IS 9845 (1998): Determination of Overall Migration of Constituents of Plastics Materials and Articles Intended to Come in Contact with Foodstuffs - Method of Analysis [PCD 12: Plastics]

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भारतीय मानक

खाद्य सामग्री के संपर्क में आने वाली प्लास्टिक-सामग्री और वस्तुओं के संघटकों की समग्न गतिशीलता ज्ञात करना — विश्लेषण पद्धति

(दूसरा पुनरीक्षण)

Indian Standard

DETERMINATION OF OVERALL MIGRATION OF CONSTITUENTS OF PLASTICS MATERIALS AND ARTICLES INTENDED TO COME IN CONTACT WITH FOODSTUFFS — METHOD OF ANALYSIS

(Second Revision)

ICS 83.140.10 ; 55.120

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Price Group 3

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The ingredients in the plastics packaging materials may cause toxicity as a result of their migration to the foodstuffs in which the later are packed. Prior to categorizing such plastics as toxic, evidence regarding degree of migration of their constituents has to be ascertained. In general, migration and extraction studies need to be simultaneously conducted on actual foodstuffs under conditions which are slightly more stringent than those encountered in normal usage. It is, however, not always possible to analyse actual foodstuffs for nature and quantity of migrants from the plastics. In order to simplify such assessment, food simulants/extractants have to be substituted for the actual foodstuffs. Further, it is also very difficult to estimate all the migrants individually. Hence, as a good measure, the overall migration of all the migrants put together is considered for safe use, unless they are especially toxic and their limits fixed.

This standard was first published in 1981 and prescribed the method for single layer plastic films only, which was subsequently revised in 1986 enlarging its scope by including method for determination of overall migration of single or multilayer composites in the form of a pouch or container. The extraction method prescribed in the standard embraces all factors likely to be encountered in actual practice in packaging with plastic materials which are likely to come into contact with foodstuffs. Method I was essentially based on Commission of European Communities and experimentally modified by Dr A. S. Aiyar of Bhabha Atomic Research Centre, Bombay and Method II was developed by Central Food Technological Research Institute (CFTRI), Mysore. Two tables were included — Table 1 Classifying food products into seven major types and covering all the food products listed in IS 10171 : 1986 'Guide on suitability of plastics for food packaging', and Table 2 Listing the simulants and test conditions (duration and temperature) for extraction depending on type of food and condition of its use. It was also felt that in due course test methods for determining specific migration of plastic ingredients may have to be devised.

The Committee responsible for the preparation of this standard, while reviewing noted that the test conditions (time and temperature) and food simulants extractants stipulated by the Commission of European Communities (namely, the various EEC-Directives) and the Codes of Federal Regulations (FDA), USA correspond most closely to the normal or forseeable conditions of contact for the plastics materials or articles. Accordingly the Committee decided to revise the standard and align it completely with the various council Directives (EEC) laying down the basic rules necessary for testing migration of the constituents of plastic materials and articles intended to come into contact with foodstuffs, namely, 82/711/EEC dated 18 October 1982, 85/572/EEC dated 19 December 1985, 90/128/EEC dated 23 February 1990, and 92/39/EEC dated 14 May 1992 respectively. Assistance has also been drawn from the Code 21 CFR 175.300 of FDA (USA) and method published in Deutsche Lebensmittel Rundschau/88 Jahrg./Heft 5/1992, while preparing the second revision of this standard.

In this revision, as many as five test methods depending upon the shape/form of the material for the determination of overall migration of constituents of plastics materials and articles intended to come into contact with foodstuffs have been prescribed — Method I and III having been developed by CFTRI, Mysore. Tables 1 and 2 have been revised and test conditions (time and temperature) and the list of simulants appropriate for the foodstuffs or group of foodstuffs have been modified and aligned with the EEC-Directives and Code of Federal Regulations (FDA), USA.

In reporting the result of a test or analysis, made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

DETERMINATION OF OVERALL MIGRATION OF CONSTITUENTS OF PLASTICS MATERIALS AND ARTICLES INTENDED TO COME IN CONTACT WITH FOODSTUFFS — METHOD OF ANALYSIS

(Second Revision)

1 SCOPE

This standard prescribes the methods of analysis for determination of overall migration of constituents of single or multi-layered heat-sealable films, single homogenous non-sealable films, finished containers and closures for sealing as lids, in the finished form, preformed or converted form.

2 NORMATIVE REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
9833 : 1981	List of pigments and colourants for use in contact with foodstuffs, pharmaceuticals and drinking water
10171 : 1986	Guide on suitability of plastics for food packaging

3 SIMULANTS

The determination of migration in simulants is to be carried out using the simulants laid down as under:

Simulant 'A' — distilled water or water of equivalent quality.

Simulant 'B' — 3 percent acetic acid (*w/v*) in aqueous solution (using the simulant 'A')

Simulant 'C^1' — 10 percent ethanol (*v/v*) in aqueous solution for foodstuffs having alcohol less than 10 percent (*v/v*) (using the simulant 'A').

Simulant 'C^2' — 50 percent ethanol (*v/v*) in aqueous solution for foodstuffs having alcohol more than 10 percent and less than 50 percent (*v/v*) (using the simulant 'A').

Simulant 'D' — *n*-heptane — shall be freshly distilled before use.

Simulant 'E' — Rectified olive oil or mixture of synthetic triglycerides or sunflower oil.

NOTE — This simulant 'E' suggested by EEC for fatty foods need not be considered at present as the methodology of estimation is not yet developed.

4 SELECTION OF STANDARD TEST CONDITIONS AND SIMULANTS FOR DIFFERENT FOODS

4.1 The choice of simulating solvents and test conditions (time-temperature) depends on the type of food and condition of use of food products. Food products have been now classified into seven major groups as per Table 1. The food products listed in IS 10171 have been fully covered in this table (further additions shall be made whenever necessary). This table has been prepared on the lines of accepted classification of foodstuffs for such purposes in developed countries. Table 1 also gives suitable simulants to be used for different types of foods.

4.2 Table 2 lists the simulants and test conditions (time-temperature) for extractability studies to be carried out as above depending on the type of food and conditions of use.

Table 1 Classification of Foods and Selection of Simulant
(*Clause* 4.1)

Sl No.	Type	Description	Example	Simulants
(1)	(2)	(3)	(4)	(5)
i)	I	Aqueous, non acidic foods (pH > 5) without fat	Honey, mineral water, sugar syrups molasses, skimmed milk, *rasgulla*, infusions, *murabba*, yeast paste etc	'A'
ii)	II	Aqueous, acidic foods (pH ≤ 5) without fat	Fruit juices, squashes, fruit chunks or puree or paste, vinegar, jams, jellies, carbonated beverages, lemonade, processed vegetables, rennet, preparations of soups, broths, sauces, RTS beverages etc	'B'
iii)	III	Alcoholic beverages:		
		i) Alcohol concentration less than 10 percent	Beer and some pharmaceuticals syrups	'C^1'
		ii) Alcohol concentration above 10 percent	Wine, brandy, whiskey, arrack and other alcoholic drinks	'C^2'
iv)	IV	Oils, fats and processed dry foods with surface fat or volatile oil	Vegetable oils, *ghee*, *vanaspati*, cocoa butter, lard, biscuits, spice powder, snacks and savoury, chocolate, caramels, malted foods, egg powder, tea, coffee powder confectionery, fried and roasted nuts etc	'D'
v)	V	Nonacidic foods (pH > 5) or high fat and having high moisture content	Butter, bread, pastry, *shreekand* with low cakes, milk based sweets, ice-cream, moist and fatty confectionery products	'A and D'
vi)	VI	Acidic foods (pH < 5) or high fat and having high moisture content	Pickles, ketchup, cheese, with low curd, fresh and processed meat and fish products, sauces having fat, frozen foods, mayannaise etc	'B and D'
vii)	VII	Dry processed foods without fat	Cereals and pulses, dehydrated vegetable and fruits, dried yeast, corn flakes, salt, sugar, milled products, barley powder, oats, vermicelli, spaghetti etc	No end test

Table 2 Simulating Solvents for Different Types of Food and Temperature — Time Conditions
(*Clause* 4.2)

Sl No.	Conditions of Use	Type of Food	Water (Time-Temp)	3 Percent Acetic Acid (Time-Temp)	10% Alcohol (Time-Temp)	50% Alcohol (Time-Temp)	*n*-Heptane (Time-Temp)
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	High temperature heat sterilized (Retorting)	I, II, IV V and VI	121°C, 2 h	121°C, 2 h	—	—	66°C for 2 h
ii)	Hot filled or pasteurized above 66°C, below 100°C	I, II, IV V and VI	100°C, 2 h	100°C, 2 h	—	—	49°C for 30 minutes
iii)	Hot filled or pasteurized below 66°C	I to VI	70°C, 2 h	70°C, 2 h	70°C, 2 h	70°C,	38°C for 30 minutes
iv)	Room temperature filled and stored (no thermal treatment in container) and also in refrigerated and frozen condition	do	40°C, 10 days	40°C, 10 days	40°C, 10 days	40°C, 10 days	do

NOTES
1 Heptane simulant not to be used on wax lined containers.
2 Heptane extractivity results must be divided by a factor of five in arriving at the extractivity of a food product.

5 METHOD I : FOR FINISHED CONTAINERS (WITHIN 2 LITRES CAPACITY) OR SEALABLE SINGLE/MULTI-LAYERED FLEXIBLE FILMS (ONE-SIDE EXPOSURE)

5.1 Apparatus

5.1.1 Electric oven/water bath equipped with thermostat to maintain the desired temperature up to ± 1°C accuracy.

5.1.2 Electric hot plate with temperature control regulator.

5.1.3 Analytical balance with a sensitivity of 0.1 mg.

5.1.4 Glass beakers, pyrex of 1 000 ml capacity or equivalent.

5.1.5 Stainless steel evaporating dish of 100 ml capacity.

5.1.6 Stainless steel tongs.

5.2 Selection of Samples

Minimum triplicate samples representing the lot/batch have to be selected. Samples in each replicate shall not consist of a number of containers (preformed or converted products) with nearest exposed area of 1 000 cm^2. In the case of films representative sample shall be of sufficient size to convert into 2 pouches of size 125 mm width and 200 mm length (inner dimension excluding seal area) with 1 000 cm^2 surface area coming in contact.

5.3 Preparation of Test Specimen

The containers/pouches used shall be carefully rinsed with water (25-30°C) to remove extraneous materials prior to actual migration test.

5.4 Simulant Quantity

Equal to nominal filling capacity or at least 1 ml/cm^2 of contact area.

NOTE — Glassware, laboratory apparatus which come into contact with simulants and/or the sample during the test shall be thoroughly washed and dried prior to test.

5.5 Procedure

Fill the container/pouch to their filled capacity with preheated simulant at test temperature and close it. In case of pouches, exclude air as much as possible before sealing and expose the filled container/pouch to specified temperature maintained in oven/waterbath/ pressure cooker/autoclave for the specified duration of time. After exposure for the specified duration, remove the container/pouch and transfer the contents immediately into a clean pyrex beaker along with three washings of the specimen with small quantity of the fresh simulant.

5.6 Determination of Amount of Extractive

Evaporate/distill the contents in pyrex beaker to about 50-60 ml and transfer into a clean tared stainless steel dish along with 3 washings of pyrex beaker with small quantity of fresh simulant and further evaporate the concentrate in the dish to dryness in an oven at 100 ± 5°C. Cool the dish with extractive in a desiccator for 30 minutes and weigh to nearest 0.1 mg till constant weight of residue is obtained. Calculate the extractives in mg/dm^2 and mg/kg or mg/l or ppm of the foodstuff with respect to the capacity of container/pouch to be used. Blank shall also be carried out without the sample.

$$\text{Amount of extractive } (Ex) = \frac{M}{A} \times 100 \text{ mg/dm}^2, \text{ and}$$

$$\frac{M}{V} \times 1\,000 \text{ mg/kg or mg/l}$$

or ppm

where

M = mass of residue in mg minus blank value,

A = total surface area in cm^2 exposed in each replicate, and

V = total volume in ml of simulant used in each replicate.

NOTES

1 For irregular shaped containers, nearest surface area is obtained by superimposing the graph sheet on the container and getting the surface area by increments in each segment.

2 In case of heptane as solvent divide Ex by a factor of five in arriving at the extractivity for a food product.

6 METHOD II : FOR LARGER CONTAINERS MADE OF SINGLE HOMOGENOUS MATERIAL ABOVE 2 LITRES CAPACITY

6.1 Selection of Sample

Minimum 3 containers representing the lot/batch are to be selected.

6.2 Test Specimen

Cut 5 pieces each of size 10 cm × 10 cm from each container at different places (each piece exposing about 200 cm^2 surface area both sides). In the case of thick material area corresponding to thickness of the sample shall also be included.

6.3 Procedure

Immerse 5 thoroughly cleaned pieces cut from each container into a clean glass container (2 litre capacity beaker) containing preconditioned simulant at test temperature such that no two pieces touch each other by placing a 2 to 3 mm dia glass rod in between the specimens and cover the beaker with glass plate/watch glass and keep the set at specified temperature maintained in oven/waterbath/pressure cooker for the specified duration of time. After exposure for the specified time, remove the test specimen from the extracted simulant with the help of clean tongs and wash the pieces with small amount of fresh simulant and combine with the extracted simulant. Blank shall also be carried out without the sample.

6.4 Determination of Amount of Extractive

Calculate the extractive in mg/dm^2 and mg/kg or mg/l or ppm with respect to capacity of the container in accordance with the procedure specified in **5.6**.

$$\text{Amount of Extractive } (Ex) = \frac{M}{A} \times 100 \text{ mg/dm}^2$$

$$Ex \text{ in ppm} = M \times TSA \times \frac{1\,000}{A \times V}$$

where

M = mass of residue in mg minus blank value,

A = surface area in cm^2 exposed in each replicate,

TSA = total surface area of the container in cm^2, and

V = total volume of the container (cc).

NOTE — Heptane extractive to be divided by factor of five.

7 METHOD III : BOTH SIDE EXPOSURE FOR SINGLE HOMOGENOUS FILM, WHICH CAN NOT BE HEAT SEALED

7.1 Apparatus

7.1.1 *Cylindrical Glass Jar,* inner dimension of 10 cm diameter and 14 cm height with 1 000 ml capacity (or 1 litre beaker).

7.1.2 *Water Bath/Electrical Oven,* equipped with thermostat to maintain the desired temperature up to $\pm 1^{\circ}C$.

7.1.3 *Glass/Stainless Steel Pins,* of 7.5-8.00 cm working length with extra bends at both the ends.

7.1.4 *Electric Hot Plate,* with temperature regulator.

7.2 Specimen Size

A film sample of 1 000 cm^2 surface area both sides with width not more than 10 cm and an appropriate length to get the required area (10 cm × 50 cm × 2 sides = 1 000 cm^2).

7.3 Simulant Quantity

Not less than 1 000 ml to immerse the sample completely.

7.4 Preparation of Specimen

The film sample is rolled in the form of a coil in different concentric rings such that no two layers shall touch each other, and held in shape with the help of glass or stainless steel (SS) pin (*see* Fig. 1).

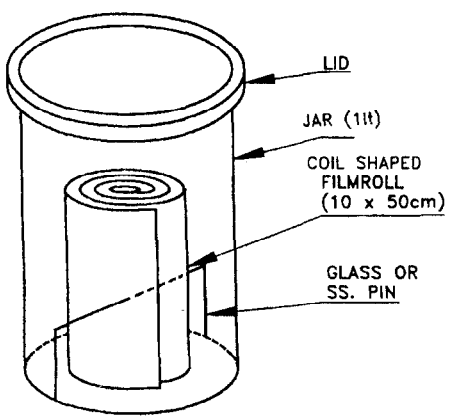

FIG. 1 EXTRACTION CELL

7.5 Procedure

Fill the cylindrical jar of 1 000 ml capacity with the required quantity of preheated simulant at the test temperature. Immerse the test specimen in the simulant completely. Cover with a glass plate and place the jar with sample immersed in simulant at the prescribed temperature for the prescribed length of time. At the end of the test period remove the sample with the help of glass rod and wash the sample with small quantity of fresh simulant and combine with the extractants. Concentrate the extracted simulant to 50-60 ml, by evaporating on a hot plate under low heat (*n*-heptane shall be concentrated by distillation). Transfer the concentrate into a clean tared stainless steel dish along with three washings with small amount of fresh simulant and further evaporate the concentrate to dryness in an oven at $100 \pm 5^{\circ}C$. Cool this in a desiccator for 30 minutes and weigh to nearest 0.1 mg till constant weight of residue is obtained. Calculate the extractive in mg/dm^2. Blank shall also be carried out without the sample.

$$\text{Amount of extractive } (Ex) = \frac{M}{A} \times 100 \text{ mg/dm}^2$$

where

M = mass of residue in mg minus blank value, and

A = total surface area in cm^2 exposed in each replicate.

NOTE — Heptane extractive value to be divided by factor of five.

8 METHOD IV : FOR CLOSURES, SEALING GASKETS, LINERS AND LIKE MATERIALS

8.1 Selection of Sample

At least triplicate samples each consisting of a number of closures/sealing gaskets/liners with the lids exposing about 100 cm^2 contact area (or ten lids) per replicate in each representing a lot or batch shall be selected.

8.2 Procedure

Smallest size glass bottles/jars actually being intended for use with closures can be used as containers to contain the simulant. Fill the glass container to their nominal capacity or 100 ml, whichever is lower with simulant preheated to test temperature and closed tight with the closures/lids lined with the test specimen. Place the closed containers upside down (to ensure the contact of the closures with the simulant) in an oven maintained at test temperature. After the exposure to the stipulated time, the closure from the containers are opened and the contents from each replicate is pooled together in a glass beaker along with the washings of the exposed closures with small amount of fresh simulant. Blank shall also be carried out without the sample.

8.3 Determination of Amount of Extractive

Proceed with the determination amount of extractive by method described at **5.6**.

Calculate the amount of extractive in ppm for the particular size of container being tested.

$$\text{Amount of extractive } (Ex) = \frac{M}{V} \times 1\,000 \text{ ppm}$$

where

M = mass of residue in mg minus blank value, and

V = volume of the container in ml in a replicate in actual use.

NOTES

1 If the extractive values for a smaller size container are within the prescribed limits, it may be taken that extractive values for a larger size container of the same material and same shape will definitely be less than the smaller container used.

2 Heptane extractive to be divided by factor of five.

9 METHOD V : MATERIALS OF ARTICLES INTENDED TO COME INTO REPEATED CONTACT WITH FOODSTUFFS

The migration test(s) shall be carried out three times on a same sample one after the other in accordance with the conditions laid down already using fresh simulant(s) in each occasion, following any one of the method applicable to it described earlier. Its compliance shall be checked on the basis of the level of the migration found in the third test. However, if there is conclusive proof that the level of the migration does not increase in the second and third tests and if the migration limit(s) is/are not exceeded on the first test, no further test is necessary.

10 EVALUATION OF RESULTS

The materials and articles are regarded as confirming to the specifications if in the migration tests for each simulant used, the average of at least three results does not exceed the value of overall migration limit specified in the relevant standards.

NOTE — Before carrying out the test, make sure that sample is free from all traces of dust, fats and other impurities. If necessary it shall be thoroughly wiped with filter paper (Whatman No. 1). The sample shall be handled carefully to avoid any contamination.

11 COLOUR MIGRATION

In the case of coloured plastic materials, colour migrated to simulant or decolourised coconut oil or food packed shall not be apparent to naked eye. If the colour migrated is clearly visible, such materials are not suitable for food contact applications, even though the extractive value is within the limit (*see* IS 9833).

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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