भारतीय मानक

Indian Standard

IS 16983 : 2024

सफ़ेद ठोस सल्फेट बोर्ड — विशिष्टि

(पहला पुनरीक्षण)

Solid Bleached Sulphate Board — Specification

(First Revision)

ICS 85.060; 85.080

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Paper Based Packaging Materials Sectional Committee, CHD 16

FOREWORD

This Indian Standard (First 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.

A range of various board constructions, such as solid bleached sulphate (SBS) boards, in the form of a coherent sheet or web, are used for printing, packaging, decorating etc. They have a lower stiffness ratio to allow more flexibility in carton design and can easily be cut, creased, hot foil stamped and embossed. These boards possess a consistent bright white appearance, have excellent printability and are used for printings, retail packaging, high impact graphic corrugated containers, secondary packaging for food and pharmaceuticals, consumer electronics, folding carton boards (mono cartons) and for packaging other consumable items.

In view of fast emerging market of SBS boards and entry of different national and international manufacturers in this field, coupled with technological developments, the need to outline a standard for the product was felt. In the absence of any existing standard, data from different manufacturers on extensive study of different parameters desirable by the consumers were considered while preparing this standard.

This standard is intended to define the quality of SBS board to assure supply of proper quality of such boards to consumers. It is expected that this standard would help manufacturers and consumers to communicate from a single platform, especially for those who prefer to adhere with a pre-defined standard for procurement as per need of their requirements. It is also expected that this standard will assist the manufacturers to control the quality of their products and the consumers to obtain material of proper quality.

This standard was first published in 2018 taking considerable assistance from the data made available by various organizations like JK Paper, ITC, Century Paper, Divya Shakti, Siddarth, Khanna, Murali Agro, Rainbow, etc, and PAPRI, where a number of tests were carried out for the purpose.

In this revision, the following changes have been made:

a) The references clause has been updated;
b) Construction clause has been modified;
c) Requirements for squareness, thickness, grammage, bulk, cobb (60 sec), moisture, stiffness, brightness, gloss, IGT dry pick and roughness have been updated;
d) New requirement and method of test for Print resistance and ink adhesion of printed cartons has been incorporated;
e) Requirements of delamination and surface oil absorbency test (SOAT) have been removed;
f) Requirements for products in contact with food items which were earlier optional have been made as specific requirements;
g) Bendtsen smoothness requirement has been specified for material when bottom is coated or uncoated;
h) The optional requirement for ECO-Mark has been deleted; and
j) Amendment 1 has been amalgamated.

This standard contains **5.3**, **5.4**, **5.5**, **5.11** and **6.1** which call for agreement between the purchaser and the supplier.

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

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

SOLID BLEACHED SULPHATE BOARD — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and test for solid bleached sulphate (SBS) board.

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 4661 (Part 5) shall apply, in addition to the following:

3.1 SBS Board — This is solid bleached sulphate (SBS) board made of bleached virgin fibre grade of wood pulp. It is a medium density board and is perfectly white, both inside and out.

3.2 Chrome Paper — A paper or board coated on one side with material containing adhesive, china clay (Kaolin) etc. The coating on one side could be glossy or matt as per requirement of the customer. Used mainly for self-adhesive stickers, calendars, posters, labels and for application where only one side has to be printed.

3.3 Mixed Waste Board — Pieces of board that may be reclaimed after use or from a converting process for re-pulping and making again into board.

3.4 Intermittent Machine — A machine for forming sheets of board. It consists of either a fourdrinier former or one or more cylinder moulds or vats. The wet web is wound on a drum forming a continuous mat of several layers.

3.5 Delamination or Interlaminar Strength — Delamination strength or interlaminar strength is usually defined by a number of methods designed to measure the force or energy required to separate or delaminate the interior structure of paperboard, that is, the bonding within or between the plies, not the interface between the fibres and coating or within the coating itself.

3.6 Interlayer Strength, Ply-bond — As a multi-ply paperboard is built from several layers of fibres, it is important that these layers are well bonded together. Interlayer strength is the expression used to quantify this property. By ply-bond test, the energy needed to delaminate a sample by applying a perpendicular force to the paperboard surface is quantified. The test result is expressed in J/m².

4 CONSTRUCTION

The construction of solid bleached sulphate board shall comprise of following layers:

a) Top coating — The coating grammage (combined pre-coat and post-coat) shall be (20 ± 6) gsm;

b) Top layer — It shall be from virgin chemical pulp;

c) Fillers — It may be from 60 percent virgin chemical pulp and 40 percent specialized customized pulp or as suitable for board application;

d) Back layer — It shall be from virgin chemical pulp; and

e) Bottom coating — It may be of 6 gsm to 10 gsm, but it is optional based on customer requirements.

5 REQUIREMENTS

5.1 General

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

5.1.2 *Slitting and Cutting*

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

5.2 Squareness

The tolerance on squareness shall be such that the shorter diagonal of the sheet shall not differ from the longer by more than 1 mm of the former.

5.3 Thickness

Normally the thickness for solid bleached sulphate board is between 270 micron to 750 micron. However, the thickness of SBS 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).

5.4 Grammage

Normally, the grammage for SBS board is between 200 gsm to 450 gsm. However, the nominal grammage of SBS 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).

5.5 Sizes

The sizes of the board shall be as agreed to between the purchaser and the supplier. The tolerance on the sizes shall be on the positive side with a maximum of 1 mm when tested in accordance with IS 1060 (Part 5/Sec 3).

5.6 Print Resistance and Ink Adhesion of Printed Cartons

The material shall pass for the print resistance and ink adhesion when tested in accordance with the Annex B and Annex C of this standard.

5.7 Stiffness (Bending Resistance)

The average L&W Bending stiffness value for different grammages of board shall be as given in Table 1 and shall be determined for machine direction (MD) and cross direction (CD) by the method prescribed in IS 1060 (Part 5/ Sec 8). The tolerance on each mean value shall be within ± 15 percent for both MD and CD. The ratio of MD to CD(MD/CD) shall be between 1.8 to 2.4 for all grammage.

5.8 Strength

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

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

5.10 Specific Requirements for Items in Contact with Food

The paper and paper boards packaging materials shall be manufactured from the following raw materials:

5.10.1 In case of direct food contact usage, the paper board shall confirm to IS 16983 or IS 1776 or IS 12999 except for the requirement of grammage. In addition, the paper board shall be OBA (Optical brightening agent) free, and anthraquinone shall not be used in pulp which is used for making paper board. The paper board shall be of virgin material.

5.10.2 Paper and paper boards used for Consumer packaging shall be manufactured from virgin pulp and shall be free from dioxins and furans (<1ppb, measured as per USEPA 1613). If it is used for food packaging, Printed surfaces of 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 D.

5.11 Optional Requirements

When agreed to between purchaser and the supplier, the board shall also comply with the requirements given in Table 3.

6 PACKING AND MARKING

6.1 Packing

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

6.2 Marking

6.2.1 Each package shall be marked with the following particulars:

a) Description of the material;
b) Size of the board;
c) Net mass of contents;
d) Batch number;
e) Date of manufacture; and
f) Manufacturer’s name and/or recognized trade name.

6.2.2 *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.

Table 1 Requirement for Solid Bleached Sulphate Boards

(*Clause* 5.7, 5.9 *and* 7.2)

Sl No.	**Characteristic**	**Requirements**		**Methods of Tests, Ref to**
(1)	(2)	(3)		(4)
i)	Bulk, cc/g	1.15 to 1.25		IS 1060 (Part 5/Sec 3)
ii)	Moisture, percent	6.0 to 9.0		IS 1060 (Part 5/Sec 2)
iii)	Cobb (60 sec) g/m^2			IS 1060 (Part 5/Sec 4)
	a) Top	25.0 to 40.0		
	b) Back	30.0 to 45.0		
iv)	Stiffness (Bending Resistance), L&W, mN	MD Tolerance ± 15 percent	CD Tolerance ± 15 percent	IS 1060 (Part 5/Sec 8)
	a) 200 gsm	90	45	
	b) 250 gsm	160	80	
	c) 300 gsm	250	130	
	d) 350 gsm	400	200	
	e) 400 gsm	540	270	
	f) 450 gsm	720	372	
v)	Brightness, Indoor C/2°, percent, *Min*	88		IS 1060 (Part 4/Sec13)
vi)	Gloss 75°, percent, *Min*	35		IS 1060 (Part 5/Sec 12)
vii)	Ply bond strength, J/m^2 , *Min*	130		T-569 pm-00
viii)	IGT dry pick, m/s, *Min*	1.0		IS 1060 (Part 5/Sec 9)
ix)	Roughness (pps), *Max*	2.2		IS 1060 (Part 5/Sec 17)

Table 2 Limits of Contaminants in Paper

(*Clause* 5.10.2)

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

Table 3 Optional Requirements for SBS Board

(*Clause* 5.11)

Sl No.	Characteristic	Requirements	Methods of Tests, Ref to
(1)	(2)	(3)	(4)
i)	Surface *p*H	5.5 to 7.5	IS 1060 (Part 4/Sec 7)
ii)	Burst Factor, *Min*	15	IS 1060 (Part 7/Sec 1)
iii)	Wax pick number	No pick at 14A	IS 1060 (Part 3)
iv)	Bendtsen smoothness, ml/min, *Max*		IS 1060 (Part 5/Sec 20)
	Top side	50	
	Back side,		
	If Bottom side is coated	500	
	If bottom side is uncoated	1 000	
v)	Water soluble chlorides [(as sodium chloride (NaCl)], percent by mass, *Max*	0.08	IS 1060 (Part 4/Sec 8)
vi)	Water soluble sulphate [(as sodium sulphate (Na_2 SO_4)], percent by mass, *Max*	0.25	IS 1060 (Part 4/Sec 9)
vii)	Fatty and/or similar acids (as $C_{17}H_{33}COOH$), percent by mass, *Max*	0.25	IS 1060 (Part 2)

7 SAMPLING AND CRITERIA FOR CONFORMITY

7.1 Sampling

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

7.2 Number of Tests

From each of the packets, selected from the lot, the board shall be taken out at random. These boards shall constitute the sample. The boards selected shall be tested for requirement given in **5.1** to **5.5**. One test piece shall be cut from each selected board and tested for each of the characteristics prescribed in **5.6** to **5.8** and Table 1. Individual sample of board not meeting the requirements for any one or more characteristics shall be considered as defective.

7.3 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. The acceptance number shall depend upon the size of the sample (*see* **7.2**) and shall be zero if the size of the sample is less than 13 and one if it is greater than or equal to 13.

ANNEX A

(*Clause* 2)

LIST OF REFERRED STANDARDS

IS No/Other Standards.	*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 2
(Part 3) : 1969	Methods of sampling and test for paper and allied products: Part 3
(Part 4)	Methods of test for paper, board and pulps,
(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*)
(Sec 8) : 2024/ ISO 2493-2 : 2020	Determination of bending resistance — Taber-type tester (*first revision*)
(Sec 9) : 2014/ ISO 3783 : 2006	Determination of resistance to picking — Accelerated speed method using the IGT-type tester (electric model)
(Sec 12) : 2021/ ISO 8254-1 : 2009	Measurement of specular gloss — 75 degree gloss with a converging beam, TAPPI method (*first revision*)
(Sec 17) : 2024/ ISO 8791-4 : 2021	Determination of roughness/smoothness (air leak methods) — Print-Surf method (*first revision*)
(Sec 20) : 2018/ ISO 8791-2 : 2013	Determination of roughness/smoothness (air leak methods) — Bendtsen method
(Part 7/Sec 1) : 2014/ISO 2759 : 2001	Methods of test for board, Section 1 Determination of bursting strength of board
IS 4661 (Part 5) : 2022/ISO 4046-5 : 2016	Paper, board, pulps and related terms — Vocabulary: Part 5 Properties of pulp, paper and board (*third revision*)
T-569 pm-00	TAPPT — Internal bond strength (Scott type)

To access Indian Standards click on the link below:

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ANNEX B

(*Clause* 5.6)

TEST FOR PRINT RESISTANCE OF PRINTED CARTONS

B-1 Leave the paper based multilayer laminated/extruded composite cartons to stand for at least 24 h after printing.

B-2 Smear the paper based multilayer laminated/extruded composite cartons, or representative section cut from the printed area with liquid food intended to be packed in at ambient conditions and leave it for 1 h.

B-3 Wash the paper based multilayer laminated/extruded composite cartons or its representative section with cold water.

B-4 Rub each paper based multilayer laminated/extruded composite carton or representative section firmly with hard paper tissue ten times.

B-5 There shall be no significant removal of the print from the surface of the paper based multilayer laminated/extruded composite carton and the print shall be legible to the naked eye after the test.

ANNEX C

(*Clause* 5.6)

TEST FOR INK ADHESION OF PRINTED CARTONS

C-1 Apply two strips of 25 mm wide transparent pressure sensitive taps or cello-tape to the printed area of the paper based multilayer laminated/extruded composite carton. One piece down the length of the carton and the other along the width.

C-2 Press the tape firmly on to the paper based multilayer laminated/extruded composite carton and leave for 15 s.

C-3 Remove the tape by pulling slowly at about 10 mm/s from one end at about 90° to the paper based multilayer laminated/extruded composite carton surface.

C-4 There shall be no significant removal of the print from the surface of the paper based multilayer laminated/extruded composite carton and the printed material shall be still legible.

ANNEX D

(*Clause* 5.10.2)

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

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

D-1.1 Principle

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

D-1.2 Apparatus

D-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.

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

D-1.3 Reagents

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

D-1.3.2 *Nitric Acid* — concentrated

D-1.3.3 *Sulphuric Acid* — approximately 0.2 N

D-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.

D-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+}).

D-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+}).

D-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+}).

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

D-1.4 Procedure

D-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+} ug
(1)	(2)	(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

D-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.

D-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.

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

D-1.4.2 *Determination*

D-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.

D-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.

D-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.

D-1.4.2.4 *Photometric measurement*

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

D-1.4.3 *Calculation*

By means of the calibration curve (*see* **D-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.

D-2 DETERMINATION OF MERCURY (as Hg)

D-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.

D-2.2 Apparatus

D-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.

D-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.

D-2.2.3 *Mercury Hollow Cathode Lamp*

D-2.2.4 *Recorder/Printer/Display Meter*

Any multi-range variable recorder that is compatible with the UV detection system is suitable.

D-2.3 Reagents

D-2.3.1 *Sulphuric Acid* — concentrated

D-2.3.2 *Nitric Acid* — concentrated

D-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.

D-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.

D-2.3.5 *Potassium Permanganate Solution*

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

D-2.3.6 *Potassium Persulphate Solution*

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

D-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.

D-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 (**D-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.

D-2.4 Procedure

D-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.

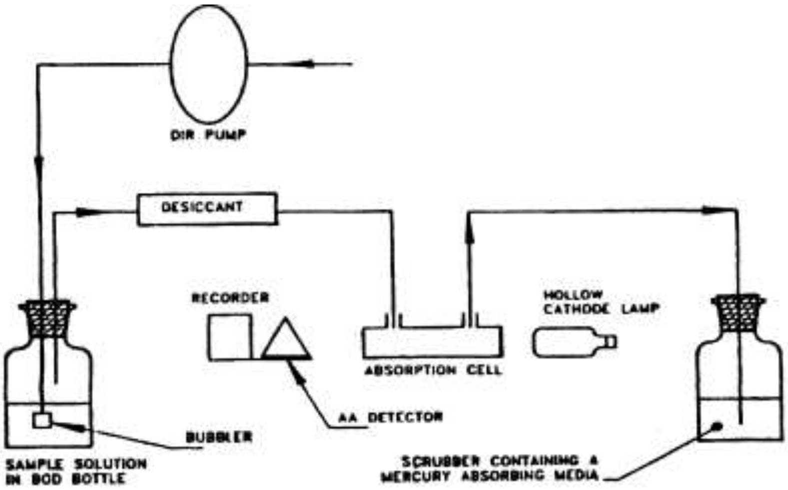

FIG.1 SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR MEASUREMENT OF MERCURY BY COLD VAPOUR ATOMIC ABSORPTION TECHNIQUE

D-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.

D-2.4.3 *Determination*

D-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.

D-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.

D-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 D-2.4.2.

D-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.

D-3 DETERMINATION OF LEAD (as Pb)

D-3.1 Principle

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

D-3.2 Apparatus

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

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

D-3.2.3 *Hot Plate*

D-3.3 Reagents

D-3.3.1 *Nitric Acid* — concentrated

D-3.3.2 *Nitric Acid* — 1 : 1

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

D-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).

D-3.3.5 *Intermediate Lead Solution*

D-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).

D-3.4 Procedure

D-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.

D-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.

D-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.

D-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.

D-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 (**D-3.4.3**). Inject a suitable portion of each standard solution in order of increasing concentration. Analyze each standard solution and measure the absorbances.

D-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.

D-4 DETERMINATION OF CADMIUM (as Cd)

D-4.1 Principle

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

D-4.2 Apparatus

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

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

D-4.2.3 *Hot Plate*

D-4.3 Reagents

D-4.3.1 *Nitric Acid* — concentrated

D-4.3.2 *Nitric Acid* — 1 : 1

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

D-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).

D-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).

D-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).

D-4.4 Procedure

D-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.

D-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.

D-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.

D-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.

D-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.

D-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:

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

where

C = concentration of cadmium from the calibration curve;

F = dilution factor; and

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

D-5 DETERMINATION OF PENTACHLOROPHENOL (PCP)

D-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).

D-5.2 Apparatus

D-5.2.1 *Round Bottom Flask* — 250 ml

D-5.2.2 *Soxhlet Extractor*

D-5.2.3 *Water Bath*

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

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

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

D-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.

D-5.3 Reagents

D-5.3.1 *Acetone*

D-5.3.2 n-*Hexane*

D-5.3.3 *Acetic Anhydride*

D-5.3.4 *Sodium Sulphate Anhydrous*

D-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.

D-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.

D-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.

D-5.3.8 *Internal Standard Solution*

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

D-5.4 Procedure

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

D-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.

D-5.4.3 *Clean Up*

Transfer the extract (D-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.

D-5.4.4 Treat the residue (D-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.

D-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.

D-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 (**D-5.4.3** to **D-5.4.5**). Tabulate peak height or area responses against calculated equivalent mass of underivatized pentachlorophenol injected. Prepare a calibration curve.

D-5.5 Calculation

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

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

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.

D-6 DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)

D-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.

D-6.2 Apparatus

D-6.2.1 *Erlenmeyer Flask* — 200 ml

D-6.2.2 *Water Bath*

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

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

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

D-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.

D-6.3 Reagents

D-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.

D-6.3.2 n-*Hexane*

D-6.3.3 *Sodium Sulphate Anhydrous*

D-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.

D-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.

D-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.

D-6.3.7 *Internal Standard Solution*

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

D-6.4 Procedure

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

D-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.

D-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.

D-6.4.4 *Clean Up*

Transfer the hexane extract (**D-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.

D-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.

D-6.4.6 *Calibration*

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

D-6.5 Calculation

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

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

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 E

(*Foreword*)

COMMITTEE COMPOSITION

Paper Based Packaging Materials Sectional Committee, CHD 16

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