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*Draft Indian Standard*  
**CELLULOSE FILM- SPECIFICATION**  
*(Second Revision of IS 5012)*

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ICS 85.080

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Paper based packaging materials Sectional  
Committee, CHD 16

**Last date of comments:** 15 March 2023

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#### FOREWORD

*(Formal text shall be added later)*

Cellulose film is used for flexible packaging and also for pressure sensitive adhesive tapes. The material consists of regenerated cellulose film, softened by additional agents, such as glycerine, polyethylene, glycol, urea and other suitable recognized softeners or their mixtures. The moisture-proof material consists of a base film of regenerated cellulose, suitably softened, coated on one or both sides with a heat sealable or adhesive-sealable moisture resisting medium. The base film may be colorless or dyed and may also be printed.

Cellulose film is a sensitive material and its quality deteriorates by excessive pressure, heat or moisture. It, therefore, becomes necessary that proper precautions are taken during its storage. Precautions required to be taken during storage are given in Annex A for information.

The standard was first published in 1968. In first revision, requirement to determine the continuity of coated film was incorporated and requirement of water vapor permeability was modified which shall be applicable to coated varieties of cellulose film only.

In view of latest practices and market demand for thinner variety of cellulose film, additional grades i.e grade 200, grade 220, grade 240 and grade 260 have been incorporated in the standard during this second revision. Apart from this, the amendment issued so far to the standard has been amalgamated. In addition, editorial changes have also been done to bring the standard in the latest style and format of Indian Standards.

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).

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'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## 1 SCOPE

This standard prescribes the requirements and methods of sampling and test for commonly used cellulose film.

## 2 REFERENCES

**2.1** The standards listed in Annex B 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 B.

## 3 TERMINOLOGY

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

## 4 GRADES

**4.1** Cellulose film shall be of seven grades, namely, Grade 200, Grade 220, Grade 240, Grade 260, Grade 300, Grade 400 and Grade 600. Each grade may be plain coloured or coated or both.

NOTE — These grades, denote the normal mass in grams for 10 sq. metre of the material.

## 5 REQUIREMENTS

**5.1 Substances** — Each grade of cellulose film shall have the substance as given below:

<i>Grade</i>	<i>Weight in g/m<sup>2</sup></i>
Grade 200 plain	19 to 24
Grade 200 coated	20 to 24.5
Grade 220 plain	21 to 25
Grade 220 coated	22 to 26
Grade 240 plain	23 to 27
Grade 240 coated	24 to 28
Grade 260 plain	25 to 29
Grade 260 coated	26 to 30
Grade 300 plain	29 to 34
Grade 300 coated	32 to 37
Grade 400 plain	38 to 45
Grade 400 coated	40 to 47
Grade 600 plain	56 to 64
Grade 600 coated	60 to 68

**5.2** Cellulose film shall also comply with the requirements given in Table 1, when tested according to the methods given in col 10 of Table 1.

**5.2.1** All test specimens shall be conditioned at a temperature of  $27 \pm 2^\circ\text{C}$  and a relative humidity of  $65 \pm 2$  percent for a maximum period of one hour in the case of plain films and for

24 hours in the case of coated films.

**5.3 Flexibility** — The film, when folded, under the maximum pressure of thumb and the first finger shall not show any sign of splitting or rupture.

**5.3.1** The film after heat sealing at 130°C and conditioning for 24 hours at a temperature of 27 ± 2°C and 65 ± 2 percent relative humidity shall also behave as in **5.3**.

**Table 1 Requirements for Cellulose Film**  
(Clause 5.2)

Sl. No.	Characteristic	Requirement							Method of Test
		Grade 200	Grade 220	Grade 240	Grade 260	Grade 300	Grade 400	Grade 600	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
i)	Moisture content, percent by mass	7.5 to 10.5	7.5 to 10.5	7.5 to 10.5	7.5 to 10.5	7.5 to 10.5	7.5 to 10.5	7.5 to 10.5	IS 1060 (Part 5/Sec 2)
ii)	Tensile strength, kg/cm, <i>Min:</i>								IS 1060 (Part 5/Sec 6)
	MD	0.80	0.90	1.00	1.30	1.45	2.00	2.90	
	CD ( <i>see note</i> )	0.36	0.42	0.48	0.56	0.72	0.90	1.08	
iii)	Elongation at break, percent, <i>Min:</i>								IS 1060 (Part 5/Sec 6)
	MD	10	10	10	10	13	13	13	
	CD	22	22	23	23	25	25	25	
iv)	Bursting strength, kg/cm <sup>2</sup> , <i>Min</i>	1.50	1.60	1.70	1.80	2.25	2.5	3.5	IS 1060 (Part 7/Sec 1)
v)	pH	5.0 to 8.0	5.0 to 8.0	5.0 to 8.0	5.0 to 8.0	5.5 to 8.0	5.5 to 8.0	5.5 to 8.0	IS 1060 (Part 4/Sec 7)
vi)	Water soluble chlorides (as NaCl), percent by mass, <i>Max</i>	0.2	0.2	0.2	0.2	0.2	0.2	0.2	IS 1060 (Part 4/Sec 8)
vii)	Water soluble sulphates (as Na <sub>2</sub> SO <sub>4</sub> ), percent by mass, <i>Max</i>	0.3	0.3	0.3	0.3	0.3	0.3	0.3	IS 1060 (Part 4/Sec 9)
viii)	Water vapor permeability, g/m <sup>2</sup> at 38°C and 90 ± 2 percent RH for 24 h (only for coated grades of cellulose film), <i>Max</i>								Cl. 14 of IS 1060 (Part 2)

	Creased	30	30	30	30	30	30	30	
	Uncreased	15	15	15	15	15	15	15	
ix)	Blocking resistance at 30 °C and 75 percent RH for 24 h (only for coated grades of cellulose film)	Nil	Nil	Nil	Nil	Nil	Nil	Nil	Cl. 10 of IS 4006 (Part 1)

Note: The term CD is sometimes also referred to as TD

**5.4 Heat Seal** — When required by the agreement between the purchaser and the supplier, the material shall also pass the following test for heat seal:

A strip of film shall be cut to a size 4 x 25 cm in such a manner that the machine direction of the film is parallel to the length of the strip. The strip shall then be cut in halves laterally and one half shall be superimposed on the other in such a manner that opposite faces of the film are in contact.

The strips shall then be sealed together or apart of their length and the whole of their width by applying a moderate pressure with a metal bar heated to 130°C for a suitable predetermined contact time, usually between 1 and 3 seconds. This sealing can be done under controlled pressure by passing a roller 2 cm wide, 2.5 cm in diameter and weighing 700 g three times across the strip placed on a platen at 130°C. The temperature may be checked with the aid of a thermocouple and pyrometer.

The strip shall be removed and allowed to cool. A 60 g weight suitably attached to one of the 4 cm strips shall then be lifted by raising the free end of the other 4 cm strip. The joint shall not peel on lifting.

**5.5 Coating Continuity** — In the case of coating grades of cellulose film, the material shall pass the following tests:

Lay the sample with the coated size exposed on a white background. Apply methylene blue dye solution (1%) evenly and thoroughly over the coated surface of the film. Allow the solution to remain on the film for one minute. Examine the film for blue stains. Cellulose film itself gets stained by the dye solution while the coating does not. Hence any uncoated spot/area show absence of coating on this area. The specimen shall show no spots or stains.

**5.6 Additional Requirements for ECO Mark**

**5.6.1 General Requirements**

**5.6.1.1** The product shall conform to the requirements for quality and performance prescribed under **5.1** to **5.5**.

**5.6.1.2** 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.

### 5.6.2 Specific Requirements

**5.6.2.1** The material shall be of the following 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.
- b) Manufactured from pulp made from 100 percent waste paper or agricultural/industrial waste.
- c) 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 4 when tested according to the methods given in Annex C.

**Table 3 Maximum Permissible Limits of Contaminants in Paper**  
(Clause 5.6.2.2)

Contaminant (1)	Paper Intended to Come into Contact with Dry Food (mg/kg of paper) (2)	Paper Intended to Come into Contact with Wet Food and Food with Fatty Surface (mg/kg of paper) (3)	Paper for Filtration (mg/kg of paper) (4)
Cadmium (Cd)	—	0.5	0.5
Chromium(Cr <sup>6+</sup> )	—	0.1	0.1
Lead (Pb)	—	3.0	3.0
Mercury (Hg)	—	0.3	0.3
Pentachlorophenol (PCP)	0.05	0.05	0.05
Polychlorinated biphenyls (PCBs)	2.0	2.0	0.5

## 6 PACKING AND MARKING

### 6.1 Packing

**6.1.1** The film shall be packed securely and suitable as agreed to between the purchaser and the supplier.

**6.1.2** For ECO Mark, cellulose film shall be packed in such packages which shall be recyclable/reusable or biodegradable.

### 6.2 Marking

**6.2.1** Each package shall be marked with the following information:

- a) Description grade of material;
- b) Mass of package;
- c) Batch number;
- d) Month and year of manufacture; and
- e) 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 BIS 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 based on which the cellulose film has been labelled as environment friendly and marked with ECO Mark.
- b) The cellulose film 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 contents 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**

**7.1.1** Representative samples of packets or rolls of cellulose film shall be selected from each lot as prescribed in 4 of IS 1060 (part 1).

**7.1.2** The samples selected for test should be kept flat, free from folds and wrinkles and should not be taken from outer shuts of a package not from the outer layers of a reel. The samples should be protected from exposure to direct sunlight, extremes of temperature, humidity and other harmful influences, and in the case of plain film, from contact with paper or other absorbent material.

### **7.2 Number of Tests**

**7.2.1** From each of the packets selected from the lot (see 7.1), take out the described number of sheets at random. These sheets shall constitute the sample for determining the characteristics, namely, substance, tensile strength, elongation at break, bursting strength. Tests for these characteristics shall be conducted individually on each of the sample sheets. A sheet not meeting the requirements for any one or more of these characteristics shall be considered as defective.

### **7.3 Criteria for Conformity**

**7.3.1** A lot shall be considered as conforming to the requirements for substance, tensile strength, elongation at break and bursting strength, if the number of defective sheets does not exceed the

acceptance number. This acceptance number shall depend on the size of the sample (see 7.1) 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** For other characteristics which are assessed on one-test basis, the lot shall be declared as conforming to the requirements of the specification if the sheets selected for this purpose pass the tests corresponding to these characteristics.

**ANNEX A**  
(Foreword)

**PRECAUTIONS DURING STORAGE OF CELLULOSE FILM**

**A-1 STORAGE**

**A-1.1** Regenerated cellulose film contains softeners of a hygroscopic nature. For this reason it is important, especially with plain film, that special care should be taken in storage; these precautions are even more necessary than those for ordinary fibrous papers.

**A-1.2** All grades of film shall be protected from exposure to extremes of temperature or humidity.

**A-1.2.1** In all cases it is advisable that film should be stored in the original wrappers away from any source of local heating and from direct sunlight. Recommended conditions for storage are:

Temperature  $20 \pm 2$  °C

Relative Humidity  $40 \pm 10$  percent

**A-1.3** The packages shall be protected from exposure excessive moisture and especially from water.

**A-1.4** It is advisable that the material shall not be subjected to excessive pressures either by reason of stacking more than 10 reams high or by placing any other weight on the packages.

**A-1.4.1** Whenever possible, reels shall be suspended on a bar or pole through the core. When this is not possible, they shall rest on a flat surface.

**ANNEX B**  
(Clause 2)

**LIST OF REFERRED INDIAN STANDARDS**

<i>IS No.</i>	<i>Title</i>
IS 1060 (Part 1) : 2022	Methods of Sampling and Test for Paper and Allied Products Part 1 Test Methods for General Purpose
IS 1060 (Part 2) : 1960	Methods of sampling and test for paper and allied products Part 2

- IS 1060 ( Part 4 / Sec 7) : Methods of sampling and test for paper and allied products: 2018  
Part 4 methods of test for paper, board and pulp: Sec 7 determination of pH of aqueous extracts - Hot extraction method
- IS 1060 (Part 4/Sec 8) :Methods of sampling and test for paper and allied products Part 4 2014  
methods of test for paper board and pulp Sec 8 determination of water soluble chlorides
- IS 1060 (Part 4/Sec 9) :Methods of sampling and test for paper and allied products Part 4 2014  
methods of test for paper board and pulp Sec 9 determination of water soluble sulfates
- IS 1060 (Part 5/Sec 2) : Methods of Sampling and Test for Paper and Allied Products 2021  
Part 5 Methods of Test for Paper and Board Section 2 Determination of moisture content of a lot Oven-drying method
- IS 1060 (Part 5/Sec 6) :Methods of sampling and test for paper and allied products Part 5 2014  
methods of test for paper and board Sec 6 determination of tensile properties - Constant rate of elongation method 20 Mm min
- IS 1060 (Part 7/Sec 1) :Methods of sampling and test for paper and allied products Part 7 2014  
methods of test for board Sec 1 determination of bursting strength of board
- IS 4006 (Part 2) : 1985 Methods of test for paper and pulp based packaging materials Part 2
- IS 4261 : 2001 Glossary of terms relating to paper and pulp based packaging materials

### ANNEX C

(Clause 5.6.2.2 and Table 2)

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

### C-1 DETERMINATION OF CHROMIUM (as Cr<sup>6+</sup>)

**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  $30 \pm 2$  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  $\mu\text{g}$  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  $\mu\text{g}$  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  $\mu\text{g}$  of chromium (as  $Cr^{6+}$ ).

**C-1.3.8** *Indicator Paper* — Covering the pH 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:

<i>Standard Chromium Solution</i> ml	<i>Corresponding to <math>Cr^{6+}</math></i> $\mu\text{g}$
1.0	0.05
2.0	0.10
3.0	0.15
4.0	0.20
5.0	0.25
6.0	0.30
7.0	0.35
8.0	0.40
9.0	0.45
10.0	0.50

**C-1.4.1.1** Add sulphuric acid to adjust the solution pH to  $1.0 \pm 0.3$  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  $18 \pm 2$  h using shaker rotating/moving at  $30 \pm 2$  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  $\mu\text{g}$  of chromium to a 100-ml volumetric flask. Adjust the pH of the solution to  $1.0 \pm 0.3$  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 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 of chromium determined in the aliquot of the sample solution,  $\mu\text{g}$ ;

$M$  = 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 hollow 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 ( $\text{SnCl}_2$ ) 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  $(\text{NH}_2\text{OH})_2 \text{H}_2\text{SO}_4$  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.

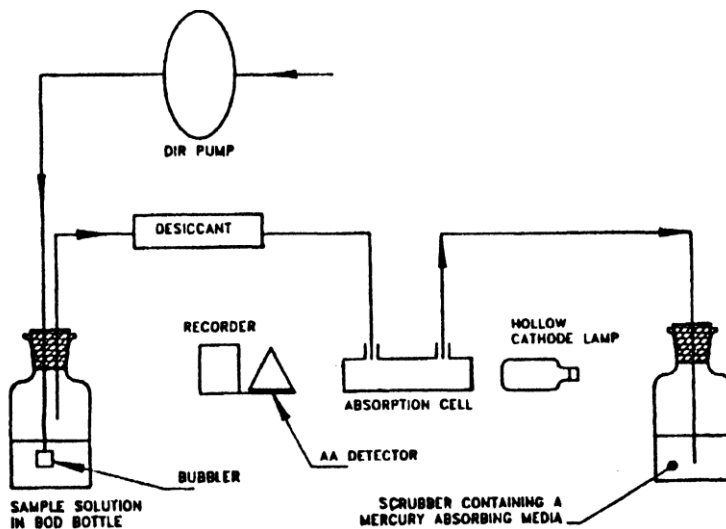
**C-2.3.7 Stock Mercury Solution** — Dissolve 1.354 g of mercuric chloride in about 700-ml of distilled water. Add 10-ml of concentrated nitric acid and make up to 1 000 ml. One millilitre of the solution contains 1 mg of mercury as Hg.

**C-2.3.8 Standard Mercury Solution** — Prepare a series of standard mercury solutions containing 0 to 5  $\mu\text{g/l}$  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<sub>2</sub>S<sub>2</sub>O<sub>8</sub>) 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 ± 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:

$$\text{Mercury (as Hg), ppm} = \frac{C \times V}{M \times 1000}$$

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 1.5999 g of  $\text{Pb}(\text{NO}_3)_2$  in a mixture of 10 ml of concentrated  $\text{NO}_3$  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  $18 \pm 2$  h using shaker rotating/ moving at  $30 \pm 2$  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 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:

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

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.

**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  $18 \pm 2$  h using shaker rotating/moving at  $30 \pm 2$  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 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:

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

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 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 column 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:

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

where

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

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

*V<sub>i</sub>* = 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<sub>t</sub>* = 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 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 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:

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

where

- $A$  = PCB content in  $\mu\text{g}$  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.