## **DUS DEAS 985-1**

## DRAFT UGANDA STANDARD

First Edition 2019-mm-dd

# Hermetic storage bags —Specification - Part 1; Woven polypropylene outer bags

Reference number DUS DEAS 985-1: 2019

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This Draft Uganda Standard, DUS DEAS 985-1: 2019, *Hermetic storage bags* — *Specification* – *Part 1; Woven polypropylene outer bags,* is identical with and has been reproduced from an East African Standard, DEAS 985-1: 2019, *Hermetic storage bags* — *Specification* – *Part 1; Woven polypropylene outer bags,* and is being proposed for adoption as a Uganda Standard.

The committee responsible for this document is Technical Committee UNBS/TC 4, Mechanical engineering and metallurgy.

Wherever the words, "East African Standard " appear, they should be replaced by "Uganda Standard."

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ICS 55.120

## **DRAFT EAST AFRICAN STANDARD**

Hermetic storage bags — Specification – Part 1; Woven polypropylene outer bags

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## Foreword

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The committee responsible for this document is Technical Committee EASC/TC 066, Packaging

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# Hermetic storage bags — Specification – Part 1; Woven polypropylene outer bags

#### 1 Scope

This Draft East African Standard specifies the requirements, methods of sampling and test for hermetic bags for storage of dried food commodities, derived products and seeds.

This standard covers hermetic bags whose outer bags are made from woven polypropylene

#### 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 7023, Packaging -- Sacks -- Method of sampling empty sacks for testing

ASTM D 398-13, Determination of water vapour transmission rate (WVTR) -- Gravimetric (dish) method ISO 4591 Plastics -- Film and sheeting -- Determination of average thickness of a sample, and average thickness and yield of a roll, by gravimetric techniques (gravimetric thickness)

ISO 2556, Plastics -- Determination of the gas transmission rate of films and thin sheets under atmospheric pressure -- Manometric method

ISO 7765, Plastic film and sheeting-Determination of Impact resistance by free falling dart method part 2: Instrumented punctured test

ISO 6383-2, Plastic film and sheeting-Determination of tear resistance part 2: Elmendorf-method

ISO 7965-2, Sack-drop test part 2: -Sacks made from thermoplastic flexible film

ISO 22198, Textile Fabrics-Determination of width and length

ISO 7211-5, Textile-woven Fabrics-Construction-methods of analysis-Part 5: Determination of linear density of yearn removed from fabrics.

ISO 3801, Textile-woven Fabrics-Determination of mass per unit length and mass per unit area

ISO 13934-1, Textiles -- Tensile properties of fabrics -- Part 1: Determination of maximum force and

elongation at maximum force using the strip method

ASTM D-3985-95 Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using a Coulometric Sensor

ISO 2062, Textiles -- Yarns from packages -- Determination of single-end breaking force and elongation at break using constant rate of extension (CRE) tester

ASTM F 1927 - 07 Standard Test Method for Determination of Oxygen Gas Transmission Rate, Permeability and Permeance at Controlled Relative Humidity Through Barrier Materials Using a Coulometric Detector

ASTM F1306 – 90, Standard Test Method for Slow Rate Penetration Resistance of Flexible Barrier Films and Laminates

ASTM D882-18, Standard Test Method for Tensile Properties of thin Plastic Sheeting

#### 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1998 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

ISO Online browsing platform: available at <u>http://www.iso.org/obp</u>.

#### 3.1

#### hermetic storage bag

bags with the requisite liner that limits exchange of oxygen, carbon dioxide and water vapour with the environment when sealed

#### 3.2

#### food grade material

packaging material, made of substances which are safe and suitable for the intended use and which will not impart any toxic substances or undesirable odour or flavor to the products.

#### 3.3

#### oxygen transmission rate (OTR)

quantity of oxygen transmitted through unit area of a test specimen in unit time under specified conditions of temperature, humidity and thickness

#### 3.4

#### water vapour transmission rate (WVTR)

quantity of water vapour transmitted through unit area of a test specimen in unit time under specified conditions of temperature, humidity and thickness

#### 3.5

#### dried food commodity

grains (cereals, pulses, nuts) and other dried food products whose moisture content is within acceptable limits as specified in the relevant product standards

#### 3.6

#### derived products

processed products from grains and other dried food commodities

#### 4 Requirements

#### 4.1 General requirements

#### 4.1.1 General

The bag shall be made from woven polypropylene tapes of virgin resin of food grade material.

#### 4.1.2 Stitching thread

The stitching thread shall be made from either polypropylene or polyester fibre.

#### 4.1.3 Edge sealing

All raw edges of the bags shall be heat sealed or hemmed to prevent fraying.

#### 4.1.4 Base Closure

The closure shall be effected either by a turned-over and stitched seam or by bonding. Where the base closure is bonded, the seam shall be effected by applying capping tape over the ends of the sack and securing by means of an adhesive.

#### 4.1.5 Longitudinal Seams

Longitudinal seams shall not be used in the manufacture of hermetic storage bags.

#### 4.2 General requirements for hermetic liner

- 4.2.1 The liner shall be made from virgin polymer resins that are food grade material
- 4.2.2 The joints of the liners shall be heat sealed.

#### 4.3 General requirements for closures of hermetic liners

The liner shall have a closing mechanism to secure and maintain the hermetic condition.

#### 4.4 General requirements for closures of woven polypropylene bag

The bag shall have a closing mechanism to secure and maintain the contents without puncturing the liner.

#### 4.2 Specific requirements

#### 4.2.1 Specific requirements for woven polypropylene bags

Woven Polypropylene bags shall meet the physical requirements as specified in table 1.

S/No.	Characteristic		Requirements	Test method
a)	Degree of	Warp	100	Annex A
	coverage, %, min.	weft	100	
b)	Breaking Force of the	warp	600	ISO 13934-1
	sacking,N, min.	weft	600	
c)	Break strengt	h of the tape	20	ISO 2062
	( yarn),N, min			X
d)	Elongation at break of the tape,%, min		20	ISO 2062
e)	Linear density, denier, min.		780	ISO 7211 Part 5
f)	Mass per unit area of the sacking ,g/m <sup>2</sup> ,min		70	ISO 3801
g)	Stitch density per 10 cm, min.		12	Annex B
h)	Seam strengt	h, N. min.	480	Annex C
i)	Turned-over a	and stitched seam	The turn-over shall be 2 cm minimum and the stitch line shall be $1 \pm 0.3$ cm from the base so formed and shall pass through all the four thicknesses of the fabrics.	Annex B

Table 1 — Specific requirements for polypropylene bags

#### 4.2.2 Hermetic liner

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When tested in accordance with the methods specified in Table 2, the hermetic liner shall comply with the requirements specified therein.

S/No.	Characteristic	Requirements	Test method
a)	Thickness , μm ,min.	65	ISO 4591
b)	OTR, (cc/m²/day), max		ISO 2556
	single liner	50	
	multi liners	250	
c)	WVTR, (g/m²/day),max	10	ISO 2528
d)	Impact resistance, g,min.	350	ISO 7765-2
e)	Puncture resistance, N, min.	50	ISO 6383-2
			ASTM F 1306-90

 Table 2 — Specific requirements for hermetic liner

#### 4.2.3 Specific requirements for fully assembled hermetic storage bag

#### 4.2.3.1 Drop test

#### 4.2.3.1.1 Butt dropping

When tested in accordance with ISO 7965-2 at a height of 1.20m on the bottom and the top of the bag, after each drop, there shall be no rupture or loss of contents.

#### 4.2.3.1.2 Flat dropping

When tested in accordance with ISO 7965-2 at a height of 1.60m twice on one flat face and twice on the opposite flat face., there shall be no rupture or loss of contents.

#### 4.2.3.1.3 Dimension and capacity

When tested in accordance with ISO 22198 the nominal dimensions of the bags shall be as declared subject to a tolerance of  $\pm 2$  cm and capacity as stated.

#### 4.3 Food grade requirements

#### 4.3.1 Overall migration

When tested in accordance with method specified in Annex D, the hermetic storage bag shall also comply with the overall migration limits. 60 mg/kg (max.) of the foodstuff; and for liquid foodstuffs or of simulants, the limit shall be 60 mg/l (max.).

#### 4.3.2 Pigments, colorants and heavy metals

When tested in accordance with Annex E, the hermetic storage bag shall comply with the list and limits of the pigments, colorants and heavy metals specified therein.

#### 5 Packaging and labelling

#### 5.1 Packaging

Hermetic bags shall be packed in materials that prevents it from damage, contamination during normal handling, storage and transportation and the packaging shall be in accordance with relevant environmental regulations of destination country.

#### 5.2 Labelling

#### 5.2.1 Woven polypropylene

The woven polypropylene bag shall be legibly and indelibly marked with the following information on the outer.

- a) manufacturer's name, address and /or registered trade mark;
- b) description of the product, "Hermetic Storage bag".
- c) capacity of the hermetic bag in kg as dried maize equivalent, e.g. 25, 50, 90, 100;
- d) dimensions of the bag;
- e) declaration of the number of liners in the bag;
- f) batch number or code;
- g) instruction for correct use;
- h) instruction for storage and disposal of used bag; and
- i) country of origin.

#### 5.2.2 Liner

The liner shall be legibly and indelibly marked with the following information

- a) manufacturer's name, address and /or registered trade mark;
- b) declaration, "Hermetic liner";
- c) dimensions of the liners;
- d) batch number or code;
- e) country of origin; and
- f) instruction for correct use, storage and disposal of the liner.

#### 5.2.3 Bulk package

The bulk package shall be legibly marked with the following information;

a) manufacturer's name, address and /or registered trade mark;

- b) description of goods, "Hermetic Storage bags";
- c) capacity of the hermetic bags in kg as dried maize equivalent e.g. 25,50,90,100;
- d) the quantities of the bags;
- e) declaration of the number of liners in the bag;
- batch number or code; f)
- g) instruction for storage and disposal of bulk packaging material; and
- h) country of origin.

#### Sampling 6

Sampling of hermetic storage bags shall be done in accordance with ISO 7023.

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## Annex A

#### (normative)

#### Determination of degree of coverage for warp tapes and weft tapes

#### A.1 Procedure

**A1.1** Mark out separately five distinct spaces (areas), each measuring 20 cm x 20 cm, on each sample of wrapping cloth.

A1.2 Count the number of warp tapes within each space (area). Denote this by '*N*.

A1.3 Count also the number of weft tapes within each space (area) and denote this by 'n'.

**A1.4** Remove ten (10) warp tapes form each sample and measure the width of each tape to the nearest 0.10 mm. Calculate the mean warp tape width and denote this by '*a*'.

**A1.5** Remove also ten (10) weft tapes form each sample and measure the width for each tape to the nearest 0.10 mm. Calculate the mean weft tape width and denote the by 'b' mm.

A1.6 Determine the degree of warp coverage '*K*p' and weft covering *K*f as follow:

**A1.6.1**  $Kp = 1/5 (Kp_1 + Kp_2 + Kp_3 + Kp_4 + Kp_5)$ 

where

*K*p = Degree of warp coverage for the sample and

 $Kp_1 Kp_2 \dots Kp_5$  = Degree of warp coverage for each marked space (area).

*K*pt = 0.5 Na

where

t = 1, 2, 3, 4, 5

a = mean warp tape width in mm and

N = Number of warp tapes/20 cm.

**A1.6.2** ( $Kf_1 = 1/5 (Kf_1 + Kf_2 + Kf_3 + Kf_4 + Kf_5)$ )

where

*K*f = Degree of weft coverage for the sample and

 $Kf_1, Kf_2 \dots Kf_5 =$  Degree of weft coverage for each marked space (area).

Kft = 0.5nb

where

t = 1, 2, 3, 4, 5,

n =number of weft tapes/20 cm, and

b = mean tape width in mm.

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#### Annex B

(normative)

#### **Determination of stitch density**

#### **B.1** Determination of stitch density

Count the number of stitches over a length of 10 cm on each side and bottom seams (if any) of each B.1.1 bag.

B.1.2 Space the measurements so that all the seams are adequately assessed.

Calculate the mean reading and report the mean for each sack. B.1.3

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#### Annex C

(normative)

#### Determination of seam strength

#### C.1 Breaking strength of seam

**C.1.1** Cut out two specimens from each bag in the form of a double "T" with 10 cm of seam and 5 cm of sacking as shown in Figure C.1.

All dimensions in centimetres

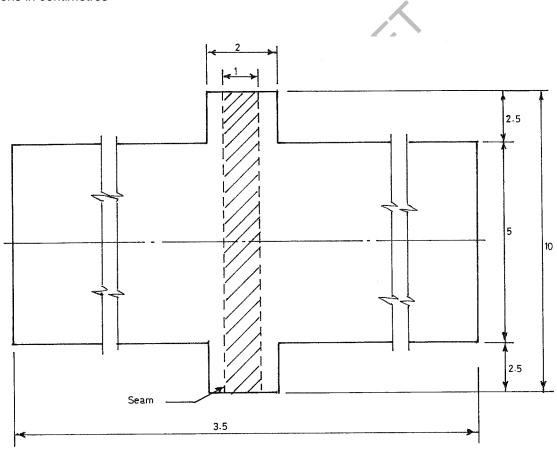


Figure C.1 — size and shape of test-specimen for seam strength

**C.1.2** Determine the average breaking strength of the seam taking 20.0 cm as the nominal gauge length of the strength-tester, with the seam near about the center using a constant-rate-of-traverse of 20 cm per minute.

#### 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.1 Principle

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

#### D.2 Simulants

The determination of migration in simulants is carried out using the simulants listed below:

D.2.1 Simulant 'A' – Distilled water or water of equivalent quality.

**D.2.2** Simulant 'B' – 3 % acetic acid (w/v) in aqueous solution (using the simulant 'A')

**D.2.3** Simulant 'C'' – 10 % ethanol (v/v) in aqueous solution for foodstuffs having alcohol less than 10 % (v/v) (using the simulant 'A')

**D.2.4** Simulant 'C<sup>2</sup>'- 50 % ethanol ( $\nu/\nu$ ) in aqueous solution for foodstuffs having alcohol more than 10 % and less than 50 % ( $\nu/\nu$ ) (using the simulant 'A').

**D.2.5** Simulant 'D' – *n*-heptane – shall be freshly distilled before use.

D.2.6 Simulant 'E' – Rectified olive oil or mixture of synthetic triglycerides or sunflower oil.

#### D.3 Selection of standard test conditions and simulants for different foods

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

SI no	Туре	Description	Example	Simulant
i)	1	Aqueous, non-acidic foods(pH >5) without fat	Honey, mineral water, sugar syrups, skimmed milk, infusions, yeast paste etc.	'A'
ii)	2	Aqueous, non-acidic foods(pH ≤5) without fat	Fruit juices, squashes, fruit chunks or puree or paste, vinegar, jams, jellies, carbonated beverages, lemonade, processed vegetables, rennet, preparations of soup, broths, sauces, RTS beverages etc.	'В'
iii)	3	Alcoholic Beverages: a) Alcohol concentration less than 10 % b) Alcohol concentration above 10 %	Beer and some pharmaceutical syrups Wine brandy, whiskey, arrack and other alcoholic drinks	'C <sup>1'</sup> 'C <sup>1'</sup>
iv)	4	Oils, fats and processed dry foods with surface fat or volatile oil	Vegetable oils, ghee, vanaspati, cocoa butter, lard biscuits, spice powder, snacks and savoury, chocolate, caramels, malted foods, egg powder, tea, coffee powder, confectionery, fried and roasted nuts	ʻD'
V)	5	Non-acidic foods(pH>5) or high fat and having high moisture content	Butter, bread pastry, milk based sweets, ice-cream, moist and fatty confectionery products	'A and D'
vi)	6	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.	'B and D'
vii)	7	Dry processed foods without fat	Cereals and pulses, dehydrated vegetables and fruits, dried yeast, corn flakes, salt, sugar, milled products, barley powder, oats, vermicelli, spaghetti etc.	No end test

**D.4** Table D.2 lists the simulants and tests conditions (time-temperature) for extractability studies to be carried out as above depending on the type of food and conditions of use.

SI No	Conditions of use	Type of food	Water(time- temp)	3% acetic acid (time- temp)	10% alcohol(time- temp)	50% alcohol (time-temp)	<i>n</i> - Heptane( time- temp)
i)	High temperature heat sterilized (retorting)	I, II, IV, V and VI	121°C 2h	121°C 2h	-	-	66 °C for 2h
ii)	Hot filled or pasteurized above 66 °C, below 100 °C	II, IV, V and VI	100°C 2h	100 °C 2h			49 °C and 30 minutes
iii)	Hot filled or pasteurized above 66 °C	I to VI	70°C 2h	70 °C 2h	70 °C 2h	70 °C	38°C and 30 minutes
iv)	Room temperature filled and stored (no thermal treatment in container) and also in refrigerated and frozen conditions	do	40 °C 10 days	40 °C 10 days	40 °C 10 days	40 ℃ 10 days	do

#### Table D.2 — Simulating solvents for different types of food and temperature – Time conditions

NOTE 1 Heptane simulant not to be used on wax lined containers

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

#### D.5 Method 1: For finished containers (within 2 litres capacity) or sealable single/multilayered flexible films (one side exposure)

D.5.1 Apparatus

**D.5.1.1 Electric oven/water bath**, equipped with thermostat to maintain the desired temperature up to  $\pm 1^{\circ}$  accuracy.

D.5.1.2 Electric hot plate, with temperature control regulator.

- **D.5.1.3** Analytical balance with a sensitivity of 0.1 mg.
- D.5.1.4 Glass beakers, Pyrex of 1000 mL capacity or equivalent.
- D.5.1.5 Stainless steel evaporating dish of 100mL capacity.
- D.5.1.6 Stainless steel tongs.

#### **D.5.2 Selection of samples**

Minimum triplicate samples representing the lot/batch have to be selected. Samples in each replicate shall not consist of a number of containers (preformed or converted products) with nearest exposed area of 1000 cm<sup>2</sup>. In the case of films representative sample shall be of sufficient size to convert into 2 pouches of size 125 mm width and 200 mm length (inner dimension excluding seal area) with 1000 cm<sup>2</sup> surface area coming in contact.

#### D.5.3 Preparation of test specimen

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

#### **D.5.4 Simulant quantity**

Equal to nominal filling capacity or at least 1mL/cm<sup>2</sup> 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 the test.

#### D.5.5 Procedure

Fill the container/pouch to their filled capacity with preheated simulant at test temperature and close it. In the 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 stimulant.

## D.5.6 Determination of amount of extractive

Evaporate/distill the contents in Pyrex beaker to about 50 ml - 60ml and transfer into a clean tared stainless steel dish along with 3 washings of Pyrex beaker with small quantity of fresh stimulant and further evaporate the concentrate in the dish to dryness in an oven at 100 °C  $\pm$  5 °C.Cool the dish with extractive in a desiccator for 30 min. and weigh to nearest 0.1 mg till constant weight of residue is obtained. Calculate the extractives in mg/dm<sup>2</sup> 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 with the sample.

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

 $\frac{M}{V}$  ×1000 mg/kg or mg/l or ppm

Where

M = mass of residue in mg minus blank value

A = total surface area in cm<sup>2</sup> exposed in each replicate, and

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

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.

# D.6 Method ii: For larger containers made of single homogenous material above 2 litres capacity

#### D.6.1 Selection of sample

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

#### D.6.2 Test Specimen

Cut 5 pieces each of size 10 cm  $\times$ 10 cm from each container at different places (each piece exposing about 200 cm<sup>2</sup> surface area both sides). In the case of thick material area corresponding to thickness of the sample shall be included.

#### D.6.3 Procedure

Immerse 5 thoroughly cleaned pieces cut from each container into a clean glass container (2-L capacity beaker) containing preconditioned simulant at test temperature such that no two pieces touch each other by placing a 2 to 3 mm diameter glass rod in between the specimens and cover the beaker with glass plate/watch glass and keep the set at specified temperature maintained in oven/waterbath/pressure cooker for the specified duration of time.

After exposure for the specified time, remove the test specimen from the extracted simulant with the help of clean tongs and wash the pieces with a small amount of fresh simulant and combine with the extracted simulant. Blank shall also be carried out without the sample.

#### D.6.4 Determination of amount of extractive

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

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

Ex in ppm =  $M \times TSA \times \frac{1000}{A \times V}$ 

Where

M = mass of residue in mg minus blank value,

A = surface area in cm<sup>2</sup> exposed in each replicate,

TSA = total surface area of the container in cm<sup>2</sup>, and

V = total volume of the container.

NOTE Heptane extractive to be divided by factor of five

# D.7 Method iii: Both side exposure for single homogenous film, which cannot be heat sealed

#### D.7.1 Apparatus

**D.7.1.1 Cylindrical glass jar**, inner dimension of 10 cm diameter and 14 cm height with 1000-mL capacity (for 1-L beaker)

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

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

D.7.1.4 Electric Hot Plate, with temperature regulator.

#### D.7.2 Specimen Size

A film sample of 1000 cm<sup>2</sup> surface area both sides with width not more than 10 cm and an appropriate length to get the required area ( $10 \text{ cm} \times 50 \text{ cm} \times 2 \text{ sides} = 1000 \text{ cm}^2$ ).

#### **D.7.3 Simulant Quantity**

Not less than 1000 ml to immerse the sample completely.

#### D.7.4 Preparation of the specimen

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

#### D.8 Procedure

Fill the cylindrical jar of 1000-mL capacity with the required quantity of preheated simulant at the test temperature. Immerse the test specimen in the simulant completely. Cover with a glass plate and place the jar with sample immersed in simulant at the prescribed temperature for the prescribed length of time. At the end of the test period remove the sample with the help of glass rod and wash the sample with small quantity of fresh simulant and combine with the extractants. Concentrate the extracted simulant 50-60 mL by evaporating on a hot plate under low heat (n-heptane shall be concentrated by distillation).

Transfer the concentrate into a clean tared stainless steel dish along with three washings with small amount of fresh simulant and further evaporate the concentrate to dryness in an oven at  $100 \pm 5^{\circ}$ C. Cool this in a dessicator for 30 min and weigh to nearest 0.1 mg till constant weight of residue is obtained. Calculate the extractive in mg/dm<sup>2</sup>. Blank shall also be carried out without the sample.

Amount of extractive 
$$Ex$$
 =  $\frac{M}{A}$  ×100 mg/dm<sup>2</sup>

Where

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

A =total surface area in cm<sup>2</sup> exposed in each replicate.

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

#### D.9 Method iv: for closures, sealing gaskets, liners and like materials

#### D.9.1 Selection of the Sample

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

#### **D.9.2 Procedure**

Smallest size glass bottles/jars actually being intended for use with closures can be used as containers to contain the simulant. Fill the glass containers to their nominal capacity or 100 ml, whichever is lower with simulant preheated to test temperature and closed tight with the closures/lids lined with the test specimen. Place the closed containers upside down (to ensure the contact of the closures with the simulant) in an oven maintained at test temperature.

After the exposure to the stipulated time, the closures from the containers are opened and the content from each replicate is pooled together in a glass beaker along with the washings of the exposed closures with small amount of fresh simulant. Blank shall also be carried out without the sample.

#### D.9.3 Determination of amount of extractive

Proceed with the determination amount of extractive by method described at D.5.6. Calculate the amount of extractive in ppm for the particular size of container being tested.

Amount of extractive (*Ex*) = 
$$\frac{M}{V}$$
 ×1000 ppm

Where

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

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

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

NOTE 2 Heptane extractives to be divided by factor of five.

#### D.9.4 Method v: materials of articles intended to come into repeated contact with foodstuffs

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

#### D.10 Evaluation of results

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

NOTE Before carrying out the test, make sure that the sample is free from all traces of dust, fats and other impurities. If necessary, it shall be thoroughly wiped with filter paper. The sample shall be handled carefully to avoid any contamination.

#### D.11 Colour migration

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

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#### Annex E (normative)

## List and limits of the pigments, colorants and heavy metals

#### E.1 Principle

**E.1.1** This annex provides a list of permitted pigments and colorants for use in plastics and shall be regarded as safe for use in contact with foodstuffs, pharmaceuticals and drinking water

**E.1.2** Pigments and colorants used shall not show visible bleeding or immigration from the finished product and shall show no signs of instability or degradation during processing.

**E.1.3** Pigments and colorants used shall have a high degree of purity. In particular, if the following impurities are present, these shall not exceed the limits specified below.

- a) Lead, per cent by mass, max. 0.01
- b) Arsenic, per cent by mass, max. 0.005
- c) Mercury, per cent by mass, max. 0.005 (soluble in N/10 HC)
- d) Cadmium, per cent by mass, max. 0.10 do
- e) Zinc, per cent by mass, max. 0.20 do
- f) Selenium, per cent by mass, max. 0.01 do
- g) Barium, per cent by mass, max. 0.01 do
- h) Chromium, per cent by mass, max. 0.025 do
- i) Antimony, per cent by mass, max. 0.025 do
- j) Total aromatic amines, per cent by mass, max. 0.05 do
- **E.1.4** Carbon black, if used shall conform to the following requirements:
- a) Benzene extract 0.1 per cent by mass, max.
- b) 3 Benzpyrene no traces.

# E.2 List of pigments and colorants for use in plastic in contact with foodstuffs, pharmaceuticals and drinking water

#### E.2.1 Organic pigments

SI No	Composition
Yellow	

	o-Nitroaniline-acetoacetanilide
	p-Natroaniline-acetoacetanilide
	4-Chloro-2-nitroaniline-o-chloro-acetoacetanilide
	o-Nitro-p-tuluine-acetoacetanilide
	2:5-Dichloraniline-3-methyl-I-phenyl-5-pyrazolone
	2:4-Dichloraniline( 2 mol)-4:4' -bis-(o-acetyl-aceto-toluidine)
	1-Amino-5-benzamindo-anthraquinone-oxalyl-chloride
	1:1-Diamino-athraquinol-terephthalate
	Bis(2"-anthraqunonyl) -6:6'-dibenzothiazolyle
	Pigment yellow-Benzidine-yellow
Orange	
	o-Nitro-p-anisidine-o-acetyl-acetotoluidine
	2:4-Dinitroanile-2-naphthol
	2-Nitro-4-toluidine-3-methyl-phenyl-5-pyrazolone
	o-Dianisidine-acetylacetoquinilide (2)
	5:5"-Dibenzoylamino-1:1-anthrimide-carbazole
	Napthalene-tetracarboxylic acid-1:2-diamino-benzene (2 mol)
Red	
a)	4-Nitraniline-2-naphthol
b)	2-Nitro-4-toluidine-2-naphthol
c)	2-Anisidine-2-naphthol
d)	5-Nitro-2-toluidine-4-chloro-3-oxy-2-naphthanilide
e)	2-Nitro-4-toluidine-3-oxy-3' -nitro-napthanilide
f)	4-Nitro-2-totuidine-3-oxy-2-napththo-8-o-toluidine
g)	N:N'-diethyl-4-methoxymetanilamide-5-chloro-3-oxy-2', 4'dimethoxy-2-napthanilide
h)	4-Nitro-2-anisidine-3-oxy-N-1-naphthyl-2-naphthamide
i)	1-Naphthylamine-I-naphol-5-sulphonic acid (calcium salt)
j)	2-Amino-naphthalene-1-sulphonic acid-2-naphthoic acid (calcium salt)
k)	6-Amino-m-toluenessulphonic acid-3-oxy-2-naphthoic acid (calcium salt)
I)	6-Amino-4-chloro-m-toluenesulphonic acid-oxy-2 naphthoic acid (calcium salt)
m)	3:3' -Dichlorobenzidine-3-carbothoxy-phenyl-5-pyrazonolone (2 mol)
n)	2-Amino-naphthalene-1-sulphonic acid-3-hydroxy-2-naphthoic acid (calcium salt)
0)	1:2-Dioxyanthranthraquinone (alizarine) calcium ferrous and alumina lakes
p)	Benzoyl-1-amino-4-hydroxyanthraquinone

q)	2-Thionaphthene-2' -acenaphthene-idigo	
r)	1-Amino-2-methoxy-5benylsuphone-oxy-naphtho-m-xylidide	
s)	1-Amino-2-methoxy-5-benzoxy-5-benzoylaniline-2:3-oxynaphtho-4- chloro-2,5-dimethoxyanilide	
t)	Dihydroquinacridone comparable with phthalo-cyanines	
u)	Bis-(1-amino)-(3-chloro-benzene)-bis(2'-oxy-3-naphthalo)-4,4-diamino- 3,3-dichlorodiphenyl	
v)	Bis-(amino)-2-methyl-5-chlorobene)-bis(2'-oxy-3-naphtho)-4'4'- diamino-3-diphenyl	
w)	Bis-(1-amino)-(2-methyl-3-chlorobenzene)-bis(2'-oxy-3-naphtho)-4':4- diaminophenyl	
Violet		
a)	6:6' -Dibromo-isoviolanthrone	7
b)	Dioxazine-2:5di(N-ethylcarbozoyl)-3'-3:6-dichloro-1:4-benzoquinone	
c)	Pigment violet red-quinacridone	
Blue		
a)	N-dihydro-1:2:1':2' anthroquinone-azine (idnanthrone)	
b)	3:3'-dichloro-indanthrene	
c)	Indigo	
d)	Phthalocyanine	
e)	Cobaltic complex of partially suphonated phthalocyanine	
f)	Cupric complex of phthalocyanine	
g)	Sulphonic derivative of cupric phthalocyanine	
Green		
a)	Ferrous complex of alpha-nitroso-beta-naphthol	
b)	Cupric complex of 4-Nitrobenzene-azo-2-naphthol	
c)	Actachlorophthaloyanine of copper	
	Pigment green oxide-chromium	

#### E.2.2 Cellulose Film Dyes

#### E.2.2.1 Yellow

Azine arising from the oxidation of dehydrothio-p-toluidine sulphonic acid orange

#### E.2.2.2 Orange

3-Carboxyl (parasulphophenyl)-5-pyrazone acid benzidine-salicylic acid (sodium salt)

#### E.2.2.3 Pink

Aniline (2 mol)-6:6-bis-1-mino-bis 1-naphthol-3-sulphonic

#### E.2.2.4 Red

Gamma acid-benzidine-salicylic acid

#### E.2.2.5 Blue

O-dinianisidine-8-amino-1-naphthol-5:7-disulphonic acid

#### E.2.2.6 Green

- Successive condensation of cyanogen chloride on 1-animo-4 p-phenylene-diamine-2-anthraquinone a) sulphonic, p-phenylene diamine-p-amino-salicylic acid aniline
- b) P-Nitraniline-8-amino-1-naphthol-3:6-disulphonic-benzidine-salicylic acid c zevilender

#### E.3 Inorganic pigments

#### E.3.1 Metals

- Aluminium a)
- b) Copper
- Silver C)
- d) Gold
- e) Tin
- Platinum and platinum group metals f)

#### E.3.2 Alloys

- a) Bronzes
- b) Brasses

#### E.3.3 White

- Bartytes (barium sulphate) a)
- Whitening (calcium carbonate) b)
- Calcium sulphate (gypsum, plaster of paris) c)
- d) Kaoline
- Titan white (titanium oxide) e)
- Alumina f)
- Aluminium strearate g)
- h) Talc

#### E.3.4 Yellow

- a) Cadmium yellow (cadmium sulphides and selenium-sulphide)
- b) Yellow iron oxide
- c) Stanic sulphide (SnS)

#### E.3.5 Brown

- a) Ferrite of magnesium
- b) Cupric ferrocyanide
- c) Umber
- d) Sienna (natural ferric oxide)

#### E.3.6 Red

- a) Red ferric oxide
- b) Cadmium red, cadmium sulpho-selenium
- c) Double sulphide of cadmium and mercury

#### E.3.7 Blue

- a) Prussian blue (ferric ferrocyanide and turnbulls blue)
- b) Ultramarine blue (complex silicate of aluminium and sodium sulphurated)

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- c) Egyptian blue (double silicate of copper and calcium)
- d) Cobalt blue (cobalt aluminate)

#### E.3.8 Green

- a) Chrome green (insoluble Cr2 O3 oxide)
- b) Aluminate of chrome
- c) Ultramarine green (complex silicate, sulphurated)
- d) Terre verte (complex silicate)

#### E.3.9 Black

- a) Carbon black
- b) Black oxide of iron (natural and artificial magnetite

#### **Bibliography**

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