

भारतीय मानक मसौदा
सेलूलोज़ फिल्म — विशिष्टि
(दूसरा पुनरीक्षण)

Draft Indian Standard
Cellulose Film — Specification
(*Second Revision*)

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ICS 85.060

Paper Based Packaging Materials Sectional
Committee, CHD 16

Last date of comments: 15th August 2024

FOREWORD

(formal text shall be added later)

Cellulose Film is a thin transparent film derived from natural wood pulp and is primarily used in the packaging industries for flexible packaging as well as for pressure sensitive adhesive tapes. The cellulose film exhibits excellent optical properties such as transparency and gloss, good physical and mechanical properties, excellent tear properties, non-toxic, and does not alter the taste and odor of the food upon contact. It is suitable for decorative packaging for flowers, gift wrapping, firecrackers, incense sticks, tea, coffee, dry food articles, confectionery items, etc. Additionally, it is chemically resistant to oils, fats, and grease and is highly permeable to water vapor with an excellent barrier to gases and aromas. The film is completely biodegradable with no disposal hazards.

The material consists of regenerated cellulose film is softened by additional agents, such as glycerin, 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 suitable biodegradable heat sealable or adhesive-sealable moisture resisting medium to impart heat sealable property and enhance moisture and other barrier properties. 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 B for information.

The standard was first published in 1968 and subsequently revised in 1987. In first revision, requirement to determine the continuity of coated film was incorporated and requirement of water vapor permeability was modified which is applicable to coated varieties of cellulose film only.

In view of latest practices and market demand for thinner varieties of cellulose film, the committee decided to revise the standard.

In this second revision, the following changes have been made:

- a) The scope has been modified to cover all type of cellulose films;
- b) The references clause has been added;
- c) Additional grades i.e. grade 200, grade 240 and grade 260 have been incorporated in the standard. Coating has been permitted on single or both sides;
- d) Requirements for moisture, tensile strength, elongation break and burst strength have been modified;
- e) Tolerance for substance has been incorporated; and
- f) Apart from this, the amendment issued so far to the standard has been amalgamated.

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

This standard contains **5.5** and **6.1.1** which call for agreement between the purchaser and the supplier

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.

Draft Indian Standard
CELLULOSE FILM — SPECIFICATION
(*Second Revision*)

1 SCOPE

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

2 REFERENCES

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 4261 shall apply.

4 GRADES

Cellulose film shall be of six grades, namely, Grade 200, Grade 240, Grade 260, Grade 300, Grade 400 and Grade 600. Each grade may be plain or coated (single or both sides) and with or without colour.

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

5 REQUIREMENTS**5.1 Substances**

5.1.1 Each grade of cellulose film shall have the substance as given below in Table 1, when tested according to the method given in IS 1060 (Part 5/Sec 5).

Table 1 Substances for Cellulose Film

(Clause 5.1.1)

Grade	Weight in g/m ²	
	Plain	Coated (Single or both side)
200	18.0 to 22.0	19.0 to 22.5
240	22.5 to 25.0	23.0 to 25.5
260	25.5 to 28.0	26.0 to 29.0
300	29.0 to 34.0	32.0 to 37.0
400	38.0 to 45.0	40.0 to 47.0
600	56.0 to 64.0	60.0 to 68.0

5.1.2 No single test result shall vary by more than ± 4 percent from the nominal grammage. Further the mean value of 10 test results shall not vary from the nominal grammage by more than ± 2.5 percent.

5.1.3 The film under test shall be conditioned as per IS 1060 (Part 4/Sec 1) before it is subjected to this test.

5.2 Cellulose film shall also comply with the requirements given in Table 2, when tested according to the methods given in col (9) of Table 2.

Table 2 Requirements for Cellulose Film

(Clause 5.2)

Sl. No.	Characteristic	Requirements						Methods of Tests, Ref to
		Grade 200	Grade 240	Grade 260	Grade 300	Grade 400	Grade 600	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
i)	Moisture content, percent by mass	8.5 to 10.5	8.5 to 10.5	8.5 to 10.5	8.5 to 10.5	8.5 to 10.5	8.5 to 10.5	IS 6213 (Part 21)
ii)	Tensile strength, kN/m, <i>Min</i>							
	MD	0.90	1.10	1.30	1.45	2.00	2.90	IS 1060 (Part 5/Sec 6)
	CD (<i>see note</i>)	0.40	0.50	0.58	0.72	0.90	1.08	
iii)	Elongation at break, percent, <i>Min</i>							
	MD	10	11	11	13	13	13	IS 1060 (Part 5/Sec 6)
	CD	22	24	24	25	25	25	
iv)	Bursting strength, kg/cm^2 kPa, <i>Min</i>	1.60	1.70	1.90	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	5.5 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	IS 1060 (Part 2)
vii)	Water soluble sulphates (as Na_2SO_4), percent by mass, <i>Max</i>	0.3	0.3	0.3	0.3	0.3	0.3	IS 1060 (Part 2)
viii)	Water vapor permeability, g/m^2 at 38 °C and 90 percent \pm 2 percent RH for 24 h (only for coated grades of cellulose film), <i>Max</i>							IS 1060 (Part 2)
	Creased	30	30	30	30	30	30	
	Uncreased	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	IS 4006 (Part 1)

NOTE — The term CD is sometimes also referred to as TD

5.2.1 All test specimens shall be conditioned at a temperature of (27 ± 2) °C and a relative humidity of 65 percent ± 2 percent for a minimum period of one hour in the case of plain films and for 24 h in the case of coated films.

5.3 Flexibility

5.3.1 The film shall show no sign of splitting or rupture when tested according to the method given in Annex D.

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

5.4 Colour — The cellulose film may be coloured. The coloured cellulose film if intended to be used for food packaging, shall use food grade dyes/colours.

5.5 Heat Seal

When required by the agreement between the purchaser and the supplier, the material shall also pass the test for heat seal when tested according to the method given in Annex E.

5.6 Coating Continuity

In the case of coating grades of cellulose film, the material shall pass the coating continuity test when tested according to the method given in Annex F.

5.7 Food Contact Materials

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

Table 3 Maximum Permissible Limits of Contaminants in Paper

(Clause 5.7 and 5.8.2.1)

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

5.8 Additional Requirements for ECO Mark

5.8.1 General Requirements

5.8.1.1 The product shall conform to the requirements for quality and performance prescribed under 5.1 to 5.7.

5.8.1.2 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.

5.8.2 Specific requirements

5.8.2.1 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; and
- 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 3 when tested according to the methods given in Annex C.

5.9 Storage and control conditions for products intended to be used in contact with food:

- a) Product intended for food contact use shall be stored separately from other materials, in closed and properly identified area;
- b) An authorized person shall supervise and control the issue of materials to the process or manufacturing area and shall maintain appropriate records of the issue of such materials;
- c) Adequate standards of hygiene (*see* IS 2491) shall be maintained at all times and plant operators and store men shall be trained in proper hygiene practices;
- d) Separate cutting, reeling and packaging area wherein the temperature and relative humidity shall be maintained at $(25 \pm 3) ^\circ\text{C}$ and 65 percent \pm 5 percent RH, respectively;
- e) No direct human contact shall be with the product; and
- f) SPM_{10} shall not be more than $100 \mu\text{g}/\text{m}^3$.

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

'Food contact safe' may be marked if food contact criteria are fulfilled.

A clear marking indicating storage conditions, as specified in A-1.2.1, shall also be provided.

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.

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; and
- 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

7.1.1 Representative samples of packets or rolls of cellulose film shall be selected from each lot as prescribed in 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.

7.1.3 All test specimens shall be conditioned at a temperature of (27 ± 2) °C and a relative humidity of 65 percent ± 2 percent for a minimum period of one hour in the case of plain films and for 24 h in the case of coated films before testing.

7.2 Number of Tests

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

(Clause 2)

LIST OF REFERRED STANDARDS

<i>IS No.</i>	<i>Title</i>
IS 1060	Methods of sampling and test for paper and allied products:
(Part 1) : 2022	Test methods for general purpose (<i>second revision</i>)
(Part 2) : 1960	Methods of sampling and test for paper and allied products: Part 2
(Part 4)	Methods of test for paper, board and pulps,
(Sec 1) : 2014/ISO 187 : 1990	Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples
(Sec 7) : 2018/ISO 6588-2 : 2012	Determination of pH of aqueous extracts — Hot extraction method
(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 (<i>first revision</i>)
(Sec 5) : 2021/ISO 536 : 2019	Determination of grammage (<i>first revision</i>)
(Sec 6) : 2014/ISO 1924-2 : 2008	Determination of tensile properties — Constant rate of elongation method (20 mm/min)
(Part 7/Sec 1) : 2014	Methods of test for board: Section 1 Determination of bursting strength of board
IS 4661 (Part 1) : 2022/ISO 4046-1 : 2016	Paper, board, pulps and related terms — Vocabulary: Part 1 Alphabetical Index (<i>third revision</i>)
IS 4261 : 2001	Glossary of terms relating to paper and pulp based packaging materials (<i>first revision</i>)
IS 6213 (Part 21) : 2019	Methods of test for pulp: Part 21 Paper, board and pulps — Determination of dry matter content — Oven-drying method (<i>first revision</i>)

ANNEX B*(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	(28 ± 5) °C
Relative Humidity	65 percent ± 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 C*(Clause 5.7, 5.8.2.1)***DETERMINATION OF CHROMIUM, LEAD, MERCURY, CADMIUM, PENTACHLOROPHENOL AND POLYCHLORINATED BIPHENYLS****C-1 DETERMINATION OF CHROMIUM (as Cr⁶⁺)****C-1.1 Principle**

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

C-1.2 Apparatus**C-1.2.1 Spectrophotometer**

Any spectrophotometer suitable for measurement at a wavelength of about 550 nm or photoelectric absorptionmeter fitted with filters giving maximum transmission near 550 nm.

C-1.2.2 Shaker

Any shaker suitable for rotating/moving at (30 ± 2) rev/min.

C-1.3 Reagents

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

C-1.3.2 Nitric Acid — concentrated

C-1.3.3 Sulphuric Acid— approximately 0.2 N

C-1.3.4 Diphenylcarbazide Solution

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

C-1.3.5 Stock Chromium Solution

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

C-1.3.6 Intermediate Chromium Solution

Take 10 ml of stock chromium solution and dilute to 1 000 ml with distilled water. One milliliter of this solution contains 5.00 μg of chromium (as Cr^{6+}).

C-1.3.7 Standard Chromium Solution

Take 10 ml of intermediate chromium solution and dilute to 1 000 ml with distilled water. One milliliter of this solution contains 0.05 μg of chromium (as Cr^{6+}).

C-1.3.8 Indicator Paper — covering the 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>Sl No.</i>	<i>Standard Chromium Solution</i> ml	<i>Corresponding to Cr^{6+}</i> 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

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

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

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

C-1.4.2 *Determination*

C-1.4.2.1 *Sample preparation*

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

C-1.4.2.2 *Preparation of test solution*

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

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

C-1.4.2.4 *Photometric measurement*

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

C-1.4.3 *Calculation*

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

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

where

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

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

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

C-2 DETERMINATION OF MERCURY (as Hg)

C-2.1 *Principle*

The flameless atomic absorption procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapour. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapour passes through a cell positioned in the light path of mercury 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 (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 milliliter of the solution contains 1 mg of mercury as Hg.

C-2.3.8 Standard Mercury Solution

Prepare a series of standard mercury solutions containing 0 $\mu\text{g/l}$ 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.

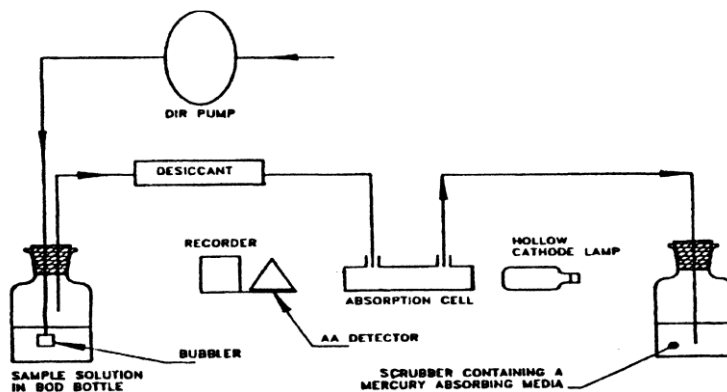


FIG.1 SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR MEASUREMENT OF MERCURY BY COLD VAPOUR ATOMIC ABSORPTION TECHNIQUE

C-2.4.2 Standardization

Transfer 100 ml of each of the 1.0 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 (K₂S₂O₈) solution to each bottle and heat for 2 h in a water bath at 95 °C. Cool and add 6 ml of sodium chloride-hydroxylamine sulphate solution to reduce the excess permanganate. After decolourization add 5 ml of stannous chloride solution and attach the bottle immediately to the aeration apparatus forming a closed system. As mercury is volatilized and carried into the absorption cell, absorbance will increase to a maximum within a few seconds. As soon as recorder returns approximately to the base line, remove stopper holding the aeration frit from the reaction bottle and replace with a bottle containing distilled water. Flush the system for a few seconds and run the next standard in the same manner. Construct a standard calibration curve by plotting absorbance (peak height) versus mercury concentration in µg.

C-2.4.3 Determination

C-2.4.3.1 Sample preparation

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

C-2.4.3.2 Preparation of test solution

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

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

C-2.5 Calculation

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

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

where

C = concentration of mercury from the calibration curve;

V = volume in ml of test solution prepared; and

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

C-3 DETERMINATION OF LEAD (as Pb)

C-3.1 Principle

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

C-3.2 Apparatus

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

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

C-3.2.3 Hot Plate

C-3.3 Reagents

C-3.3.1 Nitric Acid — concentrated

C-3.3.2 Nitric Acid — 1 : 1

C-3.3.3 Dilute Nitric Acid — 1 : 499

C-3.3.4 Stock Lead Solution

Dissolve 1.599 9 g of $\text{Pb}(\text{NO}_3)_2$ in a mixture of 10 ml of concentrated HNO_3 and 100 ml of water and dilute to 1 litre. One milliliter of this solution contains 1.0 mg of lead (as Pb).

C-3.3.5 Intermediate Lead Solution

C-3.3.6 Standard Lead Solution

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

C-3.4 Procedure

C-3.4.1 Sample Preparation

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

C-3.4.2 Preparation of Test Solution

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

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

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

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

C-3.4.6 *Calculation*

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

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

where

C = concentration of lead from the calibration curve;

F = dilution factor; and

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

C-4 DETERMINATION OF CADMIUM (as Cd)

C-4.1 Principle

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

C-4.2 Apparatus

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

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

C-4.2.3 Hot Plate

C-4.3 Reagents

C-4.3.1 Nitric Acid — concentrated

C-4.3.2 Nitric Acid — 1 : 1

C-4.3.3 Dilute Nitric Acid — 1 : 499

C-4.3.4 Stock Cadmium Solution

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

C-4.3.5 Intermediate Cadmium Solution

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

C-4.3.6 Standard Cadmium Solution

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

C-4.4 Procedure

C-4.4.1 Sample Preparation

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

C-4.4.2 Preparation of Test Solution

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

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

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

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

C-4.4.6 Calculation

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

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

where

C = concentration of cadmium from the calibration curve;

F = dilution factor; and

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

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 liter of acetone. One millilitre of this solution contains 1 mg of dibromophenol.

C-5.3.8 *Internal Standard Solution*

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

C-5.4 Procedure

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

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

C-5.4.3 *Clean Up*

Transfer the extract (**C-5.4.2**) to the silica gel packed 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 min and let the layers be separated. Collect the hexane layer, dry over anhydrous sodium sulphate and evaporate nearly to dryness. Cool for at least 10 min, add 1 ml of internal standard solution and adjust the volume to 5.0 ml with *n*-hexane.

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

C-5.4.6 Calibration

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

C-5.5 Calculation

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

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

where

A = PCP content in μg from the graph;

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

V_i = volume, in ml, of eluent injected;

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

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

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

X = moisture content, percent by mass.

C-6 DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)

C-6.1 Principle

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

C-6.2 Apparatus

C-6.2.1 Erlenmeyer Flask — 200 ml

C-6.2.2 Water Bath

C-6.2.3 Separating Funnels — 60 ml and 100 ml

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

C-6.2.5 Gas Chromatograph with ECD — capillary columns

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

C-6.3 Reagents

C-6.3.1 Ethanolic Potassium Hydroxide Solution

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

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

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

C-6.3.2 *n-Hexane***C-6.3.3** *Sodium Sulphate Anhydrous***C-6.3.4** *PCB Stock Solution*

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

C-6.3.5 *PCB Standard Solution*

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

C-6.3.6 *Internal Standard Stock Solution*

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

C-6.3.7 *Internal Standard Solution*

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

C-6.4 Procedure

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

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

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

C-6.4.4 *Clean Up*

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

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

C-6.4.6 *Calibration*

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

C-6.5 Calculation

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

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

where

A = PCB content in µg from the graph;

V_i = volume, in ml, of eluent injected;

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

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

X = moisture content, percent by mass.

Annex D

(Clause 5.3.1)

Determination of Flexibility

D-1 Determination of flexibility

Cut a paper in 4×2 cm size, fold it and keep between two parallel surfaces. Apply pressure of 2.0 kg for 30 min. This film shall not show any sign of splitting or rupture.

Annex E

(Clause 5.5)

Determination of Heat Sealing

E-1 Determination of Heat Sealing

E-1.1 A strip of film shall be cut to a size 4×25 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.

E-1.1 Sealing machine fitted with a heated upper sealing jaw, and either an unheated lower sealing bed (HS-2 Single) or a heated lower sealing bed (HS-2 Dual) with optional interchangeable sealing jaws can be used

E-1.1 The sealer may have a set of sealing jaws, with temperature control and display. A pneumatic piston drive system to deliver the sealing pressure, a pressure transducer to display the sealing pressure, a precision pressure regulator for adjustment of sealing pressure and a digital dwell time controller.

E-1.1 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.

E-1.1 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.

Annex F

(Clause 5.6)

Determination of coating continuity

F-1 Determination of coating continuity

Lay the sample with the coated size exposed on a white background. Apply methylene blue dye solution (1 percent) evenly and thoroughly over the coated surface of the film. Allow the solution to remain on the film for 1 min. 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.