procedure is to exhaustively extract test specimens of the plastics with a liquid which can penetrate the plastics matrix and is a strong solvent for the substance under test. The substance is again quantitatively determined in the solvent using a suitable analytical method. The result is expressed in milligrams of substance per kilogram plastics (mg/kg).

5.4.2 "QMA" test

The "QMA" test is a similar test to the "QM" test in that it also measures the total quantity of the substance under test, in the plastics material or article sample. In the "QMA" test the result is expressed in milligrams of substance per 6 square decimetres of surface area of plastics intended to come into contact with foodstuff (mg/6 dm^2). Using similar procedures described for the "QM" tests, a known area of homogeneous plastics is dissolved or extracted under severe conditions and the test substance is determined using a suitable analytical method.

5.4.2.1 "QMA" test for thick samples

It is generally accepted that migration into real foodstuffs occurs mainly from the first 0,25 mm of the plastics thickness and, that, therefore, the contribution from the inner parts of thickness $\geq$ 0,25 mm is relatively insignificant, and can be neglected. Therefore only the quantity of substance(s) released by the first 0,25 mm of plastics thickness shall be considered in calculating the "QMA" value, when an extraction test is carried out.

For samples having a thickness, D, greater than 0,25 mm, if the plastics is homogenous, the total sample can be subjected to an extraction test, but the total quantity of substance shall be divided by D/0,25, e.g. if the sample has a thickness of 1 mm the total quantity released shall be divided by 4.

5.4.2.2 "QMA" test for multilayer materials and articles

For multi-layer plastics materials and articles (laminates), a known area of the sample is extracted to determine the "QMA" value. When determining the "QMA" value for a substance in a multi-layer sample, only that portion of the layer(s) which is/are within 0,25 mm of the foodstuff contact surface shall be used in the analysis and/or calculation.

5.4.2.3 "QMA" test for plasticized materials and articles

For plasticized materials, for 'foamed' plastics (e.g. foamed/expanded polystyrene) and for other plastics which have open structures produced by a physical process, the maximum thickness rule of 0,25 mm shall not be applied because migration from depths greater than 0,25 mm can occur.

6 Food simulants, test media and reagents

6.1 Aqueous food simulants

The aqueous food simulants shall be of the following specification:

- distilled water or water of equivalent quality, simulant A;

- 3 % acetic acid (w/v) in aqueous solution, simulant B;

For the purposes of this standard this means a solution prepared by diluting 30 g of acetic acid with distilled water to a volume of 1 l;

- 10 % ethanol (v/v) in aqueous solution, simulant C.

For the purposes of this standard this means a solution prepared by diluting 100 ml of 100% ethanol with distilled water to a volume of 1 litre;

For liquids or beverages with an ethanol content greater than 10 % (v/v) the test is carried out with aqueous solutions of ethanol of a similar strength.

NOTE Many of the test methods described in other parts of this European Standard were developed for the determination of substances in 15 % (v/v) aqueous ethanol, as required by the regulations in force at the time the development work was carried out. However, there is no reason why these methods developed for 15 (v/v) aqueous ethanol shall not be applicable to 10 (v/v) aqueous ethanol.

6.2 Fatty food simulants

The fatty food simulants are as follows:

- rectified olive oil, "reference simulant D".

This "reference simulant D" can be replaced by a synthetic mixture of triglycerides or sunflower oil or corn oil with standardized specifications (characteristics). These are known as "other fatty food simulants" and called "simulant D".

For the characteristics of olive oil, a synthetic mixture of triglycerides, sunflower oil and corn oil, see annex B. The simulant D shall meet the specifications listed in annex B and shall also be checked in advance to ensure that it is free from any significant interference with the method of analysis for the substance that is to be tested for in the specific migration test.

NOTE 1 Experience has shown that olive oil "light" or "mild" usually meets the specifications mentioned and can easily be obtained. The unsaponifiable part in this light version of olive oil is lower than in standard olive oil.

NOTE 2 When these fatty food simulants are used to simulate some classes of food, reduction factors can be used, see 7.3 and Table 2.

6.3 Test media

6.3.1 Test media for substitute tests

The test media to be used in substitute tests are iso-octane, 95 % ethanol in aqueous solution and a modified polyphenylene oxide (MPPO). The characteristics of modified polyphenylene oxide are to be found in annex B.

For the purposes of this standard, 95 % ethanol means a solution prepared by diluting 950 ml of 100% ethanol with distilled water to a volume of 1 l.

6.3.2 Test media for alternative tests

In general, these are volatile media such as iso-octane and 95 % ethanol in aqueous solution or any other volatile solvent or mixtures of solvents. The use of solid test media are not common but can appear suitable in special cases and are therefore not excluded.

NOTE Unless otherwise specified, food simulants, substitute and alternative test media as well as extraction or dissolution solvents should be of analytical quality. The simulants, test media or solvents should be free from components interfering in the determination of the specific migration or the residual content.

6.4 Reagents

During the analysis, unless otherwise stated, only reagents of recognized analytical grade and only distilled water of equivalent purity shall be used.

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 can 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.

7 Selection of food simulants

7.1 General

NOTE European Commission Directive 85/572/EEC [6] specifies the use of 15 % ethanol (v/v) in aqueous solution as simulant C. This has been superseded in European Commission Directive 97/48/EC [5] the second amendment to Council Directive 82/711/EEC [3], which specifies 10 % ethanol (v/v) in aqueous solution.

7.2 Simulating contact with all food types

Where a plastics material or article is intended for use in contact with all types of food it shall be tested with 3 % acetic acid (w/v) in aqueous solution, simulant B, 10 % ethanol (v/v) in aqueous solution, simulant C and a fatty food simulant, simulant D, without reduction factors. If when using any of the other fatty food simulants, see 6.2, the migration limit is exceeded, for the judgement of non compliance with the migration limit a confirmation of the result by using olive oil is obligatory, when technically feasible. If this confirmation is not technically feasible and the migration from the material or article exceeds the limit, it shall be deemed not in compliance with the migration limit.

7.3 Simulating contact with specific food types

Provision for plastics materials and articles intended to come into contact with specific food types has been made in the following situations:

a) when the material or article is already in contact with a known foodstuff;

b) when the material or article is accompanied by a specific indication stating with which food types it can or cannot be used, for example "only for aqueous foods";

c) when the material or article is accompanied by a specific indication stating with which foodstuff(s) or group(s) of foodstuffs they can or cannot be used. This indication shall be expressed:

1) at the marketing stage other than retail stage, by using the " reference number" or "description of foodstuffs" ;

2) at the retail stage using an indication which shall refer to only a few foods or groups of food, preferably with examples which are easy to understand.

In situation b) the simulants to be used in the migration tests are specified in Table 1.

Table 1 — Food simulants to be selected for testing food contact materials in special case

Contact foods	Simulant
Only aqueous foods	Simulant A
Only acidic foods	Simulant B
Only alcoholic foods	Simulant C
Only fatty foods	Simulant D
All aqueous and acidic foods	Simulant B
All alcoholic and aqueous foods	Simulant C
All alcoholic and acidic foods	Simulants C and B
All fatty and aqueous foods	Simulants D and A

All fatty and acidic foods	Simulants D and B
All fatty and alcoholic and aqueous foods	Simulants D and C
All fatty foods and alcoholic and acidic foods	Simulants D, C and B

In situation a) and c) the tests are carried out using the food simulants mentioned in Table 2.

In Table 2 for each foodstuff or group of foodstuffs, only the simulant(s) indicated by an 'X' is (are) to be used, using for each simulant, a new sample of the plastics material or article. Where no 'X' appears, no migration test with any of the simulants is specified for the class of foodstuffs under the heading or sub-heading concerned, however, see 7.4 on dry foods and frozen foods.

When 'X' is followed by an oblique stroke and a figure, the result of the migration test shall be divided by the number indicated. In the case of certain types of fatty foodstuffs, this figure, known as the 'reduction factor', is conventionally used to take account of the greater extractive capacity of the simulant for such foodstuffs.

Where a letter 'a' is shown in brackets after the 'X' only one of the two simulants given shall be used:

- if the pH value is higher than 4,5, simulant A shall be used;
- if the pH value is 4,5, or less, simulant B shall be used.

Where a foodstuff is listed under both a specific heading and a general heading, only the simulant(s) indicated under the specific heading is (are) to be used.

Where the foodstuff(s) or group(s) of foodstuffs are not included in Table 2, select the item from the table of food simulants to be selected for testing food contact materials in special cases, which corresponds most closely to the foodstuff(s) or group of foodstuff(s) under examination.

Table 2 — List of simulants to be used in the migration test with a particular foodstuff or group of foodstuffs

Reference number	Description of foodstuffs	Simulants to be used			
		A	B	C	D
01	**Beverages**				
01.01	Non-alcoholic beverages or alcoholic beverages of an alcoholic strength lower than 5 % vol.:				
	Waters, ciders, fruit or vegetable juices of normal strength or concentrated, musts, fruit nectars, lemonades, and mineral waters, syrups, bitters, infusions, coffee, tea, liquid chocolate, beers and other	X (a)	X (a)		
01.02	Alcoholic beverages of an alcoholic strength equal to or exceeding 5 % vol.:				
	Beverages shown under heading 01.01 but with an alcoholic strength equal to or exceeding 5 % vol.:				
	Wines, spirits and liqueurs		X [a]	X [b]	
01.03	Miscellaneous: undenatured ethyl alcohol		X [a]	X [b]	
02	**Cereals, cereal products, pastry, biscuits, cakes and other bakers' wares**				
02.01	Starches				
02.02	Cereals, unprocessed, puffed, in flakes, (including popcorn, corn flakes and the like)				
02.03	Cereal flour and meal				
02.04	Macaroni, spaghetti and similar products				
02.05	Pastry, biscuits, cakes and, other bakers' wares, dry:				
	A. With fatty substances on the surface				X/5
	B. Other				
02.06	Pastry, cakes, and, other bakers' wares, fresh:				
	A. With fatty substances on the surface				X/5
	B. Other	X			
03	**Chocolate, sugar and products thereof Confectionery products**				
03.01	Chocolate, chocolate-coated products, substitutes and products coated with substitutes				X/5
03.02	Confectionery products:				
	A. In solid form:				
	I. With fatty substances on the surface				X/5
	II. Other				

[a] This test shall be carried out only in cases where the pH is 4,5 or less.

[b] This test can be carried out in the case of liquids or beverages of an alcoholic strength exceeding 10 % vol. with aqueous solutions of ethanol of a similar strength.

Table 2 (continued)

Reference number	Description of foodstuffs	Simulants to be used			
		A	B	C	D
03.02	B. In paste form				
(continued)	I. With fatty substances on the surface				X/3
	II. Moist	X			
03.03	Sugar and sugar products				
	A. In solid form				
	B. Honey and the like	X			
	C. Molasses and sugar syrups	X			
04	**Fruit, vegetables and products thereof**				
04.01	Whole fruit, fresh or chilled				
04.02	Processed fruit:				
	A. Dried or dehydrated fruit, whole or in the form of flour or powder				
	B. Fruit in the form of chunks, purée or paste	X (a)	X (a)		
	C. Fruit preserves (jams and similar products - whole fruit or chunks or in the form of flour or powder, preserved in a liquid medium):				
	I. In an aqueous medium	X (a)	X (a)		
	II. In an oily medium	X (a)	X (a)		X
	III. In an alcoholic medium (≥ 5 % vol.)		X [a]	X	
04.03	Nuts (peanuts, chestnuts, almonds, hazelnuts, walnuts, pine kernels and other):				
	A. Shelled, dried				
	B. Shelled and roasted				X/5 [b]
	C. In paste or cream form	X			X/3 [b]
04.04	Whole vegetables, fresh or chilled				
04.05	Processed vegetables:				
	A. Dried or dehydrated vegetables whole or in the form of flour or powder				
	B. Vegetables, cut, in the form of purées	X (a)	X (a)		
	C. Preserved vegetables;				
	I. In an aqueous medium	X (a)	X (a)		
	II. In an oily medium	X (a)	X (a)		X
	III. In an alcoholic medium (≥ 5 % vol.)		X [a]	X	
05	**Fats and oils**				
05.01	Animals and vegetable fats and oils, whether natural or treated (including cocoa butter, lard, resolidified butter)				X
	Margarine, butter and other fats and oils made from water emulsions in oil				X/2

[a] This test is to be used only where the pH is 4,5 or less.
[b] If it can be demonstrated by means of an appropriate test that there is no 'fatty contact' with the plastic, the test with simulant D can be dispensed with.

Table 2 (continued)

Reference number	Description of foodstuffs	Simulants to be used			
		A	B	C	D
06	Animal products and eggs				
06.01	Fish:				
	A. Fresh, chilled, salted, smoked	X			X/3 [a]
	B. In the form of paste	X			X/3 [a]
06.02	Crustaceans and molluscs (including oysters, mussels, snails) not naturally protected by their shells	X			
06.03	Meat of all zoological species (including poultry and game):				
	A. Fresh, chilled, salted, smoked	X			X/4
	B. In the form of paste, creams	X			X/4
06.04	Processed meat products (ham, salami, bacon and other)	X			X/4
06.05	Preserved or part preserved meat and fish:				
	A. in an aqueous medium	X (a)	X (a)		
	B. In an oily medium	X (a)	X (a)		X
06.06	Eggs not in shell:				
	A. Powdered or dried				
	B. Other	X			
06.07	Egg yolks:				
	A. Liquid	X			
	B. Powdered or frozen				
06.08	Dried white of egg				
07	Milk products				
07.01	Milk:				
	A. Whole	X			
	B. Partly dried	X			
	C. Skimmed or partly skimmed	X			
	D. Dried				
07.02	Fermented milk such as yoghourt, buttermilk and such products in association with fruit and fruit products		X		
07.03	Cream and sour cream	X (a)	X (a)		
07.04	Cheeses:				
	A. Whole, with rind				
	B. Processed cheeses	X (a)	X (a)		
	C. All others	X (a)	X (a)		X/3 [a]

[a] If it can be demonstrated by means of an appropriate test that there is no 'fatty contact' with the plastic, the test with simulant D can be dispensed with.

Table 2 (continued)

Reference number	Description of foodstuffs	Simulants to be used			
		A	B	C	D
07.05	Rennet:				
	A. in liquid or viscous form	X (a)	X (a)		
	B. Powdered or dried				
08	**Miscellaneous products**				
08.01	Vinegar		X		
08.02	Fried or roasted foods:				
	A. Fried potatoes, fritters and the like				X/5
	B. Of animal origin				X/4
08.03	Preparations for soups, broths, in liquid, solid or powder form (extracts, concentrates); homogenized composite food preparations, prepared dishes:	X			X/4
	A. Powdered or dried				
	I. With fatty substances on the surface				X/5
	II. Other				
	B. Liquid or paste				
	I. With fatty substances on the surface	X (a)	X (a)		X/3
	II. Other	X (a)	X (a)		
08.04	Yeasts and raising agents:				
	A. In paste form	X (a)	X (a)		
	B. Dried				
08.05	Salt				
08.06	Sauces:				
	A. Without fatty substances on the surface	X (a)	X (a)		
	B. Mayonnaise, sauces derived from mayonnaise, salad creams and other oil in water emulsions	X (a)	X (a)		X/3
	C. Sauce containing oil and water forming two distinct layers	X (a)	X (a)		X
08.07	Mustard (except powdered mustard under heading 08.17)	X (a)	X (a)		X/3 [a]
08.08	Sandwiches, toasted bread and the like containing any kind of foodstuff:				
	A. With fatty substances on the surface				X/5
	B. Other				
08.09	Ice-creams	X			
08.10	Dried foods:				
	A. With fatty substances on the surface				X/5
	B. Other				

[a] If it can be demonstrated by means of an appropriate test that there is no 'fatty contact' with the plastic, the test with simulant D can be dispensed with.

Table 2 (concluded)

Reference number	Description of foodstuffs	Simulants to be used			
		A	B	C	D
08.11	Frozen or deep-frozen foods				
08.12	Concentrated extracts of an alcoholic strength equal to or exceeding 5 % vol.		X [b]	X	
08.13	Cocoa:				
	A. Cocoa powder				X/5 [a]
	B. Cocoa paste				X/3 [a]
08.14	Coffee, whether or not roasted, decaffeinated or soluble, coffee substitutes, granulated or powdered				
08.15	Liquid coffee extracts	X			
08.16	Aromatics herbs and other herbs:				
	camomile, mallow, mint, tea, lime blossom and others				
08.17	Spices and seasonings in the natural state:				
	cinnamon, cloves, powdered mustard, pepper, vanilla, saffron and other				

[a] If it can be demonstrated by means of an appropriate test that there is no 'fatty contact' with the plastics, the test with simulant D can be dispensed with.

[b] This test is to be used only where the pH is 4.5 or less.

NOTE This list of simulants to be used in the migration test with a particular foodstuff or group of foodstuffs is as specified in Council Directive 85/572/EEC [6].

7.4 Simulating contact with dry foods and frozen food

Plastics intended to come into contact with dry foodstuffs, such as cereals and dried eggs, or with frozen foods, need not be tested for migration with the food simulants A, B, C and D, listed in clause 5, because these liquid food simulants are not appropriate models (mimics) for dry and frozen foods. However, volatile substances in particular can migrate into dry and frozen foods, especially if there is likely to be a long period of contact with the plastics. Therefore these food contact materials shall be tested for the release of volatile substances. This can be determined in the relevant food or in a substitute food simulant. In this respect MPPO as an absorbent for the volatile substances can be used while applying test conditions as indicated in Table 3 of 8.1.3.2. Another simulant that has been used for non-polar organic substances is powdered charcoal. A third simulant that has been used to test plastics intended for contact with dry and frozen foods is silica gel which has been partially saturated with water and which can be suitable to determine the release of polar volatile substances. None of these three simulants has yet been fully validated and standardised for use in testing plastics for intended contact with dry and frozen foods.

7.5 Testing for fatty contact

The simulants have been specified according to the type of foodstuff the plastics is intended to contact in actual or foreseeable use. Fatty food simulants, simulant D, are used for testing plastics intended to contact fatty foodstuffs. For certain specified food types, testing with simulant D can be dispensed with if it can be demonstrated, by means of an appropriate test, that there is no 'fatty contact' between the plastics and the foodstuff with which it comes into contact.

A method for determining whether a food makes fatty contact was prepared by a Subcommittee (SC1) of TC 194 'Utensils in contact with food' under EN 14481.

The principle of the method is that food, of a similar nature to that which will contact the plastics in actual use, is placed in contact with a polyethylene test film into which has been incorporated a fat-soluble fluorescent dye. After exposure to the film, the dye is extracted from the food and the quantity transferred from the film is determined by high performance liquid chromatography with fluorescence detection. The degree of transfer indicates whether the food has made fatty contact with the plastics or not and hence determines whether the plastics shall be tested with simulant D or not.

8 Migration test, substitute test and alternative test conditions and conditions of residual content determination

8.1 Test conditions for migration tests

8.1.1 General

NOTE Basic rules necessary for testing the migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs are laid down (reference Council Directive 82/711/EEC and its subsequent amendments [3], [4], [5]).

The test times and temperatures are chosen according to conditions of contact in actual use. Tolerances on contact times and contact temperatures for migration tests are detailed in Tables C.1 and C.2.

8.1.2 Introduction

The migration tests are carried out selecting the times and temperatures, specified in Table 3, which correspond to the worst foreseeable conditions of intended contact for the plastics material or article, and to any labelling information on maximum temperature for use. Therefore, if the final plastics material or article is intended for a food contact application covered by a combination of two or more times and temperatures taken from the table, the migration test shall be carried out subjecting the test specimen successively to all the applicable worst foreseeable conditions appropriate to the sample, using the same portion of food simulant.

8.1.3 Contact conditions generally recognized as ‘more severe’

8.1.3.1 General

In the application of the general criteria that the determination of the migration shall be restricted to the test conditions which, in the specific case under examination, are recognised to be the most severe on the basis of scientific evidence, some specific examples for the test conditions are given below.

8.1.3.2 Contact with foodstuffs at any condition of time and temperature

Many articles can be used at a variety of temperatures and for varying times, or their conditions of use might not be known. Where the plastics material or article can in actual use be employed under any conditions of contact time, and no labelling or instructions are given to indicate contact temperature and time expected in actual use, depending on food type(s), simulants(s) A and/or B and/or C shall be used for 4 h at 100 °C or for 4 h at reflux temperature and/or simulant D shall be used only for 2 h at 175 °C.

Table 3 — Conventional conditions for migration tests with food simulant

Conditions of contact in worst foreseeable use	Test conditions
Contact time	Test time
t ≤ 5 min	see the conditions in 8.1.7
5 min < t ≤ 0,5 h	0,5 h
0,5 h < t ≤ 1 h	1 h
1 h < t ≤ 2 h	2 h
2 h < t ≤ 4 h	4 h
4 h < t ≤ 24 h	24 h
t> 24 h	10 d
Contact temperature	Test temperature
T ≤ 5 °C	5 °C
5 °C < T ≤ 20 °C	20 °C
20 °C < T ≤ 40 °C	40 °C
40 °C < T ≤ 70 °C	70 °C
70 °C < T ≤ 100 °C	100 ° C or reflux temperature
100 °C < T ≤ 121 °C	121 °C [a]
121 °C < T ≤ 130 °C	130 °C [a]
130 °C < T ≤ 150 °C	150 °C [a]
T > 150 °C	175 °C [a]
[a] This temperature shall be used only for simulant D. For simulants A, B, or C the test can be replaced by a test at 100 °C or at reflux temperature for a duration of four times the time selected according to the general rules of 8.1.2	

NOTE These conventional conditions for migration tests with food simulants are specified in European Council Directive 82/711/EEC [3] as amended by [4] and [5].

8.1.3.3 Contact with foodstuffs at room temperature or below for an unspecified period

Where the materials and articles are labelled for use at room temperature or below or where the materials and articles by their nature are clearly intended for use at room temperature and below, the test shall be carried out at 40 °C for 10 days. These conditions of time and temperature are conventionally considered to be the more severe.

8.1.4 Contact for less than 15 min at temperatures between 70 °C and 100 °C

If the plastics material or article can in actual use be employed for periods of less than 15 min at temperatures between 70 °C and 100 °C, e.g. hot fill, and is so indicated by appropriate labelling or instructions, only the 2 h test at 70 °C shall be carried out. However if the material or article is intended to be used also for storage at room temperature, the test at 70 °C for 2 h is replaced by a test at 40 °C for 10 d, this being conventionally the more severe test.

8.1.5 Contact in a microwave oven

For materials and articles intended for use in microwave ovens, migration testing can be carried out in either a conventional oven or a microwave oven provided the appropriate time and temperature conditions are selected.

A method has been prepared by a Subcommittee (SC1) of TC 194 'Utensils in contact with food' to measure the temperature, during microwave heating and during heating in a conventional oven, at the interface of food with packaging materials, see EN 14233.

In principle, the temperature is recorded during the heating time using a fluoroptic thermometer. The temperature probes are positioned at the interface of the food with the plastics material or article. The highest temperature recorded is taken to establish the appropriate test temperature in a conventional migration test.

8.1.6 Contact conditions causing changes in physical or other properties

If it is found that carrying out the test under the chosen contact conditions causes physical or other changes in the test specimen which do not occur under worst foreseeable conditions of use of the plastics material or article under examination, the migration tests shall be carried out under the worst foreseeable conditions of use in which these physical or other changes do not take place.

In general a decrease of temperature will suffice, but also reduction of contact time can be required. The new test conditions have not necessarily to comply with the temperatures or time conditions in Table 3. Intermediate conditions are allowable. It shall be reconsidered whether the new established exposure conditions are still representative for the intended purpose.

8.1.7 Contact not covered by the conventional condition for migration tests

In those instances where the conventional conditions for migration tests do not adequately cover the conditions in actual use, for instance contact at temperatures greater than 175 °C or contact times of less than 5 min, other contact conditions can be used which are more appropriate to the case under examination, provided that the selected conditions represent the worst foreseeable conditions of contact.

8.1.8 Testing at low temperatures

When testing with fat simulants, simulant D, at 5 °C the simulant can partially or totally solidify. As many fatty foods are solids or partial solids at this temperature, the state of the simulant can replicate the state of the foodstuff when in contact with the plastics material or article at 'low' temperatures, e.g. chilled storage temperature. Solidification or partial solidification of the simulant however, can lead to variable and unreliable test results. The following are possible solutions for this problem:

- a sunflower oil, which is free of components which solidify at the 5°C temperature of test, i.e. a "dewaxed" oil, can be used;
- fat simulant is heated just to a liquid, and after bringing the simulant into contact with the test specimen the simulant is cooled down immediately to the intended temperature;
- in case of film or sheet samples, a slice of solid fat simulant, a mixture of synthetic triglycerides, is sandwiched between two pieces of test sample.

Testing by total immersion or in a cell or in a pouch or by filling is practicable at low temperatures. If a cell is used where it is difficult to make visual checks for solidification of the simulant, a dewaxed simulant shall be used.

With olive oil and sunflower oil the test is usually without this problem at 10 °C. However, this is a 'more severe' test, but if the migration does not exceed the specified limit when tested at 10 °C, then it would also not have exceeded the limit at 5 °C.

8.1.9 Testing at high temperature

Experience from overall migration testing indicates that it is difficult to obtain consistent, comparable and reliable test results in interlaboratory trials with the test conditions for simulating exposure at temperatures of use in excess of 100 °C over brief periods of time. The main source of inconsistency appears to be due to variation in the time required to achieve the test temperature with olive oil (simulant D) and other fatty food simulants. Various options such as exposure of sample tubes in electrically heated cells, or migration cells provided with controlled electrical heating etc. are under investigation as possible solutions to the problem.

8.1.10 Caps, gaskets, stoppers or similar sealing devices and lids

In many cases lids and closures can be expected to come into contact with foodstuffs and are tested under similar conditions to the rest of the container. However in some high temperature applications the lid can only be exposed to water vapour and this condensed vapour can be returned to the bulk of the foodstuff. In such cases the lids and closures tested with simulant A at reflux temperature is appropriate.

8.1.11 Tubing, taps, valves, filters

Defining the time of exposure can be difficult for articles such as tubing, taps, valves, filters etc. as they can be in contact with flowing foodstuff. However, this exposure can be considered to be repeated brief contact for the purposes of migration testing. Such articles can be tested by repeated total immersion or by repeated filling. Tubing can be stoppered with an inert stopper. To select the exposure time for tubing, the retention time of the foodstuff, which is subject to the flow rate of the foodstuff, as well as length and diameter of the tubing, shall be taken into account. Also the stop flow time can influence the final contact time.

8.2 Test conditions for substitute fat tests

Corresponding conventional conditions for the substitute tests have been agreed. For examples of the most important conventional migration test conditions for substitute tests, see Table 4.

Table 4 — Conventional conditions for substitute tests

Test conditions with simulant D	Test conditions with iso-octane	Test conditions with ethanol 95 %	Test conditions with MPPO [a]
10 d at 5 °C	0,5 d at 5 °C	10 d at 5 °C	-
10 d at 20 °C	1 d at 20 °C	10 d at 20 °C	-
10 d at 40 °C	2 d at 20 °C	10 d at 40 °C	-
2 h at 70 °C	0,5 h at 40 °C	2 h at 60 °C	-
0,5 h at 100 °C	0,5 h at 60 °C (**)	2,5 h at 60 °C	0,5 h at 100 °C
1 h at 100 °C	1,0 h at 60 °C(**)	3,0 h at 60 °C [b]	1 h at 100°C
2 h at 100°C	1,5 h at 60 °C(**)	3,5 h at 60 °C [b]	2 h at 100 °C
0,5 h at 121 °C	1,5 h at 60 °C(**)	3,5 h at 60 °C [b]	0,5 h at 121 °C
1 h at 121 °C	2,0 h at 60 °C(**)	4,0 h at 60 °C [b]	1 h at 121 °C
2 h at 121 °C	2,5 h at 60 °C(**)	4,5 h at 60 °C [b]	2 h at 121 °C
0,5 h at 130 °C	2,0 h at 60 °C(**)	4,0 h at 60 °C [b]	0,5 h at 130 °C
1 h at 130 °C	2,5 h at 60 °C(**)	4,5 h at 60 °C [b]	1 h at 130 °C
2 h at 150 °C	3,0 h at 60 °C(**)	5,0 h at 60 °C [b]	2 h at 150 °C
2 h at 175 °C	4,0 h at 60 °C(**)	6,0 h at 60 °C [b]	2 h at 175 °C

[a] MPPO = modified polyphenylene oxide

[b] The volatile tests media are used up to a maximum temperature of 60 °C. A precondition of using the substitute tests is that the material or article will withstand the test conditions that would otherwise be used with simulant D. Immerse the test specimen in olive oil under the appropriate conditions. If the physical properties are changed (e.g. melting, deformation) then the material is considered unsuitable for use at that temperature. If the physical properties are not changed then proceed with the substitute tests using new specimens.

NOTE 1 These conventional conditions for substitute tests are specified in Commission Directive 97/48/EC [5] the second amendment to Council Directive 82/711/EEC [3].

NOTE 2 Since conducting a 12 h test can pose organizational problems to a laboratory, a prolonged test, for example of a more manageable 16 h, can be applied. This is acceptable as long as the migration limit is not exceeded under such more severe test conditions.

Other combinations of time and temperature can be used. In such cases the examples detailed above shall be taken into account as well as existing experience for the type of plastics under examination.

8.3 Test conditions for alternative fat tests

8.3.1 Alternative fat test with volatile media

The test conditions for alternative tests using volatile test media such as iso-octane and 95 % ethanol in aqueous solution or other volatile solvents or mixtures of solvents are chosen so that:

a) the 'alternative test' result obtained is equal to or greater than that obtained in a corresponding migration test with a fatty food simulant (simulant D);

b) the 'alternative test' result does not exceed the specific migration limit, after application of appropriate reduction factors.

If either or both conditions are not fulfilled, then a migration test with a fatty food simulant shall be performed.

8.3.2 Extraction tests

The test conditions are selected so that the result obtained using these extraction tests is equal to or higher than that obtained with simulant D.

Residual content determinations

For the determination of the residual content, to establish compliance with QM or QMA limits, the plastics shall be treated in such a way that all of the substance under test is removed from the test specimen.

9 Apparatus

9.1 Specimen supports

In the methods for determining migration by total immersion, cruciform specimen supports, see Figure D.1, are specified, but other supports can be used providing they are capable of holding and keeping the test pieces apart and at the same time ensuring complete contact with the simulant. An example of a type of support that has been used successfully, particularly for thick and very thin samples, which are wound around the support, is shown in Figure D.2. This type of support when loaded with the specimens is exposed to the simulants in 100 ml beakers. The beaker is then covered with a watch glass.

9.2 Tubes, glass rods and glass beads

In several of the methods for determining migration by total immersion the samples are tested at a fixed ratio of surface area of test specimen to food simulant volume. In order to ensure that all parts of the test specimen are in contact with the food simulant, glass tubes of the appropriate diameter are used. The dimensions of the suitable tubes are specified in the individual methods. However, minor adjustments to the level of the simulant in the tubes can be made by adding glass rods or glass beads sufficient to ensure complete immersion of all of the surfaces of the test specimen. Again the dimensions of suitable glass rods and glass beads are specified in the individual methods.

9.3 Cells

In the procedures described in this part the availability of the cell, type A Mark 2 as shown in Figure D.3, has been assumed. However the alternative cells shown in annex D produce results which, within experimental error, are similar. Alternative cells shall be of such design to give satisfactory performance, particularly freedom from leakage, with all four food simulants and with minimum area of the test specimen not in direct contact with the food simulant. Also, the cell design and material(s) of constructions shall not allow transfer of substances which contaminate the food simulant, particularly substances which subsequently interfere in the analysis of the substance under examination. Examples of other cells that are available are type A, type B, type C; type D, type E, and type F these are shown in Figures D.4, D.5, D.6, D.7, D.8, and D.9 respectively. Suitability of the various types of cells for the determination of the migration of volatile substances depends on the design of the cell, the food simulant and the physical properties of the subject substance. Suitability of a cell shall be established by filling the cell with a portion of simulant of equal volume to that to be used in the migration test. The simulant shall be spiked with the substance under test at a level at which migration is expected. After storage under relevant test conditions and determination of the substance, the recovery will show suitability of the cell for determination of the volatile substance. Cells without gas tight closures and with large headspace volume are by definition not suitable for volatile substances.

9.4 Thermostatically controlled ovens or incubators

Experience has shown that close temperature control is essential to obtain repeatable results. Therefore care has to be exercised in selecting ovens or incubators to ensure that the temperature control is that specified in Table C.2 throughout the volume of air encompassing the sample tubes, cells or pouches. In particular, refrigerators and ovens of the type used in the home to store and cook foods usually have inadequate temperature control and are consequently unsuitable for use in conducting migration tests.

10 Samples and sample geometry

10.1 Samples

The sample taken for testing for compliance shall be the final article, in its ready-for-use state. In some cases this can be impracticable and specimens can be taken from the material, article or, where appropriate, specimens representative of this material or article can be used.

Tests are also carried out on components and precursors of final articles, i.e. a film intended to be laminated to another film or an unprinted film. These tests are carried out in order to provide guidance on the suitability of the material for use in the manufacture of the final article.

An example is where an article is filled with food at the time it is formed. In this case the test can be carried out on a test article prepared especially for testing purposes. A further example is where the sample to be tested is of inhomogeneous construction, is too large to be tested by filling and no flat surfaces can be cut from the sample for testing in a cell. In this case the test can be carried out on a test article prepared especially for testing purposes. The article shall be as representative as possible of the article in actual use.

Where samples are taken at random from a production batch, this shall be indicated when reporting the result. The samples shall be representative of normal production material. Similarly if the sample was not a random sample, and it was selected according to some other parameter, e.g. thickness variation, this shall also be reported.

Samples can be inhomogeneous, e.g. varying in crystallinity or in molecular orientation, or of irregular shape or thickness, e.g. sections cut from bottles, trays, work surfaces, cutlery etc., or so small that several samples are required to constitute a test specimen. Replicate samples as similar as possible to each other and proportionally representing the sample article shall be tested and the sampling details shall be included in the final report.

Samples shall be clean and free from surface contamination; dust can be removed by wiping the sample with a lint-free cloth or brushing with a soft brush.

If articles are accompanied with an instruction that they shall be cleaned before use, then this instruction shall be followed before testing.

10.2 Surface-to-volume ratio

Where the surface-to-volume ratio to be used in contact with food is known this shall be used in the migration test. An example of this is where a bottle or other container is intended to contain a specified volume of contents, even if this does not completely fill the article. In this case the article shall be tested with the specified volume of simulant.

Where the surface-to-volume ratio to be used in contact with foodstuff is not known, conventional exposure conditions shall be used, i.e. 0,6 dm² of surface area of plastics in contact with 100 g of foodstuff or 100ml of food simulant.

NOTE In Commission Directive 2002/72/EC[2] the specific migration limits (SML) have been set with the assumption that 6 dm² of surface area of plastics comes into contact with 1 kg of food. Under the rules specified for migration testing, the specific gravity of all simulants conventionally is assumed to be '1'. 1 kg of food simulant is therefore taken to occupy the volume of 1 l.

With some migration tests where there are difficulties in achieving an adequate detection limit with the analytical method for the substance under test, it can be possible to obtain a more favourable concentration of the substance in the simulant for analysis by varying the surface-to-volume ratio. However, valid results can only be achieved if the migration values obtained, expressed in milligrams per square decimetre, are the same for both the standard and altered surface-to-volume ratios. This cannot be so if there are solubility limitations. Experience has shown that for many substances, the surface-to-volume ratio can be reduced to a maximum ratio of 1 dm²/20 ml in migration tests with fatty food simulants, and can be reduced to a maximum ratio of 1 dm²/50 ml in migration tests with aqueous simulants.

Before commencing a migration test with a different surface-to-volume ratio, the solubility of the substance in the simulant to be used for the migration test, at the expected migration level, shall be determined before commencement of the test, at both the test temperature and ambient temperature. Substances which are soluble at the test temperature but are not fully soluble at ambient temperature can lead to unreliable analytical test results.

Changes to the surface-to-volume ratio can also be made in 'Substitute Tests' with test media, when similar detection limit difficulties are experienced.

10.3 Single surface versus double surface testing (by total immersion)

Specific migration tests shall be performed in such a way that only those parts of the sample intended to come into contact with foodstuffs in actual use shall be in contact with the foodstuff or simulant. However, it is permissible to demonstrate compliance with a specific migration limit by the use of a more severe test.

In the total immersion test both the surface which is intended to come into contact with the foodstuff and the outside surface are in contact with the food simulant. As only the surface area of the foodstuff contact side is used in the calculation of migration per unit of surface area, no allowance is made for any migration of the substance from the outside surface. It is therefore a more severe test than testing in a pouch or in a cell or by filling.

However, for symmetrical samples only, it is sometimes possible to demonstrate that including both surfaces in the calculation is valid. This is when it can be shown that the specific migration value obtained in the total immersion test including both surfaces in the calculation is, allowing for analytical tolerance, the same as that obtained by single surface testing.

Conventionally, if the thickness exceeds 0,5 mm, the surface area of both sides is taken into account in determining the migration value.

Test specimens with cut edges can give higher migration results than those obtained with test specimens without cut edges. In use the plastics would not normally have cut edges in contact with the foodstuff. Conventionally, in calculating the specific migration result as milligrams of the migrant per square decimetre, the area of the cut edges can be taken into account only if the thickness exceeds 2 mm.

Testing samples with the test specimens prepared by cutting sections from the plastics and totally immersing in the food simulant, is a more severe test. If migration from samples sensitive to increased values due to cutting, exceed the migration limit, then the test shall be repeated while avoiding any contact with cut edges, e.g. by filling, or single sided testing.

The surface-to-volume ratio in the total immersion test is conventionally 6 dm² of food contact area to 1000 ml of food simulant.

The procedure for exposure by total immersion is given in clause 15.

10.4 Single surface testing using a cell type A Mark 2

Where single surface testing is the preferred procedure, particularly important for multi-layer materials and articles, this can be carried out in a cell type A Mark 2 or in an equivalent cell. For samples that can be obtained in flat form, e.g. film, foil or sheet, testing in the cell has the advantage of readily reproducible sample geometry. The cell is described in annex D.

Other cells which can be suitable are referred to in 9.3 and described in annex D.

The surface-to-volume ratio in the Cell type A Mark 2 is conventionally 6 dm² of food contact area to 1000 ml of food simulant.

When using a cell, particular care has to be exercised to minimize loss of volatiles and this can be checked using a simulant "spiked" with the analyte at a known concentration.

The procedure for the exposure in a cell type A Mark 2 is given in clause 17.

10.5 Single surface testing using a pouch

For flat materials and articles which can be formed into durable pouches with seals of sufficient strength to maintain the integrity for the duration of the migration test with the particular simulant, testing in a pouch can be preferred as only equipment to produce the pouch seals is required and there is more efficient use of oven space.

The surface-to-volume ratio of the pouch is conventionally 2 dm² of food contact area to 100 ml of food simulant. If the analyte is determined in milligrams per millilitre (mg/ml), then this can be recalculated for the conventional surface-to-volume ratio of 6 dm² to 1 kg (l) of food or food simulant by multiplying by the factor of 300, the result is then in milligrams per kilogram (litre).

NOTE For test temperatures above 40 °C, it is permissible to fill the pouches with food simulant at ambient temperature and pre-heat the pouches in a microwave oven to reach the test temperature. A procedure that has been found to be suitable is to insert into the simulant of one of the pouches a fibre optic probe or to check the temperature after heating, by thermometer. The filled pouches are placed in a microwave oven and heated until the simulant has attained the test temperature. The pouches are then transferred to a thermostatically controlled oven or incubator that has been pre-heated to the test temperature. This part of the operation should be carried out in the minimum time to prevent undue heat loss. The pouches are left in the oven or incubator for the selected test period.

The procedure for exposure in a pouch is given in clause 18.

10.6 Single surface testing using a reverse pouch

As an alternative to using a pouch, a reverse pouch can be used. In this case the surface intended to come into contact with the foodstuff is made the outer surface of the pouch, which is then exposed to the food simulant by total immersion.

The use of a reverse pouch offers advantages over the pouch. As the ‘normal’ pouches are filled with the simulant the sealed edges have to be capable of bearing the weight of that simulant and maintaining integrity for the duration of the test period. If a seal fails, the simulant will tend to leak from the pouch. With the reverse pouch the seals do not have to withstand the pressure of the simulant and consequently are less likely to fail and leak. In addition, the sealed area can be reduced which permits a more accurate measurement of

the surface area exposed to the food simulant. It is however, possible that simulant can leak into the reverse pouch thus increasing the area exposed to simulant. A means of checking if leaks have occurred with pouches made from transparent/translucent film is to seal into each reverse pouch a piece of filter paper which is of similar dimensions to the pouch. If the pouch leaks, the paper will absorb the simulant and this will be visible. Any pouch that leaks shall be discarded and the test repeated.

A further advantage of reverse pouches is that the surface-to-volume ratio can, if required, be adjusted to coincide with that which will arise when the plastics material or article comes into contact with the foodstuff.

Where the surface-to-volume ratio to be used in contact with foodstuff is not known, the conventional exposure conditions shall be used, i.e. 6 dm² of surface in contact with 1000 ml.

10.7 Single surface testing by filling

For articles in container form, e.g. bottles and trays, it is often most convenient to test them by filling with food simulant. For very large containers testing by filling can be impracticable and it can be necessary to fabricate smaller test specimens representing the article to be tested. Any special fabrications to facilitate testing shall be disclosed in the test report.

The procedure for exposure by article fill is given in clause 19.

10.8 Articles intended for repeated use

It is accepted that where an article is intended to come into repeated contact with foodstuffs, tests shall be carried out three times on a single sample using fresh simulant on each occasion. Its compliance shall be checked on the basis of the level of the migration found in the third test. However, if there is conclusive proof that the level of migration does not increase in the second and third test and if the migration limit is not exceeded on the first test, no further test is necessary. No increase in migration is deemed to have occurred if the mean of the results for the third exposure does not exceed the mean of the results for the second exposure.

Unless previous testing has demonstrated that the type of plastics under test does not give rise to increased migration in the second and third exposure periods, all types of articles intended for repeated use are required to be tested for repeated use.

10.9 Caps, closures and other sealing devices

Caps, sealing gaskets and other sealing devices shall be tested under conditions which, as far as possible, simulate actual conditions of use.

The test is carried out on closures in the state and form in which they are intended to be used.

The simulants are placed in jars/containers, known to give consistently low migration, and the jars/containers closed with the test closures. The jars/containers are then inverted and subjected to the test conditions appropriate for the actual conditions of use. In case of high temperature with aqueous food simulants it can be necessary to carry out the treatment with the jar/container in the upright position. If this treatment is followed by storage at room temperature, then the jar/container shall be inverted during that period.

With substitute tests, test media can pass the closure area by diffusion. This will result in loss of test medium and a decrease of migration. However, also the opposite is possible when the test medium extracts the substance from an area outside the jar/container. In this situation the actual contact area shall be determined and the migration of the total closure be determined. After correction for the examined surface, the migration from the closure in actual conditions can be established

The surface to volume ratio used shall be the same as that intended for use.

For containers where the migration needs to be expressed in terms of milligrams per kilogram, the migration from the closure is added to that of the container when assessing compliance with the specific migration limit.

Calculation of the migration in mg/dm² has no meaning as the contact area is difficult to establish and usually relatively small. The migration shall be expressed in mg/closure and related to the content of the jar, container or bottle. An example is a lid intended to be assembled on a 350 ml jar. If X = migration in milligrams per lid, then if the value $X \times 1000/350 < SML$, the lid is in compliance with the restriction and thus suitable for contact with foodstuff.

10.10 Large containers

Large containers can be tested by cutting test specimens from them and testing these by total immersion or by the cell method or using an equivalent cell. Alternatively, the containers can be filled and portions taken for the analytical measurements, after thorough mixing. A third alternative is to fabricate smaller test sample containers representing the large containers, which are then tested by filling.

10.11 Tubing, taps, valves and filters

Articles such as tubing, taps, valves etc. can be in contact with flowing foodstuff, which can be considered to be repeated brief contact for the purposes of migration testing. Such articles can be tested by repeated total immersion or by repeated filling. Tubing can be stoppered with an inert stopper.

10.12 Fibres and cloths

Polymeric fibres and cloths are used to make such articles as sacks, filters, conveyor belting and bags for the infusion of beverages. For these materials, do not calculate the total surface area of the fibres. For example, a 10 cm by 10 cm square specimen of the material has a one-sided contact area of 1 dm² and the contact area for migration calculations is not the total area of all fibres contained with in this square specimen.

10.13 Articles of irregular shape

Many articles which require to be tested are of irregular shape or dimensions, e.g. thickness. Examples of these are sinks and work surfaces, eating and cooking utensils, shaped bottles and containers. When portions of these samples are taken for test by total immersion or in a cell care has to be exercised to ensure that the test specimens selected are representative of the whole of those parts of the article intended to come into contact with food. Also, care shall be taken to ensure that replicate test specimens are sufficiently dimensionally similar, one to another, to allow valid replication of results.

11 Sampling

11.1 Sampling of test articles

When testing for substances subject to either a specific migration limit (SML) or a compositional limit (QM or QMA), plastics materials and articles which have not been in contact with foodstuffs shall be sampled in such a way that the portion taken for testing is representative of the whole food contact area.

With plastics materials or articles containing a volatile substance which is the subject of the test, care shall be taken to ensure that when the sample is tested it is in a state, with respect to the presence of the volatile substance, which is representative of that when the plastics comes into contact with the foodstuff. When considered necessary, loss of the volatile substance from the plastics sample before commencement of the test shall be minimized by storage of the sample at a low temperature, or by hermetic sealing.

Cutting of plastics samples to obtain test specimens can introduce a source of error in certain monomer specific migration tests, as it is known that with some plastics the monomer is re-formed by heat generated in the cutting process. Examples are plastics manufactured with the monomers acrylonitrile and butadiene from the respective acrylonitrile polymers/co-polymers and butadiene polymers/co-polymers. Migration testing of these plastics types with the simulant in contact with cut edges shall be avoided where possible. If it is necessary to perform the specific migration test on these plastics types with cut test specimens, it is recommended that cutting is performed under cryogenic conditions.

Cutting of samples can also induce changes in morphology of the plastics which in turn can influence migration of substances and the validity of the test result. Also, with some plastics samples there can be a difference in morphology between the surfaces and the 'core'.

Migration testing of test specimens with cut edges by the total immersion procedure, usually implies that it is a 'more severe' test.

In determinations for compliance with a QM or QMA limit, the formation of monomer upon cutting of the plastics sample can produce test results which are even more unreliable than those incurred in migration tests. If the plastics samples are relatively small, whole samples shall be used in the solvent dissolution/extraction or any other procedure used to isolate the monomer, and portions of the solvent taken for the analysis. Where it is necessary to cut or grind the plastics to produce test specimens, the generation of heat during the cutting/grinding operation shall be minimized, and where possible performed under cryogenic conditions.

In less critical situations, the whole of the food contact area of the plastics article shall be cut into small pieces which are subsequently mixed and a randomized portion taken for analysis.

11.2 Sampling of foodstuffs

Where a plastics material or article has been in contact with a foodstuff it is not practicable to test with food simulants and therefore, compliance can only be demonstrated by analysis of the foodstuff. Care has to be taken in sampling of the foodstuff to avoid loss of volatile substances and also to ensure that portions taken for analysis are representative of the foodstuff as a whole. Wherever possible the entire contents of the article shall be homogenized under cooled conditions and portions of the homogenized material taken for subsequent analysis.

12 Precision

Precision data enables an assessment of the significance of a test result obtained from tests performed with the standard test method, and the significance of the result in comparison with a result obtained by another analyst in a different laboratory.

The basic precision data which are required for each test method are:

'r' - repeatability value;

'R' - reproducibility value.

For the calculation of *'r'* and *'R'* refer to ISO 5725.

13 Expression of results

13.1 General - specific migration test results

13.1.1 Introduction

For articles with a volume of less than 500 ml and more than 10 l the specific migration test results shall be expressed as milligrams per square decimetre (mg/dm^2). In these cases the specific migration limits, expressed in milligrams per kilogram (mg/kg), shall be divided by the conventional conversion factor of 6 in order to express them in milligrams per square decimetre (mg/dm^2).

The various circumstances are detailed in 13.1.3, 13.1.4 and 13.1.5.

13.1.2 For unknown surface-to-volume ratios

When the surface-to-volume ratio in actual use is not known the results obtained under the test conditions shall be reported in milligrams per square decimetre (mg/dm^2).

13.1.3 For known surface-to-volume ratios and tested under these conditions

When the surface-to-volume ratio in actual use is known, and the tests have been carried out under these conditions and the plastics articles are articles which are containers or are comparable to containers or which can be filled, with a capacity of less than 500 ml and more than 10 l, the results shall be expressed in milligrams per square decimetre (mg/dm^2).

When the surface-to-volume ratio in actual use is known, and the tests have been carried out under these conditions and the plastics articles are not articles which are containers or are comparable to containers or which can be filled, with a capacity of less than 500 ml and more than 10 l, the results shall be expressed in milligrams per kilogram (mg/kg).

13.1.4 For known surface-to-volume ratios and not tested under these conditions

When the surface-to-volume ratio in actual use is known, but the tests have not been carried out under these conditions and the plastics articles are articles which are containers or are comparable to containers or which can be filled, with a capacity of less than 500 ml and more than 10 l, the results shall be recalculated to the actual conditions of use and expressed in milligrams per square decimetre (mg/dm^2).

When the surface-to-volume ratio in actual use is known, but the tests have not been carried out under these conditions and the plastics articles are not articles which are containers or are comparable to containers or which can be filled, with a capacity of less than 500 ml and more than 10 l, the results shall be recalculated to the actual conditions of use and expressed in milligrams per kilogram (mg/kg).

Where results are expressed in milligrams per square decimetre (mg/dm^2) they shall be compared to the limits recalculated in milligrams per square decimetre (mg/dm^2) obtained by dividing the substance limits in milligrams per kilogram (mg/kg) by the conventional conversion factor of 6.

13.1.5 Conversion recalculation

The equation for the conversion of milligrams per kilogram (mg/kg) to milligrams per square decimetre (mg/dm^2) is:

$$M = \frac{c \times V}{a1 \times 1000} \qquad (1)$$

where:

- M is the migration in milligrams per square decimetre;
- C is the concentration in milligrams per kilogram of substance released to the food simulant by the sample as determined by the migration test;
- V is the volume of food simulant used in the migration test in millilitres;
- $a1$ is the surface area in square decimetres of the sample in contact with the foodstuff or simulant during the migration test.

This assumes that the migration expressed in milligrams per square decimetre is independent of surface-to-volume ratio.

13.2 Reduction factors with the fat simulant

In Table 2 when "X" is followed by an oblique stroke and a figure, the results of the migration tests shall be divided by the number indicated. In the case of certain types of fatty foodstuffs this figure, known as the "reduction factor", is conventionally used to take account of the greater extractive capacity of the simulant for such foodstuffs.

Where tests have been carried out in fat simulant and where the article is intended to be used with a variety of fatty foods, the migration shall be calculated and reported for all possible factors.

The procedure for calculating the migration value when reduction factors are permissible for certain types of food will vary according to the legal limitation for that substance. Some limitations are expressed as numerical values for a single substance. Some limitations are expressed as a total numerical value for a combination of substances. Other limitations are that a substance shall not be detected by an analytical method with a stated limit of detection. In addition some limitations are that a substance shall not be detected by an analytical method with a stated limit of detection, analytical tolerance included.

In the first case, that is when the limit is expressed as a numerical value for that substance, the procedure is as follows:

The migration shall be calculated without factors and reported using no factors; and with a factor of 2; and with a factor of 3; and with a factor of 4; and with a factor of 5. However, if the foodstuff with which the plastics material or article is intended to come into contact is known then the migration can be calculated and reported using only the factor appropriate to that known foodstuff.

In all other cases, the procedure for calculating the migration value with reduction factors is given in the relevant part of the standard describing the method for each specific substance.

13.3 Calculation of QA for compliance with QMA

When a specified area is used in the procedure, then the equation for the calculation of the QA is as follows:

$$QA = c \times VA \qquad (2)$$

where:

QA is quantity in milligrams per square decimetre;

c is the concentration in milligrams per millilitre of solvent as determined in the analysis;

V is final volume of the extract of the test specimen in millilitres;

A is area in square decimetre (dm^2) of the test specimen.

If the article was thicker than 0,25 mm then the *QA* shall be corrected for the maximum thickness of 0,25 mm.

$$QA = c \times (VA) \times (0{,}25 / w) \qquad (3)$$

where

w is the thickness of the test specimen taken for the analysis, in millimetres.

When a specified weight of polymer has been taken in the procedure then the calculation of the QA is as follows:

$$QA = c \times V \times 100 \times d \times (L / 10W) \qquad (4)$$

$$QA = c \times V \times 100 \times d \times (L / W) \qquad (5)$$

where:

QA is quantity in milligrams per square decimetre;

c is the concentration in milligrams per millilitre of solvent as determined in the analysis;

V is final volume of the extract of the test specimen in millilitres;

d is density of the polymer in grams per cubic centimetre;

L is thickness of the test article with a maximum of 0,25 mm;

W is the weight of the sample used in the analysis in gram.

13.4 Validity of results

Samples of the substance of known purity, or solutions of the substance of known strength, where stability permits, available from the Measurement and Testing Programme of Directorate General Research of the European Communities, can be used for calibration purposes and for establishing that the standard methods are being satisfactorily applied to yield valid results.

The performance characteristics for most of the methods for the determination of specific substances, or groups of substances, in food simulants have been established. The performance characteristics for a specific substance determination are given in the performance section for each part of this standard. Each laboratory carrying out specific substance migration testing has to demonstrate that it is carrying out the procedure competently. This can be shown, for example by the results obtained using solutions fortified with known levels of the substance.

NOTE When determinations are carried out in actual foodstuffs the performance characteristics could be inapplicable for the analysis of food. The laboratory carrying out the tests should assure itself that it can carry out valid tests with the food and that there are no adverse effects such as interfering substances or reactions with the food which would make the method invalid.

13.5 Confirmation of results

In cases where the substance migration from the plastics material or article into foodstuff or food simulant or where the substance level in the plastics material or article itself exceeds restriction limit, the identity shall be confirmed by the specified confirmation procedure. This procedure shall include quantification, where appropriate.

The substance level found by the standard method described shall be used when reporting the determination.

13.6 Group limits

There are some groups of substances which because they contain similar chemical moieties, or functional groups, within their molecular structures and their toxicological effects are similar and additive, have been given group migration limits, SML(T), or group composition limits QM(T). For each group the sum of the migration values or compositional contents for the individual substances might not exceed the SML(T) or QM(T) value for the group. An example is the SML(T) for the glycols - ethylene glycol, diethylene glycol and stearic esters of ethylene glycol. The SML(T) value applies to the migration value of each individual glycol where only one of the glycols is present in the plastics sample, or to the combined migration values of the three glycol substances where all are present. Other examples are the group of isocyanates, where the QM(T) group limit is expressed as a concentration of NCO in the final product, and the pair of monomers caprolactam and caprolactam sodium salt which have an SML(T).

For caprolactam, and its sodium salt, and the glycols, where the same analytical method can be used for all substances in the group and where each of the substances can be reliably determined at levels well below the total for the group, there are few problems.

When summing the total levels of moiety present, all contributions less than 10 % of the group limit and all contributions below the detection limit are conventionally disregarded.

Details on how to calculate and report the results for these groups are given in the appropriate method in other parts of this standard.

14 Test reports and statements of compliance

14.1 Test reports

The test report shall include the following particulars, where known:

a) all information necessary for complete identification of the sample, e.g. chemical type, trade mark, grade, batch number, thickness, etc.;

b) form of the plastics, e.g. film, bottle, pot, etc.;

c) use/class of food for which the sample is intended to contact, where known, and where possible food classification reference number as listed in Table 2;

d) intended conditions of use, where known e.g. time/temperature;

e) conditions of the test;

1) part(s) of this standard used;

2) foodstuffs or food simulants used;

3) duration and temperature, and relation with "Conditions of contact in worst foreseeable use", as defined in Table 3;

4) area and geometry of the test specimen;

5) volume of foodstuff or food simulant used where appropriate;

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

g) particular requirements of the parts of this standard;

h) relevant comments on the test results;

i) details of any confirmation procedure(s);

k) individual triplicate or quadruplicate test results, and the mean of these results expressed, for SMLs, in units of milligrams per kilogram of foodstuff or food simulant (mg/kg) or, in the units milligrams per square decimetres of surface area (mg/dm^2) and, for QMs, milligrams per kilogram (mg/kg) of final product and for QMAs in the units milligrams per square decimetres of surface area (mg/dm^2).

15 Exposure by total immersion in a thermostatically controlled oven, incubator or refrigerator

NOTE These methods are most suitable for plastics in the form of films and sheets, but can be applied to a wide range of articles or containers from which test pieces of suitable size can be cut.

15.1 Introduction

The method describes the exposure of plastics which are intended to come into contact with foodstuffs, by total immersion to food simulants or test media for the subsequent analysis of substances. The method is applicable for exposure to aqueous food simulants at temperatures up to but not including 100 °C, exposure to aqueous food simulants at reflux temperatures and exposure to fat simulants at all temperatures up to 175 °C and exposure to the substitute test media, iso-octane and 95 % (v/v) aqueous ethanol, at temperatures up to and including 60 °C.

15.2 Principle

The selection of the conditions of test will be determined by the conditions of use, see clause 8.

Test specimens are immersed in food simulant or test medium for the specified period at a set temperature then removed. The food simulant or test medium is analysed using an appropriate method. Methods for the determination of some substances are to be found in the subsequent parts of this standard.

15.3 Reagents

15.3.1 Distilled water or water of equivalent quality (simulant A).

15.3.2 Acetic acid 3 % (w/v) in aqueous solution (simulant B).

15.3.3 Ethanol 10 % (v/v) in aqueous solution (simulant C).

15.3.4 Alcoholic simulants for liquids or beverages of an alcoholic strength exceeding 10% (v/v).

NOTE In the case of materials and articles intended to come into contact with liquids or beverages of an alcoholic strength exceeding 10 % (v/v) the test can be carried out with aqueous solutions of ethanol of a similar strength.

15.3.5 Olive oil, simulant D as specified in clause 8.

15.3.6 Dewaxed sunflower oil for determinations at low temperatures.

15.3.7 Test media for substitute tests

15.4 Apparatus

15.4.1 Cutting slab: clean smooth glass, metal or plastics slab of suitable area to prepare test specimens

NOTE 250 mm x 250 mm is suitable.

15.4.2 Stainless steel, blunt nosed tweezers.

15.4.3 Cutting implement: scalpel, scissors or sharp knife or other suitable device.

15.4.4 Metal templates for preparing test pieces and specimens:

a) 100 mm $\pm$ 0,2 mm x 60 mm $\pm$ 0,2 mm (regular rectangle);

b) 20 mm $\pm$ 1 mm x 100 mm $\pm$ 1 mm (regular rectangle).

15.4.5 Rule, graduated in millimetres, with an accuracy of 0,1 mm.

15.4.6 Specimen supports, see Figure D.1, constructed of stainless steel with cross arms attached by welding or silver soldering. Stainless steel X4 CrNi 18 10 according to EN 10088-1 or of composition, chromium 17 %, nickel 9 %, carbon 0,04 % is suitable. These shall be of low thermal capacity. Before initial use the supports shall be thoroughly cleaned first with a degreasing solvent and then with dilute nitric acid. For the aqueous acetic acid food simulant, supports constructed out of glass are recommended, as there is a tendency for the acetic acid to corrode stainless steel supports, particularly if the joints are silver soldered.

NOTE The method has been written for the supports shown in Figure D.1 which have been found to be suitable for holding thin film and sheet test pieces. However, other supports could be used providing they are capable of holding and keeping the test pieces apart and at the same time ensuring complete contact with the simulant or test medium. For rigid samples, supports with a single cross arm can be used.

15.4.7 Gauze, pieces of fine stainless steel gauze approximately 25 mm x 100 mm or, glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long to be used with the acetic acid food simulant for insertion between the test pieces. Before initial use the gauze shall be thoroughly cleaned first with a degreasing solvent and then with dilute nitric acid.

15.4.8 Glass tubes, ground necks and stoppers, for retaining the food simulant or test medium and test specimens. The tubes shall have an internal diameter of approximately 35 mm and length of in the range of 120 mm to 200 mm, excluding the ground neck, with B34/35 stopper fitted with a gas tap. The tubes shall

have a volume of 125 ml excluding the stopper but with a minimum headspace. For the testing of non-volatile substances a stopper without tap can be used.

15.4.9 Glass beads, 2 mm to 3 mm diameter or glass rods, 2 mm to 3 mm in diameter and approximately 100 mm long.

15.4.10 Thermostatically controlled oven, incubator or refrigerator capable of maintaining the set temperature, within the tolerances specified in Table C.2.

15.4.11 Measuring cylinder, of 100 ml capacity, complying with the minimum requirements of ISO 4788.

15.5 Preparation of test specimens

NOTE Under no circumstances should the sample be washed with water or solvent. If it is specified in the instructions of use that it should be washed or cleaned before use it is recommended to refer to 10.1.

15.5.1 Number of test specimens

Three test specimens are required for samples in the form of thin films, sheet, pots, containers or similar articles. For samples where the specific migration limit is expressed in milligrams per square decimetre five test specimens, similar dimensionally one to another, are required for samples of articles of irregular shape.

These test specimens are utilized as follows:

a) three specimens for the migration test;

b) two specimens for determination of the surface area, in the case of samples of irregular shape;

15.5.2 Thin films and sheet materials

Lay the sample on the cutting slab (15.4.1) and cut the test specimens, each 0,6 dm², using the 100 mm x 60 mm template (14.4.4a). Check, using the rule (15.4.5), that the dimensions of the specimen are within the tolerance (± 1 mm).

Cut each test specimen into three test pieces 20 mm x 100 mm using the appropriate template (15.4.4b). Assemble one test specimen onto the support by piercing suitable holes in the test pieces and placing one test piece on one cross arm and two test pieces on the other cross arm. Repeat this procedure for all remaining test specimens.

15.5.3 Containers and other articles

Cut sections from the walls of the pot, container or article to give test specimens each of area approximately 0,6 dm². For articles with individual areas less than 0,6 dm², use a number of articles to provide each test specimen. Measure the dimensions of each test specimen to the nearest 1 mm, using the rule. Only the surface area of the sample which is intended to come into contact with foodstuffs shall be measured, that is, cut edges and any surfaces not intended to come into contact with foodstuffs are ignored. The area of the cut edges can be taken into account only if the thickness exceeds 2 mm (see 10.3). Calculate the area of each test specimen to the nearest 0,01 dm² and record. If necessary, cut each test specimen into smaller pieces to enable them to fit into the glass tubes (15.4.8). The test specimens or pieces are placed on the specimen supports if these are appropriate or, if the test specimens or pieces are sufficiently rigid, they can be tested unsupported.

15.5.4 Articles of irregular shape

Select representative portions of the article, or multiples of the article for small articles, to give five dimensionally similar test specimens each with a surface area of approximately 0,6 dm². Measure the surface area of two of these specimens to the nearest 0,05 dm² using the Schlegel Method (EN ISO 8442-2:1997, annex B), or any other suitable method. Only the surface area of the sample which is intended to come into contact with foodstuffs shall be measured, that is cut edges and any surfaces not intended to come into contact with foodstuffs are ignored (see 10.3). Record the surface area of each test specimen.

15.5.5 General

Ensure that test pieces are well separated and that the surfaces are freely exposed to the foodstuff or food simulant or test medium during the period of the test. For thin films: insert a piece of fine stainless steel gauze, or glass rods with the acetic acid simulant, between the test pieces. For thick samples not placed on the supports: insert glass rods between the test pieces after immersion in food simulant or test medium. Where specimen supports are used label the supports with a tag bearing the test specimen identification.

15.6 Procedure

NOTE 1 The procedure described here takes the full precautions for volatile substances, for non-volatile substances the procedures can be modified where appropriate.

Take four of the glass tubes (15.4.8), measure by measuring cylinder 100 ml ± 1 ml of the aqueous food simulant or test medium for substitute tests into each tube and stopper the tube. In the case of olive oil and simulant D measure by weighing 100 g ± 1 g into each tube and stopper the tube. Place the four tubes in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature, and leave until the test temperature has been attained.

Place a test specimen into three of the tubes containing food simulant or test medium and stopper the tube and where appropriate ensure that the gas tap is closed. Mark the tubes for identification. Ensure that the test specimens are totally immersed in the simulant or test medium; if they are not then add either pre-heated glass beads or rods (15.4.9) to raise the level of the simulant or test medium until total immersion is achieved.

NOTE 2 This part of the operation should be carried out in the minimum time to prevent undue heat loss from the simulant or test medium.

Replace all of the tubes in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature and observe the temperature, leave the tubes for the selected test period, taking into account the tolerances specified in Table C.1.

Remove from the oven or refrigerator and cool to 10 °C for olive oil or 25 °C for a synthetic mixture of triglycerides.

NOTE 3 Cooling should be as rapid as possible. The time taken will depend upon the exposure temperature. For those substances which have "low solubility" in the simulant or test medium, precipitation of the substance could occur when the simulant or test medium is cooled resulting in a false low migration value.

Retain the food simulant or test medium and take portions as appropriate for analysis.

16 Exposure by total immersion at reflux temperatures

16.1 Introduction

The method describes the exposure of plastics which are intended to come into contact with foodstuffs, by total immersion in food simulants for the subsequent analysis of non-volatile substances. The method is applicable for exposure to aqueous simulants that are subjected to reflux temperatures. This method is not suitable for the exposure of samples for the determination of the specific migration of volatile substances.

16.2 Principle

The selection of the conditions of test will be determined by the conditions of use, see clause 8.

Test specimens are immersed in aqueous food simulant for the specified period at a set temperature then removed. The food simulant is analysed using an appropriate method. Methods for the determination of some substances are to be found in the subsequent parts of this standard.

16.3 Reagents

The reagents shall be the same as in 15.3.

16.4 Apparatus

The apparatus in 15.4 shall be used, with the addition of:

a) flange flasks, 250 ml;

b) condensers to fit the flange flasks;

c) heating mantle for maintaining the simulants at reflux during the exposure.

16.5 Preparation of test specimens

The preparation of the test specimens shall be as in 15.5, but the specimens shall be cut into test pieces 20 mm x 20 mm and supports are not used.

16.6 Procedure

Take four flange flasks (16.4a), measure by measuring cylinder 100 ml of the aqueous food into each flask. Place a test specimen into three of the flasks containing 100 ml food simulant. Mark the flasks for identification. Ensure that the test specimens are totally immersed in the food simulant, glass rods can be used to ensure total immersion.

Place the four flange flasks in the heating mantle and connect the condensers (16.4b).

Turn on the water supply to the condensers.

Switch on the heating mantle (16.4c) and heat to reflux as quickly as possible. When reflux is achieved adjust heat to maintain gentle reflux. Observe the food simulant in the flask, leave for the selected test period, taking into account the tolerances specified in table C.1, following the onset of reflux. Turn off the heating mantle, turn off the water to the condenser and remove the flask from the mantle.

WARNING: The flasks and contents will be hot.

Remove the sample from the flask as soon as possible.

Retain the food simulant and take portions as appropriate for analysis.

17 Single-side exposure in a cell in a thermostatically controlled oven, incubator or refrigerator

17.1 Introduction

The method describes the exposure for the determination of specific migration from one surface only of plastics in the form of sheet and film, which are intended to come into contact with foodstuffs. The method is applicable for exposure to aqueous food simulants at temperatures up to but not including 100 °C, when reflux has to be used, for exposure to substitute test media, up to and including 60 °C, and for fat simulants at all temperatures up to 175 °C.

17.2 Principle

The selection of the conditions of test will be determined by the conditions of use, see clause 8.

Prepared test specimens are placed in the cells and an area of 2,5 dm² of each test specimen is exposed to the food simulant or test medium, when using the cell type A, Mark 2. The food simulant or test medium is

analysed using the appropriate method. Methods for the determination of some substances are to be found in the subsequent parts of the standard. The procedure with the cell type A, Mark 2, described here, takes full precautions for volatile substances. For non-volatile substances either the type A Mark 2 cells or any of the cells referred to in 9.3 can be used.

NOTE The ratio of aqueous food simulant and substitute test medium to exposed area of test specimen should be 100 ml to 0,6 dm² and for olive oil and simulant D 100 g to 0,6 dm².

17.3 Reagents

The reagents shall be as in 15.3.

17.4 Apparatus

17.4.1 Cutting slab: clean smooth glass, metal or plastics slab of suitable area to prepare test specimens.

NOTE 250 mm x 250 mm is suitable.

17.4.2 Stainless steel, blunt nosed tweezers.

17.4.3 Cutting implement: scalpel, scissors, or sharp knife or suitable device.

17.4.4 Cells, Cell type A - Mark 2 as shown in Figure D.3. The diameter of the "O" ring seal from the centre point of the seal on the sealing ring shall be 178,4 mm ± 0,1 mm, to give an area of the test specimen exposed to the food simulant or test medium of 2,5 dm². For details of equivalent cells see 9.3.

17.4.5 Oven, incubator or refrigerator capable of maintaining a temperature of capable of maintaining the set temperature, within the tolerances specified in Table C.2.

17.4.6 Measuring cylinder, 500 ml, conforming to the minimum requirements of ISO 4788.

17.4.7 Conical flasks with ground neck and stoppers, of 500 ml capacity, for pre-heating the simulant or test medium.

17.4.8 Temperature measuring instrument.

17.5 Preparation of the test specimens

NOTE Under no circumstances should the sample be washed with water or solvent. If it is specified in the instructions of use that it should be washed or cleaned before use it is recommended to refer to 10.1.

17.5.1 Number of test specimens

Three test specimens are required in the form of thin films, sheet, and flat sections cut from containers or similar articles.

17.5.2 Cutting test specimens

Lay the sample on the cutting slab (17.4.1) with the surface to be in contact with the simulant or test medium uppermost. Take a sealing ring from a cell (17.4.4) and place on the surface of the sample. Cut out the test specimen by cutting round the outer edge of the ring, using the cutting implement (17.4.3).

NOTE If cells of other designs are used different procedures for cutting the test specimens might have to be used.

17.6 Procedure

NOTE 1 The procedure described here takes the full precautions for volatile substances, for non-volatile substances the procedures can be modified where appropriate.

Take three cells (17.4.4), mark for identification, dismantle and place in the thermostatically controlled oven, incubator or refrigerator (17.4.5), which is set at the test temperature. Connect the thermocouple probes to the sealing rings and leave until the cells reach the test temperature.

Take three 500 ml conical flasks (17.4.7), measure by measuring cylinder 450 ml ± 5 ml of aqueous simulants or substitute test media into each flask. In the case of olive and simulant D measure 450 g ± 5 g into the cells by weighing. Place the flasks in the thermostatically controlled oven, incubator or refrigerator set at the test temperature and leave until the simulant or test medium in each flask reaches the test temperature. Remove the cells from the thermostatically controlled oven, incubator or refrigerator. Place on the base of each cell one of the test specimens. Reassemble the cells, ensuring the clamping screw is well tightened down. Remove the flasks containing the simulant or test medium from the thermostatically controlled oven, incubator, or refrigerator and transfer the simulant or test medium from each flask to each of the test cells through the filler hole until the level of simulant or test medium reaches the base of the filler hole. Return the cells to the thermostatically controlled oven, incubator or refrigerator set at the test temperature.

NOTE 2 The period of time covering the removal of the test cells from the thermostatically controlled oven, incubator or refrigerator, the loading of the test specimens into the cells and the transfer of the simulant or test medium from the flasks to the cells should be kept as short as possible to minimize heat loss of the cells and simulant or test medium.

Monitor the temperature of each of the cells (see NOTE 2) by means of the temperature measuring instrument attached to the thermocouple probes until all three cells have reached the set test temperature, taking into account the tolerance specified in Table C.2 and leave the tubes for the selected test period, taking into account the tolerances specified in Table C.1

NOTE 3 For exposure time of 24 h or more it is acceptable to monitor the temperature of the airbath of the thermostatically controlled oven or incubator or refrigerator, instead of the temperature of the simulant or test medium. However, because of the thermal mass (inertia) of the sample along with the simulant or test medium it is possible that the temperature fluctuation of the free air is greater than the temperature fluctuation (due to the operation of the thermostat) of the sample. If the fluctuation of the air temperature is found to be outside the range specified in Table C.2 then it is recommended that the temperature profile of the sample be monitored since it can itself be within the tolerance value.

Leave for the duration of the test period. Take the cells from the thermostatically controlled oven, incubator or refrigerator and cool to 10 °C for olive oil and sunflower oil and 25 °C for a mixture of synthetic triglycerides.

NOTE 4 Cooling should be as rapid as possible. The time taken will depend upon the exposure temperature. For those substances which have "low solubility" in the simulant or test medium, precipitation of the substance could occur when the simulant or test medium is cooled resulting in a false low migration value.

Remove samples of the simulant or test medium from each cell using a syringe inserted through the septum in the filler plug and analyse as instructed in the method of analysis for the substance to be determined.

18 Single-side exposure with a pouch in a thermostatically controlled oven, incubator or refrigerator

18.1 Introduction

The method describes the exposure of plastics, which are intended to come into contact with foodstuffs, by single surface contact with food simulants or test media in pouches and for the subsequent analysis of the simulants.

The method is applicable for exposure to aqueous food simulants at temperatures up to but not including 100 °C, when either reflux or a sealed system has to be used, for substitute test media at temperatures up to and including 60 °C and for fatty food simulants at all temperatures up to 175 °C.

18.2 Principle

The selection of the conditions of test will be determined by the conditions of use, see clause 8.

Test specimens in the form of pouches are filled with the food simulant or test medium and exposed for the specified period at a set temperature. The food simulant or test medium is analysed using an appropriate method. Methods for the determination of some substances are to be found in the subsequent parts of this standard.

18.3 Reagents

The reagents shall be the same as in 15.3.

18.4 Apparatus

18.4.1 Cutting slab: clean smooth glass, metal or plastics slab of suitable area to prepare test specimens.

NOTE 250 mm x 250 mm is suitable.

18.4.2 Stainless steel, blunt nosed tweezers.

18.4.3 Cutting implement: scalpel, scissors or sharp knife or other suitable device.

18.4.4 Rule, graduated in millimetres, and with an accuracy of 0,1 mm.

18.4.5 Metal template 120 mm ± 1 mm x 120 mm ± 1 mm (square).

18.4.6 Analytical balance capable of determining a change in mass of 0,1 mg.

18.4.7 Pouch holder, the example shown in Figure D.7 has been shown to be suitable, constructed from aluminium or other suitable material or an equivalent holder, plus clips to secure corners of pouches.

18.4.8 Pipettes, conforming to the minimum requirements of ISO 648, of 50 ml and 100 ml capacity.

18.4.9 Glass tubes, ground necks and stoppers, for retaining the food simulant or test medium and test specimens. Tubes with an internal diameter of approximately 35 mm and length in the range of 120 mm to 200 mm, excluding the ground neck, have been found to be satisfactory.

18.4.10 Thermostatically controlled oven, incubator or refrigerator capable of maintaining the set temperature, within the tolerances specified in Table C.2.

18.4.11 Heat or pressure sealing device, for use in forming pouches.

18.4.12 Measuring cylinders, of 100 ml capacity, conforming to the minimum requirements of ISO 4788.

18.5 Preparation of test specimens

NOTE Under no circumstances should the sample be washed with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before use - see 10.1. It is recommended to minimize handling of the samples and where necessary, to wear cotton gloves.

18.5.1 Number of test specimens

Three test specimens are required.

18.5.2 Cutting and preparation of specimens

Lay the sample on the cutting slab (18.4.1) with the surface to be in contact with the food simulant or test medium uppermost and cut the test specimens using the 120 mm x 120 mm template (18.4.5).

Place pairs of the test pieces together with the surfaces to be in contact with the food simulant facing. Using the heat or pressure sealer, join to form pouches with four seals parallel to all four edges, 10 mm from the edge. Measure the distances between the inner edges of the seals to the nearest 1 mm and calculate the

total surface area of the test specimen which will be exposed to the food simulant or test medium, to the nearest 0,01 dm². This shall be approximately 2 dm². Using the cutting implement (18.4.3), remove excess film from the sealed area (to reduce area of film not directly exposed to food simulant or test medium whilst leaving enough to withstand the test conditions without leaking).

Mark each pouch for identification. Cut off one corner of the pouch to leave a hole sufficiently large to insert the tip of a 100 ml pipette.

18.6 Procedure

Take four glass tubes (18.4.9), measure by measuring cylinder 100 ml $\pm$ 1 ml of the aqueous food simulant or test medium for substitute tests into each tube and stopper the tubes. In the case of olive oil and simulant D measure 100 g $\pm$ 1 g, by weighing, into each tube and stopper the tubes. Place the four tubes and the pouch holder in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature and leave until the test temperature has been attained.

Remove the pouch holder from the thermostatically controlled oven, incubator or refrigerator and place the test specimen pouches between the spacers.

Remove the three tubes containing the food simulant or test medium from the thermostatically controlled oven, incubator or refrigerator and into the three test specimens pouches pipette sufficient food simulant or test medium to just fill the pouch, about 100 ml or 100 g, but for thick/semi-rigid materials the quantity is less. Secure the open corner with a clip. If all the simulant or test medium is not used to fill the pouch, retain the tube and residual contents. Measure and record the volume of the residual aqueous food simulant and substitute test medium. In the case of olive oil and simulant D record the weight. Measure and record the area of the pouch in contact with the simulant or test medium. This part of the operation shall be carried out in the minimum time to prevent undue heat loss.

Replace the pouch holder, containing the test specimen pouches, in the thermostatically controlled oven incubator or refrigerator, set at the test temperature, leave for the selected test period , taking into account the tolerances specified in Table C.1.

Take the pouch holder and the tubes containing the blank food simulant from the thermostatically controlled oven, incubator or refrigerator.

If an evident leak has occurred with more than one pouch, the test is invalid and has to be repeated using fresh pouches.

If no evident leaks have occurred in at least two of the pouches, then cool to 10 °C for olive oil or 25 °C for a synthetic mixture of triglycerides.

NOTE Cooling should be as rapid as possible. The time taken will depend upon the exposure temperature. For those substances which have "low solubility" in the simulant or test medium, precipitation of the substance could occur when the simulant or test medium is cooled resulting in a false low migration value.

Retain the food simulant or test medium in the pouch and take portions as appropriate for analysis.

19 Single-side exposure by article fill in a thermostatically controlled oven, incubator or refrigerator

19.1 Introduction

This method is most suitable for plastics in the form of containers and articles that can be filled.

19.2 Principle

Test specimens are filled with the food simulant or test medium, with the minimum of headspace and an aluminium lid is sealed in place using a suitable adhesive. Where it is impracticable to seal a lid then the

entire article filled with simulant or test medium has to be contained in a gas tight vessel. The method is applicable for exposure to aqueous food simulants at temperatures up to, but not including 100 °C, when either reflux or a sealed system has to be used, for exposure to test media for substitute tests, up to and including 60 °C, and for fatty food simulants at all temperatures up to 175 °C.

19.3 Reagents

The reagents shall be the same as in 15.3.

19.4 Apparatus

19.4.1 Beaker, 2 l.

19.4.2 Thermostatically controlled oven, incubator or refrigerator capable of maintaining the set temperature, within the tolerances specified in Table C.2.

19.4.3 Glass jars with wide screw necks with lids and screwbands.

19.5 Preparation of the test specimens

NOTE Under no circumstances should the sample be washed with water or solvent. If it is specified in the instructions for use of the article that it should be washed or cleaned before see 10.1.

19.5.1 Number of test specimens

Three test specimens are required.

Determine and record the volume of aqueous food simulant or substitute test medium required to fill a test specimen. In the case of olive oil and simulant D determine and record the weight of fatty food simulant required to fill a test specimen

For articles with a small volume a number of articles could be required to provide a test specimen. A test specimen shall be made up of a number of articles, so that their combined contents provide sufficient volume for subsequent analysis.

19.5.2 Articles with a capacity of less than 500 ml or more than 10 l

It is necessary to determine the surface area of these articles, which is exposed to food simulant or test medium, since the migration has to be expressed in milligrams per square decimetre of surface area.

19.6 Procedure

NOTE 1 The procedure described here takes the full precautions for volatile substances, for non-volatile substances the procedures can be modified where appropriate.

Mark each of the articles making up each test specimen with an identification code.

Place, in a beaker, a sufficient volume of the food simulant or test medium to fill the three test specimens and to provide one blank in the thermostatically controlled oven, incubator or refrigerator, set at the test temperature and leave until the test temperature has been attained.

Where appropriate seal an aluminium foil lid on the article.

NOTE 2 The adhesive used to seal an aluminium foil lid should be checked to ensure that there are no interferences.

Remove the beaker containing the food simulant or test medium from the thermostatically controlled oven, incubator or refrigerator. Fill the three test specimens with simulant or test medium by injection through the aluminium lid by syringe to within 0,5 cm of the top. If the container has a specified nominal volume of

contents see 9.2. Seal the injection point with adhesive. This part of the operation shall be carried out in the minimum time to prevent undue heat loss from the simulant or test medium.

Where it is inappropriate to seal a lid in place, carry out the test in the open article but placing in an appropriate gas-tight vessel (wide neck glass jar with a septum fitted in the lid for sampling).

Place the test specimens and food simulant or test medium contained in a gas-tight vessel in the thermostatically controlled oven, incubator or refrigerator set at the test temperature, leave for the selected test period taking into account the tolerances in Table C.1

Remove from the oven and cool to 10 °C for olive oil or 25 °C for a synthetic mixture of triglycerides.

NOTE 3 Cooling should be as rapid as possible. The time taken will depend upon the test specimen and the volume of simulant or test medium. For those substances which have "low solubility" in the simulant or test medium, precipitation of the substance could occur when the simulant or test medium is cooled resulting in a false low migration value.

Retain the food simulant or test medium for subsequent analysis. For volatile substances portions can be taken by syringe through the aluminium lid or via the septum if a wide neck glass jar is used.

Annex A
(normative)

Criteria for classification of non-volatility

A.1 Volatile substances

The loss of volatile substances is potentially influenced by a large number of factors. Some are intrinsic properties of the substance e.g. vapour pressure and boiling point. Some are a function of the substance/plastics combination e.g., diffusion rate for the substance through the plastics or partition coefficients for the substance between the plastics and liquids or gases. Some are a function of the exposure conditions, e.g. period of exposure, temperature of exposure, surface-to-volume ratio. In actual use additional factors could influence loss of volatiles e.g., effectiveness of closures, stoppers, or seals; also losses frequently occur when containers are opened prior to consumption of the food.

The testing under sealed conditions are intended to ensure that any loss of volatiles in tests simulating use is always less than that occurring under actual conditions of use. The non-volatile substances are categorized according to the criteria in this annex and for these substances testing under sealed conditions is not essential. The method for the determination of each substance, contained in other parts of this standard, states if the substance is conventionally considered volatile or non-volatile.

A.2 Criteria for conventional classification of non-volatility

A solution of the substance in food simulant or test medium at a concentration of ten times the SML value is conventionally classified as non-volatile if it does not lose more than 10 % of the substance when exposed for the time and temperature specified in the test procedure compared to an identical sample under sealed conditions.

Annex B
(normative)

Characteristics of fatty food simulants and test media

Characteristics of rectified olive oil, reference simulant D

iodine value (Wijs)	= 80 to 88
refractive index at 25 °C	= 1,4665 to 1,4679
acidity, expressed as % oleic acid	= 0,5 % maximum
peroxide number, expressed as oxygen milliequivalent per kg of oil	= 10 maximum
unsaponifiable matter	= < 1 %

Composition of the mixture of synthetic triglycerides, simulant D

Fatty acid distribution

Number of C-atoms in fatty acid moiety

	6	8	10	12	14	16	18	Others
GLC-area-%	~1	6 to 9	8 to 11	45 to 52	12 to 15	8 to 10	8 to 12	≤ 1

Purity

content of monoglycerides (enzymatically)	≤ 0,2%
content of diglycerides (enzymatically)	≤ 2,0%
unsaponifable matter	≤ 0,2%
iodine value (Wijs)	≤ 0,1%
acid value	≤ 0,1%
water content (K. Fischer)	≤ 0,1%
melting point	28 °C ± 2 °C

Typical absorption spectrum (thickness of layer d=1cm, reference: water, 35 °C)

wavelength (nm)	290	310	330	350	370	390	430	470	510
transmittance (%)	~2	~15	~37	~64	~80	~95	~97	~95	~98

at least 10 % light transmittance at 310 nm (cell of 1 cm, reference: water, 35 °C)

Characteristics of sunflower oil, simulant D

iodine value (Wijs)	= 120 to 145
refractive index at 20 °C	= 1,474 to 1,476
saponification number	= 188 to 193
relative density at 20 °C	= 0,918 to 0,925
unsaponifiable matter	= < 0,5 %
acidity, expressed as oleic acid	= < 0,5%

Characteristics of corn oil, simulant D

iodine value (Wijs)	= 110 to 135
refractive index at 20 °C	= 1,471 to 1,473
acidity, expressed as oleic acid	= < 0,5%
peroxide number	= < 10
unsaponifiable matter	= < 0,5%

Characteristics of modified polyphenylene oxide (MPPO)

molecular weight	500 000 to 1 000 000
size	60 mesh to 80 mesh
T_{max}	350 °C
specific mass	0,23 g/ml

Annex C
(normative)

Tolerances on contact times and contact temperatures applicable to all parts of this standard

Table C.1 — Contact times and tolerances

Contact times and tolerances
+1 30 0 min
+1 60 0 min
+3 90 0 min
+5 120 0 min
+5 150 0 min
+7 180 0 min
+8 210 0 min
+9 240 0 min
+10 270 0 min
+12 300 0 min
+15 360 0 min
+0,5 24 0 h
+0,5 48 0 h
+5 240 0 h

Table C.2 — Contact temperatures and tolerances

Contact temperatures and tolerances
5 °C ± 1 °C
20 °C ± 1 °C
30 °C ± 1 °C
40 °C ± 1 °C
50 °C ± 2 °C
60 °C ± 2 °C
70 °C ± 2 °C
80 °C ± 3 °C
90 °C ± 3 °C
100 °C ± 3 °C
121 °C ± 3 °C
130 °C ± 5 °C
140 °C ± 5 °C
150 °C ± 5 °C
160 °C ± 5 °C
170 °C ± 5 °C
175 °C ± 5 °C

Annex D
(informative)

Supports and cells

Dimensions in millimetres

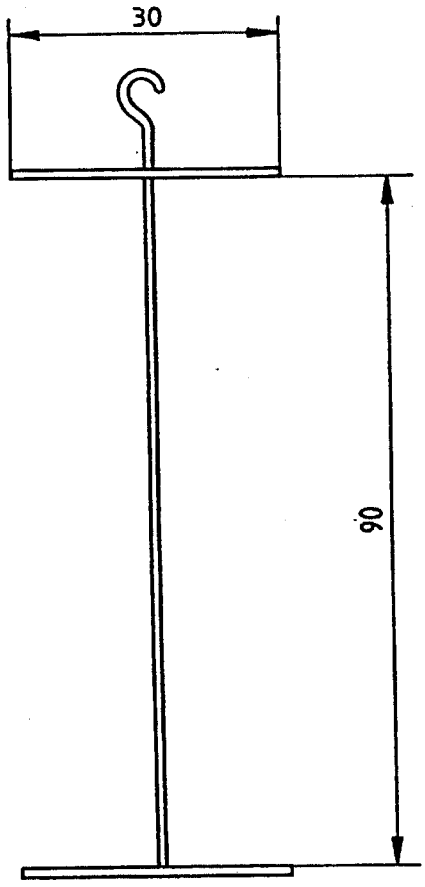


Figure D.1 — Example of support

Dimensions in millimetres

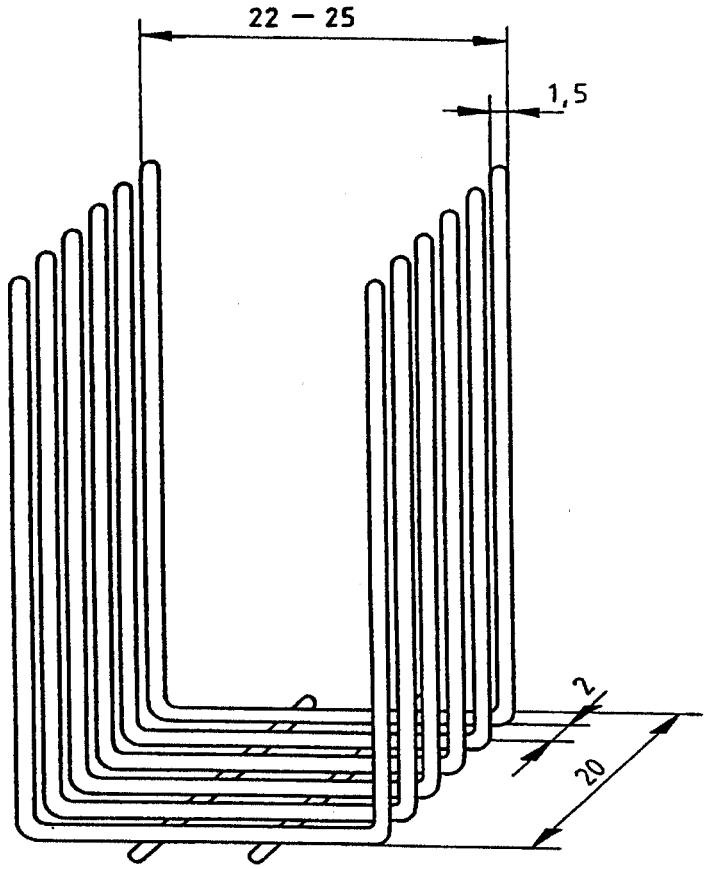
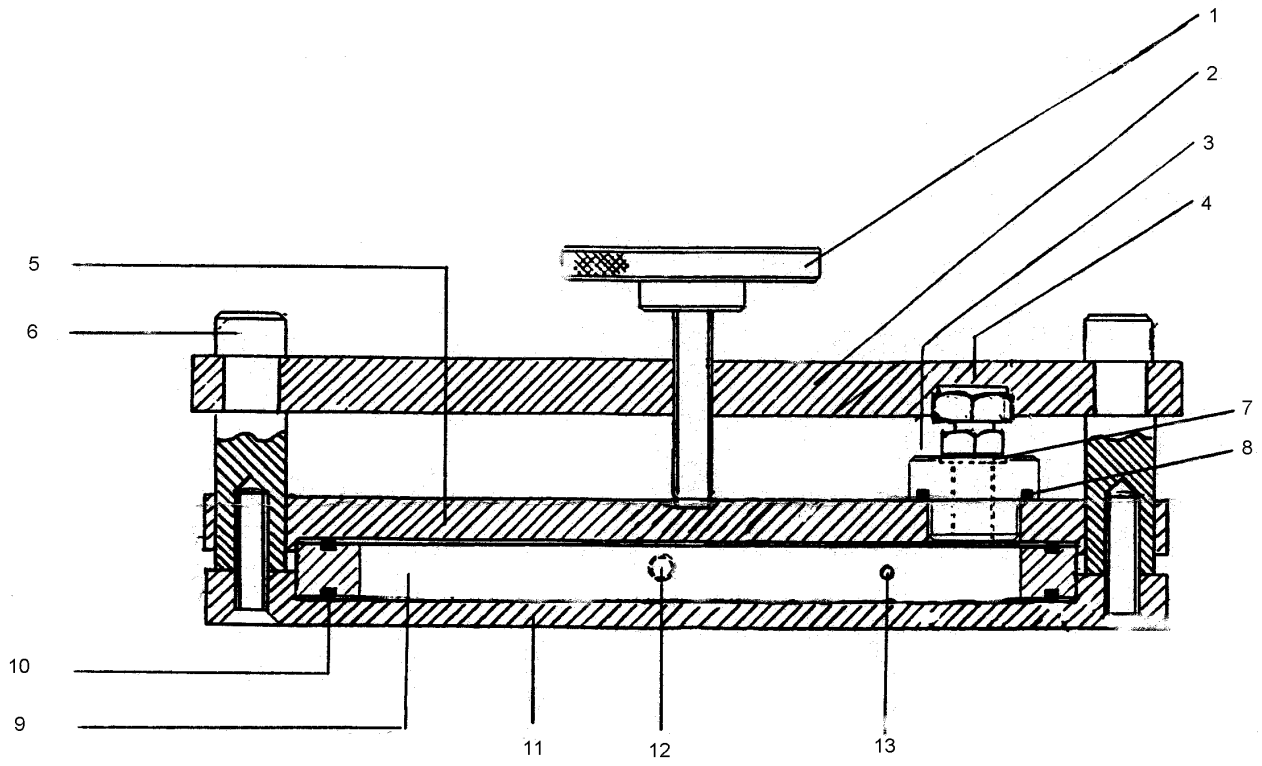


Figure D.2 — Example of larger support

Dimensions in millimetres

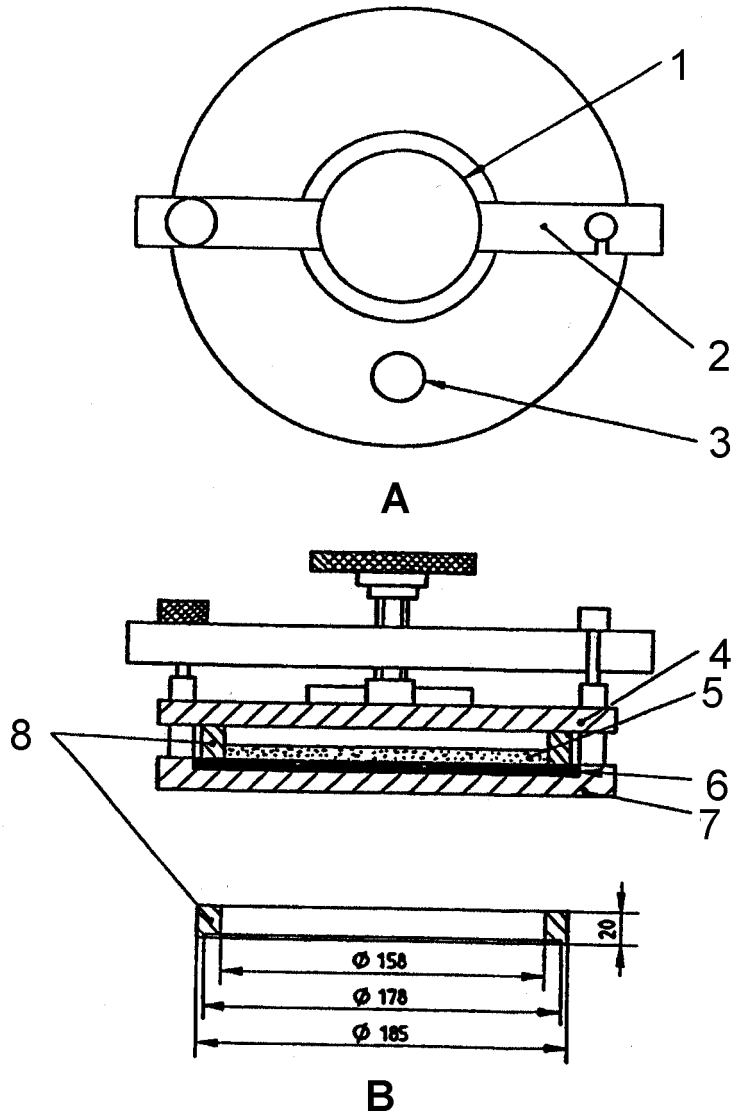


Key

1	Clanp screw	7	Sealing washer
2	Clamp bar	8	‘O’ ring
3	Filler plug	9	Cell ring
4	Septum	10	Cell ‘O’ rings two off
5	Top plate	11	Base plate
6	Clamping pillars two off	12	Drain hole
		13	Thermocouple attachment

Figure D.3 — Cell type A, Mark 2

Dimensions in millimetres

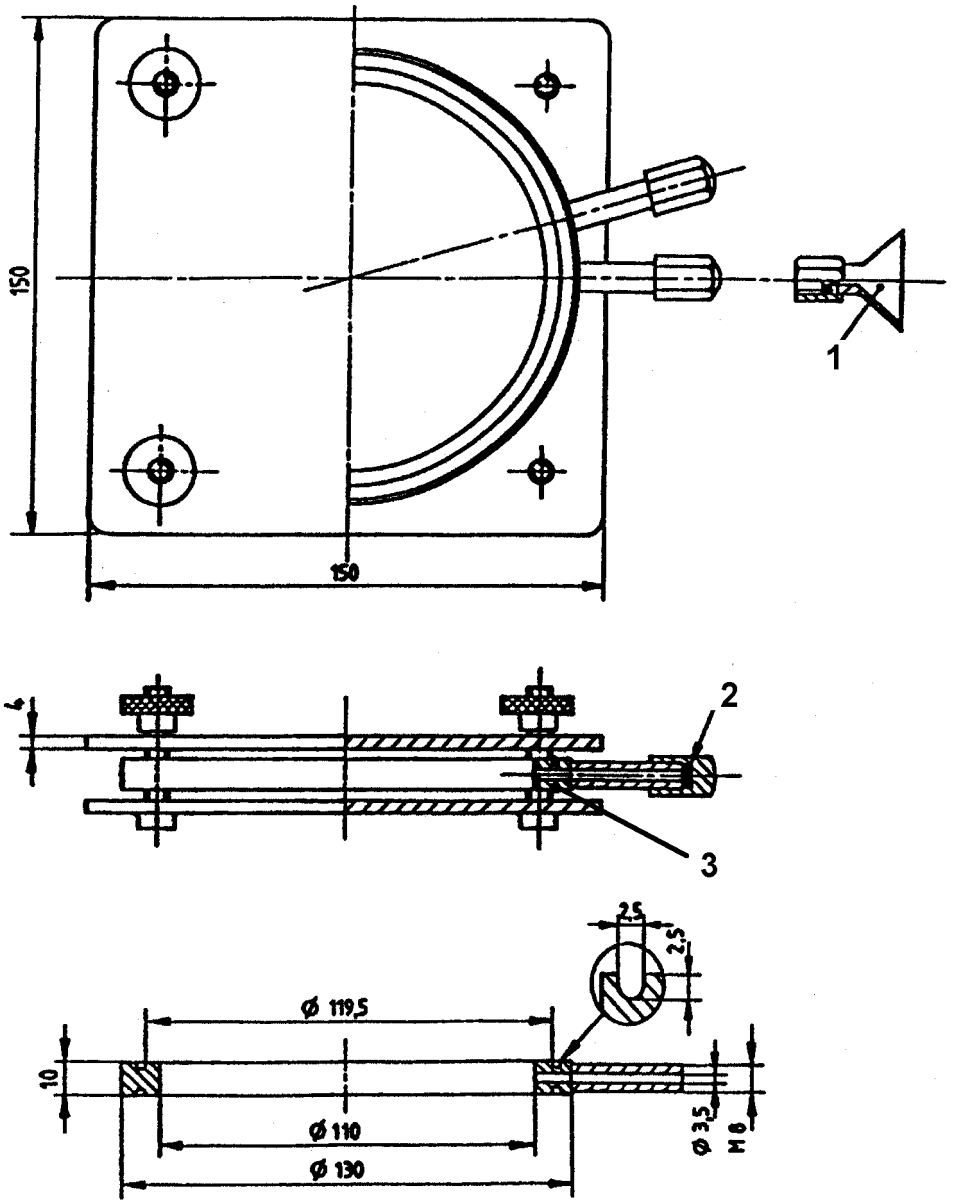


Key

A	**Plan elevation**	**B**	**Side elevation**
1	Clamp screw	4	Lid
2	Clamp bar	5	Food simulant
3	Filler plug	6	Rubber mat
		7	Base plate
		8	Sealing ring

Figure D.4 — Cell type A

Dimensions in millimetres



Key

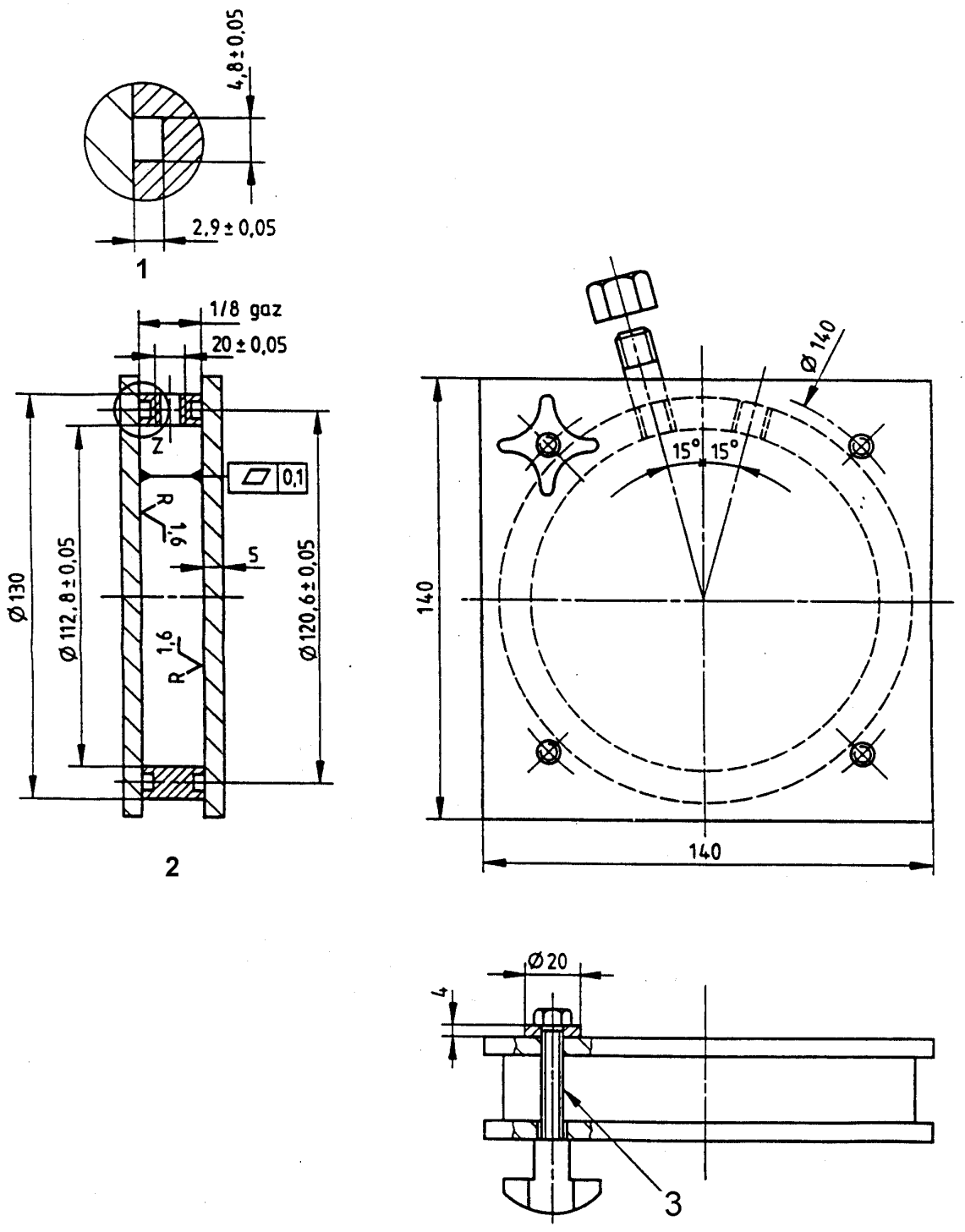
1 Funnel for filling

2 PTFE disk

3 PTFE 'O' ring (119,5 x Ø 3)

Figure D.5 — Cell type B

Dimensions in millimetres



Key

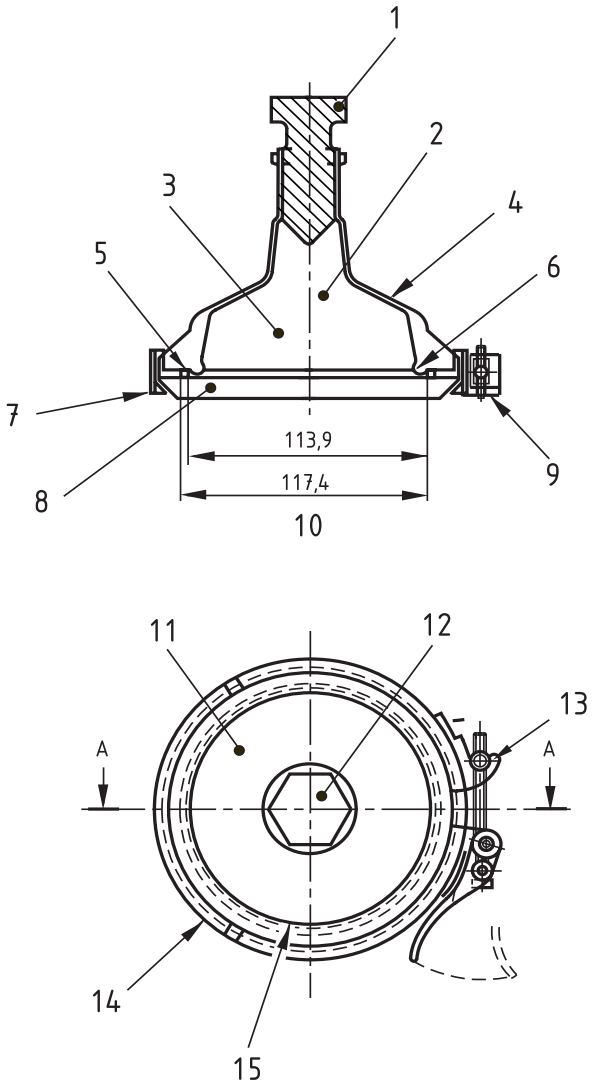
1 Detail Z

2 'O' – ring Ø 117,07 / 124,13 / 3,53

3 Screw HM8-50

Figure D.6 — Cell type C

Dimensions in millimetres



Key

1 Glass stopper
2 Total inner volume:296 ml (maximum volume of simulant: 250 ml)
3 Exposed surface area of circular test specimens: 1,019 dm²
4 Glass bell
5 Sealing ring ('O' ring) (silicon rubber sheathed in PTFE)
6 Raised edge to fix the 'O' ring in place
7 Tension ring (stainless steel)
8 PTFE plate
9 Tension seal (stainless steel)
10 Sectional view A-A
11 Glass bell
12 Glass stopper
13 Tensioning seal (stainless steel)
14 Tension ring (stainless steel)
15 Sealing ring

Figure D.7 — Cell type D

Dimensions in millimetres

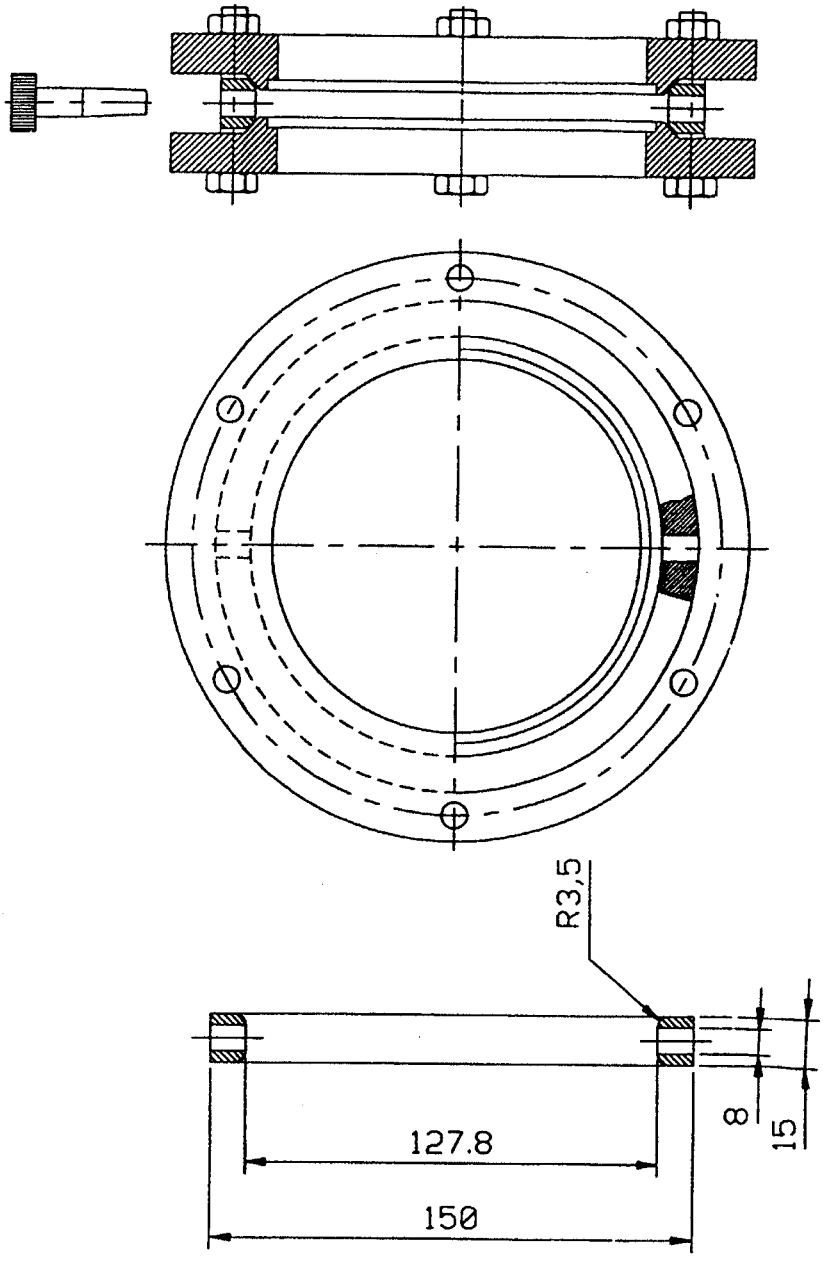
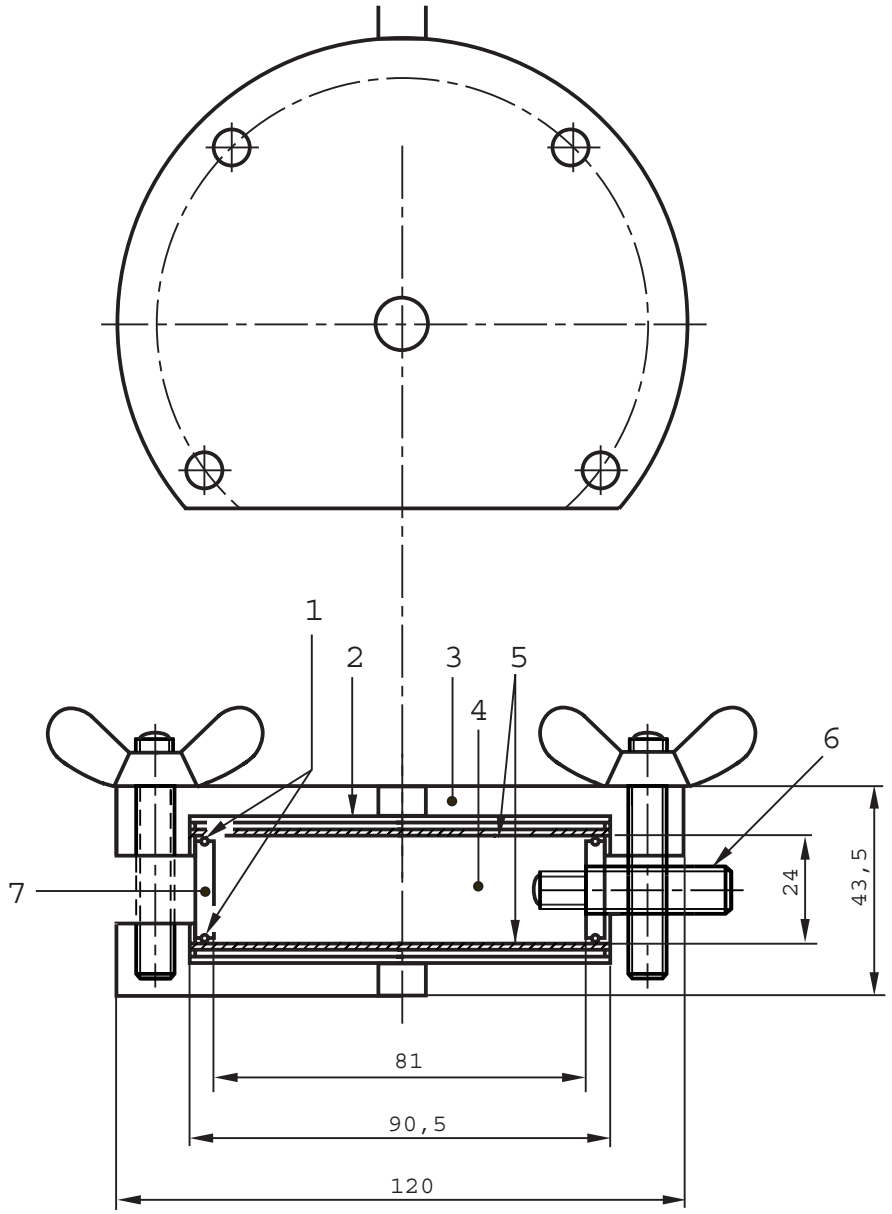


Figure D.8 — Cell type E

Dimensions in millimetres



Key

1 Sealing ring

2 Lid (stainless steel)

3 Body (aluminium)

4 (simulant)

5 Test sample

6 Stopper (PTFE)

7 Ring (stainless steel)

Figure D.9 — Cell type F

Annex E
(informative)

Relationship of this European Standard with Council Directive 89/109/EEC and Commission Directive 2002/72/EC and associated Directives

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association (EFTA).

WARNING: Other requirements and other EU Directives may be applicable to products falling within the scope of this standard.

The clauses of this standard are likely to support, Directives 89/109/EEC [1], 2002/72/EC [2], 82/711/EEC [3] and its amendments 93/8/EEC [4] and 97/48/EC [5], and 85/572/EEC [6].

Compliance with this standard provides one means of conforming to the migration requirements of the Directive concerned and associated EFTA regulations.

European Commission Directive 2002/72/EC relating to plastics materials and articles intended to come into contact with foodstuffs, [2] specifies in article 2:

“Plastics materials and articles shall not transfer their constituents to foodstuffs in quantities exceeding 10 milligrams per square decimetre of surface area of materials or articles (overall migration limit). However this limit shall be 60 milligrams of constituents released per kilogram of foodstuff in the following cases.

a) articles which are containers or are comparable to containers or which can be filled, with a capacity of not less than 500 ml and not more than 10 l;

b) articles which can be filled and for which it is impracticable to estimate the surface area in contact with foodstuffs;

c) caps, gaskets, stoppers or similar devices for sealing.”

European 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 [3], and the subsequent amendments (Directives 93/8/EEC [4] and 97/48/EC [5]), recognizes that there are difficulties in the determination of the migration in food products and allows use of food simulants with conventional test conditions, which reproduce, as far as possible, the migration phenomena which may occur with contact between the article and foodstuffs. There are four food simulants:

- simulant A, distilled water or water of equivalent quality
- simulant B, 3% acetic acid (w/v) in aqueous solution
- simulant C, 10% ethanol (v/v) in aqueous solution
- simulant D, rectified olive oil, or other fatty food simulants

European Directive 82/711/EEC and the subsequent amendments also contain the conventional test conditions (time and temperature) for migration tests with food simulants. European Commission Directive 97/48/EC, the second amendment to European Council Directive 82/711/EEC, also contains test media and conventional test conditions for 'substitute tests'. Substitute tests may be performed in place of migration tests with simulant D, if it has been shown that for technical reasons connected with the method of analysis it is not feasible to obtain a valid test result in a migration test with simulant D.

European Council Directive 85/572/EEC laying down the list of simulants to be used for testing of constituents of plastics materials and articles intended to come into contact with foodstuffs [6] has a table in the Annex which contains a non-exhaustive list of foodstuffs and which identify the simulants to be used in migration tests on those plastics materials and articles intended to come into contact with a particular foodstuff or group of foodstuffs.

This standard contains information on the selection of test methods for the measurement of migration from plastics materials to food simulants, or test media, using conventional contact test conditions of time and temperature, to determine compliance with the legislative migration limit specified in article 2 of European Commission Directive 2002/72/EC.

These test methods may also be used for the verification of compliance with the specific migration limits provided for in Commission Directive 2002/72/EC, if it can be established that the overall migration limits laid down in Article 2 of Commission Directive 2002/72/EC are not exceeded.

Commission Directive 2002/72/EC also specifies that the migration tests using rectified olive oil or substitutes shall not be carried out to check compliance with the migration limit in cases were there is conclusive proof that the specified analytical method is inadequate from the technical standpoint.

In any such case, for substances exempt from specific migration limits or other restrictions in the list provided in Annex II of Commission Directive 2002/72/EC, a generic specific migration limit of 60 mg/kg or 10 mg/dm², according to the case, is applied.

Bibliography

[1] 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.

[2] 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.

[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 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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