Post-consumer polyethylene terephthalate (PET) containers — Specification — Part 1: Food grade PET recyclates, preforms and containers
In order to match with technological development and to keep continuous progress in industries, standards are subject to periodic review. Users shall ascertain that they are in possession of the latest edition.
## Contents

<table>
<thead>
<tr>
<th></th>
<th>Scope</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>Normative references</td>
<td>1</td>
</tr>
<tr>
<td>3</td>
<td>Terms and definitions</td>
<td>2</td>
</tr>
<tr>
<td>4</td>
<td>Requirements</td>
<td>4</td>
</tr>
<tr>
<td>5</td>
<td>Packaging</td>
<td>8</td>
</tr>
<tr>
<td>6</td>
<td>Marking</td>
<td>8</td>
</tr>
<tr>
<td>7</td>
<td>Sampling</td>
<td>9</td>
</tr>
</tbody>
</table>

Annex A (normative) Determination of overall migration of constituents of plastics materials and articles intended to come in contact with foodstuffs - Methods of analysis ....................... 11
Foreword

Rwanda Standards are prepared by Technical Committees and approved by Rwanda Standards Board (RSB) Board of Directors in accordance with the procedures of RSB, in compliance with Annex 3 of the WTO/TBT agreement on the preparation, adoption and application of standards.

The main task of technical committees is to prepare national standards. Final Draft Rwanda Standards adopted by Technical committees are ratified by members of RSB Board of Directors for publication and gazettment as Rwanda Standards.

DRS 499-1 was prepared by Technical Committee RSB/TC 020, Packaging and packaging materials.

In the preparation of this standard, reference was made to the following standards:

1) MS 250:2021 Plastics – Post-consumer polyethylene terephthalate (PET) bottle – Specifications for food grade PET recyclates and preforms

The assistance derived from the above source is hereby acknowledged with thanks.

DRS 499 consists of the following parts, under the general title Post-consumer polyethylene terephthalate (PET) containers — Specification

— Part 1: Food grade PET recyclates, preforms and containers
— Part 2: Non-food grade PET recyclates, preforms and containers

Committee membership

The following organizations were represented on the Technical Committee on Packaging and packaging materials. (RSB/TC 020) in the preparation of this standard.

BRALIRWA Plc

Cleaner Production and Innovation and Climate Innovation Centre (CPCIC/NIRDA)

Horizon Sopyrwa

Rwanda Forensic Laboratory

Rwanda Harambee

Rwanda Inspectorate, Competition and Consumer Protection Authority (RICA)

Rwanda Investigation Bureau (RIB)
The Coca-Cola Company

University of Rwanda-College of Education (UR-CE)

University of Rwanda-College of Science and Technology (UR-CST)

Rwanda Standards Board (RSB) – Secretariat
Introduction

Polyethylene terephthalate (PET) has become the preferred packaging material worldwide over glass and metal for water and soft drinks bottles. With the increasing demand for packaged and processed food, the risk of the amount of plastic waste accumulated will be too large for landfilling. To prevent degradation of the environment, manufacturers are encouraged to dispose post-consumer plastic bottles and containers properly for ease of recycling.

Recycling of PET waste is one type of material recovery process intended to save resources while minimizing harmful emissions into air, water and soil as well as human health.

The environmental impact of recycling has to be assessed over the whole life cycle of the recycling system (from the waste generation point to the disposal of final residues). To ensure that recycling constitutes the best environmental option for treating the available waste, some prerequisites should preferably be met:

— recycling scheme being contemplated should generate lower environmental impacts than alternative recovery options;

— existing potential market outlets should be identified that would secure a sustainable industrial recycling operation;

— collection and sorting schemes should be properly designed to deliver recyclable plastics waste fractions fitting reasonably well with the available recycling technologies and with needs of the identified market outlets.

Plastics packaging waste may contain residues from previous use, contaminants from misuse and contaminants from non-authorised substances which could migrate into the food the material contacts. Manufacturers of food contact articles made from recycled PET are responsible for ensuring that, like virgin material, recycled PET material is of suitable purity for its intended use and will meet all existing specifications for the virgin material.
Post-consumer polyethylene terephthalate (PET) containers — Specification — Part 1: Food grade PET recyclates, preforms and containers

1 Scope

1.1 This Working Draft specifies requirements, sampling and test methods for post-consumer polyethylene terephthalate (PET) recyclates (flakes and pellets) for use in PET preforms and PET containers intended for food packaging excluding alcoholic beverages.

1.2 The standard also specifies requirements for the PET preform and container material that characterize it as safe for direct food contact.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ASTM E1347-06, Standard test method for color and color-difference measurement by tristimulus colorimetry

ASTM F1877-16, Standard practice for characterization of particles

ASTM F2013-10, Standard Test Method for Determination of Residual Acetaldehyde in Polyethylene Terephthalate Bottle Polymer Using an Automated Static Head-Space Sampling Device and a Capillary GC with a Flame Ionization Detector


ASTM D6290, Standard test method for colour determination of plastic pellets

ASTM D7486, Standard Test Method for Measurement of Fines and Dust Particles on Plastic Pellets by Wet Analysis

ISO 472, Plastics — Vocabulary

ISO 1133-2, Plastics — Determination of the melt mass-flow rate (MFV) and melt volume flow rate (MVF) of thermoplastics - Part 2: Methods for materials sensitive to time-temperature history and/or moisture
ISO 1628-5, Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 5: Thermoplastic polyester (TP) homopolymers and copolymers

ISO 1628-1, Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 1: General principles

RS ISO 2859-1, Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection

RS ISO 9001, Quality management systems — Requirements

ISO 12418-2, Plastics — Post-consumer poly(ethylene terephthalate) (PET) bottle recyclates — Part 2: Preparation of test specimens and determination of properties

ISO 12418-1, Plastics — Post-consumer poly(ethylene terephthalate) (PET) bottle recyclates — Part 1: Designation system and basis for specifications

ISO 13106, Plastics — Blow-moulded polypropylene containers for packaging of liquid foodstuffs

ISO 14001, Environmental management systems — Requirements with guidance for use

ISO 14782, Plastics — Determination of haze for transparent materials

ISO 15270, Plastics — Guidelines for the recovery and recycling of plastics waste

ISO 15512, Plastics — Determination of water content

ISO 22000, Food safety management systems — Requirements for any organization in the food chain

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 472, ISO 15270 and the following apply:

3.1 challenge test
demonstration of the effectiveness of a recycling process to remove chemical contamination from input

3.2 Flake
plate-like regrind

NOTE The shape of regrind depends both on the plastics being processed and the manner of processing.
3.3
regrind

regrind

shredded and/or granulated recovered plastics material in the form of free-flowing material

3.3
pellet

classification of preformed moulding material, having relatively uniform dimensions, in a given lot, used as feedstock in moulding and extrusion operations

3.4
recycled PET (rPET)

recycled materials resulting from industrial packagings and post-consumer PET materials that have been cleaned, prepared and processed into new recyclates

3.5
bulk density

weight per unit volume of plastic flakes or pellets

3.6
recyclate

plastics material resulting from the recycling of food grade PET waste

3.7
post-consumer

descriptive term covering material, generated by the end-users of products, that has fulfilled its intended purpose or can no longer be used (including material returned from within the distribution chain)

3.8
intrinsic viscosity

limiting value of the reduced viscosity or the inherent viscosity at infinite dilution of the polymer is a common descriptor of PET flow ability
3.9

food

any substance, whether processed, semi-processed or raw, which is intended for human consumption, and
includes drinks, chewing gum and any substance which has been used in the manufacture, preparation or
treatment of "food" but does not include cosmetics or tobacco or substances used only as drugs.

4 Requirements

4.1 General requirements

4.1.1 The resins shall be made from food grade flakes, pellets or chips which may be natural or coloured.

4.1.2 The recycling process shall be done in accordance with ISO 15270. The input to the recycling process
shall originate from a product loop which is in a closed and controlled chain ensuring that only PET materials
are used and any contamination can be ruled out.

NOTE Management systems including ISO 9001, ISO 22000 and ISO 14001 is a suitable guarantee to consistent
recyclate quality. Records of the quality control carried out, including incoming materials, processes and finished products
are maintained in order that the purchaser of the recyclate can have confidence in the quality of the product.

4.1.3 The recyclates, preform and container shall be manufactured in accordance with good manufacturing
practice (GMP) so that, under normal or foreseeable conditions of use, they do not transfer their constituents
to food in quantities which could endanger human health; bring about an unacceptable change in the
composition of the food; bring about deterioration in the organoleptic characteristics (taste, odour and texture)
thereof.

4.1.4 The designation system and sizes for the post-consumer PET recyclates shall be done as described
in ISO 12418-1.

4.1.5 Recyclates may be in various shapes/forms namely flakes or pellets.

4.2 Specific requirements for recycled PET recyclates (pellets or flakes), preforms and
containers

4.2.1 Physical and chemical requirements

4.2.1.1 Recycled PET flakes

Recycled PET flakes shall comply with the physical and chemical requirements of Table 1 when tested in
accordance with the test methods specified therein.

<table>
<thead>
<tr>
<th>S/No.</th>
<th>Parameters</th>
<th>Requirement</th>
<th>Test methods b</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mandatory</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

©RSB 2022 - All rights reserved
### 4.2.1.2 Recycled PET pellets

Recycled PET pellets shall comply with the physical and chemical requirements of Table 2 when tested in accordance with the test methods specified therein.

#### Table 2 — Parameters of rPET pellets

<table>
<thead>
<tr>
<th>S/No.</th>
<th>Parameters</th>
<th>Requirements</th>
<th>Test methods</th>
</tr>
</thead>
<tbody>
<tr>
<td>i.</td>
<td>Fine particle content, % (max)</td>
<td>≤ 0.1</td>
<td>ASTM D7486</td>
</tr>
<tr>
<td>ii.</td>
<td>Intrinsic viscosity (IV), dl/g</td>
<td>0.74 – 0.84</td>
<td>ISO 1628-5 in combination with ISO 1628-1</td>
</tr>
<tr>
<td>iii.</td>
<td>Contamination due to label and other visible contaminants, mg/kg, max</td>
<td>≤ 300</td>
<td>ISO 12418-2</td>
</tr>
<tr>
<td>iv.</td>
<td>Contamination due to Polyvinyl Chloride (PVC), mg/kg</td>
<td>&lt; 5</td>
<td>ISO 12418-2</td>
</tr>
<tr>
<td>v.</td>
<td>Contamination due to Polyolefins (including adhesives), mg/kg, max</td>
<td>50</td>
<td>ISO 12418-2</td>
</tr>
<tr>
<td>vi.</td>
<td>Metal (aluminium, steel), mg/kg</td>
<td>&lt; 5</td>
<td>Spectroscopic analysis: FTIR or XRF</td>
</tr>
<tr>
<td>vii.</td>
<td>Water content, %</td>
<td>≤ 0.5</td>
<td>ISO 15512, Method B</td>
</tr>
<tr>
<td>viii.</td>
<td>Bulk density, kg/m³</td>
<td>As specified by manufacturer</td>
<td>ISO 12418-2,</td>
</tr>
</tbody>
</table>

#### Optional

<table>
<thead>
<tr>
<th>S/No.</th>
<th>Parameters</th>
<th>Requirements</th>
<th>Test methods</th>
</tr>
</thead>
<tbody>
<tr>
<td>ix.</td>
<td>Alkalinity</td>
<td>7 - 8</td>
<td>ISO 12418-2</td>
</tr>
<tr>
<td>x.</td>
<td>Melt volume-flow rate (MVR), cm³/10min</td>
<td>As specified by manufacturer</td>
<td>ISO 1133-2</td>
</tr>
<tr>
<td>xi.</td>
<td>Filterability, 10 MPa/h/cm²</td>
<td>As specified by manufacturer</td>
<td>ASTM D4532-15</td>
</tr>
</tbody>
</table>
4.2.1.3 Recycled PET preforms or containers

Recycled PET preforms and containers shall comply with the physical and chemical requirements of Table 3 when tested in accordance with the test methods specified therein.

| v. | Haze 3mm thick plaques, %, max at 550nm | 13 | ISO 14782 |
| vi. | Acetaldehyde content, ppm (max) | 1.0 | ASTM F2013 10 |
| vii. | Limonene, μg/kg (max) | < 20 | GC/MS |
| viii. | Water content, % | < 0.5 | ISO 15512 |
| ix. | Optional |  |  |
| x. | Melt volume flow rate, cm³/10min | As specified by manufacturer | ISO 1133-2 |
| xi. | Filterability, 10 MPa/h/cm² | As specified by manufacturer | ASTM D4532-15 |
| xii. | Colour | ASTM D6290 |

**Coloured 3mm thick plaques**

| Colour L*, min | 70 |
| Colour a*, min | -3 |
| Colour b*, max | +10 |

**Coloured amorphous pellets (indicative values only, interim processing step in processes)**

| Colour L*, min | 45 |
| Colour a*, min | -10 |
| Colour b*, max | +1 |

**Coloured crystallized pellets**

| Colour L*, min | 67 |
| Colour a*, min | -3 |
| Colour b*, max | +3 |

*Solvent shall be a mixture of phenol and 1,1,2,2-tetrachloroethane (6:4)*

* A certificate of analysis, including the test results for each batch of recyclate, the sampling method and the number of replicates, shall be provided by the supplier to the purchaser on request. The sampling method and the number of replicates are important in determining the quality of the material. Quality levels can vary within the batch and from batch to batch, depending on the source of the recyclate.*
Table 3 — Physical and chemical requirements of recycled PET preforms or containers

<table>
<thead>
<tr>
<th>S/No.</th>
<th>Parameter</th>
<th>Requirements</th>
<th>Test Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>i.</td>
<td>Acetaldehyde, ppm, max</td>
<td>4</td>
<td>ASTM F2013-10</td>
</tr>
<tr>
<td>ii.</td>
<td>Intrinsic Viscosity, dL/g</td>
<td>0.76 - 0.86</td>
<td>ISO 1628-5</td>
</tr>
<tr>
<td>iii.</td>
<td>Colour L* (CIE), min</td>
<td>70</td>
<td>ASTM E1347-06</td>
</tr>
<tr>
<td>iv.</td>
<td>Colour a* (CIE), min</td>
<td>- 4</td>
<td></td>
</tr>
<tr>
<td>v.</td>
<td>Colour b* (CIE), max</td>
<td>1.4</td>
<td></td>
</tr>
<tr>
<td>vi.</td>
<td>Bulk density, kg/m³</td>
<td>820 ± 30</td>
<td>ASTM D4602</td>
</tr>
<tr>
<td>vii.</td>
<td>Water content, %</td>
<td>&lt; 0.2</td>
<td>ISO 15512</td>
</tr>
</tbody>
</table>

4.2.2 Overall migration of constituents

When tested in accordance with Annex A the rPET recyclates and preform material shall not transfer their constituents to food and beverage simulants in quantities exceeding an overall migration limit of 10 milligrams of total constituents (non-volatile substances) released per square decimetre (10 mg/dm²) of food or beverage contact surface, equivalent to 60 mg of total constituents released per kg (60 mg/kg) of food simulants.

4.2.3 Specific migration of chemicals

4.2.3.1 Specific migration of compound

The specific migration limit of compound from the recyclates or preform material shall not exceed the limit specified in Table 4 when determined in accordance with the test method specified therein.

Table 4 — Specific migration limits for compounds used in the manufacture of recyclates and preform

<table>
<thead>
<tr>
<th>S/No.</th>
<th>Compound</th>
<th>Specific Migration Limit, mg/kg of food, max.</th>
<th>Test method</th>
</tr>
</thead>
<tbody>
<tr>
<td>i.</td>
<td>Mono- and diethylene glycol (including the ester of stearic acid with ethylene glycol)</td>
<td>30</td>
<td>ASTM D4754-98</td>
</tr>
<tr>
<td>ii.</td>
<td>Terephthalic acid</td>
<td>7.5</td>
<td></td>
</tr>
<tr>
<td>iii.</td>
<td>Isophthalic acid</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>iv.</td>
<td>Antimony trioxide(calculated as antimony)</td>
<td>0.04</td>
<td></td>
</tr>
<tr>
<td>v.</td>
<td>2-Aminobenzamide (anthranilamide)</td>
<td>0.05</td>
<td></td>
</tr>
</tbody>
</table>

4.2.3.2 Restrictions on release of inorganic substances

The preform and container material shall not release contaminants in quantities exceeding their migration limits given in Table 5 when determined in accordance with spectroscopic analysis such as FTIR or XRF or any other equivalent method.
Table 5 — Specific migration limit of inorganic substances

<table>
<thead>
<tr>
<th>S/No.</th>
<th>Substance</th>
<th>Specific migration limit mg/kg of food, beverage or food stimulant, max.</th>
</tr>
</thead>
<tbody>
<tr>
<td>i.</td>
<td>Aluminium</td>
<td>1</td>
</tr>
<tr>
<td>ii.</td>
<td>Barium</td>
<td>1</td>
</tr>
<tr>
<td>iii.</td>
<td>Cobalt</td>
<td>0.05</td>
</tr>
<tr>
<td>iv.</td>
<td>Copper</td>
<td>5</td>
</tr>
<tr>
<td>v.</td>
<td>Iron</td>
<td>48</td>
</tr>
<tr>
<td>vi.</td>
<td>Lithium</td>
<td>0.6</td>
</tr>
<tr>
<td>vii.</td>
<td>Manganese</td>
<td>0.6</td>
</tr>
<tr>
<td>viii.</td>
<td>Zinc</td>
<td>5</td>
</tr>
</tbody>
</table>

4.2.4 The dimensional and mechanical properties of the PET containers

The dimensional and mechanical properties of the PET containers shall be as specified in ISO 13106.

5 Packaging

The recyclates, preforms and containers shall be packaged in suitable packaging materials that maintain their integrity prevent the ingress of dust, moisture and other foreign matters during transportation and storage.

6 Marking

The following information shall be legibly and indelibly marked on the packaging, container or in accompanying documents:

a) name, trade mark or other means of identification of the manufacturer;

b) batch number

c) date of manufacture;

d) words “for food contact” or symbol for food contact material
e) symbol indicating that it is made from recycled PET material;

f) if necessary, special instructions to be observed for safe and appropriate use; and

g) recycled content, %

7 Sampling

The sampling criteria shall be in accordance with ISO 2859-1
Annex A
(normative)

Determination of overall migration of constituents of plastics materials and articles intended to come in contact with foodstuffs - Methods of analysis

A.1 Types of simulants

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

a) Simulant ‘A’ - distilled water or water of equivalent quality.

b) Simulant ‘B’ - 3 percent acetic acid (w/v) in aqueous solution (using the simulant ‘A’).

c) Simulant ‘C1’ - 10 percent ‘ethanol (v/v) in aqueous solution for foodstuffs aqueous solution for foodstuffs having alcohol less than 10 percent (v/v) (using the simulant ‘A’).

d) Simulant ‘C2’ - 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’).

e) Simulant ‘D’ - n-heptane - shall be freshly distilled before use.

f) 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.

A.2 Selection of standard test conditions and simulants for different foodstuffs

A.2.1 The choice of simulating solvents and test conditions (time-temperature) depends on the type of foodstuff and its condition of use. Food products have been classified into seven major groups suitable simulants to be used for different types of foods as per Table A.1.

<table>
<thead>
<tr>
<th>S/N</th>
<th>Type of food</th>
<th>Description</th>
<th>Examples</th>
<th>Simulant</th>
</tr>
</thead>
<tbody>
<tr>
<td>i.</td>
<td>I</td>
<td>Aqueous, non-acidic foods without fat (pH &gt; 5)</td>
<td>Honey, mineral water, sugar syrups molasses, skimmed milk, rusgulla, infusions, murabba, yeast, paste etc yeast paste etc</td>
<td>‘A’</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Aqueous, acidic foods without fat (pH ≤ 5)</td>
<td>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</td>
<td>‘B’</td>
</tr>
</tbody>
</table>

Table A.1 — Classification of foods and selection of simulant
### Alcoholic beverages:

i. Alcohol concentration less than 10 percent
   - Beer and some pharmaceuticals syrups
   - Wine, brandy, whiskey, arrack and other alcoholic drinks

ii. Alcohol concentration above 10 percent
   - Vegetable oils, ghee, cocoa butter, lard, biscuits, spice powder, snacks and savoury, chocolate, caramels, malted foods, egg powder, tea, coffee powder confectionery, fried and roasted nuts etc

### Oils, fats and processed dry foods with surface fat or volatile oil

- Vegetable oils, ghee, cocoa butter, lard, biscuits, spice powder, snacks and savoury, chocolate, caramels, malted foods, egg powder, tea, coffee powder confectionery, fried and roasted nuts etc

### 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

### 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, mayonnaise etc

### 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

---

### Table A.2 — Simulating solvents for different types of food, temperature and time conditions

<table>
<thead>
<tr>
<th>S/N</th>
<th>Conditions of use</th>
<th>Type of food</th>
<th>Water (time-temperature)</th>
<th>3 % Acetic acid (time-temperature)</th>
<th>10 % alcohol (time-temperature)</th>
<th>50 % alcohol (time-temperature)</th>
<th>n-Heptane</th>
</tr>
</thead>
<tbody>
<tr>
<td>i.</td>
<td>High temperature heat sterilized (Retorting)</td>
<td>I, II, IV, V and VI</td>
<td>121 °C, 2 h</td>
<td>121 °C, 2 h</td>
<td>-</td>
<td>-</td>
<td>66 °C for 2 h</td>
</tr>
<tr>
<td>ii.</td>
<td>Hot filled or pasteurized above 66 °C, 100 °C</td>
<td>I, II, IV, V and VI</td>
<td>100 °C, 2 h</td>
<td>100 °C, 2 h</td>
<td>-</td>
<td>-</td>
<td>49 °C for 30 min</td>
</tr>
<tr>
<td>iii.</td>
<td>Hot filled or pasteurized below 66 °C</td>
<td>I to VI</td>
<td>70 °C, 2 h</td>
<td>70 °C, 2 h</td>
<td>70 °C, 2 h</td>
<td>70 °C, 2 h</td>
<td>38 °C for 30 min</td>
</tr>
<tr>
<td>iv.</td>
<td>Room temperature filled and stored (no thermal treatment in container) and also in refrigerated and frozen condition</td>
<td>I to VI</td>
<td>40 °C, 10 days</td>
<td>40 °C, 10 days</td>
<td>40 °C, 10 days</td>
<td>40 °C, 10 days</td>
<td>38 °C for 30 min</td>
</tr>
</tbody>
</table>
NOTE 1  Heptane simulant not to be used on wax lined containers.

NOTE 2  Heptane extractivity results must be divided by a factor of five in arriving at the extractivity of a food product

A.3 Method I: For finished container (within two litres capacity) or sealable single/multi-layered flexible films (one side exposure)

A.3.1 Apparatus

A.3.1.1 Electric oven/water bath, equipped with thermostat to maintain the desired temperature within ± 1 ˚C accuracy

A.3.1.2 Electric hot plate, with temperature control regulator

A.3.1.3 Analytical balance, with a sensitivity of 0.1 mg

A.3.1.4 Glass beakers, Pyrex of 1 000 ml capacity or equivalent

A.3.1.5 Stainless steel evaporating dish of 100 ml capacity

A.3.1.6 Stainless steel tongs

A.3.2 Selection of Sample

Minimum triplicate samples representing the lot/batch have to be selected. The films representative sample shall be of sufficient size to convert into two pouches of size 125 mm width and 200 mm length (inner dimension excluding seal area) with 1 000 cm² surface area coming in contact.

A.3.3 Preparation of the test specimen

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

A.3.4 Simulant Quantity

Equal to nominal filling capacity or at least 1 mL/cm² 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.

A.3.5 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/water bath/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.

**A.3.6 Determination of Amount of Extractive**

**A.3.6.1** Evaporate/distil the contents in Pyrex beaker to about 50-60mL 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 °C ± 5 °C. Cool the dish with extractive in a desiccator for 30 minutes and weigh to nearest 0.1mg till constant weight of residue is obtained. Calculate the extractives in mg/dm² 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.

**A.3.6.2** Calculate the amount of extractive in ppm for the particular size of container being tested.

Amount of extractive (Ex) = \( \frac{M}{V} \times 100 \) mg/kg or mg/l

or

Ex = \( \frac{M}{A} \times 100 \) mg/dm²

where

M = mass of residue in mg minus blank value;

A = surface area in cm² exposed in each replicate;

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

**NOTE 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.

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

**A.4 Method II: For larger containers made of single homogenous material above 2 litre capacity**

**A.4.1 Selection of Sample**

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

**A.4.2 Test Specimen**

Cut 5 pieces each of size 10 cm x 10 cm from each container at different places (each piece exposing about 200 cm² surface area both sides). In the case of thick material area corresponding to thickness of the sample shall also be included.
A.4.3 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 3mm 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/water bath/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.

A.4.4 Determination of amount of extractive

Follow the procedure in A.3.6.1 and calculate the amount of extractive in mg/dm$^2$ and mg/kg or mg/l with respect to capacity of the container.

Amount of extractive (Ex) = $M \times \frac{1000}{A \times V}$ mg/kg or mg/l

or

$Ex = \frac{M \times A}{V} \times 100$ mg/dm$^2$

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$^2$;

$V$ = total volume of the container in ml or cm$^3$ in

NOTE Heptane extractive to be divided by factor of five
Bibliography


