DD CEN/TS 14235:2002

Materials and articles in contact with foodstuffs — Polymeric coatings on metal substrates — Guide to the selection of conditions and test methods for overall migration

ICS 67.250

British Standards

National foreword

This Draft for Development is the English language version of CEN/TS 14235:2002.

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature because of the need to develop a method of test dealing with the coatings of materials and articles in contact with food, in order to satisfy the proposed European legislation. It should be applied on this provisional basis, so that information and experience of its practical application may be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the European organization responsible for its conversion to a European standard. A review of this publication will be initiated 2 years after its publication by the European organization so that a decision can be taken on its status at the end of its 3-year life. Notification of the start of the review period will be made in an announcement in the appropriate issue of *Update Standards*.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into a European standard, to extend the life of the Technical Specification or to withdraw it. Comments should be sent in writing to the Secretary of BSI Subcommittee CW/47/1, Migration from plastics, 389 Chiswick High Road, London W4 4AL, giving the document reference and clause number and proposing, where possible, an appropriate revision of the text.

A list of organizations represented on this committee 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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English version

Materials and articles in contact with foodstuffs — Polymeric coatings on metal substrates — Guide to the selection of conditions and test methods for overall migration

Werkstoffe und Gegenstände in Kontakt mit Lebensmitteln – Polymerebeschichtungen auf Substraten aus Metall – Leitfaden zur Auswahl der Bedingungen und Prüfverfahren zur Bestimmung der Gesamtmigration

This Technical Specification (CEN/TS) was approved by CEN on 28 July 2002 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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Contents

Foreword

This document (CEN/TS 14235:2002) has been prepared by CEN/TC 194 "Utensils in contact with food", the secretariat of which is held by BSI.

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

In this Technical Specification the annexes A, B and C are normative.

This Technical Specification includes a Bibliography.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

No single test method has been devised which can be used to determine overall migration, at all temperatures, in all food simulants. Indeed, owing to the practical difficulties inherent in testing with involatile extractants such as fats and the multitude of applications in which polymeric coatings on metal substrates come into contact with food, there are many methods and permitted variations to methods in this Technical Specification.

This Technical Specification is intended to give advice on the selection of the most appropriate type of test, test conditions and test method for a given application of a polymeric coating on a metal substrate and is intended to be read in its entirety before testing protocols are finalized. A test method for overall migration into aqueous simulants by article filling from polymeric coatings on food and beverage cans and non-stick coatings is given in clause 12. For many polymeric coated articles methods in EN 1186-2 to EN 1186-9 are suitable, according to the form in which the article is tested.

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 45001, EN 45002 and EN 45003. It is recommended that laboratories using this Technical Specification validate their procedures by testing certified reference samples and 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 Technical Specification gives guidelines for the selection of the appropriate conditions and test methods for the determination of overall migration into food simulants and test media from polymeric coatings on metal substrates which are intended to come into contact with foodstuffs and a test method for overall migration into aqueous simulants by article filling from polymeric coatings on food and beverage cans and non-stick coatings.

NOTE Polymeric coatings on metal substrates are not yet included in the scope of any European Union Directive. This Technical Specification has been prepared to assist in the development of such a Directive.

2 Normative references

This Technical Specification 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 Technical Specification only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1186-2, *Materials and articles in contact with foodstuffs - Plastics - Part 2: Test methods for overall migration into olive oil by total immersion.*

EN 1186-3, *Materials and articles in contact with foodstuffs - Plastics - Part 3: Test methods for overall migration into aqueous food simulants by total immersion.*

EN 1186-4, *Materials and articles in contact with foodstuffs - Plastics - Part 4: Test methods for overall migration into olive oil by cell.*

EN 1186-5, *Materials and articles in contact with foodstuffs - Plastics - Part 5: Test methods for overall migration into aqueous food simulants by cell.*

EN 1186-6, *Materials and articles in contact with foodstuffs - Plastics - Part 6: Test methods for overall migration into olive oil using a pouch*

EN 1186-7, *Materials and articles in contact with foodstuffs - Plastics - Part 7: Test methods for overall migration into aqueous food simulants using a pouch.*

EN 1186-8; *Materials and articles in contact with foodstuffs - Plastics - Part 8: Test methods for overall migration into olive oil by article filling.*

EN 1186-12, *Materials and articles in contact with foodstuffs - Plastics - Part 12: Test methods for overall migration at low temperatures.*

EN 1186-13, *Materials and articles in contact with foodstuffs - Plastics - Part 13: Test methods for overall migration at high temperatures.*

ISO 648 *Laboratory glassware - One-mark pipettes*

3 Terms and definitions

For the purposes of this Technical Specification the following terms and definitions apply.

3.1
polymeric coating
organic material applied in the form of a continuous film on a substrate in such a way as to form a protective layer and/or a functional barrier between food and substrate. They may be applied to the substrate in the form of solution, dispersions, powders, or solvent free preparations.

NOTE This may be different to the definition which is expected to be included in Commission Directive 90/128/EEC when its scope is extended to include polymeric coatings

3.2
final article
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 final article 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 3 and 4)

3.8
migration test
test for the determination of overall migration using food simulants under conventional test conditions

3.9
substitute test
test carried out which uses test media under conventional substitute test conditions when the use of a migration test is not feasible

3.10
test media
substances used in "substitute tests", iso-octane, 95 % ethanol in aqueous solution and modified polyphenylene oxide

3.11
alternative test
tests, with volatile media, that may be used instead of migration tests with fatty food simulants

3.12
extraction tests
tests in which media having strong extraction under very severe test conditions are used

3.13
overall migration; global migration
mass of material transferred to the food simulant or test media as determined by the relevant test method

3.14
reduction factor
numbers, 2 to 5, which may 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

3.15
pouch
receptacle of known dimensions manufactured from film to be tested, which when filled with food simulant exposes the food contact side of the film to the food simulant or test medium

3.16
reverse pouch
pouch, which is fabricated such that the surface intended to come into contact with foodstuffs is the outer surface. All of its sides are sealed to prevent the inner surfaces coming into contact with the food simulant. The reverse pouch is intended to be totally immersed in food simulant or test medium

3.17
inert substrate
material (e.g. silver, platinum, stainless steel, tantalum) which is non-reactive in the food simulant and which can simulate the intended metal substrate when coated with the test material. It is necessary that the inert substrate does not interfere with the overall migration from the coating under the applied test conditions

3.18
pressure retort or autoclave
pressure vessel (complying with appropriate pressure vessel regulations) in which hermetically sealed containers (e.g. metal plastics, glass) may be safely heated in a controlled manner, typically up to 122 °C and above, and which has suitable controlled cooling facilities

3.19
cell
device in which a film or flat sheet to be tested can be mounted which when assembled and filled with food simulant, exposes the food contact side of the film to the food simulant or test medium

3.20
repeatability value 'r'
value below which the absolute difference between two single test results obtained under repeatability conditions may be expected to lie with a probability of 95 %

3.21
reproducibility value 'R'
value below which the absolute difference between two single test results obtained under reproducibility conditions may be expected to lie with a probability of 95%.

3.22
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

3.23
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 Types of test

4.1 Migration tests

"Migration" tests for the determination of overall migration are carried out using the "food simulants" and "conventional migration test conditions", see 4.1, 4.2 and Table 1.

4.2 Substitute tests

If the migration test using fatty food simulants is not feasible, for technical reasons connected with the test method, "substitute tests" which use test media under the conventional substitute test conditions may 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 5. To ascertain compliance with the overall migration limit the highest value obtained using all of the test media is selected.

4.3 Alternative tests

4.3.1 "Alternative tests" with volatile media

The results of alternative tests, using volatile test media such as iso-octane and 95 % ethanol in aqueous solution or other volatile solvents or mixtures of solvents may be used to demonstrate compliance with the legislative limit, provided that:

a) the result obtained in a comparison test shows that the value is equal to or greater than those obtained in the migration test with a fatty food simulant;

b) the migration in the alternative test does not exceed the overall migration limit, after application of appropriate reduction factors.

If either or both conditions are not fulfilled, then the migration tests (3.1) have to be performed.

4.3.2 Extraction tests

Other tests are permitted which use other test media having very strong extractive power under severe test conditions, if it is generally recognized, on the basis of scientific evidence, that the results obtained using these extraction tests are equal to or higher than those obtained with simulant D.

4.4 Criteria for the use of substitute tests

The use of substitute tests is justified, when the migration test carried out with each of the possible simulants D is found to be inapplicable due to technical reasons connected with the migration test, e.g. interferences, incomplete extraction of oil, absence of stability of the weight of the polymeric coating, excessive absorption of fatty food simulant, reaction of components with the fat.

5 Food simulants, test media and reagents

5.1 Aqueous food simulants

The aqueous food simulants shall be of the following quality:

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

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

- For the purposes of this Technical Specification 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 liquids or beverages with an ethanol content greater than a volume fraction of 10 % the test is carried out with aqueous solutions of ethanol of a similar strength.

Each of the above food simulants shall give a non-volatile residue of less than 5 mg/l, when evaporated to dryness and dried to constant weight at 105 °C to 110 °C.

For overall migration testing of a coating material which is applied to substrates which are not resistant to acid, 3 % acetic acid is an unsuitable simulant because of the interfering corrosion products. 10 % aqueous ethanol should be used instead, since intensive studies have demonstrated that they provide sufficient information to evaluate coating materials for the overall migration properties even under the condition of contact with acidic food.

Only in certain cases would it appear necessary to use 3 % acetic acid as simulant, e.g. for samples containing inorganic constituents and testing specific migration of certain additive such as pigments, siccatives, amines and similar. In these cases the finished product can be tested with acetic acid only if the corrosion products do not interfere with the determination of the particular specific migrant. Otherwise it is recommended either to apply the coating on to an inert substrate prior to testing, or to test the unsupported film in the case of laminated metal.

5.2 Fatty food simulants

The fatty food simulants are as follows:

- rectified olive oil, "reference simulant D".

This "reference simulant D" may be replaced by a synthetic mixture of triglycerides or sunflower oil or corn oil with standardized specifications. 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 A.

NOTE When these fatty food simulants are used to simulate some classes of food, reduction factors may be used, see 5.3 and Table 2.

When determining the overall migration from materials and articles the mixtures of fatty acid triglycerides conventionally chosen to simulate fatty food have a number of shortcomings, as follows:

- they are not a single chemical compound and composition can vary even within the permitted specification. The naturally occurring unsaturated fatty acid triglycerides can change upon storage. This allows the possibility of inconsistent results;

- they cannot be evaporated to dryness without decomposition and therefore direct determination of the overall migration by weighing the residue after evaporation is not feasible. An inherently less accurate and more time consuming indirect determination is required;

- they are absorbed by the polymeric coating under test to a varying extent. If the absorption is excessive, this leads to gross inaccuracies in the determination. If the olive oil is not readily extractable errors also can occur. Errors can also occur if the different components of the fatty acid triglyceride are absorbed and extracted from the polymeric coating to a varying degree. Furthermore, substances present in the polymeric coating can interfere with the gas chromatographic determination of olive oil as described in EN 1186-2, EN 1186-4, EN 1186-6 and EN 1186-8;

- fatty acid triglycerides pose safety hazards when used at high temperatures both from the possibility of fire and the dangers of handling hot liquids;

- testing of thin layer coatings, such as used as protective coatings in cans, collapsible tubes and similar articles, does not give reliable results. The overall migration which could be expected under worst case conditions if of about the same order of magnitude as the accuracy of the method (3 mg/dm^2). The olive oil procedure is difficult to apply to cans with double seamed or heat sealed ends, and it cannot be applied to finished articles of greater dimension or weight.

For these reasons alternatives are desirable which should, as far as possible, avoid the deficiencies arising with triglycerides while still giving levels of migration comparable with, but not less than, that obtained with fatty acid triglycerides. Alternative fatty food simulants when compared with fatty food or existing fatty food simulants should possess the following characteristics:

- they should penetrate polymeric coating to the same extent;
- they should be as good a solvent for ingredients of polymeric coatings;
- they should be single substances or clearly defined mixtures.

5.3 Test media

5.3.1 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 A.

5.3.2 Test media for alternative tests

These are volatile media such as iso-octane and 95 % ethanol in aqueous solution or other volatile solvents or mixtures of solvents.

5.4 Reagents

Unless otherwise required, reagents shall be of analytical quality.

NOTE Specifications for solid reagents, used as such in discrete quantities, may not address suitability for use in methods of analysis in this Technical Specification. Solid reagents may not be homogeneous with respect to contaminants not addressed by specifications, therefore it may be necessary to demonstrate that such reagents are suitable for use.

6 Selection of food simulants

6.1 General

NOTE Commission Directive 85/572/EEC [6] specifies the use of a volume fraction of 15 % ethanol in aqueous solution as simulant C. This has been superseded by Commission Directive 97/48/EC [5} the second amendment to Council Directive 82/711/EEC [3] that specifies a volume fraction of 10 % ethanol in aqueous solution.

6.2 Simulating contact with all food types

Where a polymeric coating on a metal article is intended for use in contact with all types of food it shall be tested with a volume fraction of 3 % acetic acid in aqueous solution, simulant B, a volume fraction of 10 % ethanol in aqueous solution, simulant C and a fatty food simulant, simulant D, without reduction factors, except when the coating material is applied to a substrate which is not resistant to acid then testing with a volume fraction of 3 % acetic acid is not required, see 4.1.

If, when using any of the other fatty food simulants, see 4.2, the migration limit is exceeded, for the judgement of non compliance with the overall 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 overall migration limit.

6.3 Simulating contact with specific food types

Provision for 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 may or may not 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 may or may not 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 overall 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 materials and subject concerned. Where no 'X' appears, no migration test is required for the heading or subheading concerned , see 5.4 on dry foods and frozen foods.

When 'X' is followed by an oblique stroke and a figure, the result of the migration tests should 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 should be used:

- if the pH value is higher than 4,5, simulant A should be used;

- if the pH value is 4,5, or less, simulant B should 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 the 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 [a]	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 a volume fraction of 5 % :				
	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 others	X(a)	X(a)		
01.02	Alcoholic beverages of an alcoholic strength equal to or exceeding a volume fraction of 5 % :				
	Beverages shown under heading 01.01 but with an alcoholic strength equal to or exceeding a volume fraction of 5 % :				
	Wines, spirits and liqueurs		X [b]	X [c]	
01.03	Miscellaneous: undenatured ethyl alcohol		X [b]	X [c]	
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				

Table 2 *(continued)*

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

Table 2 *(continued)*

Reference number [a]	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 [d]
	B. In the form of paste	X			X/3 [d]
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 pastes or creams	X			X/4
06.04	Processed meat products (ham, salami, bacon and other)	X			X/4
06.05	Preserved and 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 yoghurts, 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 [d]

Table 2 *(continued)*

Reference number [a]	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 food:				
	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 preparation, prepared dishes:				
	A. Powdered or dried				X/5
	I. With fatty substances on the surface				
	II. Other				
	B. Liquid or paste:	X(a)	X(a)		X/3
	I. With fatty substances on the surface	X(a)	X(a)		
	II. Other				
08.04					
	Yeasts and raising agents:	X(a)	X(a)		
	A. In paste form				
	B. Dried				
08.05					
	Salt				
08.06					
	Sauces:	X(a)	X(a)		
	A. Without fatty substances on the surface				
	B. Mayonnaise, sauces derived from mayonnaise, salad	X(a)	X(a)		X/3
	creams and other oil in water emulsions	X(a)	X(a)		X
	C. Sauce containing oil and water forming two distinct				
08.07	layers	X(a)	X(a)		X/3 [d]
08.08	Mustard (except powdered mustard under heading 08.17				
					X/5
	Sandwiches, toasted bread and the like containing any kind of foodstuff:				
08.09	A. With fatty substances on the surface	X			
	B. Other				
08.10					
	Ice-creams				X/5
	Dried foods:				
	A. With fatty substances on the surface				
	B. Other				

Table 2 *(concluded)*

Reference number [a]	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 a volume fraction of 5 %		X [b]	X	
08.13	Cocoa: A. Cocoa powder B. Cocoa paste				 X/5 [d] X/3 [d]
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 seasoning in the natural state: cinnamon, cloves, powdered mustard, pepper, vanilla, saffron and other				

[a] The source of the reference number is Council Directive 85/572/EEC [6]
[b] This test shall be carried out only in cases where the pH is 4,5 or less.
[c] This test may be carried out in the case of liquids or beverages of an alcoholic strength exceeding a volume fraction of 10 % with aqueous solutions of ethanol of a similar strength.
[d] If it can be determined by means of an appropriate test that there is no 'fatty contact' with the plastics, the test with simulant D may be dispensed with.

6.4 Simulating contact with dry foods

Polymeric coatings 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 4, because these liquid food simulants are not appropriate models (mimics) for dry and frozen foods. However, volatile substances in particular may migrate into dry and frozen foods, especially if there is likely to be a long period of contact with the plastic. Therefore these food contact materials should 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 6.12.1. 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 may 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.

6.5 Testing for fatty contact

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

NOTE A method for determining whether a food makes fatty contact is being prepared by a Subcommittee (SC1) of TC194 'Utensils in contact with food' under work item 194077.

The principle of the method is that food, of a similar nature to that which will contact the plastic 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 plastic or not and hence determines whether the plastic shall be tested with simulant D or not.

The method described is suitable for direct use for a wide variety of foods. For some foods, it can be necessary to modify the method in order to obtain results which are representative of the food/plastic contact which occurs in actual use. Examples of such foods include crisps and snack foods where the food/plastic contact area in actual use can be small and irregular. In this instance it can be necessary to use a larger food/plastic contact area for the test. In situations where in actual use the food can consist of different surfaces and only one surface is to contact the food, it can be necessary to modify the method. Suitable modifications can involve altering the food so that only the surface that will contact the plastic in use is used for the test.

7 Migration tests, substitute tests and alternative test conditions

7.1 Test conditions for migration tests

NOTE 1 The basic rules necessary for testing the overall migration of the constituents of plastics materials and articles intended to come into contact with foodstuffs are laid down in Council Directive 82/711/EEC and its subsequent amendments, [3], [4] and [5]).

NOTE 2 The test times and temperatures are chosen according to conditions of contact in actual use. Tolerances on contact times and contact temperatures applicable to all Parts of this Technical Specification are detailed in Tables B.1 and B.2.

7.1.1 General

The migration tests are to be carried out, selecting from the times and temperatures specified in table 3 those which correspond to the worst foreseeable conditions of contact for the polymeric coating and to any labelling information on maximum temperature for use. Therefore, if the final polymeric coating 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.

7.1.2 Contact conditions generally recognized as more severe

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

7.1.2.1 Contact with foodstuffs at any condition of time and temperature

Many articles may be used at a variety of temperatures and for varying times, or their conditions of use may not be known. Where the polymeric coating may 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 6.1.6
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]
NOTE These conventional conditions for migration tests with food simulants are specified in Council Directive 82/711/EEC [3] as amended by D.4 and D.5.	
[a] This temperature shall be used only for simulant D. For simulants A, B, or C the test may 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 6.1.1.	

7.1.2.2 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.

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

If the polymeric coated material or article may 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 days, this being conventionally the more severe test.

7.1.4 Contact in a microwave oven

For materials and articles intended for use in microwave ovens, migration testing may be carried out in either a conventional oven or a microwave oven provided the appropriate time and temperature conditions are selected. In some circumstances where arcing might occur testing of metal articles in microwave ovens may be impossible or unsafe. In these circumstances the nearest corresponding conditions of time and temperature in a conventional oven may be appropriate. Alternatively, the coatings may be applied to an inert non metal and then tested in a microwave oven.

NOTE A method is being prepared by a Subcommittee (SC1) of TC194 'Utensils in contact with food', under work item 194078, as a European Standard, to measure the temperature, during microwave heating and during heating in a conventional oven , at the interface of food with packaging material.

7.1.5 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 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.

7.1.6 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 may be used which are more appropriate to the case under examination, provided that the selected conditions represent the worst foreseeable conditions of contact.

7.1.7 Testing at low temperatures

Testing with fats at 5 °C can lead to technical problems if the fat partially solidifies or, in the case of the synthetic triglyceride mixture, totally solidifies.

A sunflower oil, which is free of components which solidify at the temperature of test (i.e. a "dewaxed" oil), may be used.

However, with olive oil and sunflower oil the test is usually without this problem at 10 °C. If the overall migration does not exceed the limit when tested at 10 °C this indicates that it would not have exceeded the limit at 5 °C.

Testing by total immersion is practicable at low temperatures,

The method of test for the determination of overall migration from plastics at low temperatures (5 °C and 20 °C) is given in EN 1186-12.

7.1.8 Testing at high temperature

In practice, severe difficulties have been found in obtaining consistent and comparable results in interlaboratory trials with the test conditions for simulating exposure at temperatures of use in excess of 121 °C. The main source of inconsistency appears to be due to variation in the time required to achieve the test temperature with olive oil and other fatty food simulants. Various options such as exposure of sample tubes in electrically heated cells, etc. are under investigation as possible solutions to the problem and have been incorporated in methods described in EN 1186-13, see also 6.2 for substitute tests and 6.3 for alternative tests.

7.1.9 Food and beverage cans

By derogation from the conditions provided in the table 3, if the material or article is metal packaging, which in actual use be heat processed for two or more hours or at temperatures of 130 °C or above, it is appropriate to apply only the real conditions of use as the test conditions. The materials and articles shall be labelled accordingly.

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

Closure sealants shall also be subjected to these conditions, although they should be tested as part of the final article. Alternatively, they can be tested in a simulated manner, e.g. closed on to the neck of an article manufactured from an inert substance. This ensures that the closure sealant is only exposed to the simulant as would occur in practice.

For metal ends for processed foodstuffs, the end lining compound is entrapped in the mechanically formed double seam and, in most cases, is not in contact with food. It is, therefore, not necessary to carry out overall migration tests. Only were it is demonstrated that food contact occurs is testing required under the conditions of actual use.

7.1.11 Tubing, taps, valves, filters

Defining the time of exposure may be difficult for articles such as tubing, taps, valves, filters etc. as they may be in contact with flowing foodstuff. However, this exposure may be considered to be repeated brief contact for the purposes of migration testing. Such articles may be tested by repeated total immersion or by repeated filling. Tubing may 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.

7.2 Test conditions for substitute tests

Corresponding conventional conditions for the substitute tests have been agreed for examples of the most important conventional migration test conditions, 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 [b]	2,5 h at 60 °C	0,5 h at 100 °C
1 h at 100 °C	1,0 h at 60 °C [b]	3,0 h at 60 °C [b]	1 h at 100°C
2 h at 100°C	1,5 h at 60 °C [b]	3,5 h at 60 °C [b]	2 h at 100 °C
0,5 h at 121 °C	1,5 h at 60 °C [b]	3,5 h at 60 °C [b]	0,5 h at 121 °C
1 h at 121 °C	2,0 h at 60 °C [b]	4,0 h at 60 °C [b]	1 h at 121 °C
2 h at 121 °C	2,5 h at 60 °C [b]	4,5 h at 60 °C [b]	2 h at 121 °C
0,5 h at 130 °C	2,0 h at 60 °C [b]	4,0 h at 60 °C [b]	0,5 h at 130 °C
1 h at 130 °C	2,5 h at 60 °C [b]	4,5 h at 60 °C [b]	1 h at 130 °C
2 h at 150 °C	3,0 h at 60 °C [b]	5,0 h at 60 °C [b]	2 h at 150 °C
2 h at 175 °C	4,0 h at 60 °C [b]	6,0 h at 60 °C [b]	2 h at 175 °C

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

[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.

Other test conditions may be used. In this case the examples detailed above shall be taken into account as well as existing experience for the type of polymer under examination.

7.3 Test conditions for alternative tests

7.3.1 Alternative 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 result obtained in a comparison test shows that the value is equal to or greater than those obtained in the migration test with a fatty food simulant;

b) the migration in the alternative test does not exceed the migration limits, after application of appropriate reduction factors, see clause 5.

If either or both conditions are not fulfilled, then the migration tests with fatty food simulants have to be performed.

7.3.2 Extraction tests

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

8 Apparatus

8.1 Specimen supports

In the methods for determining overall migration by total immersion, cruciform specimen supports, see Figure C.1, are specified, but other supports may 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 C.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.

8.2 Tubes, glass rods and glass beads

In several of the methods for determining overall 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 may 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.

8.3 Cells

In the methods recommended this Technical Specification, the availability of cell type A, as shown in Figure C.3, has been assumed. Alternative cells shall be of such design to give satisfactory performance, particularly freedom from leakage with all four food simulants to prevent contamination of the food simulant with non-volatile substances, and with minimum area of the test specimen not in direct contact with the food simulant. Examples of other cells that are available, and have been found to be suitable, are type B, type C, type D, type E and type F; these are shown in Figures C.4, C.5, C.6, C.7 and C.8 of EN 1186-1:2001 respectively.

8.4 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 the Table B.2 throughout the volume of air encompassing the sample tubes, cells or pouches.

9 Samples and sample geometry

9.1 Samples

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

An example is where an article is filled with food at the time it is formed. In this case the test may be carried out on a test article prepared especially for testing purposes. This article shall be as representative as possible of the article in actual use.

A further example is where the sample to be tested is of inhomogeneous construction and 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 may be carried out on a test article prepared especially for testing purposes. This article shall be as representative as possible of the article in actual use.

Tests are also carried out on components and precursors of final articles, i.e., a coating on an inert substrate intended for can coating. 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.

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 may 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 may be removed by wiping the sample with a lint-free cloth or brushing with a soft brush.

Some articles may be sold with instructions that they should be cleaned or treated with oils or cleaning solutions before first use. In these cases the samples should be tested both before and after the treatment or cleaning process and the results evaluated.

In the case of treatments or cleaning carried out by the manufacturer, prior to sale, the samples should be tested after the treatment or cleaning process.

As part of a supplier-customer agreement, a regime of good manufacturing practice may include a provision for empty unused coated metal containers intended for processed foodstuffs to be pre-rinsed with water. When this is the standard operating procedure, then the cans should be treated similarly - either on the line or simulated in the laboratory - prior to the standard migration tests being done.

9.2 Surface to volume ratio

Where the surface to volume ratio to be used in contact with food is known this is used in the migration testing. 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 is tested with the specified volume of simulant.

Where the surface to volume ratio to be used in contact with food is not known conventional conditions are used, as described in 8.3 to 8.13.

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

Overall 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 will be in contact with the foodstuff or simulant. However, it is permissible to demonstrate compliance with an overall 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. No allowance is made for this in the calculation of migration per unit of surface area. Although the total surface exposed is 2 dm^2, only 1 dm^2, i.e. the food contact surface, is taken into account in the calculation. It is therefore a more severe test than testing in a pouch or in a cell or by filling.

However, if it is possible to demonstrate experimentally that the value obtained in a total immersion test is double that obtained in a single surface test, the value obtained in the total immersion test shall be divided by the total surface area exposed.

The method for determining overall migration by total immersion with olive oil is given in EN 1186-2 and with aqueous food simulants in EN 1186-3.

9.4 Single surface testing using a cell

Where single surface testing is the preferred procedure, particularly important for multi-layer articles, this may be carried out in a cell. For samples that may be obtained in flat form, e.g. film or sheet, testing in the cell has the advantage of readily reproducible sample geometry.

However, if testing polymeric coatings on acid resistant substrates with a volume fraction of 3 % aqueous acetic acid ensure that the materials of the cell do not influence the final result, e.g. cells constructed from aluminium may not be suitable in contact with a volume fraction of 3 % aqueous acetic acid.

The method for the determination of overall migration in the cell into olive oil is given in EN 1186-4 and into aqueous food simulants in EN 1186-5.

As an example the use of cell type A is described in EN 1186-4. The surface to volume ratio in the type A cell is conventionally 2,5 dm^2 of food contact area to 125 ml of food simulant.

Interlaboratory trials carried out by experienced laboratories have shown that consistent overall migration results can be obtained using cell type A.

Comparative studies carried out on the performance of cells type A, B, C, D, E and F revealed that these cells gave similar results. Therefore the cells referred to in Figures C.3, C.4, C.5, C.6, C.7 and C.8 are considered equivalent.

9.5 Single surface testing by pouch

For plastics films coated with metals, which have sufficient seal strength to form durable pouches, single surface testing in a pouch may be preferred as this does not require specialized apparatus and allows more efficient use of oven space. Inter-laboratory collaborative testing studies using pouches of precisely specified dimensions have shown that variations in pouch geometry (particularly varying areas outside the seals) can lead to significant variability in the final result.

The surface to volume ratio in the pouch is conventionally 2 dm^2 of food contact area to 100 ml of food simulant.

The method for the determination of overall migration in a pouch into olive oil is given in EN 1186-6 and into aqueous simulants in EN 1186-7.

NOTE For test temperatures above 40 °C it is permissible to fill the pouches with food simulant at ambient temperature and preheat the test specimens 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 test specimens a fibre optic probe or to check the temperature after heating by a thermometer. The filled pouches are placed in a microwave oven and heated until the simulant has attained the test temperature. Remove the test specimens to a thermostatically controlled oven or incubator that is preheated to the test temperature. This part of the operation should be carried out in the minimum time to prevent undue heat loss. Leave the pouches for the selected test period.

9.6 Single surface testing using a reverse pouch

For thin metals coated both sides with plastics films an alternative to using a pouch, a reverse pouch may be used. In this case the surface intended to come into contact with the foodstuff is the outer surface and the pouch is exposed to the food simulant by total immersion.

The use of a reverse pouch offers advantages over the pouch. Since pouches are filled with simulant, the sealed edges have to be capable of bearing the weight of that simulant; if they are not the seals give way and the pouches are prone to leakage. With the reverse pouch the seals do not have to withstand the pressure of the simulant and consequently are less likely to leak and the sealed area can be reduced. The use of a reverse pouch permits a more accurate determination of the area exposed to food simulant. However, it is possible that simulant may leak into the reverse pouch thus increasing the area exposed to simulant. A way of checking if leaks have occurred is to seal into the 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. This method may not be applicable for overall migration into fatty food simulants, as the weight of the inserted paper may change during storage due to loss of water. Any pouch that leaks shall be discarded and the test repeated.

Where the surface to volume ratio to be used in contact with food is not known, the conventional conditions are used, i.e. 2 dm^2 of surface in contact with 100 ml of simulant.

9.7 Single surface testing by filling

For articles in container form, e.g. cans, bottles and trays, it is often most convenient to test them by filling with food simulant, see clause 12, also methods for the determination of overall migration by filling with olive oil and by filling with aqueous food simulants are given in EN 1186-8 and EN 1186-9 respectively. For very large containers testing by filling cannot be practicable and it can be necessary to fabricate smaller test specimens representing the article to be tested.

9.8 Articles intended for repeated use

9.8.1 Criteria for testing

It is accepted that where a material or article is intended to come into repeated contact with foodstuffs, the migration tests are carried out three times on the same test sample in accordance with the conditions laid down, using a fresh sample of the food simulant on each occasion. The compliance of the material 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.

Experience has shown that some thermosetting polymers, e.g. melamine/formaldehyde resins, can give rise to increasing levels of migration on second and subsequent exposure to foodstuffs. However, for the majority of polymers migration levels will fall in the second and subsequent extracts. Proof of this may be found from past experience with similar polymer types. For these polymeric coatings it is only necessary to show that the migration limit is met in the first extract

9.8.2 Aqueous simulants

For aqueous simulants, no increase in migration is deemed to have occurred if the mean of the results for the second and third test do not exceed the mean of the result for the first extract by more than the permitted analytical tolerance.

9.8.3 Fatty food simulants

With fatty food simulant, the repeated exposure of the same test specimen to fresh portions of food simulant is not a feasible procedure, since the procedure requires solvent extraction to remove the fatty simulant. Therefore, the test is carried out on three sets of test specimens from the same sample of the material or article. One of these is subjected to the test appropriate for articles intended for single use by the standard procedure and the mean result calculated (M_1). The second and third samples are exposed in a manner identical in every respect to the first sample except for the period of exposure. The second sample is exposed for a period of twice that of sample one and sample three is exposed for a period three times that of sample one. The mean result for sample 2 is calculated (M_2) as is that for sample 3 (M_3).

The migration as a result of the second or third period is calculated as follows:

- migration caused by first period = M_1 ;
- migration caused by the second period = M_2 - M_1 ;
- migration caused by the third period = M_3 - M_2 .

No increase in migration into fatty food simulant is deemed to have occurred if the results (M_3 - M_2) and (M_2 - M_1) do not exceed M_1 by more than the analytical tolerance.

The true values for M_1, M_2 or M_3 are subject to uncertainty owing to the lack of precision inherent in the method. Systematic errors in the determination of the overall migration are likely to apply equally to the determination of M_1, M_2 or M_3 and therefore need not be allowed for. Random errors do need to be recognized and allowed for.

When repeated testing is used to determine the overall migration into a fatty food simulant the individual results for each set of the determinations (M_1, M_2 or M_3) shall be deemed valid if at least three results are obtained in each set which do not differ from the mean for that set by more than 30% for results above 10 mg/dm² or by more than 3 mg/dm² for results below 10 mg/dm². Results which exceed this tolerance shall be discarded according to the procedure given in 11.3.2.

When the polymeric coated material or article is intended for use with a class of foodstuff where a reduction factor may be used, this shall be applied to the individual determinations before the mean of M_1 or M_2 or M_3 is calculated.

The material and articles are deemed to be in compliance with the overall migration limit provided that either M_1 or M_3 - M_2 do not exceed the specified overall migration limit.

9.9 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, see 6.1.10.

The simulants are placed in jars, known to give only consistently low migration, and the jars closed with the test closures. The jars are then inverted and subjected to the test conditions appropriate for the actual conditions of use.

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

For articles where the overall migration will be limited in terms of milligrams per kilogram the migration from the closure is added to that of the container when assessing compliance with the limit.

9.10 Food and beverage cans

The finished article should be used. When closure equipment is not readily available in the laboratory, cans with double-seamed or heat-sealed ends may be tested using ends in which a hole has been carefully drilled. The closing of the empty container can be done with the equipment of the actual user. Such empty closed cans can be subsequently filled with simulants through the hole, which can then be closed by an inert stopper.

For a test method for overall migration into aqueous simulants by article filling from polymeric coatings on food and beverage cans and non-stick coatings see clause 12.

If flat sheet material is available from which the finished articles are formed or assembled, this can be used for single sided tests in a suitable test cell.

In many situations, the shape, size and/or weight of the finished article are inappropriate for testing. In these cases it is advisable to apply the coating material on to an inert substrate which can be tested by total immersion, or in the case of laminated metal to test the unsupported polymeric film. This procedure is also recommended for testing the coating material itself independently from the application on or for the finished article. The application of the coating material to an inert substrate should follow, as closely as possible, the conditions and specifications commensurate with the expected production coating process.

9.11 Non-stick coatings

In the case of some coatings on thick metal substrates, e.g. non-stock cookware, it is not practicable to determine the migration into fat simulants according to EN 1186-2 to EN 1186-8, because weight differences attributable to the migration and absorption of fat simulant are very small in comparison to the mass of the test specimens and thus substantial errors can be expected.

In these cases, the test shall be carried out on specially prepared test specimens that are representative of the polymeric coating on the material or article, but where the metal substrate has a mass that permits the practicable use of the methods EN 1186-2 to EN 1186-8. Alternatively, compliance with the overall migration can be demonstrated by the use of substitute tests using the test media iso-octane, 95 % aqueous ethanol and modified polyphenylene oxide or a 'more severe test', such as extraction with solvents, such as iso-octane or 95 % aqueous ethanol.

For a test method for overall migration into aqueous simulants by article filling from polymeric coatings on food and beverage cans and non-stick coatings see clause 13.

9.12 Flexible films

With the exception of the use of 3 % aqueous acetic acid flexible plastics films coated with thin layers of metals should be tested in the same way as flexible plastics films and tested in accordance with the relevant Parts of EN 1186.

9.13 Large containers

Large containers, where filling is not practicable, may be tested by cutting test specimens from them and testing these by total immersion or by the cell method or using an equivalent cell.

When cutting the container is impracticable because of its size, composition, design or construction, specially prepared test specimens that are representative of the article or the relevant parts of the article shall be tested in a cell or by total immersion.

In the case of aqueous simulants a large container may be filled and portions taken after thorough mixing, to determine the residue. Alternatively, smaller test samples representing the large container may be fabricated and tested by filling.

9.14 Inert substrates

The application of the coating material to an inert substrate should follow, as closely as possible, the conditions and specifications commensurate with the expected production coating process.

9.15 Tubing, taps, valves and filters

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

9.16 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.

10 Overall migration test methods with fatty food simulants

NOTE For many polymeric coated articles the test methods in EN 1186-2 to EN 1186-9 are suitable, according to the form in which the article is tested. However, the test methods detailed in EN 1186-2 to EN 1186-12 may not give reliable results in certain circumstances; these are described 9.1 to 9.10.

10.1 Extraction solvents

In previous methods for determining overall migration into fats and fatty food simulants the solvent 1,1,2, trichloro-trifluoroethane has been used to extract fat from polymeric coatings. This solvent is a member of the chloro-fluorocarbon (CFC) class of chemicals and, since every effort has been made to prevent release of this solvent to the atmosphere, an alternative has been sought.

Pentane is the recommended extraction solvent for non-polar polymeric coatings, such as polyethylene and polypropylene. A 95/5 by volume azeotropic mixture of pentane and ethanol is recommended as the extraction solvent for polar polymeric coatings, such as polyamide and polyacetal

Do not discard used solvent. Re-distilled solvent, free of fat, can be used.

10.2 Incomplete extraction of fat

Incomplete extraction of absorbed fatty food simulant from some polymeric coatings occurs despite prolonged soxhlet extraction with pentane. This is known to give falsely low results in the standard test procedure. This difficulty can be overcome by subjecting the test specimens to a second extraction, this time with diethyl ether, or to the dissolution/precipitation method set out in EN 1186-10. The amount of oil obtained in the diethyl ether extract or in the solution after precipitation of the polymer coating is added to the amount of oil obtained in the pentane extract. To obtain reliable results the migration test shall be repeated using the dissolution/precipitation method.

10.3 Substances which interfere with gas chromatography

Some substances which may migrate from polymeric coatings are capable of interfering with the gas chromatographic method for the determination of olive oil, e.g. glyceryl oleates. When testing articles containing these substances they may be tested with other fatty food simulants, such as sunflower oil, corn oil or synthetic triglyceride mixtures.

Other migrating substances can give rise to peaks in the gas chromatogram which interfere with the internal standard peak. Alternative internal standards such as hydrocinnamic acid, ethyl ester or trinonadecanoin may be used in such cases.

10.4 Loss of volatile substances

During exposure of the test specimens to food simulants volatile substances such as water, solvents, monomers, oligomers etc. may be lost from the plastic. In the test procedures with aqueous food simulants further loss of volatile substances will occur upon evaporation of the food simulants. When the overall migration into aqueous simulants is reported no possible loss of volatile substances is taken into account. For consistency reasons, it is conventionally agreed that also for the fat test the migration of non-volatile substances only is determined.

NOTE For the specific purpose to meet eventual health concerns about migration of organic volatiles from polymeric coatings other analytical methods such as gas chromatographic determinations of the polymeric coating headspace or solvent extracts may be applied. This is currently not within the scope of conventional overall migration testing.

In the test procedures with a fatty food simulant, total or partial loss of volatile substances may occur, particularly at high temperature. An indication of this possible loss may be deduced from:

- the loss of weight, after conditioning to constant weight at 50 % relative humidity, of the test specimens which have not been exposed to the fatty food simulant but have been subjected to the test temperature for the test period i.e. those that have been in empty tubes or pouches or test specimens which have not been filled.

- vacuum drying tests carried out for one hour at 60 °C according to the procedure set out in Annex D of various Parts of EN 1186 concerned with overall migration testing using fatty food simulants.

When a loss of volatile substances is indicated, i.e. the tolerance for weight change permitted in the appropriate section of the test method has been exceeded, then results with correction for loss of volatile substances may be reported. The corrected overall migration figure is calculated by subtracting the mean weight loss per square decimetre of the test specimens not exposed to fatty food simulant from each uncorrected value calculated according to the procedure in the appropriate test method. Reduction factors may then be applied (see 11.2) and the validity of the corrected values assessed (see 11.3.2). Reported results may be based on the values with correction for loss of volatile substances.

10.5 Gas chromatographic columns

In the relevant Parts of EN 1186 concerning the determination of the overall migration into olive oil different types of gas chromatographic columns are mentioned, polar and non-polar.

Column 1 is a column with a polar stationary phase that allows separation of the individual methyl esters of fatty acids according to their carbon number as well as their number of double bonds in the chain, e.g. the methyl esters of stearic acid is separated from the methyl esters of oleic acid and this is separated from the methyl esters of linoleic acid.

Column 2 is a column with a non-polar coating which allows only separation of the carbon number, e.g. no separation is obtained between the methyl esters of oleic acid and the methyl esters of stearic acid.

Both types of columns have their own specific advantages and disadvantages. A gas chromatogram obtained with column 1 will reveal more information on the distribution of fatty acid in the olive oil extracted from the test specimen than with column 2. To determine the total area of the fatty acids using column 1, the area of at least 5 peaks may be measured and summed. With column 2 only 2 peaks have to be measured. On the other hand the determination will be more sensitive to interferences when using column 2. In the case where interferences occur on one of the minor peaks, when using column 1, it is possible to exclude that peak and to adapt the calibration graph for the excluded peak. It is even possible to measure only the major peak of oleic acid to quantify the total amount of oil, provided the calibration graph is constructed in the same way.

NOTE A polar column is the preferred one.

Column 3, referred to in the relevant Parts of EN 1186 concerning the determination of the overall migration into olive oil is a polar column.

10.6 Changes in the C18/C16 ratio

A difference in C18:1/C16:0 ratio (using column 1) between the olive oil extracted from the test specimen and the olive oil applied as the fatty food simulant in the migration test indicates that the composition of the extracted oil for some reason is different from the composition of the oil that has not been in contact with a test specimen. Possible causes for the changes of the composition are:

- reaction of olive oil constituents with polymeric coating constituents;
- oxidation of unsaturated constituents of the olive oil. This has been observed to occur when rather long periods for conditioning the test specimen after contact with the oil are necessary;
- incomplete methylation of fatty acids in the trans-esterification procedure, such difficulties arise with some types of high impact polystyrene (HIPS) and acrylonitrile-butadiene-styrene(ABS);
- selective absorption of oil constituents by test specimens. Polyolefins for example do absorb selectively mono- and diglycerides of saturated free fatty acids in some cases, whereas HIPS, ABS and nitrile-butadiene rubber (NBR) often selectively absorb diglycerides, and to a lesser extent also monoglycerides of unsaturated fatty acids;
- interface by polymeric coatings constituents having the same retention time as C16:0 or C18:1 methyl ester or forming those esters in the trans-esterification stage.

Whether a change in the C18:1/C16:0 ratio acts upon the final result of the overall migration determination to an extent which is not acceptable depends mainly on the magnitude of the change and on the amount of oil recovered from the test specimen, e.g. a 25 % change in the C18:1/C16:0 ratio may result in a 25% lower result in the amount of fat extracted, which would mean 2,5 mg when only 10 mg fat is absorbed by the test specimen but 25 mg when 100 mg of fat is absorbed. So a proportional change in C18:1/C16:0 ratio will result in an absolute difference in the amount of fat calculated, and consequently in an absolute difference in the overall migration values. Whilst an absolute difference of 2,5 mg is acceptable, because it is within the accepted analytical tolerance, one of 25 mg is not.

Whether there might be a possibility of obtaining false results because of a change in the C18:1/C16:0 ratio, can easily be established by measuring the amount of oil extracted from the test specimen using two different calibration graphs. In one graph the ratio C16:0/C17:0 is plotted versus the amount of olive oil and in the other one the ratio C18:1/C17:0. The amount of oil calculated using the C16:0/C17:0 graph shall differ from the amount calculated using the C18:1/C17:0 graph by no more than 2 mg/dm^2. In case a larger difference is observed the cause of it has to be identified and an appropriate action be taken. Remedies for problems can be:

- if reaction of oil constituents with polymeric coating constituents is suspected a less reactive oil, e.g. a synthetic mixture of triglycerides, can be used;
- if oxidation of unsaturated fatty acids is suspected a less vulnerable fatty food simulant, e.g. a synthetic mixture of triglycerides, can be used;
- if incomplete methylation of fatty acids during trans-esterification is suspected the heptane layer obtained in the normal trans-esterification procedure is subjected to an additional trans-esterification treatment;
- if selective absorption of fatty simulant constituents by the test specimen is suspected, which can be ascertained by thin layer chromatography comparison of the composition of extracted and olive oil a fatty food simulant low in free fatty acids and mono- and diglycerides can be used;
- if interference of oleic acid (C18:1) or heptadecanoic acid (C17:0) peak area measurement by plastic constituents is suspected which can be ascertained by running a blank experiment with a sample of the final article in question, the palmitic acid (C16:0) peak area of olive oil can be used as a reference. It is preferable however to use, if possible, sunflower oil or a synthetic mixture of triglycerides as the food simulant instead.

10.7 Initial mass of the test specimen

If conditioning of test specimens is not required, the initial mass of the test specimen to be used in the formula to calculate overall migration is simply the initial mass of the test specimen.

If it has been shown that test specimens require conditioning, they are subjected to the vacuum drying procedure or conditioning at constant relative humidity set out in Annex C and Annex B of the relevant parts of the standards concerned with overall migration testing using a fatty food simulant, until constant mass.

The initial mass to be used in the formula to calculate overall migration in this case is the mass of the test specimen when constant mass has been achieved.

When using the vacuum drying procedure, before initiating the migration experiments the test specimens are subsequently placed at ambient humidity or in a container at 80% relative humidity until they have regained 80 % to 120 % of the mass lost during vacuum drying.

The conditioning procedure at a relative humidity of 50 % as described in Annex B of the relevant parts of this Technical Specification can be used to establish the initial mass of the test specimen to be used in the equation to calculate the overall migration.

Sometimes, when a number of test specimens are subjected to conditioning together, not all specimens reach constant mass simultaneously. In such a case it is permissible to remove the test specimens that have achieved constant mass from the conditioning device and store them until the remaining test specimens also have achieved constant mass, before exposing all test specimens simultaneously to the food simulant.

In selection of the conditioning technique consideration may be given to the shorter conditioning periods required by the vacuum drying technique in comparison with conditioning at 50 % relative humidity. Short conditioning times are very important when the final mass of the test specimen after the migration period has been determined. Long conditioning times at room temperature in presence of oxygen will cause oxidation of the olive oil and as a consequence the composition of the olive oil absorbed by the test specimen can change (see 9.6).

In addition, volatile substances will be removed from the test specimen and they will not interfere in the calculation of the overall migration. In this way only the migration of non-volatile substances is measured as is the case in the determination of the overall migration in aqueous food simulants.

10.8 Final mass of the test specimen

If conditioning of test specimens was not required to establish the initial mass of the test specimen, then the mass of the test specimen after removal of the adhering oil is simply the final mass of the test specimen.

If the test specimen was conditioned before the migration period then the test specimen shall be conditioned after the migration period as well, using the same technique of conditioning either vacuum drying or conditioning at 50 % relative humidity. The final mass of the specimen is obtained when the difference between two consecutive weighings is less than the permitted tolerance.

10.9 Selection of the appropriate conditioning procedure

In the relevant Parts of EN 1186 concerning test methods for overall migration testing with olive oil two procedures have been described to establish the mass of the test specimen before and after the exposure time.

The vacuum drying method is fast and repeatable and changes in conditioning temperature will not influence the final mass of the test specimen. The vacuum drying method removes all volatile components and no correction for loss of volatile is required. Loss of volatiles is not usually a problem when only small amounts of volatile substances are present, e.g. residual monomers. If the allowed analytical tolerance of 3 mg/dm^2 is taken into account the removal of the volatiles will not have a significant influence on the reported overall migration. If large amounts of volatiles are present, e.g. in expanded polystyrene, then conditioning at 50 % relative humidity has to be taken into consideration. The major advantage of the vacuum method is the time required to establish the mass of the test specimen. If the conditioning after the exposure takes a long time then the oil can oxidize and the oxidized components will not be recovered, this results in an under estimation of the overall migration.

The vacuum method is not suitable for those samples that, after drying, re-absorb the water very quickly, e.g. thick polyamide samples. In such cases the mass will change constantly during weighing.

NOTE When following the vacuum drying procedure the mass lost during the initial conditioning may not be regained for the following reasons:

- loss of mass is caused by release of water from one of the under-laying layers of a multi-layer material. It may be time consuming or even impossible to regain the loss of water during reconditioning. There is no objection to continue the test without regaining the loss of mass;

- release of a small amount of water from lipophylic polymers such as polypropylene. These type of polymers are usually not capable of regaining the major part of the water lost. The conditioned samples can be used for overall migration testing;

- loss of mass is caused by the removal of volatile organic components. In this case the vacuum drying method may result in too low a migration value and another method of conditioning should be used to condition the test specimen.

Conditioning at 50 % relative humidity is suitable for most types of polymers, particularly those that are subject to only small changes in mass, and for those which are hygroscopic after vacuum drying. The procedure is also suitable for thin polyamide samples, whereas problems are foreseen with thick polyamide samples. The procedure of conditioning at 50 % relative humidity is usually time-consuming and can take 4 days or more. If the procedure takes more than 7 days, then there is a possibility of oxidation of the unsaturated fatty acids and an alternative procedure has to be considered. The conditioning method is simple and does not require any special apparatus and can therefore be applied by any laboratory with standard equipment. Control of a specific temperature is not important, but the temperature needs to be kept within a very narrow range during conditioning, before and after exposure, as the mass of test specimen is related to temperature. Correction for volatiles is allowed, but in the case where large quantities of volatiles are present the validity of the correction has to be carefully considered.

Determination of the release of water from a test specimen by means of Karl-Fisher titration is also allowed as a method of establishing the mass of the test specimen before and after exposure. Use of this method prevents the conditioning of the test specimen. This method can be useful for samples that cannot be conditioned to constant mass by one of the methods above. The method cannot be applicable to samples that release significant amounts of water resulting in over-saturation of the oil with water and subsequent loss of water through the vapour phase.

The selection of the appropriate procedure is determined by the nature of the sample and the fact that if an inappropriate procedure is adopted the result obtained may be different. The procedure used, and the reason for selecting that procedure, has to be stated in the report.

10.10 Loss of simulant due to permeation

When testing some samples by single surface testing the small amounts of simulant may permeate through the sample. For example, there may be a small loss of alcohol when testing with high strength ethanol/water simulants by filling. In this case, as the loss in ethanol from the simulant can be expected to reflect what would happen under actual conditions of use of the alcoholic beverage, this loss may be disregarded. However, if permeation occurs when testing in a cell, care has to be taken to ensure contamination does not arise from contact of the simulant with components of the cell.

11 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.

NOTE At the time of publication as a Technical Specification precision data is not available.

12 Test reports

12.1 Surface to volume ratios in actual use

12.1.1 General

Overall migration is a measure of inertness and may be expressed in different ways according to the following circumstances.

12.1.2 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 and shall be recalculated to the "conventional" surface to volume ratio of 6 dm^2 to 1 kg of food and expressed in milligrams per kilogram.

For articles which can be filled and for which it is impracticable to estimate the surface area which is in contact with the foodstuff the results shall be expressed in milligrams per kilogram.

12.1.3 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 articles are containers or articles which are comparable to containers or which can be filled, with a capacity of not less than 500 ml and not more than 10 l, the results shall be expressed in milligrams per kilogram.

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

12.1.4 For known surface to volume ratios and tested under different 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 articles are containers or articles which are comparable to containers or which can be filled, with a capacity of not less than 500 ml and not more than 10 l, the results shall be recalculated to the actual conditions of use and expressed in milligrams per kilogram.

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

12.1.5 Conversion recalculation

Where the migration tests are carried out on samples taken from the material or article or on samples manufactured for the purpose, and the quantities of foodstuff or simulant placed in contact with the sample differ from those employed in the actual conditions under which the material or article is used, the results obtained shall be corrected by applying the following equation:

$$M = \frac{m \cdot a_2 \cdot 1000}{a_1 \cdot q} \qquad (1)$$

where

M is the migration in milligrams per kilogram;

m is the mass in milligrams of substance released by the sample as determined by the migration test;

a_1 is the surface area in square decimetres of the sample in contact with the simulant during the migration test;

a_2 is the surface area in square decimetres of the material or article intended to come into contact with foodstuff in real conditions of use;

q is the quantity in grams of foodstuff in contact with the material or article in real conditions of use.

12.2 Reduction factors

Reduction factors are conventionally used for some fatty foodstuffs to take account of the greater extractive capacity of the fatty food simulant compared to particular categories of foodstuffs. Where the use of a reduction factor is appropriate, individual test results are divided by the reduction factor before applying the test of validity as defined in 11.3. The reduction factors appropriate to various food types are to be found in Table 2

12.3 Validity of results

12.3.1 Aqueous food simulants

The following analytical tolerances are allowed:

6 mg/kg or 1 mg/dm² for all aqueous food simulants.

The test result for each individual test specimen is valid if it differs from the mean of the triplicate test results by not more than the permitted analytical tolerance. If a minimum of three results is not within the analytical tolerance, then the test is repeated using fresh test specimens from the sample.

A material or article with a mean overall migration result that exceeds the overall migration limit by an amount not exceeding the analytical tolerance, shall be deemed to be in compliance with the overall migration limit.

12.3.2 Fatty food simulants for single use applications

The following analytical tolerances are allowed:

20 mg/kg or 3 mg/dm² for all fatty food simulants and substitute test media.

The tolerances are valid also after application of a reduction factor to the results of the test.

If a reduction factor does not apply, results above 10 mg/dm² shall not differ by more than 30 % from the mean of the set of results.

The determination of overall migration into the fatty food simulant is normally carried out in quadruplicate to allow three valid results to be obtained even if one determination is discarded.

Where four results have been obtained from four determinations i.e. no single determination has been rejected because of an obvious manipulative error, all four results are valid when if each individual result differs from the mean of the four results by not more than the analytical tolerance. If one of the four results is greater or less than the mean by an amount more than the tolerance, then this result can be rejected and the mean recalculated on the remaining three results. If two results are greater or less than the mean by amounts more than the tolerance, the result with the largest difference from the mean can be rejected and a new mean calculated from the remaining three results. The remaining three test results are valid if they are within the analytical tolerance.

If a minimum of three results do not meet the above criteria of being within the analytical tolerance, then the test shall be repeated using fresh test specimens from the sample.

A material or article with a mean overall migration result that exceeds the overall migration limit by an amount not exceeding the analytical tolerance shall be deemed to be in compliance with the overall migration limit.

12.3.3 Fatty food simulants for repeated use applications

The permitted tolerances are as indicated in 8.8.3.

12.4 Test report

The test report shall include the particulars, required by the relevant Part of this Technical Specification.

12.5 Statements of compliance

When the test reports from the individual tests carried out according to the various Parts of this Technical Specification are collated and related to the limits for overall migration specified, a statement of compliance with regulatory limits may be made. This may include for what types of food and under what conditions of use a article may comply with overall migration limits.

13 Test method for overall migration into aqueous simulants by article filling from polymeric coatings on food and beverage cans and non-stick coatings

13.1 Scope

This Part of this Technical Specification specifies test methods for the determination of the overall migration into distilled water and aqueous ethanol food simulants, from polymeric coatings on metal articles in the form of containers, where the polymeric coatings are intended to come into contact with foodstuffs, by filling test specimens of the articles with a selected food simulant at test temperatures up to and including 70 °C for selected test times.

NOTE 1 Experience has shown that acetic acid is not an appropriate food simulant when testing polymeric coatings on metal substrates, but 10 % v/v aqueous ethanol has been shown to be acceptable;

NOTE 2 In practice industrial processes use temperatures in excess of 70 °C, commonly 121 °C, and it is recognized that this method will need to be extended to cover these conditions or that the conditions need to be covered in a separate test method.

13.2 Principle

The overall migration of non-volatile substances from a sample of the metal article with polymeric coating is determined as the mass of non-volatile residue after evaporation of the food simulant following filling and exposure of the test specimen.

The selection of the conditions of test will be determined by the conditions of use, see clauses 3, 4, and 5.

Test specimens are filled with the food simulant for the exposure time at temperatures up to and including 70 °C. At the end of the test period each test specimen is emptied. The food simulant from each test specimen is evaporated to dryness, the mass of the non-volatile residue is determined gravimetrically and expressed as milligrams per square decimetre of surface area exposed to the food simulant.

NOTE In some circumstances the procedure described in this Technical Specification may be used for exposure at temperatures above 70 °C

Overall migration is reported as the mean of three determinations on separate test specimens.

13.3 Reagents

NOTE For details of the preparation and purity of these reagents see clause 4.

13.3.1 Distilled water or water of equivalent quality (simulant A)

13.3.2 Ethanol with a volume fraction of 10 % in aqueous solution (simulant C)

13.3.3 Alcoholic simulants for liquids or beverages of an alcoholic strength exceeding a volume fraction of 10 %.

NOTE In the case of materials and articles intended to come into contact with liquids or beverages of an alcoholic strength exceeding a volume fraction of 10 %, the test may be carried out with aqueous solutions of ethanol of a similar strength.

13.4 Apparatus

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

13.4.2 Lint-free cloth or soft brush.

13.4.3 Conical flask, 2 l.

13.4.4 Glass beads, 2 mm to 3 mm diameter.

13.4.5 Thermostatically controlled oven or incubator or refrigerator capable of maintaining the set temperature within the tolerances specified in Table B.2.

13.4.6 Dishes, stainless steel, nickel, platinum, platinum alloy, gold 50 mm to 90 mm diameter and maximum weight 100 g, for evaporation of food simulants and weighing of residues. Glass, glass ceramic or ceramic dishes may be used, provided that the surface characteristics are such that the weights of the dishes after evaporation of any specified food simulants followed by conditioning in the desiccator used, achieves a constancy of ± 0,5 mg.

13.4.7 Steam bath, hot plate, distillation apparatus or rotary evaporator for evaporation of food simulant at the end of test period.

13.4.8 Desiccator with anhydrous calcium chloride or self indicating silica gel.

13.4.9 Beakers, 250 ml**.**

13.4.10 Pipette, 200 ml, complying with the minimum requirements of ISO 648.

13.5 Preparation of test specimens

13.5.1 General

It is essential that test specimens are clean and free from surface contamination. Before preparing test specimens, remove any surface contamination from the sample by gently wiping it with a lint free cloth, or by brushing with a soft brush. Under no circumstances wash the sample 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 8.1. Minimize handling of the samples and, where necessary, wear cotton gloves.

13.5.2 Number of test specimens

13.5.2.1 Volume of articles

Determine and record the volume of food simulant required to fill an article to its nominal foodstuff volume. If the nominal volume of foodstuff to fill the article is not known, determine the surface area which will be in contact with the food simulant when filled to 5 mm from the top of the test specimen.

13.5.2.2 Articles with a nominal volume of more than 200 ml

Five articles are required to provide five test specimens. These test specimens are utilized as follows:

a) Three test specimens for the migration test;

b) Two test specimens for the determination of surface area.

13.5.2.3 Articles with a nominal volume of less than 200 ml

The number of articles required to provide a test specimen is dependent on their volume. A test specimen shall be made up of sufficient articles to contain a minimum of 200 ml of the food simulant.

Five test specimens are required. These test specimens are utilized as follows:

a) Three test specimens for the migration test;

b) Two test specimens for the determination of surface area.

Record the number of articles used to provide the test specimen.

13.5.3 Surface area of test specimen exposed to food simulant

Determine and record the surface area of the test specimen which is intended to come into contact with its nominal volume of foodstuff. If the nominal volume of foodstuff to fill the article is not known, determine the surface area which will be in contact with the food simulant when filled to 5 mm from the top of the test specimen.

NOTE 1 For some articles it is recognized that it is impractical to measure the surface area intended to come into contact with foodstuff. For such articles the overall migration is measured as milligrams of substance released per kilogram of food simulant.

NOTE 2 In the case of articles with a volume of less than 200 ml this will be the surface area of one article multiplied by the number of articles used to provide a test specimen.

13.5.4 Articles with a capacity of not less than 500 ml and not more than 10 l

It is not necessary to determine the volume of these articles since the migration must be expressed in mg/kg of food simulant,

13.6 Procedure

13.6.1 Exposure to food simulant

Mark each test specimen for identification, and where more than one article has been used for a test specimen, also mark individually.

Place, in a conical flask, a sufficient volume of the food simulant to fill the three test specimens to the nominal volume, if known, or to 5 mm from the top and to provide two 200 ml blanks. Insert a thermometer or thermocouple in the simulant. Place the conical flask in the thermostatically controlled oven or incubator or refrigerator set at the test temperature and leave until the simulant has attained the test temperature.

Remove the conical flask containing the food simulant from the thermostatically controlled oven or incubator or refrigerator. Fill the three test specimens with simulant to the nominal volume of the article or to 0,5 cm from the top. Insert the thermometer or thermocouple in one of the test specimens containing simulant, if applicable - see NOTE 3. Cover the test specimens with an inert material to prevent evaporation. Stopper the conical flask containing the remaining simulant for the blanks. This filling of the test specimens should be carried out in the minimum time to prevent undue heat loss from the simulant.

Place the test specimens and food simulant for the blanks in the conical flask in the thermostatically controlled oven or incubator or refrigerator set at the test temperature. Observe the simulant temperature and leave the test specimens and the blank food simulant for the selected period of time after the temperature of the simulant has reached a temperature within the permitted tolerance for temperature, see Tables B.1 and B.2 for permitted tolerances on test times and temperature.

NOTE 1 Where the surface area of simulant is large, a check should be made to ensure that excessive loss of simulant by evaporation does not occur.

NOTE 2 Annex B includes tolerances on a wide range of contact times and contact temperatures. All of these contact times and contact temperatures are not necessarily relevant to this test method.

Take the test specimens and food simulant from the thermostatically controlled oven or incubator or refrigerator.

NOTE 3 For exposure times of 24 h or more it is acceptable to monitor the temperature of the airbath of the thermostatically controlled oven or incubator or refrigerator or instead of the temperature of the simulant.

13.6.2 Determination of migrating substances

13.6.2.1 Preparation of dishes

Take five dishes (12.5.6) marked for identification, place the dishes in an oven maintained at 105 °C to 110 °C, for a period of 30 min ± 5 min, to dry.

Remove the dishes from the oven, place in a desiccator (12.5.8) and allow to cool to ambient temperature. Weigh and record the individual masses of each dish.

Replace the dishes in the oven and repeat the cycle of heating, cooling and weighing until individual consecutive masses differ by no more than 0,5 mg, record their masses.

13.6.2.2 Evaporation method

Take the test specimens and measure from each, by pipette, 200 ml ± 2 ml of the simulant into separate 250 ml beakers, ensuring that the simulant is mixed.

Measure aliquots of 200 ml ± 2 ml of the food simulant from the conical flask which had been in the thermostatically controlled oven or incubator or refrigerator with the test specimens, into two more beakers to provide blanks.

Pour 40 ml to 50 ml from each beaker into separate dishes (12.5.6). By means of a steam bath, hot plate or other form of heating (12.5.7) evaporate to a low volume, taking care to avoid loss, in particular, by sputtering or overheating of the residues.

NOTE 1 The evaporation of ethanol solutions should be carried out in a fume cupboard.

When most of the simulant has evaporated pour the remaining simulant into the respective dishes. Wash each of the beakers with two lots of 10 ml ± 1 ml of unused simulant and pour these washings into the respective dishes. Continue the evaporation.

NOTE 2 A stream of nitrogen may be used to facilitate evaporation.

When the simulant in each dish has almost completely evaporated, place the dishes in an oven maintained at 105 °C to 110 °C, for a period of 30 min ± 5 min, to complete the evaporation and dry the residues.

Remove the dishes from the oven, place in a desiccator (12.5.8) and cool to ambient temperature. Weigh and record the individual masses of each dish and residue.

Replace the dishes in the oven and repeat the cycle of heating, cooling and weighing until individual consecutive masses differ by not more than 0,5 mg.

Determine the mass of the residue in each dish by subtracting the original stable mass of the dish from the stable mass of the dish and residue.

13.6.2.3 Distillation method

Transfer the simulants to individual round bottom flasks (250 ml are suitable). Rinse each test specimen with two lots of 10 ml ± 1 ml of unused simulant, add these rinses to the respective flasks. Measure into individual round bottom flasks two portions of the food simulant equal in volume to the food simulant that has been in contact with the test specimen plus the rinses, to provide blanks. Place each flask in an electric heating mantle and connect to a side arm distillation arrangement or rotary evaporator. Distil off the simulants until approximately 30 ml to 50 ml remains in each flask. Transfer the remaining simulant in each flask to individual evaporating dishes (12.5.6). Rinse each flask with two lots of 10 ml ± 1 ml of fresh simulant and add the rinses to the appropriate dishes. Continue the evaporation of the simulant by means of a steam bath, hot plate or other form of heating, proceeding as in 12.7.2.2.

NOTE The evaporation of ethanol solutions should be carried in a fume cupboard.

13.7 Expression of results

13.7.1 Method of calculation

Express the overall migration as milligrams of residue per square decimetre of the surface of the sample which is intended to come into contact with foodstuffs calculated for each test specimen using the following equation:

$$M = \frac{(m_a - m_b) \cdot 1000}{S} \qquad (2)$$

where

M is the overall migration into the simulant, in milligrams per square decimetre of surface area of sample;

m_a is the mass of the residue from the test specimen after evaporation of the simulant which had filled the test specimen, in grams;

m_b is the mass of residue from the blank simulant equal to the volume which had filled the test specimen , in grams;

S is the surface area of the test specimen which was in contact with the simulant during the exposure in square decimetres.

Calculate the result for each test specimen to the nearest 0,1 mg/dm² and the mean of the individual test results, to the nearest 0,1 mg/dm^2.

See 11.3 for the directions to determine whether the results are valid.

However, the overall migration shall be expressed in milligrams lost 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;

The overall migration shall be calculated for each test specimen using the following formula:

$$M_L = \frac{(m_a - m_b) \cdot 1000}{V} \qquad (3)$$

where

M_L is the overall migration into the simulant, in milligrams per kilogram of food simulant;

m_a is the mass of the residue from the test specimen after evaporation of the simulant which had filled the article, in grams;

m_b is the mass of residue from the blank simulant equal to the volume which had filled the test specimen, in grams;

V is the volume of simulant that filled of the test specimen in litres,

NOTE The specific gravities are by convention assumed to be 1. Therefore 1 litre of simulant is numerically the same as 1 kilogram.

Calculate the result for each test specimen to the nearest 1 mg/kg and the mean of the individual test results, to the nearest 1 mg/kg.

See 11.3 for the directions to determine whether the results are valid.

13.7.1.1 Articles with a volume of more than 200 ml but less than 500 ml; or of a volume greater than 10 l

$$M = \frac{(m_a - m_b) \cdot 1000 \cdot V}{S \cdot 200} \qquad (3)$$

where

- M is the overall migration into the simulant, in milligrams per square decimetre of surface area of sample;
- m_a is the mass of the residue from the test specimen after evaporation of 200 ml of the simulant which had filled the article, in grams;
- m_b is the mass of residue from the food simulant only, in grams;
- V is the volume of the food simulant which had filled the article, in millilitres.
- S is the surface area of the test specimen which was in contact with the simulant during exposure.

Calculate the result for each test specimen to the nearest 0,1 mg/dm2 d the mean of the individual test results, to the nearest 0,1 mg/dm2.

See 11.3 for the directions to determine whether the results are valid.

13.7.1.2 Articles with a volume of not less than 500 ml and not more than 10 l

$$M_L = \frac{(m_a - m_b) \cdot 1000}{0,2} \qquad (4)$$

that is

$$M_L = 5000 \cdot (m_a - m_b) \qquad (5)$$

where

- M_L is the overall migration into the simulant, in milligrams per kilogram of food simulant;
- m_a is the mass of the residue from the test specimen after evaporation of 200 ml of the simulant, in grams;
- m_b is the mass of residue from the 200 ml of blank simulant, in grams.

Calculate the result for each test specimen to the nearest 1 mg/kg and the mean of the individual test results, to the nearest 1 mg/kg.

See 11.3 for the directions to determine whether the results are valid.

13.7.1.3 Articles with a volume of less than 200 ml

$$M = \frac{(m_a - m_b) \cdot 1000}{S \cdot N} \qquad (6)$$

where:

M is the overall migration into the simulant, in milligrams per square decimetre of surface area of sample;

m_a is the mass of the residue from the test specimen after evaporation of the simulant which had filled the article, in grams;

m_b is the mass of residue from the blank simulant equal to the volume which had filled the test specimen, in grams;

S is the surface area of one test article in square decimetres;

N is the number of articles exposed to the simulant.

Calculate the result for each test specimen to the nearest 0,1 mg/dm2 and the mean of the individual test results, to the nearest 0,1 mg/dm^2.

See 11.3 for the directions to determine whether the results are valid.

13.7.2 Precision

NOTE The repeatability (r) and the reproducibility (R) values are to be determined from collaborative trial results.

13.8 Test report

The test report shall include the following (see clause 11):

a) reference to this Technical Specification and to the clause used for the test procedure;

b) all information necessary for complete identification of the sample such as chemical type, supplier, trade mark, grade, batch number;

c) conditions of time and temperature of exposure to simulants;

d) departures from the specified procedure, and reasons for these;

e) individual test results, and the mean of these, expressed as milligrams of residue per square decimetre of sample or as milligrams per kilogram of food simulant as appropriate;

f) relevant comments on the test results.

Annex A
(normative)

Characteristics of fatty food simulants and test media

A.1 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 %

A.2 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	0thers
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	~88	~95	~97	~98

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

A.3Characteristics 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%

A.4 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%

A.5 Characteristics of modified polyphenylene oxide (MPPO)

molecular weight	500,000 to 100,000
size	60 mesh to 80 mesh
T_{max}	350 °C
specific mass	0,23 g/ml

Annex B
(normative)

Limit deviations on contact times and contact temperatures applicable to all clauses of this Technical Specification

Table B.1 — Contact times and limit deviations

Contact times and limit deviations
30^{+1}_{0} min
60^{+1}_{0} min
90^{+3}_{0} min
120^{+5}_{0} min
150^{+5}_{0} min
180^{+7}_{0} min
210^{+8}_{0} min
240^{+9}_{0} min
270^{+10}_{0} min
300^{+12}_{0} min
360^{+15}_{0} min
$24^{+0,5}_{0}$ h
$48^{+0,5}_{0}$ h
240^{+5}_{0} h

Table B.2 — Contact temperatures and limit deviations

Contact temperatures and limit deviations
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 C
(normative)

Supports and cells

Dimensions in millimetres

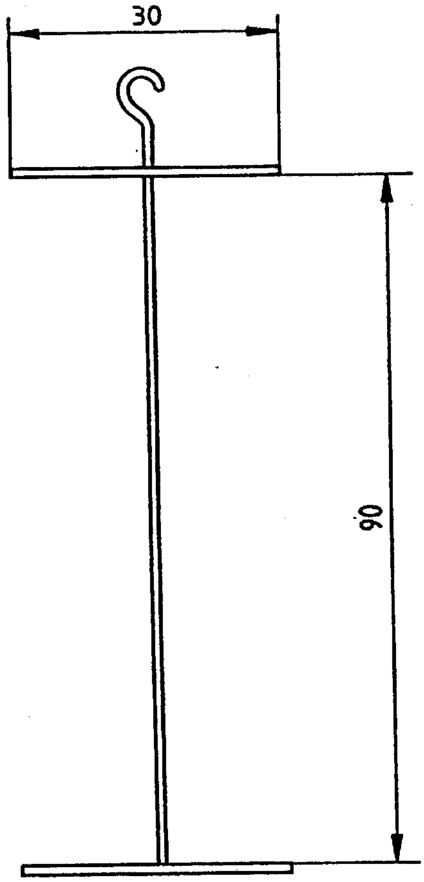


Figure C.1 — Example of support

Dimensions in millimetres

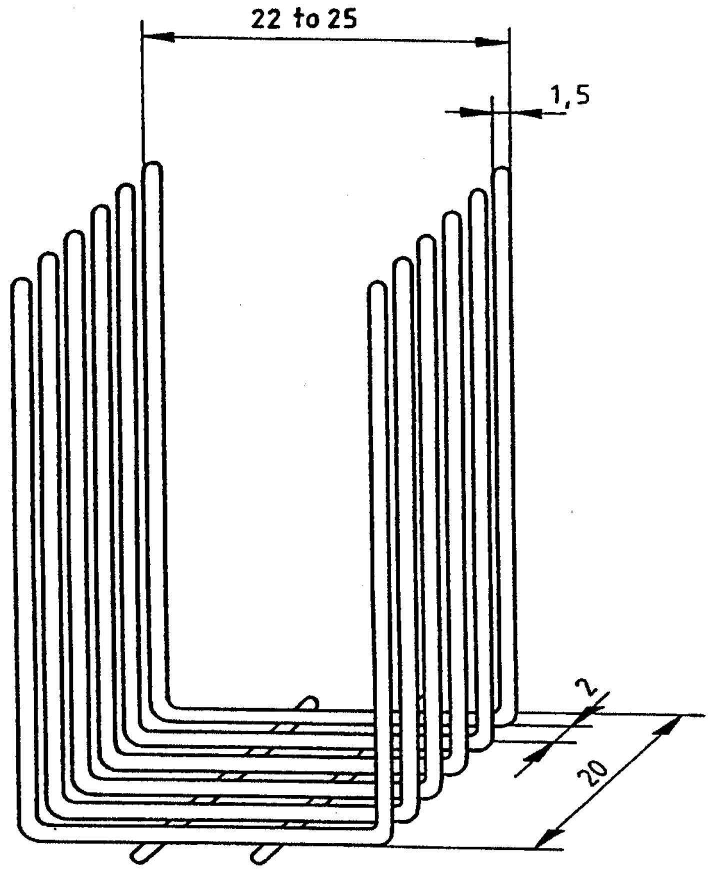
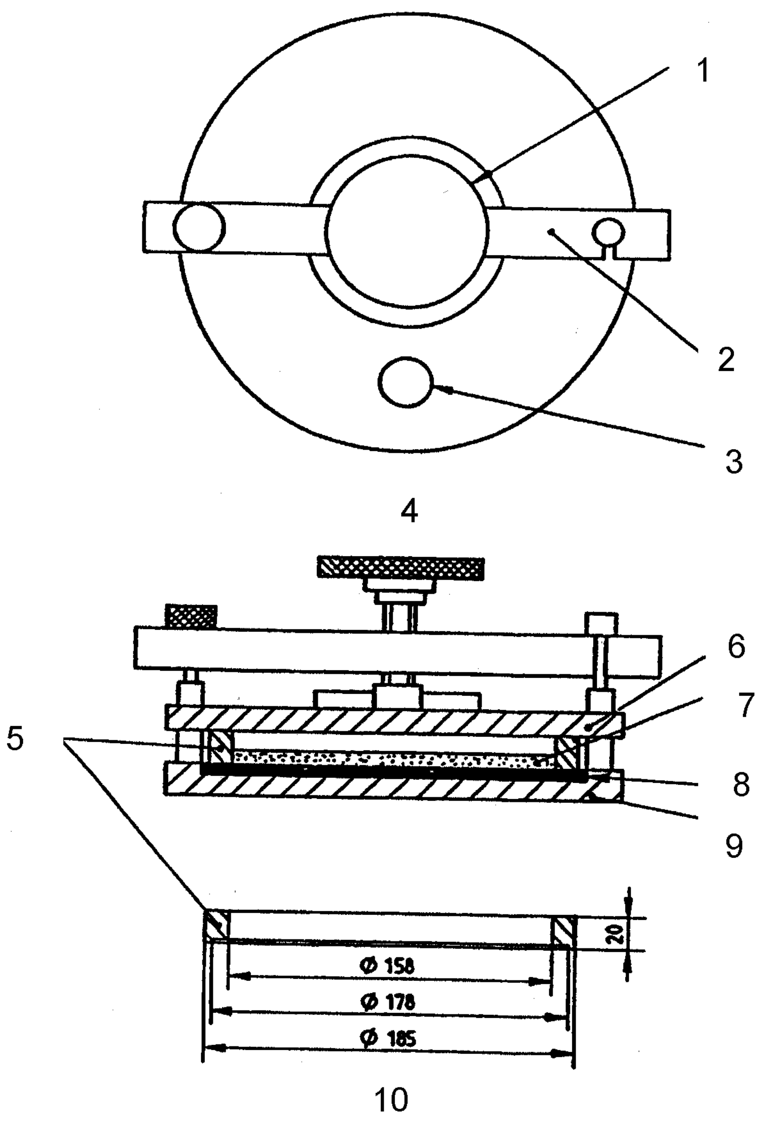


Figure C.2 — **Example of support**

Dimensions in millimetres

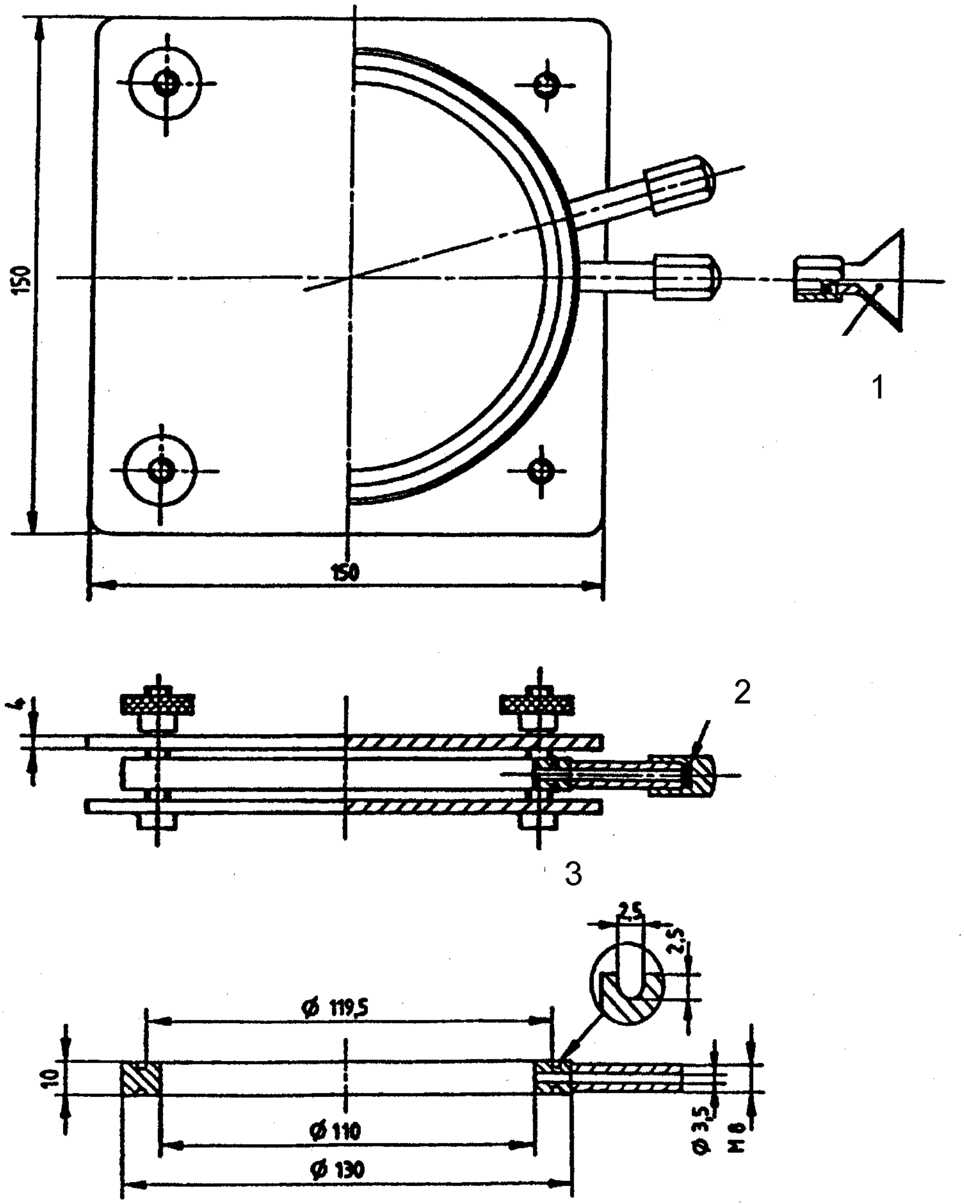


Key

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

Figure C.3 — Cell type A

Dimensions in millimetres

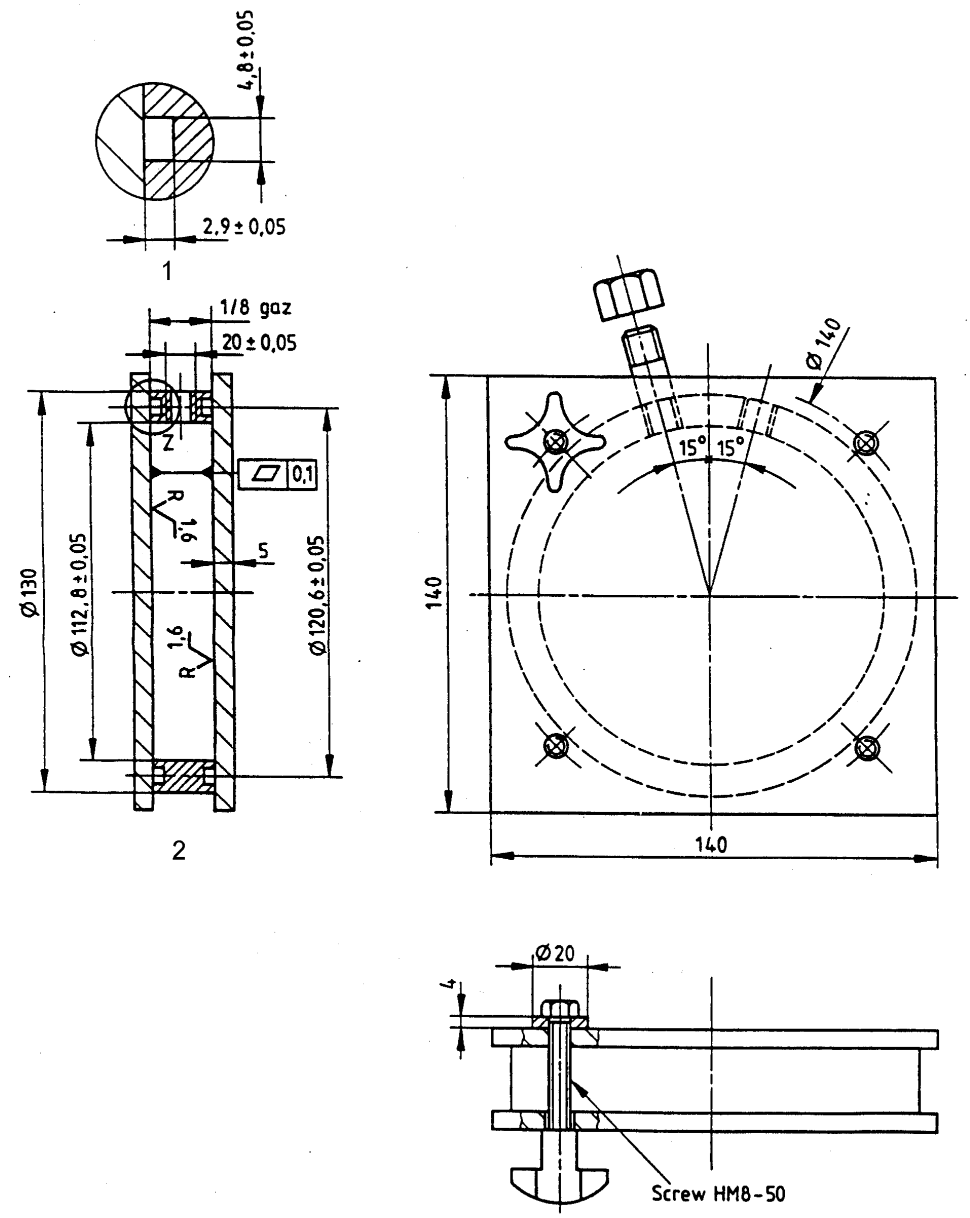


Key

1 Funnel for filling
2 PTFE disk
3 PTFE 'O'-ring (119,5 X ∅ 3)

Figure C.4 — Cell type B

Dimensions in millimetres



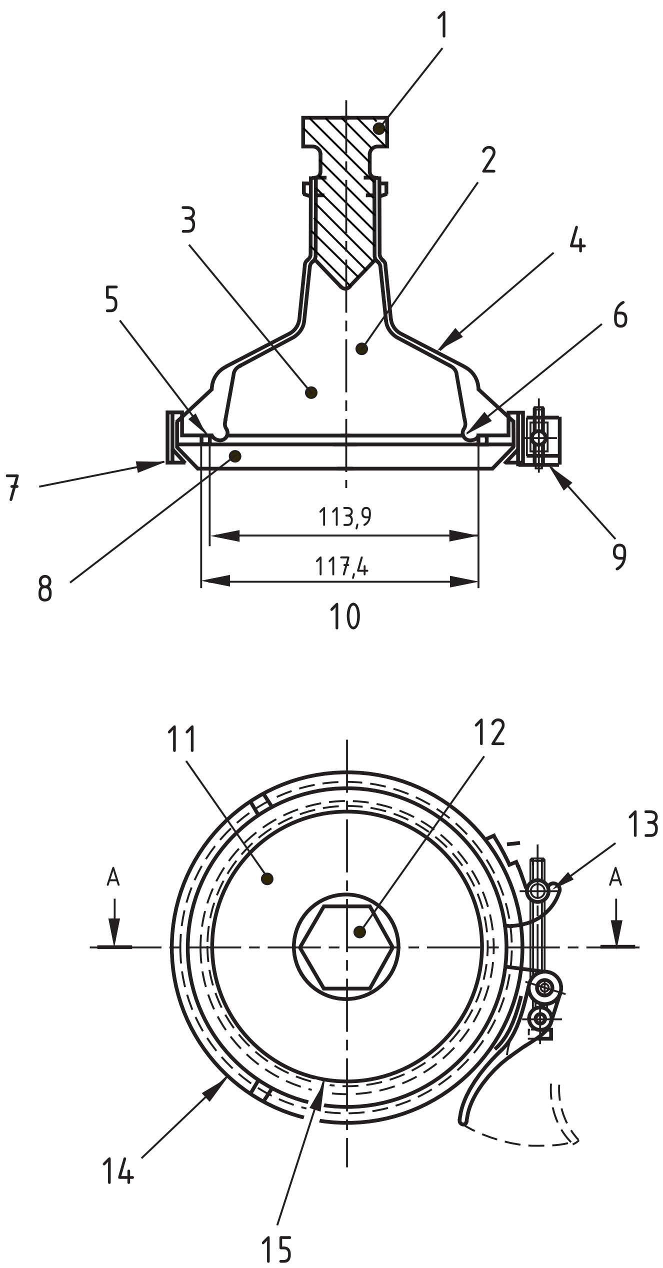
Key

1 Detail Z

2 'O'-ring ∅117,07/124,13/3,53

Figure C.5 — Cell type C

Dimensions in millimetres



Key

1 Glass stopper
2 Total inner volume 296ml (max volume of simulant 250ml)
3 Exposed surface area of circular test specimens 1,019 dm^2
4 Glass bell
5 Sealing ring ('O' ring) silicone rubber sheathed in PTFE
6 Raised edge to fix the 'O' ring in place
7 Tension ring (stainless steel)
8 PTFE plate
9 Tensioning 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 C.6 — Cell type D

Dimensions in millimetres

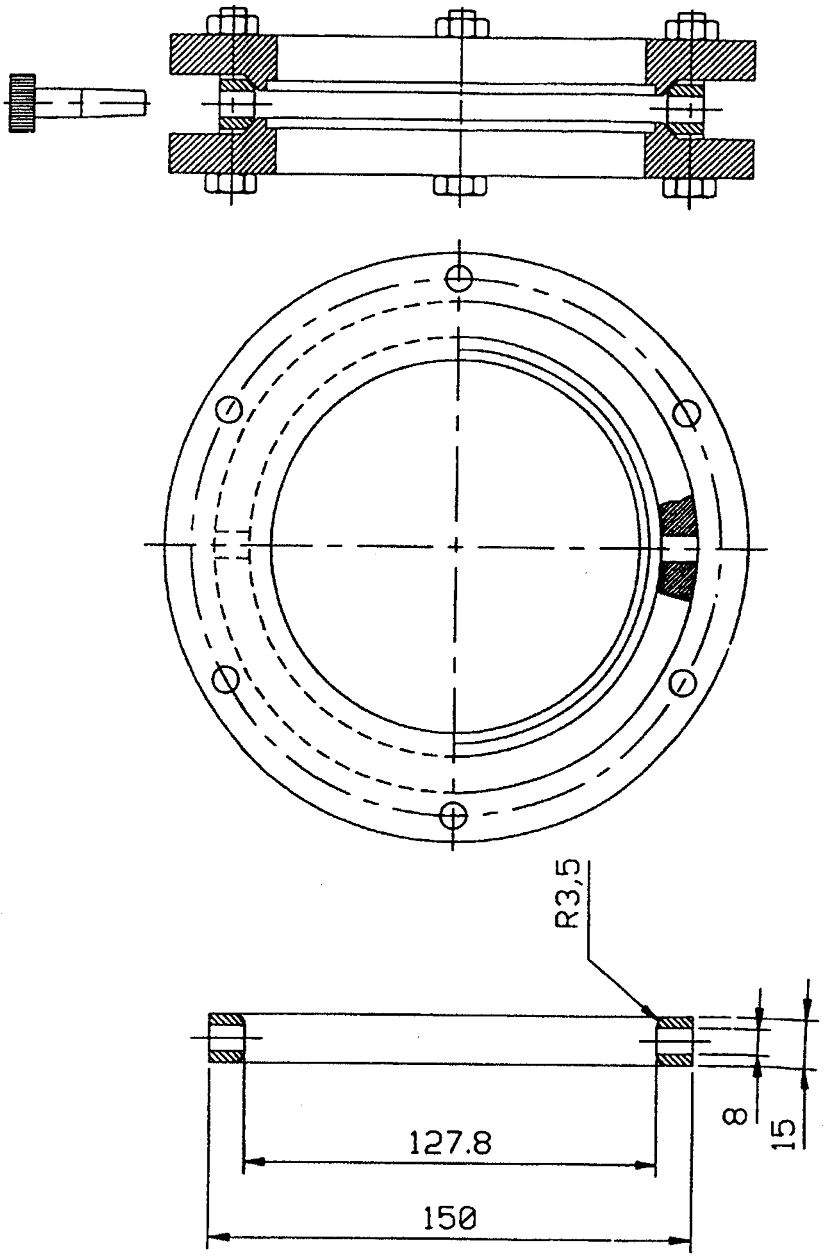
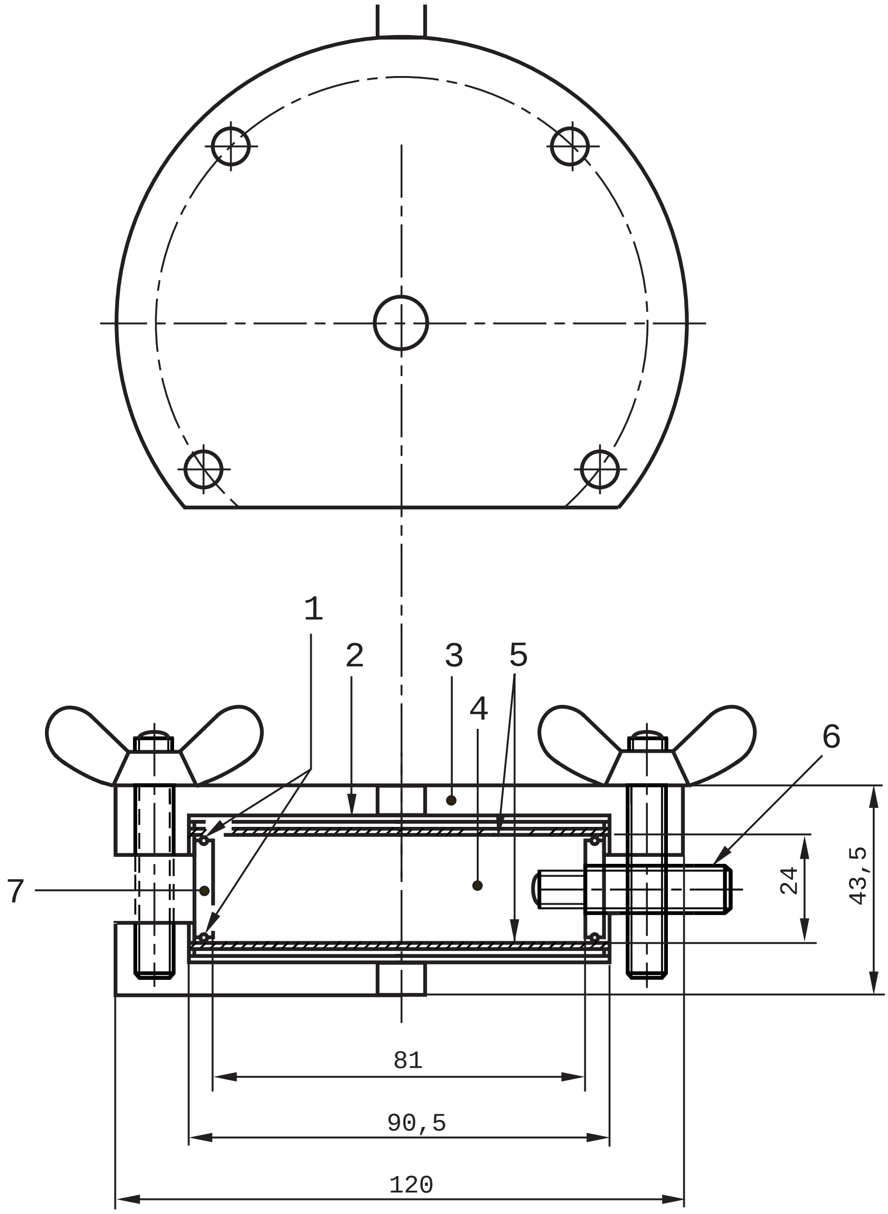


Figure C.7 — Cell type E

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 C.8 — Cell type F

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[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.

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[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

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[7] ISO 4788:1980 Laboratory glassware - Graduated measuring cylinders

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