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Draft Sri Lanka Standard
SPECIFICATION FOR REUSABLE PLASTIC FOOD CONTAINERS
(LUNCH AND SNACK BOXES)
(DSLS :)

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இவ்வரைவு இலங்கைக் கட்டளையெனக் கருதப்படவோ அன்றிப் பிரயோகிக்கப்படவோ கூடாது
This draft should not be regarded or used as a Sri Lanka Standard.

අදහස් ඊටියයුත්තේ : ශ්‍රී ලංකා ප්‍රමිති ආයතනය, 17, වික්ටෝරියා පෙදෙස, ඇල්විටිගල මාවත, කොළඹ 08.

Comments to be sent to: SRI LANKA STANDARDS INSTITUTION, 17, VICTORIA PLACE,
ELVITIGALA MAWATHA, COLOMBO 08.

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මෙම ශ්‍රී ලංකා ප්‍රමිති කෙටුම්පත , ශ්‍රී ලංකා ප්‍රමිති ආයතනය විසින් සකසන ලදුව, සියලුම උදෙසාගේ අංශ වලට තාක්ෂණික විවේචනය සඳහා යටත් ලැබේ.

අදාළ අංශ භාර කමිටු මාර්ගයෙන් ආයතනයේ මහා මණ්ඩල වෙත ඉදිරිපත් කිරීමට පෙර , ලැබෙන සියලුම විවේචන ශ්‍රී ලංකා ප්‍රමිති ආයතනය විසින් සලකා බලා අවශ්‍ය වෙනස් කෙටුම්පත සංශෝධනය කරනු ලැබේ.

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Introduction

This Draft Sri Lanka Standard has been prepared by the Sri Lanka Standards Institution and is now being circulated for technical comments to all interested parties.

All comments received will be considered by the SLSI and the draft if necessary, before submission to the Council of the Institution through the relevant Divisional Committee for final approval.

The Institution would appreciate any views on this draft which should be sent before the specified date. It would also be helpful if those who find the draft generally acceptable could kindly notify us accordingly.

All Communications should be addressed to:

The Director General
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17, Victoria Place,
Elvitigala Mawatha,
Colombo 08.

Draft Sri Lanka Standard
SPECIFICATION FOR REUSABLE PLASTIC FOOD CONTAINERS (LUNCH AND
SNACK BOXES)

SLS :

Gr.

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Draft Sri Lanka Standard
SPECIFICATION FOR REUSABLE PLASTIC FOOD CONTAINERS (LUNCH AND SNACK BOXES)

FOREWORD

This Standard was approved by the Sectoral Committee on Chemical and Polymer Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on

Some ingredients used to manufacture plastic containers may cause toxicity as a result of their migration to the foodstuffs in which the latter are packed. Thus, if ingested in large quantities, chemicals might endanger human health or change the food itself. Therefore, protection of food materials throughout the supply chain including the containment (packaging) is an integral part of food safety.

This Standard is subject to the restrictions imposed under relevant Acts and regulations framed thereunder.

For the purpose of deciding whether a particular requirement of this Standard is complied with the final value observed or calculated expressing the result of a test or an analysis shall be rounded off in accordance with **SLS 102**. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this Standard. All standard values in this Standard are given in SI units.

In the preparation of this Standard, the assistance derived from the following publications is gratefully acknowledged:

- EN 71-3: 2019 Safety of toys - Part 3: Migration of certain elements
- EN 14350: 2020 Child care articles - Drinking equipment- Safety requirements and test methods
- EN 14372: 2004 Child use and care articles - Cutlery and feeding utensils- Safety requirements and tests
- (EU) 2018/ 213 Commission Regulation - Use of bisphenol A in varnishes and coatings intended to come into contact with food
- (EU) 2011/ 10 Commission Regulation- Plastic materials and articles intended to come into contact with food
- IS 2798: 1998 Methods of test for plastics containers

1 SCOPE

- 1.1 This Standard prescribes the requirements and methods of sampling and test for reusable plastic food containers (lunch and snack boxes)
- 1.2 This Standard does not cover the single use and biodegradable plastic food containers (lunch and snack boxes).
- 1.3 This Standard does not cover plastic containers used for storage and transportation of food.

2 REFERENCES

ASTM D 1193	Standard Specification for reagent water
21 CFR 177.1520	Olefin polymers
SLS 102	Rules for rounding off numerical values
SLS 428	Random sampling methods
SLS 616	Glossary of terms for plastics
SLS 1263	Code of practice for recycling of plastics
SLS 1392	Specification for Good Manufacturing Practices (GMP) for plastic industry
SLS 1560	Generic identification and marking of plastics products
SLS 1615	Determination of overall migration of constituents of plastics materials and articles intended to come in contact with foodstuffs

3 DEFINITIONS

For the purpose of this Standard the following definition shall apply:

- 3.1 **reusable:** Plastic food containers (lunch and snack boxes) intended for repeated use.

4 TYPES

The plastic food containers (lunch and snack boxes) shall be of the following grades;

- i) Lunch boxes; and
- ii) Snack boxes.

5 REQUIREMENTS

5.1 Material requirements

5.1.1 *Polymer*

Reusable plastic food containers and their accessories (lunch and snack boxes) shall be made out from virgin and food grade polymeric materials. The polymer used in the manufacture of reusable plastic food containers (lunch and snack boxes) shall be of olefin based polymers as given in 21 CFR 177.1520. In addition to the above, the materials reviewed and determined by the FDA, to be Generally Recognized As Safe (GRAS) and materials comply with the inventories of Food Contact Substances (FCS) approved by any recognized International Organizations shall be used. The material used shall be of no health hazards to users.

5.1.2 *Compatibility tests*

Supplier of polymeric materials (Resin) and the converter shall provide documentary evidence of compliance with the International Standards. (eg: In case of FDA compliance, resin manufacturer shall provide a letter of guaranty to the effect that polymeric materials used for reusable plastic food containers are of food grade quality and suitable to be used in contact with the intended types of foods).

5.1.3 *Pigments and colorants*

Pigments and colorants used for making reusable plastic food containers (lunch and snack boxes) and their accessories shall be of food grade materials.

5.2 Physical requirements

5.2.1 Decorative prints shall not be removed from any product when tested in accordance with Appendix B. Adhesive labels shall not be used. The printed surfaces shall not come into direct contact with food.

5.2.2 *Manufacture, workmanship, finish and appearance*

5.2.2.1 The reusable plastic food containers (lunch and snack boxes) shall be manufactured by suitable processes adhering to Good Manufacturing Practices (GMP) conforming to **SLS 1392**. All accessories including materials, lubricants used in machineries that may cause possible contamination during manufacturing process shall be of food grade quality.

5.2.2.2 The body of the reusable plastic food containers (lunch and snack boxes) shall be smooth, both internally and externally, shall be free from any visual defects like cavities, crevices, hooks, embedded foreign matters, detrimental bubbles, streaks flaws and stains.

5.2.2.3 All components of reusable plastic food containers (lunch and snack boxes) when assembled for use shall be free from points and edges and any harmful extrusions, which are likely to cause injury.

5.2.3 *Design, shape and dimensions*

The reusable plastic food containers (lunch and snack boxes) shall be of suitable design, shape and required dimensions as agreed to between the purchaser and the supplier. However, the shape of the reusable plastic food containers (lunch and snack boxes) shall be such that it is properly cleanable. The reusable plastic food containers (lunch and snack boxes) shall be provided with lids made of polymeric materials suitable for food contact applications.

5.2.4 *Capacity*

The capacity of the reusable plastic food containers (lunch and snack boxes) shall be as agreed to between the purchaser and the supplier.

5.2.5 *Leakage test*

Lunch box type of reusable plastic containers shall not show any leakages when tested in accordance with Appendix C.

5.2.6 *Drop test*

The reusable plastic food containers (lunch and snack boxes) shall not show any sign of rupture or breakages when tested in accordance with Appendix D. The dropping height of the container shall be of 0.8 m.

5.3 **Chemical requirements**

5.3.1 *Migration test*

5.3.1.1 *Overall migration*

5.3.1.1.1 Representative samples of reusable plastic food containers (lunch and snack boxes) shall be subjected to overall migration test with aqueous and fatty food at (70 ± 2) °C using the simulants A and D for 24 hrs. The maximum extraction values for the reusable plastic food containers shall not exceed 10 mg/dm² or 60 mg/kg when tested in accordance with the method described in **SLS 1615**.

NOTE

The overall migration limits specified above, shall be updated with the changes in recognized International Regulations.

5.3.1.2 Migration of certain elements

Reusable plastic food containers (lunch and snack boxes) and its components made of plastics, when tested in accordance with the method described in Appendix E shall not exceed the limits for elements as given in Column 3 of Table 1. When components of reusable plastic food containers (lunch and snack boxes) are manufactured of different materials or in different colours, all components shall be separately tested and reported.

Table 1 – Limits for migration of certain elements

Sl. No. (1)	Heavy metals (2)	Maximum limits mg/kg (3)
i)	Arsenic	0.05
ii)	Chromium	0.5
iii)	Mercury	0.001
iv)	Cadmium	0.05
v)	Lead	0.5
vi)	Antimony	15

5.3.2 Phthalate content

The total content of following phthalate plasticisers of a container shall not exceed 0.1 per cent (w/w), when tested in accordance with the method described in Appendix F.

- i) Di-isononyl phthalate (DINP);
- ii) Di-(2-ethylhexyl) phthalate (DEHP);
- iii) Di-n-octyl phthalate (DNOP);
- iv) Di-iso-decyl phthalate (DIDP);
- v) Butyl benzyl phthalate (BBP); and
- vi) Di-butyl phthalate (DBP).

5.3.3 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A) release

Reusable plastic food containers (lunch and snack boxes) and their accessories made of plastics, when tested in accordance with the method described in Appendix G, the migration of the 2,2-bis(4-hydroxyphenyl)propane shall not exceed 0.05 mg/kg into aqueous food simulant.

5.3.4 4,4'-Sulfonyldiphenol (Bisphenol S) release

Reusable plastic food containers (lunch and snack boxes) and their accessories made of plastics, when tested in accordance with the method described in Appendix G, the migration of the 4,4'-Sulfonyldiphenol shall not exceed 0.05 mg/kg into aqueous food simulant.

6 PACKAGING AND MARKING/ LABELLING

6.1 Packaging

Reusable plastic food containers (lunch and snack boxes) shall be packed under hygienic conditions in suitable packages so as to protect the reusable plastic food containers (lunch and snack boxes) from dust and other contaminants. It shall be packed as agreed to between the purchaser and the supplier.

6.2 Marking/ Labeling

6.2.1 The following shall be legibly and indelibly marked on each reusable plastic food container (lunch and snack boxes):

- a) Manufacturer's/distributor's name or registered trade mark, if any;
- b) Plastic identification symbol in accordance with **SLS 1263**; and
- c) Food grade identification symbol as indicated below.



6.2.2 Each reusable plastic food container (lunch and snack boxes) or label and each package shall be marked with the following:

- a) Batch or code number;
- b) Type of the reusable plastic food container as 'lunch box' or 'snack box';
- c) Name and address of the manufacturer including country of origin
(**NOTE: Name and address of the manufacturer and the distributor should be marked on imported products**); and
- d) Month and year of manufacture.

6.2.3 Instructions for use and hygienic care of the product shall be provided with each reusable plastic food container as follows. It may be included in a separate leaflet:

- a) Information on unsuitable common methods of heating which might damage the product;
- b) Safe temperature range of usage;
- c) Statement to include 'Not suitable to be used in microwave ovens';
- d) Cleaning and drying instructions to prevent surface damages and possible growth of fungus;

- e) Warning statement as “Before each use, inspect the product and stop using at the first sign of stains, damage or weakness which leads to possible release of micro plastics”; and
- f) Any other warning statements related to cleaning, storage and usage.

NOTE Attention is drawn to certification marking facilities offered by the Sri Lanka Standards Institution. See the inside back cover of the Standard.

7 METHODS OF TEST

7.1 Tests shall be carried out as prescribed in Appendices **B** to **G** of this Standard.

7.2 Unless otherwise stated, only reagents of analytical grade and only distilled water shall be used during the analysis.

8 SAMPLING

Representative samples of the product for ascertaining conformity to the requirements of this Standard shall be drawn as prescribed in Appendix **A**.

APPENDIX A COMPLIANCE OF A LOT

The sampling scheme given in Appendix **A** should be applied where compliance of a lot to the requirements of this Standard is to be assessed based on statistical sampling and inspection.

Where compliance with this Standard is to be assured, appropriate schemes of sampling and inspection shall be adopted based on manufacturer’s control systems coupled with type tests and testing procedures.

A.1 LOT

A.1.1 In any consignment, all the containers of same material, type and size belonging to one batch of a manufacture or supply shall constitute a lot.

A.2 SCALE OF SAMPLING

A.2.1 The samples shall be inspected and tested from each lot for ascertaining conformity of the lot to the requirements of this Standard.

A.2.2 The number of containers to be selected as the sample from a lot shall be in accordance with Column (1) and Column (2) of Table 2.

A.2.3 Three (03) containers shall be drawn separately from the lot for testing raw materials to identify polymer material.

A.2.4 The number of containers to be selected as a sub sample from the primary sample selected as in **A.2.2** shall be in accordance with Column (1) and Column (3) of Table 2. Two sub samples as in Column (3) shall be drawn for testing of the physical requirements given in Clauses 5.2.1, 5.2.2, 5.2.3, 5.2.4 and 5.2.5.

TABLE 2 – Scale of sampling

No. of containers in the lot (1)	No. of containers to be selected for the primary sample (2)	No. of containers to be selected for sub sample (3)	Acceptance No. for sub sample (4)
Up to 500	13	5	0
501 to 1 200	20	5	0
1 201 to 3 200	32	8	1
3 201 and above	50	8	1

A.2.5 Ten (10) containers shall be drawn separately from the lot for inspecting drop test requirements.

A.2.6 Nine (09) containers shall be drawn separately from the lot for overall migration test.

A.2.7 Three (03) containers shall be drawn separately from the lot for migration of certain elements test.

A.2.8 Three (03) containers shall be drawn separately from the lot for phthalate content.

A.2.9 Three (03) containers shall be drawn separately from the lot for 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A) release and 4,4'-Sulfonyldiphenol (Bisphenol S) release.

A.2.10 All containers shall be selected at random. In order to ensure randomness of selection, random number tables as given in **SLS 428** shall be used.

A.3 NUMBER OF TESTS

A.3.1 Each container selected as in **A.2.2** shall be inspected for packaging and marking requirements specified in Clause 6.

A.3.2 Each container selected as in **A.2.3** shall be verified or inspected for the requirements specified in Clause 5.1.1.

A.3.3 Each container of first sub sample selected as in **A.2.4** shall be tested for the requirements specified in Clause 5.2.1.

A.3.4 Each container of second sub sample selected as in **A.2.4** shall be tested for the requirements specified in Clauses **5.2.2**, **5.2.3**, **5.2.4**, and **5.2.5**.

A.3.5 Each container selected as in **A.2.5** shall be inspected for the requirements specified in Clause **5.2.6**.

A.3.6 Each container selected as in **A.2.6** and tested for the requirements specified in Clause **5.3.1.1**.

A.3.7 One composite sample shall be drawn from the containers selected as in **A.2.7** and tested for the requirements specified in Clause **5.3.1.2**.

A.3.8 One composite sample shall be drawn from the containers selected as in **A.2.8** and tested for the requirements specified in Clause **5.3.2**.

A.3.9 Each container selected as in **A.2.9** shall be tested for the requirements specified in Clause **5.3.3** and **5.3.4**.

A.4 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this Standard if the following conditions are satisfied.

A.4.1 Each container inspected as in **A.3.1** shall satisfies the relevant requirements.

A.4.2 Each container verified or inspected as in **A.3.2** shall satisfies the relevant requirements.

A.4.3 Each container inspected as in **A.3.3** shall satisfies the relevant requirements.

A.4.4 The number of containers, not conforming to any one or more requirements as in **A.3.4** is less than or equal to the corresponding acceptance number given in Column (4) of Table 2.

A.4.5 Each container inspected as in **A.3.4** shall satisfies the relevant requirements.

A.4.6 Each container inspected as in **A.3.5** shall satisfies the relevant requirements.

A.4.7 Each container inspected as in **A.3.6** shall satisfies the relevant requirements.

A.4.8 The test results of the sample tested as in **A.3.7** shall satisfies the relevant requirements.

A.4.9 The test results of the sample tested as in **A.3.8** shall satisfies the relevant requirements.

A.4.10 Each container inspected as in **A.3.9** shall satisfies the relevant requirements.

APPENDIX B
TEST FOR PRODUCT RESISTANCE OF PRINTED REUSABLE PLASTIC FOOD
CONTAINERS

B.1 PROCEDURE

Keep the containers, or representative sections cut-out from the printed area of the containers in an oven at (40 ± 2) °C and leave it for 1 h. Wash the containers or its representative sections with cold water. Rub each container or representative section firmly with hard paper tissue ten times. Examine each container or representative section for removal of the print from the surface of the container. Print shall be legible to the naked eye after the test.

APPENDIX C
LEAKAGE TEST

C.1 CLOSURE LEAKAGE

C.1.1 Procedure

The reusable plastic food containers (lunch boxes) filled to brimful level with coloured water, coloured with water dissolving dye/ pigment or colourant at room temperature and close tight with the closures. Keep the container in 90° turned upward position on a white blotting paper for 30 minutes. Examine the container for any leakage which would be evident for visible stains on the blotting paper. The reusable plastic food containers (lunch boxes) shall be then held in a upside down position on a white blotting paper for 30 minutes. Examine the container for any leakage which would be evident for visible stains on the blotting paper.

APPENDIX D
DROP TEST

D.1 SAMPLE SIZE

The sample size shall be ten containers, taken at random from a batch, divided into two sets of 5 each, designated as set 1 and set 2.

D.2 APPARATUS

Any suitable apparatus can be used if it conforms to the following requirements:

- a) Permits accurate repositioning of the container to ensure an unobstructed fall from rest position and impact at the specified places or areas of the container;
- b) Permits accurate and convenient control of the height of drop; and

- c) Provides a solid impact surface to absorb drop impact shock without deflection.

D.3 PROCEDURE

D.3.1 Fill each container with water about 3/4 of its total capacity at (27 ± 2) °C and close tightly.

D.3.2 Drop the containers from predetermined height (0.8 m) on to a rigid flat horizontal surface under free fall conditions of set 1 squarely on their base (bottom) on to a rigid flat horizontal surface of steel or smooth concrete as the dropping surface.

D.3.3 Drop the containers under free fall condition in set 2 on their side onto the dropping surface.

D.3.4 Examine each container for signs of rupture or breakages.

APPENDIX E DETERMINATION OF THE MIGRATION OF CERTAIN ELEMENTS

E.1 PRINCIPLE

Soluble elements are extracted from the individual components of the reusable plastic food containers (lunch and snack boxes) using conditions which simulate the material remaining in contact with gastric juices for a period of time after swallowing. The concentrations of the soluble elements are determined quantitatively.

E.2 APPARATUS

E.2.1 *Equipment for measuring pH*, with an accuracy of ± 0.02

E.2.2 *Centrifuge*, capable of centrifuging at (5000 ± 500) rpm

E.2.3 *Orbital or linear shaker or shaking water bath*, capable of maintaining the migration solution in constant motion relative to the sample and agitate the mixture at a temperature of (37 ± 2) °C

E.2.4 *Plastic containers of gross volume*, between 1.6 times and 5.0 times the volume of Hydrochloric acid extract

E.2.5 *Membrane filters* with pore size of 0.45 μm

E.2.6 *Pipettes*, automatic pipettes capable of accurate delivery the reagents

E.2.7 *Analytical balance*, with an accuracy of ± 0.0001 g

E.3 REAGENTS

All reagents which have been determined to have acceptable low levels of impurity and certified to be low levels in the metals of interest to allow the analysis to be performed.

E.3.1 *Hydrochloric acid solution, (0.07 ± 0.005) mol/ L*

E.3.2 *Hydrochloric acid solution, approximately 2 mol/ L*

E.3.3 *Hydrochloric acid solution, approximately 6 mol/ L*

E.3.4 *Water conforming to the requirements of Type 1 of ASTM D1193*

E.4 SELECTION OF TEST PORTIONS

Test portions shall be taken from each individual component of the container in contact with the food. Reusable plastic food containers (lunch and snack boxes) containing components manufactured from different material that are joined together shall be tested as separate items and reported separately.

E.5 PREPARATION OF TEST PORTIONS

Whenever possible obtain a test portion of not less than 0.100 g, to the nearest 0.001 g. Prepare a test portion by cutting the laboratory sample using suitable tools. Take care to produce clean cut edges. Components shall be cut, as far as possible, into pieces of length 4 mm to 6 mm and width not exceeding 6 mm.

E.6 PROCEDURE

E.6.1 General

The volume of simulant added shall be recorded for the purpose of calculation of results. Appropriate blank solution shall be analysed so that appropriate corrections can be made when necessary (eg. contamination of reagents and materials). If the blank result exceeds half of the lowest value the laboratory intends to report (laboratory's reporting limit) at least two blank solutions shall be analysed and the analytical result shall be corrected by the average value of the blank solutions.

E.6.2 Preparation of test portions before migration testing

Weigh the test portion to the nearest 0.001 g (W_{tp}) into the container (**E.2.4**). Mix the prepared test portion at $(22 \pm 3) ^\circ\text{C}$ with 50 times of an aqueous solution of Hydrochloric acid (**E.3.1**).

Weigh the Hydrochloric acid to the nearest 0.05g. Add 5 mL of Hydrochloric acid (E.3.1) at $(22 \pm 3) ^\circ\text{C}$, if mass of the test portion is between 0.010 g and 0.100 g.

E.6.3 pH adjustment

E.6.3.1 pH adjustment – absence of buffering effect by material used for food containers

Test portion is not expected to contain a significant quantity of alkaline/ buffering materials. After adding diluted Hydrochloric acid as specified in the relevant Clause of E.6.2, shake the mixture gently for approximately 1 min to mix the contents. Measure the pH of the mixture at $(22 \pm 3) ^\circ\text{C}$ without any delay. If the pH of the mixture is greater than 1.30 add Hydrochloric acid (E.3.2) dropwise whilst mixing until the pH of the mixture is within the range of 1.10 to 1.30. Follow the migration procedure (E.6.4)

E.6.3.2 pH adjustment- presence of buffering effect by material used for food containers

Test portion is expected to contain a significant quantity of alkaline/ buffering materials. After adding diluted Hydrochloric acid as specified in the relevant Clause of E.6.2, shake the mixture gently to mix the contents. Allow the mixture to stand at $(22 \pm 3) ^\circ\text{C}$ for (5_0^{+2}) minutes. Measure the pH of the mixture at $(22 \pm 3) ^\circ\text{C}$ (pH_a). If pH_a is within the range of 1.10-1.20 follow migration procedure (E.6.4). If pH_a is greater than 1.20, add Hydrochloric acid (E.3.3) dropwise whilst mixing until the pH of the mixture is within the range 1.10-1.20. Allow the mixture to stand at $(22 \pm 3) ^\circ\text{C}$ for (5_0^{+2}) minutes. Measure the pH of the mixture at $(22 \pm 3) ^\circ\text{C}$ (pH_b). If pH_b is within the range of 1.10-1.20 proceed with the migration procedure (E.6.4). If pH_b is greater than 1.20, add 6 mol/ L HCl (E.3.3) dropwise whilst mixing until the pH is within the range 1.10-1.20. Allow the mixture to stand at $(22 \pm 3) ^\circ\text{C}$ for (10_0^{+2}) minutes. Measure the pH of the mixture at $(22 \pm 3) ^\circ\text{C}$ (pH_c). If pH_c is within the range 1.10-1.20 proceed with the migration procedure (E.6.4). If pH_c is greater than 1.20, add Hydrochloric acid (E.3.3) dropwise whilst mixing until the pH is within the range of 1.10-1.20. Allow the mixture to stand at $(22 \pm 3) ^\circ\text{C}$ for (10_0^{+2}) minutes. Measure the pH of the mixture at $(22 \pm 3) ^\circ\text{C}$ (pH_d). If pH_d is within the range of 1.10-1.20 follow the migration procedure (E.6.4). If pH_d is greater than 1.20, add Hydrochloric acid (E.3.3) dropwise whilst mixing until the pH is within the range 1.10-1.20. Then follow the migration procedure (E.6.4).

E.6.4 Migration procedure

Close the container (E.2.4) and agitate the mixture at $(37 \pm 2) ^\circ\text{C}$ for (60_0^{+5}) minutes with a speed of (150 ± 10) per minute. Stop the agitation and leave the containers to stand for a further (60_0^{+5}) minutes at $(37 \pm 2) ^\circ\text{C}$.

E.6.5 pH control after migration

pH shall be checked after completion of migration in order to ensure that the correct pH has been maintained throughout the migration procedure. If it is possible to demonstrate that there is no variation of the pH for certain materials (eg. plastic) it is not necessary to control the pH for those materials after migration. Migration solutions with a pH less than 1.10 or greater than 1.30

which have been adjusted according to **E.6.3.1** (pH adjustment without buffering effect by material used for plastic food containers) shall be discarded and a new test portion shall be analysed using the migration procedure for material with high buffering capacity (see **E.6.3.2**, pH adjustment with buffering effect by material used for reusable plastic food containers). It is sufficient to carry out the pH control check for one solution from each material type within a batch. If the pH measured is less than 1.10 or more than 1.30 the remaining solutions in the batch for test portions of that type shall also be checked.

E.6.6 Filtration

Without delay separate the solid matter from the solution by filtration through a membrane filter (**E.2.5**). If necessary, centrifuge to remove particles before filtration.

E.6.7 Stabilization and analysis of migration solutions

E.6.7.1 If the migration solutions are to be retained for more than 24 h prior to analysis, they shall be stabilized by the addition of a suitable amount of hydrochloric acid such that the Hydrochloric acid concentration of the stored solution is approximately 1 mol/ L.

E.6.7.2 Inductively Coupled Plasma Mass Spectrometry (ICP-MS) or Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method shall be used to determine the quantity of migrated elements.

APPENDIX F DETERMINATION OF PHTHALATE CONTENT

F.1 PRINCIPLE

To extract, identify and quantify monomeric phthalates (with wider application to other types of plasticizers) contained in reusable plastic food containers (lunch and snack boxes) uses soxhlet extraction apparatus with Diethyl ether. The total Diethyl ether extractable plasticizer content is calculated by mass, with Gas Chromatography-Mass Spectroscopy (GC-MS) detection to identify and quantify individual phthalates.

F.2 APPARATUS

F.2.1 Balance, accurate up to 0.000 1 g

F.2.2 Oven

F.2.3 Soxhlet extractor with siphon cup

- F.2.4 Soxhlet thimble**
- F.2.5 Condenser**
- F.2.6 Steam bath**
- F.2.7 Desiccator**
- F.2.8 Flat bottom flask, of 150-ml capacity**
- F.2.9 Volumetric flask, of 200-ml capacity**
- F.2.10 Gas Chromatography-Mass Spectrometer**

F.3 REAGENTS

- F.3.1 Diethyl ether**
- F.3.2 n-hexane**
- F.3.3 Di-isononyl phthalate (D1NP), CAS No. 28553-12-0**
- F.3.4 Di-(2-ethylhexyl) phthalate (DEHP), CAS No. 117-81-7**
- F.3.5 Di-n-octyl phthalate (DNOP), CAS No, 117-84-0**
- F.3.6 Di-iso-decyl phthalate (DIDP), CAS No. 26761-40-0**
- F.3.7 Butyl benzyl phthalate (BBP), CAS No. 85-68-7**
- F.3.8 Di-butyl phthalate (DBP), CAS No. 84-74-2**

F.4 STANDARD SOLUTIONS

Prepare a series of individual stock standard solutions of the individual phthalate esters in n-hexane as shown in Table 3

Table 3 - Stock solutions

Sl. No.	Phthalate ester	Concentration µg/mL
(1)	(2)	(3)
i)	DIDP	5000

ii)	DINP	5000
iii)	DBP	200
iv)	BBP	200
v)	DNOP	200
vi)	DEHP	200

Where appropriate from the stock standard solutions, prepare two sets of six phthalate esters GC-MS calibration solutions in n-hexane to the maximum linear concentration shown in Table 4 (Calibration Set 1) and Table 5 (Calibration Set 2). Two calibration series shall be prepared, using the intermediate stock solutions given in Table 4 and Table 5.

Table 4 - Calibration Set 1

Sl. No. (1)	Phthalate ester (2)	Concentration $\mu\text{g/mL}$ (3)
i)	DINP	500
ii)	DBP	20
iii)	BBP	20
iv)	DEHP	20

Table 5 - Calibration Set 2

Sl. No. (1)	Phthalate ester (2)	Concentration $\mu\text{g/mL}$ (3)
i)	DIDP	500
ii)	DNOP	20

F.5 PROCEDURE

F.5.1 Sampling, extraction and gravimetric analysis for phthalate plasticisers

F.5.1.1 Place sample in a pre-weighed 150 mL flat bottom flask and heat in an oven at $(105 \pm 5)^\circ\text{C}$ for (30 ± 5) minutes. Allow to cool in a desiccator.

F.5.1.2 Weigh the flask and sample. Use a scalpel or other appropriate cutting utensil to cut a representative portion from the sample into small pieces, (< 5 mm).

F.5.1.3 Weigh accurately (2 ± 0.2) g of the pieces into a soxhlet thimble and add cotton wool to the top of the thimble.

F.5.1.4 Add sufficient volume of Diethyl ether into the flask. Reflux gently for $6 \text{ h} \pm 30$ minutes. Allow sufficient time for the Diethyl ether to cool.

F.5.1.5 Evaporate the Diethyl ether completely by means of steam bath.

F.5.1.6 Place the flask in an oven at $(105 \pm 5) \text{ }^\circ\text{C}$ for (30 ± 5) minutes, Allow to cool in a desiccator and weigh.

F.5.1.7 Repeat the drying and cooling cycles until the difference between two consecutive weighings are not more than 0.0005 g. A blank solution shall be run consecutively.

F.5.2 Preparation of sample extract solution for Gas Chromatography-Mass Spectrometry (GC-MS)

F.5.2.1 To the weighed extract (**F.5.1**), add (50 ± 2) mL of *n*-hexane.

F.5.2.2 Place a stopper to the flask and swirl to completely dissolve the extract.

F.5.2.3 Decant the solution into a 200-mL volumetric flask, repeatedly rinsing the flask with *n*-hexane.

F.5.2.4 Add *n*-hexane to the volumetric flask to bring the total volume to 200 mL.

F.5.2.5 Prepare (if necessary) further diluted solutions using *n*-hexane such that the final concentration in solution is within the linear calibration concentration for phthalate present.

F.5.2.6 Transfer a portion of the *n*-hexane solution into a capped vial for GC-MS analysis.

F.5.2.7 The samples can be analysed for phthalate plasticisers by conditioning the GC-MS as follows;

Column	30 m , I.D 0.25 mm, 0.15 μm film thickness, HP 5-MS column (5%- phenyl)-methylpolysiloxane phase capillary column or equivalent column
Carrier gas	Helium
Flow rate	0.8 mL/ minute
Injector temperature	290 $^\circ\text{C}$
Injection volume	2 μl
Injection type	Splitless
Detector	MSD
Transfer line temperature	280 $^\circ\text{C}$
MSD mode	Electron impact
Temperature programme	40 $^\circ\text{C}$ for 4 minutes From 40 $^\circ\text{C}$ to 300 $^\circ\text{C}$ at 10 $^\circ\text{C}/$ minute Isothermal 4.00 minutes

Total run time is 34 minutes

F.6 CALCULATION

F.6.1 Compare the obtained GC-MS spectra to known spectra of phthalate ester standards to allow qualitative identification of phthalate ester plasticizers or any other compounds.

F.6.2 Plot a calibration graph of the response against the known standard concentrations.

F.6.3 From the calibration graph determine the response of phthalate ester found in the blank and sample. Interpolate the concentration of phthalate ester in µg/mL correcting for any dilutions as follows.

F.6.3.1 Gravimetric analysis

$$\text{Percent extract (m/m)} = \frac{W_E}{W_s} \times 100$$

where,

W_E is the weight of the extract in g; and
 W_s is the weight of the sample in g.

F.6.3.2 GC MS analysis

$$\text{Percent phthalate (m/m)} = \frac{E_s}{W_s} \times \frac{200\text{ml}}{10000} \times d_f$$

where,

E_s is the total concentration of total phthalates in the sample extract, in µg/ml;
 W_s is the weight of sample in g; and
 d_f is the dilution factor.

APPENDIX G

DETERMINATION OF 2,2-BIS (4-HYDROXYPHENYL) PROPANE (BISPHENOL A) (BPA) AND 4,4'-DIHYDROXYDIPHENYL SULPHONE (BISPHENOL S) (BPS) RELEASE

G.1 PRINCIPLE

BPA and BPS are extracted from the test articles into aqueous food simulant, identified and its level determined by High-Performance Liquid Chromatography (HPLC) with tandem mass spectrometric detection (LC-MS/MS) via electrospray ionization source.

NOTE

Alternative methodology, such as Gas Chromatography (GC), has been documented and may be used. However, in comparison with the GC method, the HPLC method has the advantage that Bisphenol A and Bisphenol S can be determined directly in the migrate without pre-concentration and derivatisation.

G.2 APPARATUS

G.2.1 LC-MS/MS, preferably equipped with an HPLC with automatic 50 μ L loop injector and electrospray ionization source coupled to a tandem mass spectrometric detector.

G.2.2 HPLC column, capable of separating BPA and BPS fully from peaks originating from simulants and/ or solvents used. The following columns and operating conditions have been found to be suitable for the determination of BPA and BPS.

G.2.2.1 Column SUPELCOSIL C18 150 mm \times 4.6 mm, 5 μ m

G.2.2.2 Column Temperature 35 $^{\circ}$ C

G.2.2.3 Mobile phase Methanol: water (70:30); isocratic

G.2.2.4 Flow 0.5 mL/ minute

G.2.2.5 Injection volume 40 μ L

G.2.2.6 Detection ESI Polarity – Negative

Multiple Ion Reaction Monitoring Transitions

Table 6 – MS Parameters for BPA and BPS

SI. No.	Quantifier mass / Qualifier mass	Analyte	Precursor ion m/z	Product ion m/z
(1)	(2)	(3)	(4)	(5)
i)	Quantifier	BPA	227	212
ii)	Qualifier			133
iii)	Quantifier	BPS	248	108
iv)	Qualifier			92

Instrument-specific temperatures and voltages needed to be optimized by users with respect to the instrument used.

G.2.2.7 Retention time BPA, approximately 5.5 minutes

or

G.2.2.8 Column Acquity UPLC BEH C18 column (1.7 μ m, 2.1 mm \times 50 mm)

G.2.2.9 Column Temperature 30 °C.

G.2.2.10 Mobile phase A – 5 per cent methanol in water

B - 2 mM NH₄OH in Methanol

The gradient programm

Table 7- Gradient elution of liquid chromatography

SI. No.	Time minutes	Mobile phase A	Mobile phase B
		5 per cent methanol in water per cent	2 Mm NH ₄ OH in methanol per cent
(1)	(2)	(3)	(4)
i)	0.0	100	0
ii)	1.0	100	0
iii)	11.0	0	100
iv)	11.5	100	0
v)	15.0	100	0

G.2.2.11 Flow 0.2 mL/ minute

G.2.2.12 Injection volume 20 μ L

G.2.2.13 Detection ESI Polarity – Negative

Multiple Ion Reaction Monitoring Transitions

Table 8- MS Parameters for BPA and BPS

SI. No.	Quantifier mass or	Analyte	Precursor ion m/z	Product ion m/z
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(1)	Qualifier mass (2)	(3)	(4)	(5)
i)	Quantifier	BPA	227	212
ii)	Qualifier		227	133
iii)	Quantifier	BPS	248	108
iv)	Qualifier		248	92

Instrument-specific temperatures and voltages needed to be optimized by users with respect to the instrument used.

G.2.2.14 Retention time

BPA, approximately 5.5 minutes

BPS, approximately 3.0 minutes

NOTES

1. Depending on the type of equipment used, the appropriate operating conditions may need to be established.
2. This information is given for the convenience of users of this Standard and does not constitute an endorsement by SLSI of the product named. Equivalent products can be used if they can be shown to lead to the same results.

G.2.3 Membrane filter, with a pore size of 0.22 µm

G.2.4 Syringe filters, NYLON or PTFE

G.2.5 Analytical balance, with sensitivity of 0.0001 g

G.2.6 Micro pipettes, 10 µL, 20 µL and 50 µL

G.3 REAGENTS

G.3.1 Water, HPLC grade

G.3.2 Methanol, LCMS grade

G.3.3 Deionized water

G.4 REFERENCE STANDARDS

G.4.1 2,2-bis(4-hydroxyphenyl)propane [Bisphenol A] (BPA) (purity greater than 98 per cent)

G.4.2 4,4'-dihydroxydiphenyl sulphone [Bisphenol S] (BPS) (purity greater than 98 per cent)

G.5 STANDARD SOLUTIONS

G.5.1 Preparation of standard solutions

G.5.1.1 *Stock standard solution of BPA in methanol at a defined concentration of approximately 1.0 mg/mL.*

Weigh to the nearest 0.1 mg, approximately 100 mg BPA (**G.4.1**) into a 100-mL volumetric flask. Dissolve the BPS in Methanol (**G.3.2**) and make up to the mark with methanol. Calculate the concentration in μg BPA/ mL solution. Repeat the procedure to obtain a second stock solution.

G.5.1.2 *Stock standard solution of BPS in methanol at a defined concentration of approximately 1.0 mg/mL*

Weigh to the nearest 0.1 mg, approximately 100 mg BPS (**G.4.2**) into a 100-mL volumetric flask. Dissolve the BPA in methanol (**G.3.2**) and make up to the mark with methanol. Calculate the concentration in μg BPS/mL solution. Repeat the procedure to obtain a second stock solution.

NOTES

1. *The solution may be stored refrigerated at +4 °C in a closed glass container, free from light for a period of less than 3 weeks.*

2. *The volume and the weight of the reference standard can be changed proportionately to prepare a sufficient amount of stock standard depending on the laboratory requirement.*

G.5.2 Preparation of an Intermediate mix standard solution of 10 μg / mL of BPA and BPS

G.5.2.1 Transfer by micropipette 1 mL from each stock standard solution (**G.5.1.1** and **G.5.1.2**) into 100 mL volumetric flask and make up to the mark with analyte-free aqueous food simulant (**G.3.3**) and mix thoroughly. This should be prepared on the date of the analysis.

G.5.3 Preparation of calibration solutions

G.5.3.1 Transfer by micropipette 0 μL , 10.0 μL , 20.0 μL , 30.0 μL , 40.0 μL , 50.0 μL of the intermediate mix standard solution (**G.5.2.1**) into a series of six 10 mL volumetric flasks and make up to the mark with analyte-free aqueous food simulant (**G.3.3**) and mix thoroughly.

G.5.3.2 Calculate the exact concentrations of BPA and BPS in the calibration samples in $\mu\text{g}/\text{mL}$. Repeat the procedure using the second stock solution (**G.5.1.1** and **G.5.1.2**).

G.6 PROCEDURE

G.6.1 Transfer 100 mL of the aqueous food simulant (deionized water) into the test article (lunch and snack boxes).

G.6.2 If this volume is too large or small, then use a known volume equivalent to 50 per cent of the capacity of the test article.

G.6.3 If the test article is an accessory place it in a 200 mL measuring cylinder (or equivalent) and adds a known volume of aqueous food simulant (**G.3.3**) sufficient to just cover the test article.

G.6.4 Store under static conditions for 24 h at 40 °C in a drying oven before transferring approximately 1 mL of the solution into a vial suitable for LC-MS/MS injection. Filter the sample via a syringe filter before loading into the vial.

G.6.5 If storage is necessary, sample solutions shall be refrigerated at +4° C in closed container free from light.

G.6.6 To determine the quantity of migrated BPA and BPS, inject the calibration solutions (**G.5.3**) into an LC-MS/MS (**G.2.1**) with LC column (**G.2.2**). Produce calibration curves of BPA and BPS µg/ mL for food simulant (**G.3.3**) using the twelve values from the two stock solutions.

NOTES

1. *The calibration curve should be rectilinear and the correlation coefficient 0.997 or better. The two sets of calibration solutions made from independently prepared stock solutions should be cross-checked by generating two calibration curves which, on the basis of peak ratio measurement, should agree to ± 5 per cent of one another.*

2. *Inject the test sample solutions (**G.6**) into the LC-MS/MS. Use the calibration curve to determine the BPA and BPS content of the test solution, either manually or with data-handling software. It is recommended that the test be carried out at least in duplicate.*
