भारतीय मानक **IS 8460 : 2023**

Indian Standard

रैपिंग टिश्यू पेपर — विशिष्टि

(पहला पुनरीक्षण)

Wrapping Tissue Paper — Specification

(First Revision)

ICS 85.080

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Paper Based Packaging Materials Sectional Committee, CHD 16

FOREWORD

This Indian Standard 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.

This standard was first published in 1977. Tissue papers are used as wrapping and packaging papers in a number of ways. As initial wrappers of merchandise and for giving cushioning effect, they may be of lighter grammage (16 to 20 g/m^2), while for some other uses the grammage is in the range of 20 g/m^2 to 25 g/m^2. These papers may be white or coloured and are made from bleached chemical pulps. For wrapping silverware and ornaments or similar other metallic items, the paper shall be tested for its staining and tarnishing property with regard to the particular metal.

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 & Climate Change (MoEF&CC). The ECO Mark is administered by the Bureau of Indian Standards (BIS).

In this first revision, requirement of moisture has been incorporated considering that binding property of the wrapping tissue paper depends on the moisture content of the paper. Further, higher cobb value will cause sticking of tissue paper layers, hence $Cobb_{60}$ value has also been included. The amendments issued so far to the standard have been amalgamated. In addition, editorial changes have also been done to bring the standard in the latest style and format of Indian Standards

The composition of technical committee responsible for formulation of this standard is listed 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

WRAPPING TISSUE PAPER — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements, methods of sampling and test for tissue paper used for wrapping and packaging purposes.

2 REFERENCES

2.1 The standards listed in Annex A contain provisions which through reference in this text, constitute provisions of and necessary adjuncts to 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 editions of the standards indicated in Annex A.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 4661 (Part 1 to 5) shall apply.

4 TYPES

4.1 Wrapping tissue paper shall be of the following two types:

a) Type A — Ordinary tissue; and
b) Type B — Neutral (non-tarnish) tissue.

5 REQUIREMENTS

5.1 Grammage

The grammage of wrapping tissue paper shall be as prescribed by the purchaser when tested as per IS 1060 (part 5/sec 5). It shall, however, not exceed 25 g/m^2 including the permissible tolerance of ± 1.5 g/m^2 on grammage.

5.2 Furnish

When tested in accordance with 10 of IS 1060 (Part I) the paper made from bleached chemical pulp shall be free from ground wood pulp.

5.3 Finish

The surface shall have an even finish on both sides (M.F.) or machine glazed (M.G.) on one side. The wrapping tissue paper shall be generally free from any visible imperfections, such as holes that may affect its serviceability.

5.4 The wrapping tissue paper shall also comply with the requirements given in Table 1.

Table 1 Requirements for Wrapping Tissue Paper
(*Clause* 5.4)

Sl No.	Characteristic	Requirement		Method of Test
		Type A	Type B	
(1)	(2)	(3)	(4)	(5)
i)	Burst index, kPa.m^2/g, *Min*	1.5	1.5	IS 1060 (Part 6/Sec 2)
ii)	Tear index, mN.m^2/g, *Min*			IS 1060 (Part 6/sec 1)
	CD	5.5	5.5	
	MD	4.5	4.5	
iii)	Tensile index, Nm/g, *Min*			IS 1060 (Part 5/Sec 6)
	CD	22	22	
	MD	25	25	
iv)	*p*H	-	6.5-7.5	IS 1060 (Part 4/Sec 7)
v)	Moisture content, Percent, *Max*	8	8	
vi)	Cobb$_{60,}$ g/ m^2, *Max,*			IS 1060 (Part 5/Sec 4)
	Top side	40	40	
	Bottom side	40	40	
vii)	Water soluble chlorides (as NaCl), percent by mass, *Max*	-	0.1	IS 1060 (Part 4/Sec8)
viii)	Water soluble sulphates (as Na_2SO_4), percent by mass, *Max*	-	0.25	IS 1060 (Part 4/Sec 9)

5.5 Type B papers (non-tarnish) shall also be tested for tarnish resistance in accordance with the procedure mentioned in Annex B and shall not leave any stain on the silver or any other metal plates used.

5.6 Size and Tolerance on Size

Wrapping tissue paper shall be in the form of either rolls or sheets as agreed to between the purchaser and the supplier. The sizes and tolerance on sizes shall be in accordance with IS 1064 unless otherwise agreed to between the purchaser and the supplier.

5.7 Additional Requirements for ECO Mark

5.7.1 *General Requirements*

5.7.1.1 The product shall conform to the requirements for quality and performance prescribed under **5.1** to **5.6**.

5.7.1.2 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.

5.7.2 *Specific Requirements*

5.7.2.1 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 80 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.

5.7.2.2 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 2 when tested according to the methods given in Annex C.

Table 2 Maximum Permissible Limits of Contaminants in Paper
(*Clause* 5.7.2.2)

Sl No. (1)	**Contaminant** (2)	**Paper Intended to Come into Contact with Dry Food** (mg/kg of paper) (3)	**Paper Intended to Come into Contact with Wet Food and Food with Fatty Surface** (mg/kg of paper) (4)	**Paper for Filtration** (mg/kg of paper) (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

6 PACKING AND MARKING

6.1 Packing

6.1.1 The wrapping tissue paper shall be packed as agreed to between the purchaser and the supplier.

6.1.2 For ECO Mark, packing/wrapping paper shall be packed in such packages which shall be recyclable/reusable or biodegradable.

6.2 Marking

6.2.1 Each package and roll shall be marked with the following information:

a) Description, type and grammage of the material;
b) Size of paper;
c) Mass in kg per ream of 500 sheets including wrapping paper; and
d) Lot number
e) Month and year of manufacture; and
f) Manufacturer's name and trade-mark, if any.

6.2.2 *BIS Certification Marking* — Each package may also be marked with the Standard Mark.

6.2.2.1 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.

6.2.3 *Additional Requirements for ECO Mark* — For ECO Mark, following additional information may also be suitably marked on the container/ package:

a) The criteria base on which the packing/wrapping paper has been labelled as environment friendly a with ECO Mark.

b) The packing/wrapping paper shall be sold along with instructions for proper use and mode of safe disposal so as to maximize its performance and minimize wastage.

c) It shall be marked that the ECO-Mark is applicable to the packing/wrapping paper 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.

7 SAMPLING AND CRITERIA FOR CONFORMITY

7.1 Sampling

Representative samples of wrapping tissue paper shall be drawn as prescribed in **4** of IS 1060 (Part 1).

7.2 Number of Tests

7.2.1 Each of the packets or rolls selected from the lot shall be examined for size according to 5.6. From each of these packets or rolls, a test sample of the quantity sufficient to carry out all the tests given in the specification, shall be taken and examined for general requirements given in **5.3**. The test sample having satisfied general requirements shall be tested for grammage, burst, tear and breaking length. A test sample failing to meet any of these requirements shall be considered as defective.

NOTE — For grammage five test specimens shall be cut from different places and average of the five test results shall be reported.

7.2.2 For furnish, *p*H, water soluble chlorides and water soluble sulphates, a composite sample weighing about 15 g shall be prepared and tested for each of these characteristics.

7.3 Criteria for Conformity

7.3.1 The lot shall be declared as conforming to the requirements for grammage, burst, tear and breaking length if the number of test samples found defective is less than or equal to the acceptance number. This acceptance number shall depend upon the size of the sample and shall be equal to 0 if the sample size is less than 13. It shall be equal to 1, if the sample size is greater than or equal to 13.

7.3.2 The lot shall be deemed as conforming to the requirements of furnish, *p*H, water soluble chlorides and water soluble sulphates, if all the test results on the composite sample meet the relevant requirements.

7.3.3 The lot shall be declared as conforming to the requirements of the specification if conditions in **7.3.1** and **7.3.2** are satisfied.

ANNEX A
(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
IS 1060	Methods of sampling and test for paper and allied products:
(Part 1) : 2022	Test methods for general purpose
(Part 4)	Methods of test for paper, board and pulp,
(Sec 7) : 2018	Determination of *p*H of aqueous extracts — Hot extraction method
(Sec 8) : 2014	Determination of water soluble chlorides
(Sec 9) : 2014	Determination of water soluble sulfates
(Part 5)	Methods of test for paper and board,
(Sec 4) : 2014	Determination of water absorptiveness — Cobb method
(Sec 5) : 2021	Determination of grammage
(Sec 6) : 2014	Determination of tensile properties — Constant rate of elongation method (20 mm/min)

IS No.	*Title*
(Part 6)	Methods of test for paper
(Sec 1) : 2014	Determination of tearing resistance — Elmendorf method
(Sec 2) : 2014	Determination of bursting strength of paper
IS 1064 : 1980	Specification for Paper sizes
IS 4661	Paper, board, pulps and related terms — Vocabulary:
(Part 1) : 2022	Alphabetical index
(Part 2) : 2022	Pulping terminilogy
(Part 3): 2022	Paper making terminilogy
(Part 4): 2022	Paper and board grades and converted products
(Part 5): 2022	Properties of pulp, paper and board

ANNEX B

(*Clause* 5.5)

METHOD OF TEST FOR TARNISH RESISTANCE OF PAPER

B- 1 APPARATUS

B-1.1 Metal Plates

Two flat plates 150 mm x 150 mm cut from commercial rolled sheet brass 6·35 thick, plated on one or both sides with pure silver to a thickness of about 0·25 mm, having a smooth and regular surface.

B-2 PROCEDURE

B-2.1 Thoroughly clean the silver surface of each plate and polish it to a high lustre free from grease, then rinse the plate with alcohol followed by either and then dry. Cut a test piece 42 sq cm in size and hold it by means of paper clips or forceps. Wet it with distilled water as uniformly as possible so that the paper is saturated but not dripping wet. Place the water saturated test piece on the polished silver surface of one plate taking care to have as few wrinkles or air bubbles as possible. Then lay the other plate with silver side on the paper. Place the test assembly in the air-oven and adjust the temperature so that the paper will dry completely in not less than 3 hours (65 °C to 75 °C is usually sufficient). When the paper is dry, examine the silvered surface of each plate for evidence of tarnishing or staining. Note the colour and character of any stain found and the number of tarnished or stained areas.

B-2.2 The method may also be used where wrapping paper is to be tested for its staining or corrosion properties with respect to other metals, such as steel, copper and nickel. In such cases it is necessary to use plates of the particular metal or alloy in question. In case of the metal that would rust like that of steel, the paper shall not be wetted; it would be necessary instead to heat it in an atmosphere of high humidity.

ANNEX C

(*Clause* 5.7.2.2 *and Table* 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 wave length 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 absorption meter fitted with filters giving maximum transmission near 550 nm.

C-1.2.2 *Shaker*

Any shaker suitable for rotating/moving at 30 ± 2 revolution/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 ml 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 ml 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 ml 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.	*Standard Chromium Solution* ml	*Corresponding to* Cr^{6+} µg
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 minutes.

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 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 minutes.

C-1.4.2.4 *Photometric measuremen* — 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:

Chromium (as Cr6+), ppm = $m \times DM$

where

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

M = Mass of paper sample taken for testing, g; and

D = Ratio of the volume of test solution to the volume of aliquot portion taken for the colour development.

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 ontinuously 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 millilitre 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 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**.

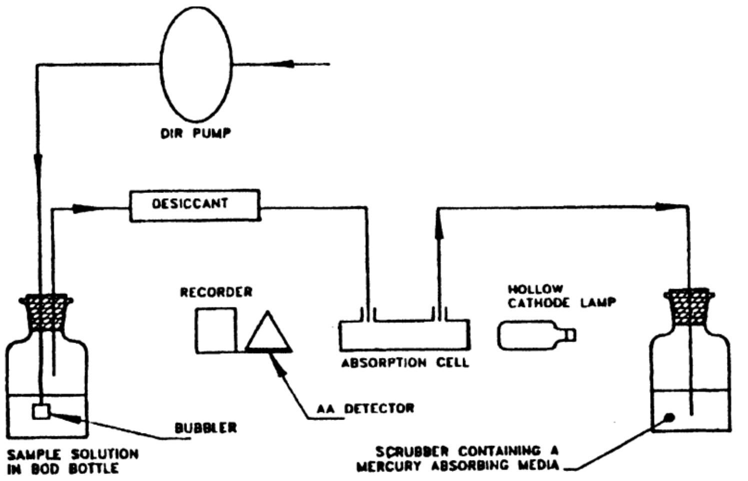

FIG.1 SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR MEASUREMENT OF MERCURY BY COLD VAPOUR ATOMIC ABSORPTION TECHNIQUE

C-2.4.2 *Standardization* — Transfer 100-ml of each of the 1.0, 2.0 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 minutes. 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 volatilised 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 h ± 2 h using shaker rotating/moving at 30 rev/min ± 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:

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

where

C = concentration of mercury from the calibration curve;

V = volume of test solution prepared, ml; and

M = mass of paper sample taken for testing; g.

C-3 DETERMINATION OF LEAD (as Pb)

C-3.1 Principle

The lead content of the sample is determined by electro thermal 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 NO_3 and 100 ml of water and dilute to 1 litre. One ml 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 h ± 2 h using shaker rotating/ moving at 30 rev/min ± 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, 7.5 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 :

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

where

C = concentration of lead from the calibration curve;

F = dilution factor; and

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

C-4 DETERMINATION OF CADMIUM (as Cd)

C-4.1 Principle — The cadmium content of the sample is determined by electro thermal 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 (*see* IS 264).

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 ml 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 ml 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 h ± 2 h using shaker rotating/moving at 30 rev/min ± 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, 7.5 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 :

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

where

C = concentration of cadmium from the calibration curve;

F = dilution factor; and

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

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-Electron Capture Detector (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 l 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 **9** of IS 1060 (Part I).

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 minutes 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 minutes, 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:

PCP content, mg/kg on dry matter

$$= \frac{A \times B \times V_i \times 100}{M \times C \times V_t \times 100 - X}$$

where

A = PCP content in µg from the graph;
B = total volume of hexane extract before derivatization, ml;
V_i = volume of eluent injected, ml;
M = mass of the paper sample taken for testing, g;
C = volume of hexane extract carried through derivatization, ml;
V_t = volume of total eluent, ml; 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 one 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 ml 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 ml 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 ml 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 ml 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 **9** of IS 1060 (Part 1).

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 minutes and wait for 15 minutes. 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:

PCB content, mg/kg on dry matter

$$= \frac{A \times V_i \times 100}{M \times V_t \times (100 - X)}$$

where

A = PCB content in µg from the graph;

V_i = volume of eluent injected, ml;

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

V_t = volume of total eluent, ml; and

X = moisture content, percent by mass.

ANNEX D
(*Foreword*)

COMMITTEE COMPOSITION
Paper Based Packaging Materials Sectional Committee, CHD 16

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