भारतीय मानक
Indian Standard

IS 1776 : 2024

मुड़ने वाला बॉक्स बोर्ड, अलेपित — विशिष्टि

(दूसरा पुनरीक्षण)

Folding Box Board,Uncoated — Specification

(Second Revision)

ICS 55.040; 85.060

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Paper Based Packaging Materials Sectional Committee, CHD 16

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Paper Based Packaging Materials Sectional Committee had been approved by the Chemical Division Council.

The manufacture of packing cartons from folding box board requires that the board should not only be mechanically sound for the purpose but also possess the qualities necessary for taking good multi-colour and bronze printing. A proper specification for the board is therefore, of importance to manufacturers of printed cartons. This standard will provide useful guidance to carton manufactures as well as the manufacturers of boards.

This standard was first published in 1961 and revised in 1989. In the first revision, Annex A and Annex B of the original standard were deleted and reference was made to the corresponding test methods in IS 1060 (Part 3) : 1969 'Methods of sampling and test for paper and allied products, Part 3' and IS 4006 (Part 1) : 1985 'Methods of test for paper and pulp based packaging materials: Part 1'. Further, SI units were prescribed in place of the existing units. A new requirement of delamination was introduced.

In this revision, the following modifications have been incorporated:

a) The references clause has been updated;

b) Requirement and test method for brightness have been updated;

c) Requirements of substance, thickness and sizes have been modified;

d) Referred standards for the test methods have been updated, as the IS 1060 (Part 1) has been revised and the test methods have been split in various parts and sections covering the different tests; and

e) Amendment No.1 and 2 have been amalgamated.

A scheme for labelling environment friendly products to be known as Eco-Mark was introduced in the standard at the instance of the Ministry of Environment, Forests and Climate Change (MoEF&CC). The Eco-Mark is administered by the Bureau of Indian Standards (BIS).

This standard contains **4.5**, **4.6**, **4.7**, **5** and **7.1** which calls for agreement between the purchaser and the supplier.

The composition of the Committee responsible for the formulation of this standard is given in Annex D.

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 analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

FOLDING BOX BOARD, UNCOATED — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements for folding box board, uncoated, white and coloured, used for multi-colour and bronze printing in line and half-tone screen and subsequent conversion into folding cartons.

2 REFERENCES

The standards listed in Annex A contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 4261 shall apply.

4 REQUIREMENTS

4.1 General

The board shall be uniform in thickness, free from holes, hard spots and lumps, shall lie flat and be dimensionally stable. The printing surface shall be smooth, of even finish, formation, absorbancy and colour. The front liner shall be opaque and free from patchy finish. Both sides of the board shall be clean and free from loosely bound fibres and loading.

4.2 Strength

4.2.1 The board shall be stiff, shall not easily delaminate, and after being properly creased, shall fold neatly through 180° without cracking.

4.2.2 The value of delamination, in terms of internal bond strength, shall be not less than 36 g/cm when tested by the method prescribed in Annex B.

4.3 Slitting and Cutting

Sheets shall be cut cleanly and square to the specified dimensions. Reels shall be evenly wound. All cut edges shall be entirely free from loose fibres and dust.

4.4 The board shall also comply with the requirements given in Table 1.

4.5 Substance

The grammage for folding box board shall be between 140 gsm to 450 gsm. However, the nominal grammage of folding box board other than this shall be as agreed to between the purchaser and the supplier. A tolerance of ± 5 percent for individual test results and ± 3 percent for the average of 10 test results shall be allowed on the nominal grammage, when tested in accordance with IS 1060 (Part 5/Sec 5).

4.6 Thickness

The thickness of folding box board shall be between 150 micron to 900 micron. However, the thickness of folding box board other than these shall be as agreed to between the purchaser and the supplier. A tolerance of ± 5 percent shall be allowed on the average thickness when tested in accordance with IS 1060 (Part 5/Sec 3).

4.7 Sizes

The sizes of folding box board in reels and sheet format shall be as agreed to between the purchaser and the supplier. The sizes of folding box board in reels or sheet format shall not vary by more than 1 mm on the positive side and zero mm on the negative side when tested in accordance with IS 1060 (Part 5/Sec 3).

5 Optional Requirements

When agreed to between the purchaser and the supplier, the board shall also comply with the requirements given in Table 2.

6 Additional Requirements for Eco-Mark

6.1 General Requirements

6.1.1 The product shall conform to the requirements for quality and performance prescribed under **4.1** to **4.7**.

6.1.2 The manufacturer shall produce to BIS, the environmental consent clearance from the concerned State Pollution Control Board as per the provisions of Water (*Prevention and Control of Pollution*) *Act*, 1974 and *Air* (*Prevention and Control of Pollution*) *Act*, 1981 along with the authorization, if required under the *Environment* (*Protection*) *Act*, 1986 and the Rules made thereunder, while applying for ECO-Mark. Additionally, the manufacturers shall also comply with the provisions under *Prevention of Food Adulteration Act*, 1954 and the Rules made thereunder wherever necessary.

6.2 Specific Requirements

6.2.1 The material shall be of the following two types depending on the raw material used in the manufacture:

a) Manufactured from pulp containing not less than 60 percent by mass of pulp made from materials other than bamboo, hardwood, softwood and reed; and

b) Manufactured from pulp made from 100 percent waste paper or agricultural/industrial waste.

6.2.2 The material if used for the packaging of food materials, shall be manufactured from virgin pulp and shall be free from dioxins. Printed surfaces of the paper shall not come into contact with the food and the maximum amounts of contaminants in paper intended to come into contact with food shall not exceed the limits prescribed in Table 3 when tested according to the methods given in Annex C.

7 PACKING AND MARKING

7.1 Packing

The boards shall be securely and suitably packed as agreed to between the purchaser and the supplier.

7.2 Marking

7.2.1 Each package shall be marked with the following particulars:

a) Description of the material;

b) Thickness and substance;

c) Type;

d) Net mass of contents;

e) Batch number;

f) Date of manufacture; and

g) Manufacturer's name and/or recognized trade name.

7.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed there under, and the products may be marked with the Standard Mark.

7.2.3 *Additional Requirements for Eco-Mark*

7.2.3.1 For Eco-Mark, folding box board shall be packed in such packages which shall be recyclable/reusable or biodegradable.

7.2.3.2 The folding box board may display in brief the criteria based on which the product has been labelled as environment friendly.

7.2.3.3 The folding box board shall be sold along with instruction for proper use and mode of safe disposal so as to maximize its performance and minimize wastage.

7.2.3.4 It shall be suitably marked that Eco-Mark label is applicable only to the folding box board if content is not separately covered under the Eco-Mark scheme.

NOTE — It may be stated that the Eco-Mark is applicable to the product or packaging material or both.

8 SAMPLING AND CRITERIA FOR CONFORMITY

8.1 Sampling

The boards shall be sampled in accordance with IS 1060 (Part 1).

8.2 Number of Tests

From each of the packets, selected from the lot (*see* **8.1**), the board shall be taken out at random. These boards shall constitute the sample. The boards selected shall first be tested for general requirements given in **4.1**. One test piece shall be cut, from each board selected and tested for each of the characteristics mentioned in **4.2** and Table 1. A board not meeting the requirements for any one or more characteristics shall be considered as defective.

8.3 Criteria for Conformity

A lot shall be declared as conforming to all the requirements of this specification if the number of defective boards found does not exceed the acceptance number. This acceptance number shall depend upon the size of the sample and shall be zero if the size is less than 13 and one if it is greater than or equal to 13.

Table 1 Requirement for Folding Box Board

(*Clauses* 4.4 *and* 8.2)

Sl No	Characteristic	Requirements	Methods of Tests, Ref to
(1)	(2)	(3)	(4)
i)	Burst index, kPa.m^2/g, *Min*	1.4	1060 (Part 7/Sec 1)
ii)	*p*H (both sides)	5.0 to 6.5	IS 1060 (Part 4/Sec 7)
iii)	Ash, at 525 °C percent by mass, *Max*	15	IS 1060 (Part 4/Sec 2)
iv)	One minute cobb test, *Max*		IS 1060 (Part 5/Sec 4)
	a) Front	25	
	b) Back	Within 30 percent of the front value	
v)	Moisture (as received), percent by mass	6 to 8	IS 1060 (Part 5/Sec 2)
vi)	Brightness of white boards, front side only, *Min*	75	IS 1060 (Part 4/Sec 13)
vii)	Stiffness factor, *Min*	14	IS 1060 (Part 3)
viii)	Wax pick number, *Min*		IS 1060 (Part 3)
	a) Front	8A	
	b) Back	6A	
ix)	Smoothness s/50 ml, *Max*	35	IS 9894

Table 2 Optional Requirements for Folding Box Board

(*Clause* 5)

Sl No.	Characteristic	Requirements	Methods of Tests, Ref to
(1)	(2)	(3)	(4)
i)	Water soluble chlorides (as NaCl), percent by mass, *Max*	0.08	IS 1060 (Part 4/Sec 8)
ii)	Water soluble sulphate (as Na_2So_4), percent by mass, *Max*	0.25	IS 1060 (Part 4/Sec 9)
iii)	Fatty and/or similar acids (as $C_{17}H_{33}COOH$), percent by mass, *Max*	0.25	IS 1060 (Part 2)

Table 3 Limits of Contaminants in Paper

(*Clause* 6.2.2)

Sl No.	**Contaminant**	**Paper Intended to Come into Contact with Dry Food** (mg/kg of paper)	**Paper Intended to Come into Contact with Wet Food and Food with Fatty Surface** (mg/kg of paper)	**Paper for Filtration** (mg/kg of paper)
(1)	(2)	(3)	(4)	(5)
i)	Cadmium (Cd)	—	0.5	0.5
ii)	Chromium (Cr^{6+})	—	0.1	0.1
iii)	Lead (Pb)	—	3.0	3.0
iv)	Mercury (Hg)	—	0.3	0.3
v)	Pentachlorophenol (PCP)	0.05	0.05	0.05
vi)	Polychlorinated biphenyls (PCBs)	2.0	2.0	0.5

ANNEX A

(*Clause* 2)

LIST OF REFERRED STANDARDS

IS No.	*Title*
IS 1060	Methods of sampling and test for paper and allied products
Part 1 : 2022	Test methods for general purpose (*second revision*)
Part 2 : 1960	Methods of sampling and test for paper and allied products: Part II
Part 3 : 1969	Methods of sampling and test for paper and allied products: Part III
Part 4	Methods of test for paper, board and pulps,
Sec 2 : 2018/ ISO 1762 : 2015	Determination of residue (ash) on ignition at 525 °C
Sec 7 : 2024/ ISO 6588-2 : 2021	Determination of *p*H of aqueous extracts — Hot extraction method (*first revision*)
Sec 8 : 2024/ ISO 9197 : 2016	Determination of water-soluble chlorides (*first revision*)
Sec 9 : 2024/ ISO 9198 : 2020	Determination of water-soluble sulfates (*first revision*)
Sec 13 : 2020/ ISO 2470-1 : 2016	Measurement of diffuse blue reflectance factor — Indoor daylight conditions (ISO brightness) (*first revision*)
Part 5	Methods of test for paper and board,
Sec 2 : 2021/ ISO 287 : 2017	Determination of moisture content of a lot — Oven-drying method (*first revision*)
Sec 3 : 2014/ISO 534 : 2011	Determination of thickness, density and specific volume
Sec 4 : 2014/ISO 535 : 1991	Determination of water absorptiveness — Cobb method
Sec 5 : 2021/ ISO 536 : 2019	Determination of grammage (*first revision*)
Part 7/Sec 1 : 2014/ ISO 2759 : 2001	Methods of test for board, Section 1 Determination of bursting strength of board
IS 4261 : 2001	Glossary of terms relating to paper and pulp based packaging materials (*first revision*)
IS 9894 : 1981	Method of test for smoothness/roughness of paper

To access Indian Standards click on the link below:
https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/knowyourstandards/Indian_standards/isdetails/

ANNEX B

(*Clause* 4.2.2)

DETERMINATION OF DELAMINATION VALUE

B-1 SCOPE

The method is measurement of the internal bond strength of paper and paper type materials using tensile testing machine, internal bond strength being defined as the transverse force required to delaminate a material**.**

B-2 APPARATUS

B-2.1 Tensile testing machine, jaw width not less than 25 mm.

B-2.2 Paper Cutter

B-2.3 Pressure or temperature sensitive tape, approx 38 mm wide.

B-3 PREPARATION OF SAMPLE

B-3.1 Cut five test specimens 38 mm wide and approximately 280 mm in length. The length direction of the specimens shall be parallel to the machine direction of the paper.

B-3.2 The samples shall be conditioned and tested as prescribed in the relevant parts of IS 1060 (Part 1).

B-4 PROCEDURE

B-4.1 Cut 2 strips of tape, approximately 150 mm in length. Seal the sample between these two strips. The tape should project beyond the end of the samples by 25 mm in the direction of length. Two pieces of note paper should be placed between two tape ends where there is no sample. This prevents the tape from sticking to itself. Cool, if necessary and cut strip to 25 mm width.

B-4.2 Start separation by hand and mount in jaws of tensile tester, folding than outward for convenience in inserting them. Clamp one of the separated ends in each of the jaws, the unseparated portion remaining at right angles to the direction of movement of the jaws. Keep the sample perpendicular to the direction of movement during the entire test.

NOTES

1 The transverse jaw separation speed shall be 300 mm/min.

2 Run the test long enough to obtain a good average. This may be estimated from a varying readout or obtained from a graph readout.

ANNEX C

(*Clause* 6.2.2)

DETERMINATION OF CHROMIUM, LEAD, MERCURY, CADMIUM, PENTACHLOROPHENOL AND POLYCHLORINATED BIPHENYLS

C-1 DETERMINATION OF CHROMIUM (as Cr^{6+})

C-1.1 Principle

The hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution at a wavelength of 550 nm.

C-1.2 Apparatus

C-1.2.1 *Spectrophotometer* — any spectrophotometer suitable for measurement at a wavelength of about 550 nm or photoelectric absorptionmeter fitted with filters giving maximum transmission near 550 nm.

C-1.2.2 *Shaker* — any shaker suitable for rotating/moving at (30 ± 2) rev/min.

C-1.3 Reagents

C-1.3.1 *Extraction Fluid* — mix 5.7 ml of acetic acid in distilled water

C-1.3.2 *Nitric Acid* — concentrated

C-1.3.3 *Sulphuric Acid*— approximately 0.2 N

C-1.3.4 *Diphenylcarbazide Solution*

Dissolve 0.25 g of diphenylcarbazide in 50 ml acetone. Store in a brown bottle. Discard when solution becomes discoloured.

C-1.3.5 *Stock Chromium Solution*

Dissolve 0.141 g $K_2Cr_2O_7$ in distilled water and dilute to 100 ml. One milliliter of this solution contains 500 µg of chromium (as Cr^{6+}).

C-1.3.6 *Intermediate Chromium Solution* — take 10 ml of stock chromium solution and dilute to 1 000 ml with distilled water. One milliliter of this solution contains 5.00 µg of chromium (as Cr^{6+}).

C-1.3.7 *Standard Chromium Solution* — take 10 ml of intermediate chromium solution and dilute to 1 000 ml with distilled water. One milliliter of this solution contains 0.05 µg of chromium (as Cr^{6+}).

C-1.3.8 *Indicator Paper* — covering the *p*H range 0.5 to 1.5

C-1.4 Procedure

C-1.4.1 *Preparation of Calibration Curve*

Into each of a series of ten 250 ml volumetric flasks, place the quantities of standard chromium solution as indicated below:

Sl No. (1)	*Standard Chromium Solution* ml (2)	*Corresponding to* Cr^{6+} ug (3)
i)	1.0	0.05
ii)	2.0	0.10
iii)	3.0	0.15
iv)	4.0	0.20
v)	5.0	0.25
vi)	6.0	0.30
vii)	7.0	0.35
viii)	8.0	0.40
ix)	9.0	0.45
x)	10.0	0.50

C-1.4.1.1 Add sulphuric acid to adjust the solution *p*H to 1.0 ± 0.3 in each flask and dilute to 100 ml. Add 2.0 ml diphenylcarbazide solution, mix thoroughly and wait for 10 min.

C-1.4.1.2 Carry out the measurement on the spectrophotometer or on a photoelectric colorimeter using appropriate filter with a 1 cm cell at a wavelength of 550 nm. As references use extraction fluid. Correct the absorbance readings of standard solution by subtracting absorbance of a reagent blank carried through the above method.

C-1.4.1.3 Construct a calibration curve by plotting corrected absorbance values against chromium content in microgram per 102 ml.

C-1.4.2 *Determination*

C-1.4.2.1 *Sample preparation*

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-1.4.2.2 *Preparation of test solution*

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for (18 ± 2) h using shaker rotating/moving at (30 ± 2) rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size. Add sufficient amount of aluminium sulphate and filter if any precipitate appears. Transfer the solution quantitatively to a volumetric flask of suitable capacity, dilute to the mark and mix well.

C-1.4.2.3 According to the expected chromium content, take an aliquot portion of the test solution containing 10 µg to 100 µg of chromium to a 100 ml volumetric flask. Adjust the *p*H of the solution to 1.0 ± 0.3 by adding 0.2 N sulphuric acid. Dilute to 100 ml. Add 2 ml diphenylcarbazide solution, mix thoroughly, and wait for 10 min.

C-1.4.2.4 *Photometric measurement*

Carry out the photometric measurements of the test solution according to the methods given in **C-1.4.1.1**.

C-1.4.3 *Calculation*

By means of the calibration curve (*see* **C-1.4.1.3**) determine the quantity of chromium present:

$$\text{Chromium (as Cr}^{6+}\text{), ppm} = \frac{m \times D}{M}$$

where

m = mass, in µg, of chromium determined in the aliquot of the sample solution;

D = ratio of the volume of test solution to the volume of aliquot portion taken for the colour development; and

M = mass, in g, of paper sample taken for testing.

C-2 DETERMINATION OF MERCURY (as Hg)

C-2.1 Principle

The flameless atomic absorption procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapour. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapour passes through a cell positioned in the light path of mercury hallow cathode lamp of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and record.

C-2.2 Apparatus

C-2.2.1 *Atomic Absorption Spectrometer* (AAS) *and Associated Equipment*

Instrument settings recommended by the manufacturer shall be followed. Instruments designed specifically for the measurement of mercury using the cold vapour technique may be substituted for the AAS.

C-2.2.2 *Mercury Vapour Generation Assembly* — Consists of an absorption cell, peristaltic pump, flow meter, aeration tubing and a drying tube containing magnesium perchlorate.

C-2.2.3 *Mercury Hollow Cathode Lamp*

C-2.2.4 *Recorder/Printer/Display Meter* — Any multi-range variable recorder that is compatible with the UV detection system is suitable.

C-2.3 Reagents

C-2.3.1 *Sulphuric Acid* — concentrated

C-2.3.2 *Nitric Acid* — concentrated

C-2.3.3 *Stannous Chloride Solution*

Dissolve 25 g of stannous chloride ($SnCl_2$) in water containing 50 ml of concentrated hydrochloric acid and dilute to 250 ml. If a suspension forms, stir reagent continuously during use.

C-2.3.4 *Sodium Chloride* — *hydroxylamine Sulphate Solution*

Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulphate $(NH_2OH)_2$ H_2SO_4 in distilled water and dilute to 100 ml.

C-2.3.5 *Potassium Permanganate Solution*

Dissolve 5 g of potassium permanganate in distilled water and dilute to 100 ml.

C-2.3.6 *Potassium Persulphate Solution*

Dissolve 5 g of potassium persulphate in distilled water and dilute to 100 ml.

C-2.3.7 *Stock Mercury Solution*

Dissolve 1.354 g of mercuric chloride in about 700 ml of distilled water. Add 10 ml of concentrated nitric acid and make up to 1 000 ml. One milliliter of the solution contains 1 mg of mercury as Hg.

C-2.3.8 *Standard Mercury Solution*

Prepare a series of standard mercury solutions containing 0 µg/l to 5 µg/l by appropriate dilution of stock mercury solution (**C-2.3.7**) with water containing 10 ml of concentrated nitric acid per litre. Prepare standards daily.

NOTE — Use mercury free distilled water for the preparation of reagents and standards.

C-2.4 Procedure

C-2.4.1 *Instrument Operation*

Follow the procedure of the manufacturer's operating manual. Connect the mercury vapour generating assembly as shown in Fig. 1.

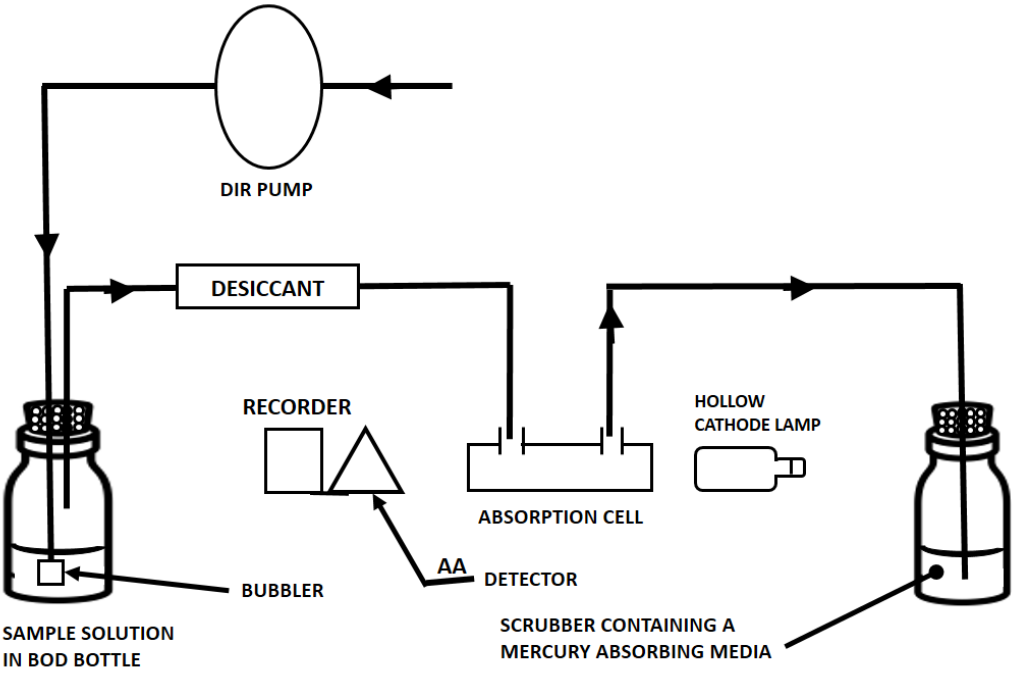

FIG.1 SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR MEASUREMENT OF MERCURY BY COLD VAPOUR ATOMIC ABSORPTION TECHNIQUE

C-2.4.2 *Standardization*

Transfer 100 ml of each of the 1.0 µg/l, 2.0 µg/l and 5.0 µg/l standard mercury solution and a blank of 100 ml water to 300 ml BOD bottles. Add 5 ml of concentrated sulphuric acid and 2.5 ml of concentrated nitric acid to each bottle. Add 15 ml of potassium permanganate solution to each bottle and let stand for at least 15 min. Add 8 ml of potassium persulphate ($K_2S_2O_8$) solution to each bottle and heat for 2 h in a water bath at 95 °C. Cool and add 6 ml of sodium chloride-hydroxylamine sulphate solution to reduce the excess permanganate. After decolourization add 5 ml of stannous chloride solution and attach the bottle immediately to the aeration apparatus forming a closed system. As mercury is volatilized and carried into the absorption cell, absorbance will increase to a maximum within a few seconds. As soon as recorder returns approximately to the base line, remove stopper holding the aeration frit from the reaction bottle and replace with a bottle containing distilled water. Flush the system for a few seconds and run the next standard in the same manner. Construct a standard calibration curve by plotting absorbance (peak height) versus mercury concentration in µg.

C-2.4.3 *Determination*

C-2.4.3.1 *Sample preparation*

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-2.4.3.2 *Preparation of test solution*

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for (18 ± 2) h using shaker rotating/moving at (30 ± 2) rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size. Transfer the solution quantitatively to a volumetric flask of suitable capacity, dilute to the mark and mix.

C-2.4.3.3 According to the expected mercury content, take an aliquot portion of the test solution containing not more than 5 µg/l of mercury to a 300 ml BOD bottle and treat as in C-2.4.2.

C-2.5 Calculation

Determine peak height of sample from recorder chart, read mercury value from standard curve and determine the mercury content of the sample using the following formula:

$$\text{Mercury (as Hg), ppm} = \frac{C \times V}{M \times 1\,000}$$

where

C = concentration of mercury from the calibration curve;

V = volume, in ml, of test solution prepared; and

M = mass, in g, of paper sample taken for testing.

C-3 DETERMINATION OF LEAD (as Pb)

C-3.1 Principle

The lead content of the sample is determined by electrothermal atomic absorption spectrometric method.

C-3.2 Apparatus

C-3.2.1 *Atomic Absorption Spectrometer* — with graphite oven technique in place of conventional burner assembly.

C-3.2.2 *Lead Hollow-Cathode Lamp or Multielement Hollow-Cathode Lamp* — for use at 283.3 nm

C-3.2.3 *Hot Plate*

C-3.3 Reagents

C-3.3.1 *Nitric Acid* — concentrated

C-3.3.2 *Nitric Acid* — 1 : 1

C-3.3.3 *Dilute Nitric Acid* — 1 : 499

C-3.3.4 *Stock Lead Solution*

Dissolve 1.599 9 g of $Pb(NO_3)_2$ in a mixture of 10 ml of concentrated HNO_3 and 100 ml of water and dilute to 1 litre. One milliliter of this solution contains 1.0 mg of lead (as Pb).

C-3.3.5 *Intermediate Lead Solution*

C-3.3.6 *Standard Lead Solution*

Dilute 100 ml of intermediate lead solution to 1 litre with dilute nitric acid (1 : 499). One milliliter of this solution contains 0.1 mg of lead (as Pb).

C-3.4 Procedure

C-3.4.1 *Sample Preparation*

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-3.4.2 *Preparation of Test Solution*

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for (18 ± 2) h using shaker rotating/ moving at (30 ± 2) rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size.

C-3.4.3 Transfer the extract to a 250 ml conical flask. Add 5 ml concentrate nitric acid and a few boiling chips or glass beads. Slowly evaporate on a hot plate to about 10 ml to 20 ml. Continue heating and adding concentrated nitric acid until digestion is complete. Wash down with water and then filter if necessary. Quantitatively transfer filtrate to a 100 ml volumetric flask, dilute to the mark and mix thoroughly.

C-3.4.4 Inject a measured portion of the digested solution into the graphite oven. Dry, char and atomize according to the preset programme. Measure the absorbance.

C-3.4.5 Prepare a reagent blank and sufficient standards containing 5.0 mg/l, 7.5 mg/l and 10.0 mg/l of lead by diluting suitable volume of the standard lead solution with nitric acid (1 : 499) and repeat as above (C-3.4.3). Inject a suitable portion of each standard solution in order of increasing concentration. Analyze each standard solution and measure the absorbances.

C-3.4.6 *Calculation*

Construct a standard calibration graph by plotting the absorbance versus mg of lead concentration of each standard. Read the concentration of the sample from the graph and determine the lead content of the sample from the calibration graph using the following formula:

$$\text{Lead (as Pb), ppm} = \frac{C \times F \times 100}{M}$$

where

C = concentration of lead from the calibration curve;

F = dilution factor; and

M = mass, in g, of paper sample taken for testing.

C-4 DETERMINATION OF CADMIUM (as Cd)

C-4.1 Principle

The cadmium content of the sample is determined by electrothermal atomic absorption spectrometric method.

C-4.2 Apparatus

C-4.2.1 *Atomic Absorption Spectrometer* — with graphite oven technique in place of conventional burner assembly

C-4.2.2 *Cadmium Hollow-Cathode Lamp or Multielement Hollow-Cathode Lamp* — for use at 228.8 nm

C-4.2.3 *Hot Plate*

C-4.3 Reagents

C-4.3.1 *Nitric Acid* — concentrated

C-4.3.2 *Nitric Acid* — 1 : 1

C-4.3.3 *Dilute Nitric Acid* — 1 : 499

C-4.3.4 *Stock Cadmium Solution*

Dissolve 1.0 g of pure cadmium metal in minimum quantity of concentrated nitric acid and dilute to 1 litre with distilled water. One millilitre of this solution contains 1 mg of cadmium (as Cd).

C-4.3.5 *Intermediate Cadmium Solution*

Add 1 ml of concentrated nitric acid to 50 ml of stock solution and dilute to 1 litre with distilled water. One milliliter of this solution contains 50 µg of cadmium (as Cd).

C-4.3.6 *Standard Cadmium Solution*

To 10 ml of cadmium intermediate solution add 1 ml of concentrated nitric acid and dilute to 1 litre with distilled water. One milliliter of this solution contains 0.5 µg of cadmium (as Cd).

C-4.4 Procedure

C-4.4.1 *Sample Preparation*

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-4.4.2 *Preparation of Test Solution*

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for (18 ± 2) h using shaker rotating/moving at (30 ± 2) rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size.

C-4.4.3 Transfer the extract to a 250 ml conical flask. Add 5 ml concentrate nitric acid and a few boiling chips or glass beads. Slowly evaporate on a hot plate to about 10 ml to 20 ml. Continue heating and adding concentrated nitric acid until digestion is complete. Wash down with distilled water and then filter if necessary. Quantitatively transfer filtrate to a 100 ml volumetric flask, dilute to the mark and mix thoroughly.

C-4.4.4 Inject a measured portion of the digested solution into the graphite oven. Dry, char and atomize according to the preset programme. Measure the absorbance.

C-4.4.5 Prepare a reagent blank and sufficient standards containing 5.0 mg/l, 7.5 mg/l and 10.0 mg/l of cadmium by diluting suitable volume of the standard cadmium solution with nitric acid (1 : 499). Inject a suitable portion of each standard solution in order of increasing concentration. Analyze each standard solution and measure the absorbances.

C-4.4.6 *Calculation*

Construct a standard calibration graph by plotting the absorbance versus mg of cadmium concentration of each standard. Read the concentration of the sample from the graph and determine the cadmium content of the sample from the calibration graph using the following formula:

$$\text{Cadmium (as Cd), ppm} = \frac{\mathrm{C} \times \mathrm{F} \times 100}{M}$$

where

C = concentration of cadmium from the calibration curve;

F = dilution factor; and

M = mass, in g, of paper sample taken for testing.

C-5 DETERMINATION OF PENTACHLOROPHENOL (PCP)

C-5.1 Principle

PCP is extracted with acetone by Soxhlet extraction. Acetone extract is evaporated to dryness and subjected to acetylation. The acetylated PCP is determined quantitatively by gas chromatograph-electroncapturedetector (GC-ECD).

C-5.2 Apparatus

C-5.2.1 *Round Bottom Flask* — 250 ml

C-5.2.2 *Soxhlet Extractor*

C-5.2.3 *Water Bath*

C-5.2.4 *Separating Funnels* — 60 ml and 100 ml

C-5.2.5 *Injection Syringes* — 1 µl, 5 µl and 10 µl

C-5.2.6 *Gas Chromatograph with ECD* — capillary columns

C-5.2.7 *Glass Columns* — length 20 cm, internal diameter 12 mm filled with 5 cm of silica gel having particle size 63 µm × 200 µm.

C-5.3 Reagents

C-5.3.1 *Acetone*

C-5.3.2 n-*Hexane*

C-5.3.3 *Acetic Anhydride*

C-5.3.4 *Sodium Sulphate Anhydrous*

C-5.3.5 *PCP Stock Solution*

Dissolve 10 mg of pentachlorophenol in 100 ml of acetone. One millilitre of this solution contains 0.1 mg of pentachlorophenol.

C-5.3.6 *PCP Standard Solution*

Dilute 10 ml of stock solution with acetone to 100 ml. One millilitre of this solution contains 0.01 mg of pentachlorophenol.

C-5.3.7 *Internal Standard Stock Solution*

Dissolve 1 g of 2,4 dibromophenol in 1 liter of acetone. One millilitre of this solution contains 1 mg of dibromophenol.

C-5.3.8 *Internal Standard Solution*

Dilute 1 ml of the stock solution (C-5.3.7) with acetone to 100 ml. One millilitre of this solution contains 10 µg of dibromophenol.

C-5.4 Procedure

C-5.4.1 Determine the moisture content of the sample as given in IS 1060 (Part 5/Sec 2).

C-5.4.2 Weigh accurately about 1 g of the paper sample pieces up to two decimal places and put into a thimble. Extract with about 150 ml of acetone by Soxhlet extraction for 6 h. Filter the acetone extract, dry over anhydrous sodium sulphate, and evaporate under vacuum to a small volume approximately to 5 ml and cool.

C-5.4.3 *Clean Up*

Transfer the extract (C-5.4.2) to the silica gel packed coloumn and elute with about 25 ml of *n*-hexane at the rate of 2 ml/min. Collect the eluent in a flask. Dry over anhydrous sodium sulphate and evaporate nearly to dryness.

C-5.4.4 Treat the residue (C-5.4.3) with 1 ml of acetic anhydride, and heat on a water bath for about 30 min. Remove the flask from the water bath and cool the acetylated product. Transfer the content in a separating funnel and add 10 ml of hexane and 5 ml distilled water. Shake well for 2 min and let the layers be separated. Collect the hexane layer, dry over anhydrous sodium sulphate and evaporate nearly to dryness. Cool for at least 10 min, add 1 ml of internal standard solution and adjust the volume to 5.0 ml with *n*-hexane.

C-5.4.5 Inject 2 µl of the solution into the gas chromatograph. Record the peak size in area and peak height units. If peak response exceeds linear range of the system, dilute the concentration of the extract and reanalyze.

C-5.4.6 *Calibration*

Prepare three calibration standards from the PCP standard solutions. Add 1 ml of internal standard solution and follow the steps as above (C-5.4.3 to C-5.4.5). Tabulate peak height or area responses against calculated equivalent mass of underivatized pentachlorophenol injected. Prepare a calibration curve.

C-5.5 Calculation

Determine the PCP content of the sample from the calibration graph using the following formula:

$$\text{PCP content, mg/kg on dry matter} = \frac{A \times B \times V_i \times 100}{M \times C \times V_t \times (100 - \mathrm{X})}$$

where

A = PCP content, in µg, from the graph;

B = total volume, in ml, of hexane extract before derivatization;

V_i = volume, in ml, of eluent injected;

M = mass, in g, of the paper sample taken for testing;

C = volume, in ml, of hexane extract carried through derivatization;

V_t = volume, in ml, of total eluent; and

X = moisture content, percent by mass.

C-6 DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)

C-6.1 Principle

PCB is extracted with boiling ethanolic potassium hydroxide solution. An aliquot of the extract is mixed with distilled water and subjected to extraction with hexane. The PCB content is determined quantitatively by GC-ECD by comparing the pattern of the peaks with the pattern of a suitable technical PCB.

C-6.2 Apparatus

C-6.2.1 *Erlenmeyer Flask* — 200 ml

C-6.2.2 *Water Bath*

C-6.2.3 *Separating Funnels* — 60 ml and 100 ml

C-6.2.4 *Injection Syringes* — 1 µl, 5 µl and 10 µl

C-6.2.5 *Gas Chromatograph with ECD* — capillary columns

C-6.2.6 *Glass Columns* — length 20 cm, internal diameter 12 mm filled with 5 cm of silica gel having particle size 63 µm × 200 µm.

C-6.3 Reagents

C-6.3.1 *Ethanolic Potassium Hydroxide Solution*

Prepare 1 N ethanolic potassium hydroxide solution by dissolving the required amount of potassium hydroxide in absolute ethanol that has been purified as follows:

Dissolve 1.5 g of silver nitrate in 3 ml of water and add it to 1 litre of alcohol. Dissolve 3 g of potassium hydroxide in the smallest amount of hot distilled water, cool, and add it to the silver nitrate solution. Shake thoroughly, allow the solution to stand for at least 24 h, filter and distill.

NOTE — Absolute alcohol denatured with 10 percent by volume of methanol may also be used.

C-6.3.2 n-*Hexane*

C-6.3.3 *Sodium Sulphate Anhydrous*

C-6.3.4 *PCB Stock Solution*

Dissolve 10 mg of any PCB technical in 100 ml of hexane. One milliliter of this solution contains 0.1 mg of PCB.

C-6.3.5 *PCB Standard Solution*

Dilute 10 ml of stock solution with hexane to 100 ml. One milliliter of this solution contains 0.01 mg of PCB.

C-6.3.6 *Internal Standard Stock Solution*

Dissolve 1 g of 2,4-dibromophenol in 1 litre of *n*-hexane. One milliliter of this solution contains 1 mg of dibromophenol.

C-6.3.7 *Internal Standard Solution*

Dilute 1 ml of the stock solution (C-6.3.6) with hexane to 100 ml. One milliliter of this solution contains 10 µg of dibromophenol.

C-6.4 Procedure

C-6.4.1 Determine the moisture content of the sample as given in IS 1060 (Part 5/Sec 2).

C-6.4.2 Weigh accurately about 1 g of the paper sample pieces up to two decimal places and take into a 200 ml Erlenmeyer flask. Add 50 ml of ethanolic potassium hydroxide solution and heat under reflux condenser for 4 h, filter and dilute to the mark with alcohol in a 250 ml volumetric flask. Take an aliquot portion of the test solution in a separating funnel, add sufficient distilled water and 15 ml hexane. Shake well for 5 min and wait for 15 min. Collect the hexane phase.

C-6.4.3 Filter the hexane extract. Dry the filtrate over anhydrous sodium sulphate, and evaporate under vacuum to approximately 5 ml. Take the residue for clean-up.

C-6.4.4 *Clean Up*

Transfer the hexane extract (C-6.4.3) to the silica gel packed column and elute with about 25 ml of *n*-hexane at the rate of 2 ml/min. Collect the eluent in a flask and evaporate to a small volume. Add 1 ml of internal standard solution and made up to 10 ml with hexane.

C-6.4.5 Inject 2 µl of the solution to the gas chromatograph. From the peaks obtained PCB and Internal Standard are identified by their retention times as well as relative retention time.

C-6.4.6 *Calibration*

Prepare three calibration standards from the PCB standard solutions and follow the steps as above (**C-6.4.3** to **C-6.4.5**). Tabulate peak height or area responses against calculated equivalent mass of underivatized pentachlorophenol injected. Prepare a calibration curve.

C-6.5 Calculation

Determine the PCB content of the sample from the calibration graph using the following formula:

$$\text{PCB content, mg/kg on dry matter} = \frac{A \times V_i \times 100}{M \times V_t \times (100 - \mathrm{X})}$$

where

A = PCB content, in µg, from the graph;

V_i = volume, in ml, of eluent injected;

M = mass, in g, of the paper sample taken for testing;

V_t = volume, in ml, of total eluent; and

X = moisture content, percent by mass.

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Paper Based Packaging Materials Sectional Committee, CHD 16

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Indian Institute of Packaging, New Delhi	Prof (Dr)Tanweer Alam **(*Chairperson*)**
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