# AFRICAN STANDARD

# DARS 1720

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## Plastics infant feeding bottles

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ICS 83.08; 55.100

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## Foreword

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This African Standard was prepared by ARSO/TC 14, Food packaging and labelling.



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# Plastics infant feeding bottles

## 1 Scope

This standard prescribes the requirements and methods of sampling and test for infant plastic feeding bottles and receptacles.

## 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.

ISO 472, *Plastics — Vocabulary*

ISO 21067-1, *Packaging — Vocabulary — Part 1: General terms*

ISO 16770, *Plastics — Determination of environmental stress cracking (ESC) of polyethylene — Full-notch creep test (FNCT)*

ASTM D6247-18, *Standard Test Method for Determination of Elemental Content of Polyolefins by Wavelength Dispersive X-ray Fluorescence Spectrometry*

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

ISO 13468-2, *Plastics — Determination of the total luminous transmittance of transparent materials — Part 2: Double-beam instrument*

## 3 Terms and definitions

For the purpose of this standard the terms and definitions given in ISO 472, ISO 21067-1 and the following definitions apply.

**3.1**
**accessories**
supplementary items added to feeding bottle t including the hood, disc/stopper, teat and cap ring to make it efficient

**3.2**
**drinking accessory**
device other than a feeding teat which permits a child to obtain fluid from a container, for example feeding spout and straw

**3.3**
**feeding bottle**
container which is capable of holding a fluid and incorporates a graduated scale suitable for visual measurement and is intended for feeding a child through a feeding teat or drinking accessory.

**3.4**
**locking ring**
component used to secure a feeding teat or drinking accessory to the container.

**3.5**
**sealing disc**
component used to create a seal between the container and the locking ring.



 

**3.6**
**protective cover**
component as safety shield to cover a feeding teat or drinking accessory.

**3.7**
**matched components**
components defined above which are used together whilst feeding a child.

**3.8**
**nominal capacity**
volume of fluid expected to be filled in the bottles at 27 ± 2°C.

**3.9**
**brimful capacity**
volume of fuid held by the container when filled to the point of overflowing while standing on a flat horizontal level with all closures removed, at 27 ± 2°C.

**3.10**
**re-usable**
component intended to be used again after first use.

**3.11**
**numbered graduations**
numbered markings which indicate the volume of fluid within the feeding bottle.

**3.12**
**Single-use drinking accessory or container**
item of drinking equipment sold for single-use.

**3.13**
**protrusions**
drinking accessory, feeding teat or spoon, excluding straws or anything extruding from physical contour of the feeding device.

**3.14**
**receptacles**
container used for holding or storing drinking equipment.

**3.15**
**fluid**
liquid that can be fed to infants with a feeding bottle, for example, water, milk or liquidised food.

## 4 Materials

4.1 The material used for plastics feeding bottles and accessories excluding teats shall be of any food-contact approved polymer or other raw material as given in Annex A for manufacture of plastic feeding bottle. The materials used should be of no health hazards to babies and shall not contain Bisphenol A (BPA) or Polyvinyl chloride (PVC) or Polyethylene terephthalate (PET).

4.2 Teats shall conform to ARS (This standard needs to be developed. REFERENCE: IS 3565: 2018 Teats for Feeding Bottles — Specification)

## 5 Requirements

## 5.1 General requirements

**5.1.1** *Design*

**5.1.1.1** The feeding bottle shall be of suitable design, shape and required dimensions as agreed to between the purchaser and the supplier.

**5.1.1.2** The shape shall be such that it is easily cleanable and does not permit the fluid remnants to remain stuck inside the feeding bottles**.**

**5.1.1.3** Figures C1 and C2 illustrate typical examples of different items of drinking equipment and their design features.

NOTE Figures 1 and 2 are illustrative and for information only.

**5.1.2** *Manufacture, Workmanship, Finish and Appearance*

**5.1.2.1** The bottles and accessories shall be manufactured by a suitable process adhering to Good Manufacturing Practice (GMP).

**5.1.2.2** The body of the bottle shall be smooth, both internally and externally, free from any visual defects like cavities, crevices, hooks, embedded foreign matters, detrimental bubbles, streaks, flaws and stains.
**5.1.2.3** Neck shall be smooth from inside.

**5.1.2.4** All components of plastic feeding bottle when assembled for use shall be free from sharp points and edges and any harmful extrusions**,** which are likely to cause injury.

**5.1.2.5** Any parts that can be detached (e.g. cleaning) shall not be able to fit inside the bottle without compression**.**

**5.1.3 Dimensions**

Plastic infants feeding bottles shall comply with the dimensional requirements given in ISO 13106 when measured in accordance with the methods specified therein.

**5.1.4** *Capacity*

**5.1.4.1** The bottles shall be manufactured in nominal capacity of 125 ml, 150 ml and 250 ml or any other capacity as agreed to between the purchaser and the supplier.

**5.1.4.2** The brimful capacity shall exceed the nominal capacity by a minimum of 15 percent when tested in accordance with ISO 13106, Annex B.

**5.1.4.3** *Capacity scale*
All feeding bottles shall be marked with graduations at least in millilitres. The feeding bottles shall be provided with the following capacity scale:

a) If the feeding bottle is unprinted, then capacity scale shall be engraved on the bottle and if the bottle is printed then the capacity scale shall be clearly printed.
b) The scale interval and the maximum indicating scale mark shall be as agreed to between the purchaser and the supplier. However, the minimum scale mark and interval marking shall be
c) not more than 20 percent of the maximum scale indicating mark.
d) The scale marks and the indicating numerical values shall be clear and shall not be affected by high temperature sterilizing treatment



 

## 5.2 Chemical Requirements

### 5.2.1 *Specific migration of Certain Elements*

When tested in accordance with the ASTM D6247-18 or any equivalent spectrophotometric test method, heavy metals in plastic components of infant feeding bottles shall not exceed the limits given in Table 1.

**Table 1 Permissible Levels of Heavy Metals in Plastic infant feeding bottle**

| SNo. | Heavy Metals | Maximum limit mg/kg *Max* |
|---|---|---|
| i) | Antimony | 15 |
| ii) | Arsenic | 10 |
| iii) | Chromium | 10 |
| iv) | Mercury | 10 |
| v) | Cadmium | 20 |
| vi) | Lead | 25 |
| vii) | Barium | 100 |
| viii) | Selenium | 100 |

### 5.2.2 *Overall migration*

When tested in accordance with Annex D the maximum extraction values for the container material shall not exceed 10 mg/dm$^2$ or 60 mg/l.

### 5.2.3 Pigments and colourants

Examples of permitted pigments and colourants to be used on hermetic bags are as listed in Annex E.
The limits and tolerances of the pigments and colourants used in the printing shall comply with the requirements given in Table 2 when tested in accordance with the test methods specified therein.

**Table 2 — Limits for heavy metals and aromatic amines in plastic infant feeding bottles**

| S/N | Heavy metals and aromatic amines | Limits, % by mass, max. | Test method |
|---|---|---|---|
| i. | Lead, %by mass, max. | 0.01 | ASTM D6247-18 or any equivalent spectrophotometric analysis |
| ii. | Arsenic, %by mass, max. | 0.005 | |
| iii. | Mercury, (soluble in N/10 HCl), %by mass, max. | 0.005 | |
| iv. | Cadmium, %by mass, max. | 0.010 | |
| v. | Zinc, %by mass, max. | 0.05 | |
| vi. | Selenium, %by mass, max. | 0.01 | |
| vii. | Barium, %by mass, max. | 0.01 | |
| viii. | Chromium, %by mass, max. | 0.025 | |
| ix. | Antimony, %by mass, max. | 0.025 | |
| x. | Polychlorinated bsphenyl reported as decachloro biphenyl, mg/kg, max. | 25 | Annex I |
| xi. | Total primary aromatic amines (calculated as aniline equivalent), %by mass, max. | 0.05 | Annex J |

| | | | |
|---|---|---|---|
| xii. | Sulphonated aromatic amines (calculated as aniline sulphonic acid), %by mass, max. | 0.05 | |
| xiii. | Carcinogenic amines listed in Annex H, mg/kg, max. | | Annex J |

## 5.3 Performance Requirements

In addition to the performance requirements specified in ISO 13106, the plastic infant feeding bottles shall comply with the requirements specified in 5.3.

**5.3.1** *Environmental stress-crack resistance*

The bottles shall be tested in accordance with ISO 16770 and shall show no evidence of stress cracking or leakage after being kept in oven for 48 h.

**5.3.2** *Transparency*

The transparency of a plastics feeding bottle shall not be less than 70 percent in any light source transmittance when tested in accordance with the method described in ISO 13468-2.

**5.3.3** *Ageing resistance*

Immerse the bottles into the boiling water for 20 min, then immediately into water at 4°C for 20 min alternately and repeat it 3 times. At the end of the test, the change in the capacity of bottles shall not be more than 1 percent and also there shall be no defective changes in the bottle. There shall be no significant changes in appearance when the accessories are tested in accordance with the method indicated above.

**5.3.4** *Compressive deformation resistance*

The bottles shall not get deformed by more than 10 percent in diameter in compressive direction at the compressive load of 19.6 N when tested in accordance with the method described in Annex G.

**5.3.5** *Ink adhesion test for printed containers*

The printed bottles when tested in accordance with the method described in Annex F (see if Annex K in ISO 13106 can apply) shall not show any significant removal of the print from the bottle surface and the print shall be legible to the naked eye after the test.

# 6 Sampling

## *6.1* ***Sampling and criterion for conformity***

The samples of the bottles shall be drawn and the criteria for conformity determined as prescribed in ISO 13106.

## 6.2 *Sample preparation*

The sample preparation applies to all tests except migration test given in **ASTM D6247-18.**

**6.2.1** Samples from re-usable products shall be immersed in boiling water for 10 min without touching the walls of the container.

NOTE — This is to remove the surface coating arising from the manufacturing processes and ensure that the materials used are stable in boiling water.



 

**6.2.2** New samples, preferably from the same batch, shall be used for each test.

**6.2.3** Samples and test portions shall only be handled with suitable (non-rubber or plastic) gloves and shall only be stored in securely fastened, migration-free (glass) containers and protected from light.

## 7 Packing and Marking

## 7.1 Packing

The bottles shall be packed as agreed to between the purchaser and the supplier.

### 7.2 Marking

**7.2.1** Each bottle shall be permanently marked with scale mark.

**7.2.2** Each carton containing the bottle shall be permanently marked with the following:

a) name of manufacturer and trade-mark, or the company responsible for placing the product in the market, if any;
b) physical locational address of the manufacturer;
c) name of product;
d) nominal capacity;
e) batch No. and Code No.;
f) month and year of manufacture;
g) type of plastics used;
h) country of origin;
i) made from plastics materials meant for food contact applications indicating material used;and
j) instructions for use and hygienic care of the product shall be printed in English/National language and may be included in a separate leaflet placed in or/on the product as given in **8.2.3.**

**8.2.3** *Instructions for Use*

**8.2.3.1** The following information shall be provided:
a) Information for the safe use of the product; and
b) Information on unsuitable common methods of heating which might damage the product.

**8.2.3.2** For re-usable products the following additional instructions shall be provided:
a) At least one method of cleaning;
b) Before first use, clean the product; and
c) Information on unsuitable common methods of cleaning, storage and use which might damage the product.

**8.2.3.3** For products with feeding accessories the following 'WARNINGS' shall be provided in the form given:
For your child's safety and health

**WARNING**
a) Always use this product with adult supervision.
b) Always check food temperature before feeding.
c) Keep all components not in use out of the reach of children.

NOTE — It is recommended that the supplier of drinking equipment include informative literature to explain the reasons and background for these warnings.

**8.2.3.4** Heating in a microwave oven may produce localised high temperatures.



 

For products where microwave heating is recommended as a suitable method of food preparation the following instructions shall be provided although alternative wording is permitted:
Take extra care when microwave heating. Always stir heated food to ensure even heat distribution and test the temperature before serving.

# Annex A
# (informative)

## List of material for manufacture of plastic feeding bottles

(Based on Malaysian Standard, MS 735 and US FDA Regulations) (1)(i) Polypropylene consists of basic polymers manufactured by the catalytic polymerization of propylene.

### A.1 21 CFR 177.1520 (a)(3)(i)

Olefin basic copolymers consist of basic copolymers manufactured by the catalytic copolymerization of:

(i) Two or more of the 1-alkenes having 2 to 8 carbon atoms. Such olefin basic copolymers contain not less than 96 weight-percent of polymer units derived from ethylene and/or propylene, except that:

(a) (1) Olefin basic copolymers manufactured by the catalytic copolymerization of ethylene and hexene-1 or ethylene and octene-1 shall contain not less than 90 weight-percent of polymer units derived from ethylene;

(2) Olefin basic copolymers manufactured by the catalytic copolymerization of ethylene and hexene-1 shall contain not less than 80 but not more than 90 weight percent of polymer units derived from ethylene.

(3) Olefin basic copolymers manufactured by the catalytic copolymerization of ethylene and pentene-1 shall contain not less than 90 weight-percent of polymer units derived from ethylene.

(4) Olefin basic copolymers manufactured by the catalytic polymerization of ethylene and octene-1 shall contain not less than 50 weight-percent of polymer units derived from ethylene.

(b) Olefin basic copolymers manufactured by the catalytic copolymerization of ethylene and 4-methylpentene-1 shall contain not less than 89 weight percent of polymer units derived from ethylene;

(c) (1) Olefin basic copolymers manufactured by the catalytic copolymerization of two or more of the monomers ethylene, propylene, butene-1, 2-methylpropene-1, and 2,4,4- trimethylpentene-1 shall contain not less than 85 weight percent of polymer units derived from ethylene and/or propylene;

(2) Olefin basic copolymers manufactured by the catalytic copolymerization of propylene and butene-1 shall contain greater than 15 but not greater than 35 weight percent of polymer units derived from butene-1 with the remainder being propylene.

(d) Olefin basic terpolymers manufactured by the catalytic copolymerization of ethylene, hexene-1, and either propylene or butene-1, shall contain not less than 85 weight percent polymer units derived from ethylene.

(e) Olefin basic copolymers manufactured by the catalytic polymerization of ethylene and octene-1, or ethylene, octene-1, and either hexene-1, butene-1, propylene, or 4- methylpentene-1 shall contain not less than 80 weight percent of polymer units derived from ethylene.

### A.2 21 CFR 177.1520 (b)

(b) Olefin basic copolymers manufactured by the catalytic copolymerization of ethylene and 4-methylpentene-1 shall contain not less than 89 weight-percent of polymer units derived from ethylene;

### A.3 CFR 177.1520 (c) Specifications

| Item | Olefin polymers | Density | Melting Point (MP) or softening point (SP) in °C | Maximum extractable fraction (expressed as percent by weight of the polymer) in n-hexane at specified temperatures | Maximum soluble fraction (expressed as percent by weight of polymer) in xylene at specified temperatures |
|---|---|---|---|---|---|
| (1) | (2) | (3) | (4) | (5) | (6) |
| 1.1a | Polypropylene described in paragraph (a)(1)(i) of this section | 0.880 – 0.913 | MP: 160-180°C | 6.4 percent at reflux temperature | 9.8 percent at 25°C |
| 3.1a | Olefin copolymers described in paragraph (a)(3)(i) of this section for use in articles that contact food except for articles used for packing or holding food during cooking; except olefin copolymers described in paragraph (a)(3)(i)(a)(3) of this section and listed in item 3.1c of this table and olefin copolymers described in paragraph (a)(3)(i)(e) of this section and listed in item 3.1b of this table | 0.85 – 1.00 | | 5.5 percent at 50°C | 30 percent at 25°C |

## Annex B
## (normative)

## Test for permanency of pigment

**B-1 General**
This test is meant only for those feeding bottles which have a printed scale and graduations.

**B-2 Reagents**

**B-2.1 Sodium Bichromate,**

**B-2.2 Concentrated Sulphuric Acid**, relative density – 1.834 approximately.

**B-3 Procedure**

**B-3.1** Weight about 20 g of sodium dichromate and dissolve in 1 500 ml of concentrated sulphuric acid and dilute to 2 500 ml with water. Immerse the bottles in the solution at room temperature for 15 min. Rinse the samples with water and dry.

**B-3.1.1** The bottles shall be taken as having satisfied the requirements of the test, if the printed impressions do not become illegible.

# Annex C
(informative)

## Typical examples of different items of drinking equipment and their design features.

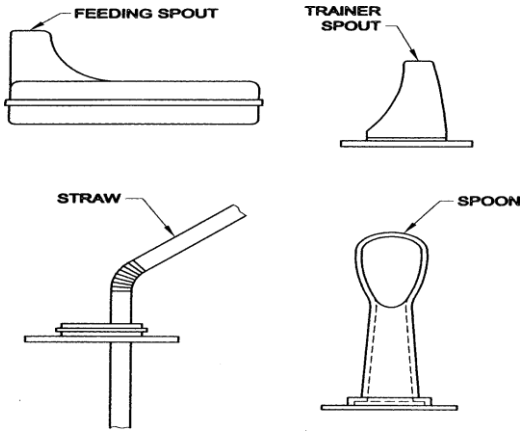


**Figure C1 – example of drinking accessories**

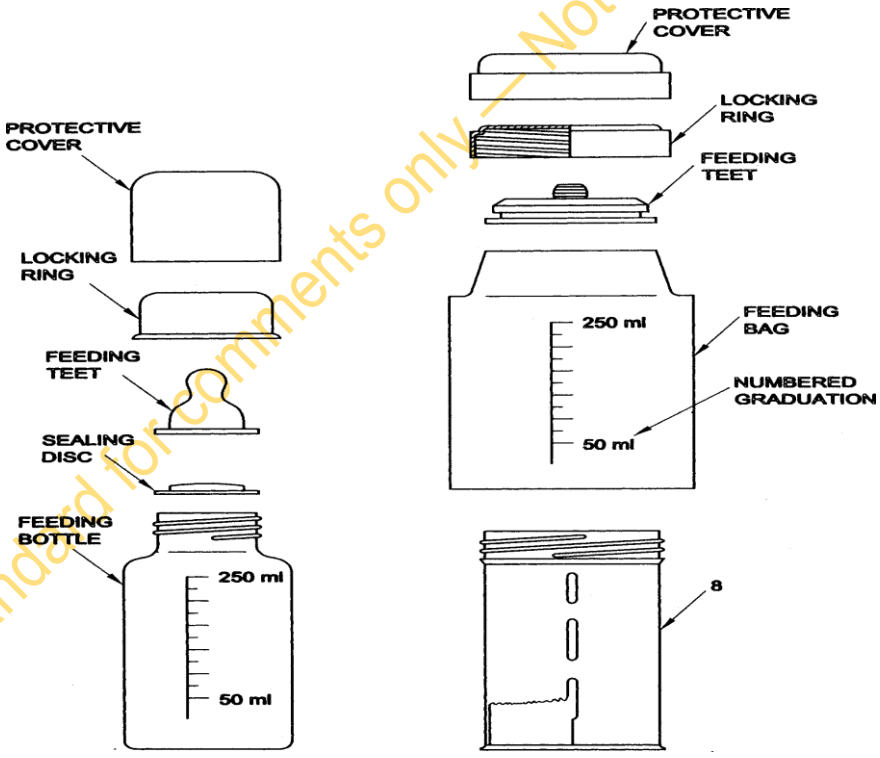


**Figure C2 – examples of containers with feeding teats**

# Annex D

## (normative)

## Determination of overall migration of constituents of plastics materials and articles intended to come in contact with foodstuffs – Method of analysis

### D.1Types 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')

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

**D.2.1** The choice of simulating solvents and test conditions (time-temperature) depends on the type of foodtuff 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 D.1.

**Table D.1 — Classification of foods and selection of simulant**

| S/N | Type of food | Description | Examples | Simulant |
|---|---|---|---|---|
| i. | I | Aqueous, non-acidic foods without fat (pH > 5) | Honey, mineral water, sugar syrups molasses, skimmed milk, rusgulla, infusions, murabba, yeast, paste etc yeast paste etc | 'A' |
| ii. | II | Aqueous. acidic foods without fat (pH ≤ 5 5) | 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' |

**D.2.2** Table D.2 lists test conditions (time-temperature) for extractability studies to be carried out as above depending on conditions of use of the food.

**Table D.2 — Test conditions of temperature and time**

| S/N | Conditions of use | Water (time- temperature ) |
|---|---|---|
| i. | High temperature heat sterilized (Retorting) | 121 °C, 2 h |
| ii. | Hot filled or pasteurized above 66 °C, 100 °C | 100 °C, 2 h |
| iii. | Hot filled or pasteurized below 66 °C | 70 °C, 2 h |
| iv. | Room temperature filled and stored (no thermal treatment in container) and also in refrigerated and frozen condition | 40 °C, 10 days |

### D.3 Apparatus

**D.3.1** Electric oven/water bath, equipped with thermostat to maintain the desired temperature within ± 1 ºC accuracy

**D.3.2** Electric hot plate, with temperature control regulator

**D.3.3** Analytical balance, with a sensitivity of 0.1 mg

**D.3.4** Glass beakers, Pyrex of 1 000 ml capacity or equivalent

**D.3.5** Stainless steel evaporating dish of 100 ml capacity

**D.3.6** Stainless steel tongs

## D.4 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 $cm^2$ surface area coming in contact.

## D.5 Preparation of the test specimen

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

## D.6 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.

## D.7 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.

## D.8 Determination of Amount of Extractive

**D.8.1** 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 $mg/dm^2$ 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.

**D.8.2** Calculate the amount of extractive in ppm for the particular size of container being tested.

Amount of extractive (Ex) = $\frac{M}{V}$x1000 mg/kg or mg/l

or Ex= $\frac{M}{A}$x100 $mg/dm^2$

where

M = mass of residue in mg minus blank value;

A= surface area in $cm^2$ 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.

# Annex E
# (informative)

## List of colourants and pigments for use in plastics in contact with foodstuffs and pharmaceuticals

### E.1 Principle

**E.1.1** This annex provides a list of permitted pigments and colourants for use in plastics intended to come in contact with foodstuffs and pharmaceuticals.

**E.1.2** Pigments and colourants used shall not show visible bleeding or migration from the dried food products and shall show no signs of instability or degradation during processing.

### E.2 List of pigments and colourants for use in plastics that come into direct contact with foodstuffs and pharmaceuticals

#### E.2.1 Organic pigments

List of organic pigments and colourants are listed in Table E1.

**Table E 1 — Organic pigments**

| Sl No. | CAS No. | C.I. No | C.I. Name |
|---|---|---|---|
| 1. | 2512-29-0 | 11680 | Pigment yellow 1 |
| 2. | 6486-23-3 | 11710 | Pigment yellow 3 |
| 3. | 5979-28-2 | 20040 | Pigment yellow 16 |
| 4. | 6370-75-8 | 65405 | C.I. Vat yellow 12 |
| 5. | 12286-66-7 | 13940 | Pigment yellow 62 |
| 6. | 5580-58-5 | 20038 | Pigment yellow 94 |
| 7. | 5280-80-8 | 20034 | Pigment yellow 95 |
| 8. | 5590-18-1 | 56280 | Pigment yellow 110 |
| 9. | 29920-31-8 | 11738 | Pigment yellow 120 |
| 10. | 79953-85-8 | 20037 | Pigment yellow 128 |
| 11. | 30125-47-4 | 56300 | Pigment yellow 138 |
| 12. | 36888-99-0 | 56298 | Pigment yellow 139 |
| 13. | 71832-85-4 | 13960 | Pigment yellow 168 |
| 14. | 96352-23-7 | 56160 | Pigment yellow 173 |
| 15. | 77804-81-0 | 21290 | Pigment yellow 180 |
| 16. | 74441-05-7 | 11777 | Pigment yellow 181 |
| 17. | 67906-31-4 | 12830 | Pigment yellow 182 |
| 18. | 65212-77-3 | 18792 | Pigment yellow 183 |
| 19. | 129433-54-7 | 18795 | Pigment yellow 191 |
| 20. | 3627-47-2 | 65410 | Vat Yellow 26 |
| 21. | 12236-62-3 | 11780 | Pigment Orange 36 |
| 22. | 4424-06-0 | 71105 | Pigment. Orange 43 |
| 23. | 40716-47-0 | 11265 | Pigment Orange 61 |
| 24. | 72102-84-2 | 12760 | Pigment Orange 64 |
| 25. | 35869-64-8 | 20060 | Pigment Brown 23 |

| | | | |
|---|---|---|---|
| 26. | 68516-75-6 | □ | Pigment Brown 41 |
| 27. | 6041-94-7 | 12310 | Pigment Red 2 |
| 28. | 2425-85-6 | 12120 | Pigment Red 3 |
| 29. | 2814-77-9 | 12085 | Pigment Red 4 |
| 30. | 6410-41-9 | 12490 | Pigment Red 5 |
| 31. | 6471-51-8 | 12420 | Pigment Red 7 |
| 32. | 6410-30-6 | 12335 | Pigment Red 8 |
| 33. | 6410-38-4 | 12460 | Pigment Red 9 |
| 34. | 6410-35-1 | 12440 | Pigment Red 10 |
| 35. | 6410-32-8 | 12385 | Pigment Red 12 |
| 36. | 3564-22-5 | 12350 | Pigment Red 18 |
| 37. | 6883-91-6 | 21205 | Pigment Red 37 |
| 38. | 6358-87-8 | 21120 | Pigment Red 38 |
| 39. | 7023-61-2 | 15865:2 | Pigment Red 48:2 |
| 40. | 15782-05-5 | 15865:3 | Pigment Red 48:3 |
| 41. | 1103-39-5 | 15630:2 | Pigment Red 49:2 |
| 42. | 17852-99-2 | 15860:1 | Pigment Red 52:1 |
| 43. | 4/9/5281 | 15850:1 | Pigment Red 57:1 |
| 44. | 6417-83-0 | 15880:1 | Pigment Red 63:1 |
| 45. | 5850-80-6 | 15525 | Pigment Red 68 |
| 46. | 72-48-0 | 58000:1 | Pigment Red 83 |
| 47. | 14295-43-3 | 73312 | Pigment Red 88 |
| 48. | 6409-74-1 | 60745 | Pigment Red 89 |
| 49. | 6535-46-2 | 12370 | Pigment Red 112 |
| 50. | 980-26-7 | 73915 | Pigment Red 122 |
| 51. | 5280-78-4 | 20735 | Pigment Red 144 |
| 52. | 5280-68-2 | 12485 | Pigment Red 146 |
| 53. | 4948-15-6 | 71137 | Pigment Red 149 |
| 54. | 56396-10-2 | 12290 | Pigment Red 150 |
| 55. | 3905-19-9 | 20730 | Pigment Red 166 |
| 56. | 2786-76-7 | 12475 | Pigment Red 170 |
| 57. | 4051-63-2 | 65300 | Pigment Red 177 |
| 58. | 5521-31-3 | 71130 | Pigment Red 179 |
| 59. | 77804-81-0 | 21290 | Pigment Red 180 |
| 60. | 2379-74-0 | 73360 | Pigment Red 181 |
| 61. | 59847-23-9 | 12486 | Pigment Red 187 |
| 62. | 3089-17-6 | 73907 | Pigment red 202 |
| 63. | 31778-10-6 | 12514 | Pigment red 208 |
| 64. | 1/1/3573 | 73905 | Pigment red 209 |
| 65. | 40618-31-3 | 20066 | Pigment red 214 |
| 66. | 68259-05-2 | 20055 | Pigment red 220 |
| 67. | 71566-54-6 | 20065 | Pigment red 221 |
| 68. | 52238-92-3 | 20067 | Pigment red 242 |
| 69. | 43035-18-3 | 15915 | Pigment red 247 |
| 70. | 84632-65-5 | 56110 | Pigment Red 254 |

| | | | |
|---|---|---|---|
| 71. | 120500-90-5 | 561050 | Pigment red 257 |
| 72. | 70833-37-3 | 56270 | Pigment red 256 |
| 73. | 88949-33-1 | 561300 | Pigment Red 264 |
| 74. | 1047-16-1 | 73900 | Pigment Violet 19 |
| 75. | 6358-30-1 | 51319 | Pigment Violet 23 |
| 76. | 81-33-4 | 71129 | Pigment Violet 29 |
| 77. | 5462-29-3 | 73385 | Pigment Violet 36 |
| 78. | 2379-75-1 | 73395 | Pigment Violet 38 |
| 79. | 147-14-8 | 74160 | Pigment Blue 15:X |
| 80. | 574-93-6 | 74100 | Pigment Blue 16 |
| 81. | 1328-50-3 | 74140 | Vat Blue 29 |
| 82. | 81-77-6 | 69800 | Pigment Blue 60 |
| 83. | 482-89-3 | 73000 | Pigment Blue 66 |
| 84. | 1328-53-6 | 74260 | Pigment Green 7 |
| 85. | 1330-37-6 | 74255 | Pigment Green 37 |
| 86. | 31837-42-0 | 13980 | Pigment Yellow 151 |
| 87. | 4118-16-5 | 60645 | Pigment Yellow 147 |
| 88. | 52238-92-3 | 20067 | Pigment Red 242 |
| 89. | 250640-08-5 | □ | Pigment Orange 79 |
| 90. | 84632-66-6/ 61951-98-2 | □ | Pigment Red 272 |
| 91. | 154946−66−4 | 18759:1 | Pigment yellow 191:1 |

**E.2. Dyestuffs**

| Sl No. | CAS No. | C.I. No | C.I. Name |
|---|---|---|---|
| 1. | 6370-85-0 | 66510 | Vat Yellow 9 |
| 2. | 6252-78-4 | 73860 | Vat Red 45 |
| 3. | 6492-68-8 | 73305 | Vat Red 47 |
| 4. | 482-89-3 | 73000 | Vat Blue 1 |
| 5. | 130-20-1 | 69825 | Vat Blue 6 |
| 6. | 1330-38-7 | 74180 | Direct Blue 86 |
| 7. | 128-80-3 | 61656 | Solvent Green 3 |
| 8. | 116-75-6 | 61568 | Solvent Blue 104 |
| 9. | 12236-03-2 | 61568 | Disperse Orange 47 |
| 10. | 17354-14-2 | 61554 | Solvent Blue 35 |
| 11. | 4702-90-3 | 48160 | Solvent Yellow 93 |
| 12. | 4851-50-7 | 625580 | Solvent Green 28 |
| 13. | 61969-44-6 | 615290 | Solvent Blue 97 |
| 14. | 6408-72-6 | 615290 | Disperse Violet 26 |
| 15. | 64696-98-6 | 48525 | Solvent Brown 53 |
| 16. | 6829-22-7 | 564150 | Solvent Red 179 |
| 17. | 61969-47-9/6925-69-5 | 564100 | Solvent Orange 60 |
| 18. | 75216-45-4/7576-65-0 | 47020 | Solvent Yellow 114/ Disperse Yellow 54 |
| 19. | 81-39-0 | 68210 | Solvent Red 52 |

| | | | |
|---|---|---|---|
| 20. | 81-48-1 | 60725 | Solvent Violet 13 |
| 21. | 83249-52-9 | 56280 | Disperse Yellow 241 |
| 22. | 80748-21-6/54079-53-7/17772-51-9 | □ | Disperse Yellow 201 |
| 23. | 20749-68-2/71902-17-5 | 564120 | Solvent Red 135 |
| 24. | 12226-78-7/81457-65-0 | □ | Solvent Blue 67 |
| 25. | 6408-72-6 | 62025 | Solvent Violet 59 |
| 26. | 72968-71-9 | □ | Solvent Red 195 |
| 27. | 23552-74-1/37229-23-5 | □ | Solvent Blue 45 |

**E.3 Inorganic pigments/alloys**

| Sl No. | CAS No. | C.I. No | C.I. Name |
|---|---|---|---|
| 1. | 7429-90-5 | 77000 | Aluminium |
| 2. | 7440-50-6 | 77400 | Copper |
| 3. | 7440-22-4 | 77820 | Silver |
| 4. | 7440-57-5 | 77480 | Gold |
| 5. | 7440-31-5 | 77860 | Tin |
| 6. | 7440-06-4 | 77795 | Platinum and platinum group metals |
| 7. | 7440-50-8 | 77400 | Bronzes of copper |
| 8. | 471-341-1 | 77220 | Whitening (calcium carbonate) |
| 9. | 10101-41-4 | 77231 | Calcium sulphate (Gypsum, plaster of Paris) |
| 10. | 1332-58-7 | 77005 | Kaolin |
| 11. | 13463-67-7 | 77891 | Titan White (titanium oxide) |
| 12. | 1344-28-1 | 77002 | Alumina |
| 13. | 637-12-7 | □ | Aluminium stearate |
| 14. | 14807-96-6 and 8005-37-6 | 77718 | Talc |
| 15. | 51274-00-1 | 77492 | Yellow iron oxide |
| 16. | 1345-27-3 | 77491 | Iron oxide |
| 17. | 57455-37-5 | 77007 | Ultramarine blue (complex silicate of aluminium and sodium sulphurated) |
| 18. | □ | 77437 | Egyptian blue (double silicate of copper and calcium) |
| 19. | 1345-16-0 | 77346 | Cobalt blue (cobalt aluminate) |
| 20. | 1333-86-4 | 77266 | Carbon black |
| 21. | 7727-43-7 | 77120 | Barytes (barium Sulphate) |
| 22. | 64294-91-3 | 77492 | Sienna (natural ferric oxide) |
| 23. | 12769-96-9 | 77007 | Pigment Violet 15 |
| 24. | 1308-38-9 | 77288 | Pigment Green 17 |
| 25. | 1309-37-1 | 77491 | Pigment Red 101 |
| 26. | 1314-13-2 | 77975 | Pigment White 4 |
| 27. | 1314-98-3 | 77975 | Pigment White 7 |
| 28. | 1317-61-9 | 77499 | Pigment Black 11 |
| 29. | 57455-37-5/ 101357-30-6 | 77007 | Pigment Blue 29 |
| 30. | 68187-51-9 | 77496 | Pigment Yellow 119 |
| 31. | 7727-43-7 | 77120 | Pigment white 21 |
| 32. | 8007-18-9 | 77788 | Pigment Yellow 53 |
| 33. | 12001-26-2 | 77019 | Pigment White 20 |
| 34. | 18282-10-5 | 77861 | □ |
| 35. | 1344-28-1 | □ | □ |
| 36. | 68186-90-3 | 77310 | Pigment Brown 24 |
| 37. | 1345-05-7 | 77115 | Pigment White 5 |

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| Sl No. | CAS No. | C.I. No | C.I. Name |
|---|---|---|---|
| 14. | 14807-96-6 and 8005-37-6 | 77718 | Talc |
| 15. | 51274-00-1 | 77492 | Yellow iron oxide |
| 16. | 1345-27-3 | 77491 | Iron oxide |
| 17. | 57455-37-5 | 77007 | Ultramarine blue (complex silicate of aluminium and sodium sulphurated) |
| 18. | □ | 77437 | Egyptian blue (double silicate of copper and calcium) |
| 19. | 1345-16-0 | 77346 | Cobalt blue (cobalt aluminate) |
| 20. | 1333-86-4 | 77266 | Carbon black |
| 21. | 7727-43-7 | 77120 | Barytes (barium Sulphate) |
| 22. | 64294-91-3 | 77492 | Sienna (natural ferric oxide) |
| 23. | 12769-96-9 | 77007 | Pigment Violet 15 |
| 24. | 1308-38-9 | 77288 | Pigment Green 17 |
| 25. | 1309-37-1 | 77491 | Pigment Red 101 |
| 26. | 1314-13-2 | 77975 | Pigment White 4 |
| 27. | 1314-98-3 | 77975 | Pigment White 7 |
| 28. | 1317-61-9 | 77499 | Pigment Black 11 |
| 29. | 57455-37-5/ 101357-30-6 | 77007 | Pigment Blue 29 |
| 30. | 68187-51-9 | 77496 | Pigment Yellow 119 |
| 31. | 7727-43-7 | 77120 | Pigment white 21 |
| 32. | 8007-18-9 | 77788 | Pigment Yellow 53 |
| 33. | 12001-26-2 | 77019 | Pigment White 20 |
| 34. | 18282-10-5 | 77861 | □ |
| 35. | 1344-28-1 | □ | □ |
| 36. | 68186-90-3 | 77310 | Pigment Brown 24 |
| 37. | 1345-05-7 | 77115 | Pigment White 5 |

## Annex F
## (normative)

## Test for permanency of pigment

**F-1 GENERAL**
This test is meant only for those feeding bottles which have a printed scale and graduations.

**F-2 REAGENTS**

**F-2.1 Sodium Bichromate,**

**F-2.2 Concentrated Sulphuric Acid**, relative density – 1.834 approximately

**F-3 PROCEDURE**

**F-3.1** Weight about 20 g of sodium dichromate and dissolve in 1 500 ml of concentrated sulphuric acid and dilute to 2 500 ml with water. Immerse the bottles in the solution at room temperature for 15 min. Rinse the samples with water and dry.

**F-3.1.1** The bottles shall be taken as having satisfied the requirements of the test, if the printed impressions do not become illegible.

# Annex G
# (normative)

## Compressive Deformation Test

### E.1 PROCEDURE

Apply the compressive load of 19.6 N in the middle part of the body or to the part having the maximum diameter of a feeding bottle by the use of compression jig as shown in Fig. 5 Measure the deflection of the part at that time, and calculate percentage deflection. The measurements shall be carried out at 27 ± 2 °C.

### E-2 CALCULATION

Percentage deflection of diameter =

$$\frac{\text{outside diameter prior to test} - \text{outside diameter at the time of compression}}{\text{outside diameter prior to test}} X100$$

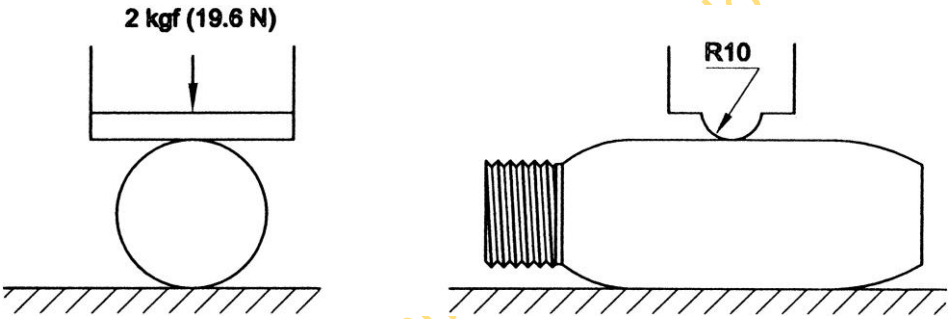


Figure G1— Compression Jig

## Annex H
## (normative)

## List of carcinogenic amine

| Sl No. | CAS No. | Substances |
|---|---|---|
| 1. | 92-67-1 | 4-Aminobiphenyl |
| 2. | 92-87-5 | Benzidine |
| 3. | 95-69-2 | 4-Chloro-o-toluidine |
| 4. | 91-59-8 | 2-Naphthylamine |
| 5. | 97-56-3 | o-Aminoazotoluene/<br>4-Amino-2,3-dimethylazobenzene/<br>4-o-Tolylazo-o-toluidine |
| 6. | 99-55-8 | 5-Nitro-o-toluidine |
| 7. | 106-47-8 | p-Chloroaniline |
| 8. | 615-05-4 | 2,4-Diamino anisole |
| 9. | 101-77-9 | 4,4'-Methylenedianiline/<br>4,4'-Diaminodiphenylmethane |
| **10.** | **91-94-1** | **3,3'-Dichlorobenzidine/**<br><br>**3,3'-Dichlorobiphenyl-4,4'-xylendiamine** |
| 11. | 119-90-4 | 3,3'-Dimethoxybenzidine |
| 12. | 119-93-7 | 3,3'-Dimethylbenzidine/<br>4,4'-Bi-o-toluidine |
| 13. | 838-88-0 | 3,3'-Dimethyl -4,4'-diaminodiphenylmethane |
| 14. | 120-71-8 | 6-Methoxy-m-toluidine-p-cresidine |
| 15. | 101-14-4 | 4,4'-Methylene-bis-(2-chloroaniline)/<br>2,2'-Dichloro-4,4'-methylenedianiline |
| 16. | 101-80-4 | 4,4'-Oxydianiline |
| 17. | 139-65-1 | 4,4'-Thiodianiline |
| 18. | 95-53-4 | o-Toluidine/<br>2-Aminotoluene |
| 19. | 95-80-7 | 4-Methyl-m-phenylenediamine |
| 20. | 137-17-7 | 2,4,5-Trimethylaniline |
| 21. | 90-04-0 | o-Anisidine-2-methoxyaniline |
| 22. | 60-09-3 | 4-Aminoazobenzene |

## Annex I
## (normative)

# Determination of polychlorinated biphenyl (PCB)

### I.1 GENERAL

This method covers determination of total polychlorinated biphenyls (PCBs) content in colourant material by low resolution gas chromatography coupled to high resolution mass spectrometer (LRGC-HRMS) using Electron Impact (EI) mode.

Application of LRGC-HRMS ensures separation/ recognition of most PCB congeners are separated or recognizable at different retention times.

NOTE — In case a specific PCB is to be reported which is different from any congener directly specified by internal standard, suitable care should be taken using external window standards for identification.

### I.2 APPARATUS

**I-2.1** Low Resolution Gas Chromatography Coupled to High Resolution Mass Spectrometer (LRGC-HRMS) Using Electron Impact (EI) Mode

**I-2.2** Rotary Evaporator

**I-2.3** Weighing Balance, nearest to 0.000 1 g.

I-2.4 Micropipette, with disposable pipette tips.

**I-2.5** Surgical Hand Gloves C-2.6 Glass Column

**I-2.6.1** Column, 30 cm long, 18 mm diameter with 250 ml top reservoir.

**I-2.6.2** Column, 30 cm long, 10 mm diameter with 250 ml top reservoir.

**I2.7** Magnetic Stirrer and Magnetic Bar

**I-2.8** Ground Joint Conical Flask — 500 ml.

### I.3 REAGENTS

**I-3.1** Concentrated Sulphuric acid — Analytical grade.

**I-3.2** n-Hexane — Analytical reagent grade.

**I-3.3** Dichloro Methane — Analytical reagent grade.

**I-3.4** Silica Gel — Technical grade, pore size 60 Å, 70-230 mesh, 63-200 µm

**I-3.5** PCB Standard Solution (EC 4058) or Equivalent

**I-3.6** n-Nonane

**I-3.7** Celite 545 (CAS No. 68855-54-9)

**I-3.8** Cesium Hydroxide

**I-3.9** Silver Nitrate

**I-3.10** Alumina (ICN Alumina B - Super I (Basic) (50-200 µm or Alternate)

**I-3.11** Sodium Sulphate, anhydrous.

**I-3.12** Toluene, analytical reagent grade.

**I-3.13** Ethanol, analytical reagent grade.

## I-4 WORK CONDITIONS

Dust free environment with positive pressure inside the laboratory by air handling unit and temperature to be maintained at 22 ± 2 °C.

## I-5 PROCEDURE

### I-5.1 Preparation of test sample

**I-5.1.**1 Weigh accurately 0.75-2.5 g sample into a conical flask and add 0.5-0.9 g ethanol to wet the colourant surface.

**I-5.1.2** Add 100 µl PCB standard (5 times diluted with n-nonane). Add 1-2 g of phosphoric acid and sodium sulphate of approximately same weight as of the sample. Add 40 g of 92 - 96 percent sulphuric acid twice, carefully with repeated shaking. Sonicate the mixture for 30-100 min until homogenous dark reddish or olive colour solution is obtained.

### C-5.2 Liquid – Liquid Extraction

**I-5.2.1** Extract sulphuric acid solution with 200 ml n-hexane and rapidly stir it by ultra-sonication for at least 30 min. In case of sample suspected of unusually high impurity content, a mixture of 50 ml n-hexane and 200 ml dichloromethane to be used.

**I-5.2.**2 Transfer the mixture to a separating funnel and collect the organic phase in a 500 ml round bottom flask. Repeat the extraction steps 2 times (more if necessary) and combine all the organic phases in a round bottom flask. In case, if possible, decant organic layer directly. In case, if oily layer found then follow the step as given as I-5.2.3. Add 1-2 ml n-nonane in organic phase and remove the solvent using rotary evaporator. Reduce the volume up to 1-2 ml in round bottom flask. Follow the cleanup procedure using 1-2 ml remaining solvent.

**I-5.2.3** Add 50 percent aqueous potassium hydroxide solution in organic phase to make its pH 7-8, and shake out the aqueous layer twice with 100 ml n-hexane, in order to control the change of temperature due to heat of neutralization. After reaching pH 8, transfer the solution into the separation funnel, rinse the flask with 10 ml of n-hexane. Combine all the clean organic phases in a round bottom flask and dry them by adding 0.5 g of anhydrous sodium sulphate. Add 1-2 ml n- nonane in organic phase and remove the solvent using rotary evaporator. Reduce the volume up to 1-2 ml in round bottom flask. Follow the cleanup procedure using 1-2 ml remaining solvent.

### I-5.3 Clean-up

**I-5.3.1** Take the neat and clean glass column (size 30 cm long, 18 mm diameter with 250 ml top reservoir). Fill the glass column with n-hexane up to 1/3 of top reservoir. Weigh the chemicals and transfer to column in following sequence. While addition of reagent shake the column externally to avoid any air bubbles left in the column for better efficiency.

| Reagent/Chemicals | Mass (g) |
|---|---|
| Silica gel | 5 |
| Celite : Sulphuric acid (1:1 mix) | 30-33 |
| Silica gel | 5-6 |
| Anhydrous sodium sulphate | 5-7 |

Let n-hexane run until top layers reached. Then pre- condition with 200 ml hexane: dichloromethane (4 : 1, v/v) and let run off to the top layer again. Discard the elute.

**I-5.3.2** Transfer the analyte from round bottom flask to column with help of transfer pipette and rinse the round bottom flask for 5 times with approximately 5 ml hexane: dichloromethane (4 : 1, v/v) and transfer to multi-



 

layer column. Add approximately 150 ml n-hexane. Collect the solvent in 500 ml round bottom flask. Recover the excess n-hexane by rotary evaporator until 5-7 ml solution is left.

### I-5.4 Alumina column

**I-5.4.1** Take the neat and clean glass column (size 30 cm long, 10 mm diameter with 250 ml top reservoir). Fill the glass column with toluene up to 1/3rd of top reservoir. Usage of n-hexane instead of toluene is also permissible. Weigh the chemicals and transfer to column in following sequence. While addition of reagent shake the column externally to avoid any air bubbles left in the column.

| Reagent/Chemicals | Mass (g) |
|---|---|
| Silica gel | 0.3-0.5 |
| Anhydrous alumina | 12.5-13 |
| Silica gel | 0.3 |
| Anhydrous sodium sulphate | 3 |

**I-5.4.2** After filling of the column, remove the toluene/n-hexane up the just above the top layer of reagent.

Load concentration — Eluate of previous step into the column. Rinse the flask twice (2 times 3 ml toluene/n hexane) and add the solvent to the column.

Pre-run — Elute off with 40 ml toluene into a calibrated cylinder in case of brominated sample matrix. Elute with hexane: dichloromethane (98 : 2, v/v), adding first 2 ml × 2 ml and finally 76 ml of the eluant and collect into the same cylinder of the pre-run till reaching a volume of 120 ml.

**I-5.4.3** Transfer the extract solution quantitatively to a flask and evaporate to a volume of 500 µl, reduce to approximately 200 µl by nitrogen blowing and transfer to a standard 1.2 ml septum-sealed glass vial. The measuring solution is obtained by rinsing the flask 2 times with 25 µl n-nonane and adding the volume to the GC-vial.

### I-5.5 Determination of analytes

Refer the instrument manual for operation and analysis of PCB by gas chromatography — Mass Spectrometry (GCMS) using auto-sampler, injection volume shall be 3-10 µl.

NOTE — Perfluorotributylamine (PFTBA) tuning is performed every two months or according to instrument performance monitoring requirement.

### I-5.6 Calculation and quantification procedure

**I-5.6.1** In case, if peaks are not appearing in desire windows then set the time accordingly.

Integration the M peaks in GC-quadrapole should be carried out manually in the re-quantification mode. Small peaks and especially those looking significant, but lacking even an approximate isotopic ratio as predetermined for the Analyte, response is considered 100 percent. Chemically and structurally most congers are to be ratio-calculated from the congener standard amount. Amount of each determined congener is divided by the sample weight.

### C-5.6.2 Lower detection limit

PCB below the range of 1 ppm cannot be measured by this method, since the internal relative standard deviation (RSD) will approach 100 percent. Report the sum of decachlorobiphenyl (DeCB) equivalent (ppm) from mono- to deca-chlorobiphenyl and apply correction factor, if any, based on the measurement of uncertainty.

### C-5.6.3 Mass Conversion Factors

| Degree of Chlorination | From CB to DeCB Equivalent | From DeCB to CB Equivalent |
|---|---|---|
| **Mono-CB** | 2.649 | 0.377 50 |
| **Di-CB** | 2.244 | 0.445 64 |
| **Tri-CB** | 1.930 | 0.518 13 |
| **Tetra -CB** | 1.706 | 0.586 17 |
| **Penta -CB** | 1.528 | 0.654 45 |



 

| | | |
|---|---|---|
| **Hexa-CB** | 1.391 | 0.718 91 |
| **Hepta-CB** | 1.264 | 0.791 14 |
| **Octa-CB** | 1.158 | 0.863 56 |
| **Nona-CB** | 1.073 | 0.931 97 |
| **Deca-CB(DeCB)** | 1.000 | 1.000 00 |

## Annex J
## (normative)

# Determination of total primary aromatic amines

### J.1 GENERAL

Analysis of primary aromatic amine (PAA) in organic colourants to ascertain quality for safe use in food contact application.

### J.2 APPARATUS

J-2.1 **Weighing Balance,** nearest to 0.000 1 g**.**

J-2.2 **Ultrasonic Bath**

J-2.3 **Centrifuge Capable** to 3 000 rpm

J-2.4 **HPLC System Equipped with Gradient Elution—DAD detector and pump**. UV detector can be use as optional.

J-2.5 **Column—**250 mm × 4 mm, 5 µm or equivalent HPLC column.

### J.3 REAGENTS

**J-3.1 Hydrochloric Acid Solution — 1N.**

**J-3.2 Sodium Hydroxide Solution — 5N.**

**J-3.3 HPLC grade methanol**

**J-3.4 pH Strips**

**J-3.5 Distilled Water**

**J-3.6 Analytical Reference Standards for Amines, to test appropriate individual colorant**.

For example, 2,5-Dichroaniline may be used as analytical reference standard for Pigment Red 2.

**J-3.7 Phosphoric Acid —**Analytical grade.

**J-3.8 Diammonium Hydrogen Phosphate —**Analytical grade.

### J.4 PROCEDURE

#### J-4.1 Sample Preparation

Weigh accurately 0.5 g sample in 250 ml capacity flask. Add 20 ml methanol and 60 ml 1N hydrochloric acid. Apply sonication for 5 min in ultrasonic bath at 37±2 °C and subsequently stir it for 25 min at 200 rpm and 37 ± 2 °C. Transfer the content to centrifuge tube and apply centrifuge for 5 min at 3 000 rpm and decant clear layer in 250 ml beaker through filter paper.

Add 30 ml 1N hydrochloric acid in centrifuge tube and stir for 25 min at 200 rpm and 37 ± 2°C. Centrifuge this at 3000 rpm and decant clear layer in 250 ml beaker through filter paper. Wash filter paper with approximately 2 ml 1N hydrochloric acid and combine the filtrate with the first aqueous phase. Adjust pH to 7 using 5N sodium hydroxide solution and transfer to 200 ml volumetric flask. Make up the volume to 200 ml with distilled water. Inject 10 µl in HPLC directly.

#### J-4.2 Standard Preparation

Weigh, to the nearest 0.1 mg, 10 ± 1 mg of each aromatic amine into a 100 ml volumetric flask. Add methanol/water 8:2 (v/v). Place it in an ultrasonic bath for 10 min to ensure complete dissolution to make standard solutions for calibration curve. The stability of the mixed stock standard solution should be checked regularly. It should



 

be stable for up to 6 months when stored in cool and dark place (27°C). Calculate the response factor (RF). Use minimum three points calibration curve.

### J-4.3 Instrument (HPLC) Set Up

**Column** **: 250 mm × 4 mm,** 5 ìm or equivalent HPLC column
**Oven temperature : 40°C**
**Mobile phase**

A :5m mol Diammonium hydrogen phosphate buffered to pH 7 with concentrated phosphoric acid
B : HPLC grade methanol

**Gradient** : 90 percent (v/v) A: 10 percent (v/v) B, in 30 min 5 percent (v/v) A and 95 percent (v/v) B
**Flow rate** : 0.9 ml/min
**Injection volume:** 10 µl or higher depending upon limit of detection.
**Injection volume:** suitable to ensure the limit of detection (LOD) (5–15 µl)
**Run time** **:** 45min
**Flow** **:** 0.3 ml/min
**DAD mode** **:** 240 ± 20 nm
**DAD range** **:** 200 to 800 nm

### J-4.4 Calculation of Response Factor for Each Concentration

$$\text{Response Factor (RF)} = \frac{\text{Area of standard}}{\text{Concentration of standard (ppm)}}$$

### J-4.5 Calculation of Individual Primary Aromatic Amine

$$\text{Individual primary aromatic amine, in ppm} = \frac{\text{Sample are x dilution factor}}{\text{RFx weight of the sample (g)}}$$

**Recalculate to aniline equivalent in case of all amines other than those listed in Annex H.**

## Bibliography

[1] IS 14625: 2015, *Plastics Feeding Bottles*

[2] IS 9845:1998, *Determination of overall migration of constituents of plastics materials and articles intended to come in contact with foodstuffs – method of analysis*

[3] IS 9833:2018, List of Colourants for Use in Plastics in Contact with Foodstuffs and Pharmaceuticals