भारतीय मानक मसौदा

तैलरोधक कागज – विशिष्टि

(*पहला पुनरीक्षण IS 6622*)

Draft Indian Standard

GREASE PROOF PAPER — SPECIFICATION

(*First Revision of IS 6622*)

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ICS 85.080

Paper based packaging materials Sectional Committee, CHD 16	**Last date of comments: 05 January 2023**

FOREWORD

Grease proof paper is used as packaging material impermeable to oil and grease in food. Grease proof paper is produced by refining the paper stock and thus creates a sheet with very low porosity. This sheet is super calendared to further improve the density, creating a paper called glassine. The glassine is treated with starches, alginates or carboxy methyl cellulose in a size press to fill pores or treat the paper chemically to make it fat repellent. Grease proof paper is produced by coating paper with starch which is impregnated with appropriate grease resistant agent.

This standard was first published in 1972.

During this revision, requirements of pH and moisture content have been incorporated. Further, microbiological requirements and limits of contaminants have also been incorporated as additional requirements for grease proof paper coming in direct contact with the food.

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 requirements, methods of sampling and test for grease proof paper. It does not cover the requirements for packing of butter or other allied materials.

2 REFERENCES

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

3 TERMINOLOGY

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

4 REQUIREMENTS

4.1 General

The paper shall be evenly machine finished. It shall be of uniform formation, thickness and substance and shall be free from visible specks, greases, cuts, holes, etc. It shall also not have an unpleasant or offensive odour when tested in accordance with IS 4006 (Part 2).

4.2 The paper shall also comply with the requirements given in table 1:

	Characteristic	**Requirement**	**Test Method**
i)	Burst factor, *Min*	20	IS 1060 (Part 6/Sec 2)
ii)	Tear factor in any direction, *Min*	40	IS 1060 (Part 6/sec 1)
iii)	Breaking length, metres, *Min*		IS 1060 (Part 5/Sec 6)
iv)	Machine direction,	5000	
	Cross direction	2500	
v)	pH	5-8	IS 1060 (Part 4/Sec7)
vi)	Moisture content, percent by mass, *Max*	4-8.5	IS 1060 (Part 5/Sec 2)
vii)	Oil transudation time, seconds	Over 1200	Annex B

4.2.1 The above requirements are for paper of grammage 35 g/m^2 and above.

4.3 Additional Requirements for Direct Contact with Food

4.3.1 Microbiological Requirements

If grease proof paper is used for direct contact with the food product, it shall also meet the microbiological parameters given in table 2.

Table 2 Microbiological Requirements

Sl No.	Characteristic	Limit	Test Method
i	Total plate count, cfu/g, Max.	300	ISO 8784-1
ii	Pseudomonas aeruginosa, cfu/g	Not detected in 1 gm of the product	
iii	Staphylococcus aureus, cfu/g		
iv	Candida albicans, cfu/g		
v	Escherichia coli, cfu/g		

4.3.2 Limits of contaminants

If grease proof paper is used for direct contact with the food product, it shall also meet the requirements given in table 3.

Table 3 Limits of Contaminants

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)	Test Method
Cadmium (Cd)	-	0.5	Annex C
Chromium (Cr^+)	-	0.1	
Lead (Pb)	-	3.0	
Mercury (Hg)	-	0.3	
Pentachlorophenol (PCP)	0.05	0.05	
Polychlorinated biphenyls (PCBs)	2.0	2.0	

4.4 Additional Requirements for ECO Mark

4.4.1 *General Requirements*

4.4.1.1 The product shall conform to the requirements for quality and performance prescribed under **4.1** to **4.3.2**.

4.4.1.2 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 land Air (Prevention and Control of Pollution) Act, 198 1 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.

4.4.2 *Specific Requirements*

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

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

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

4.4.2.2 The material if used for the packaging of food materials, shall be manufactured from virgin pulp and shall be free from dioxins. Printed surfaces of the paper shall not come into contact with the food and the maximum amounts of contaminants in paper intended to come into contact with food shall not exceed the limits prescribed in Table 4 when tested according to the methods given in Annex C.

Table 4 Limits of Contaminants in Paper

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)
Cadmium (Cd)		0.5	0.5
Chromium (Cr^+)		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

5 Packing and Marking

5.1 Greaseproof paper shall be packed securely and suitably as agreed to between the purchaser and the supplier. Five hundred sheets shall form a ream.

5.2 Each package shall also be marked with the following information:

a) Description and grammage of the material;

b) Size of paper;

c) Mass in kg per ream of 500 sheets including wrapping paper;

d) Lot number;

e) Month and year of manufacture; and

f) Manufacturer’s name or recognized trade-mark.

5.2.1 *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

5.2.1.1 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.

5.3 Additional Requirements for ECO Mark

5.3.1 For ECO Mark, greaseproof paper shall be packed in such packages which shall be recyclable/reusable or biodegradable.

5.3.2 The greaseproof paper may display in brief the criteria based on which the product has been labelled as environment friendly.

5.3.3 The greaseproof paper shall be sold along with instruction for proper use and mode of safe disposal so as to maximize its performance and minimize wastage.

5.3.4 It shall be suitably marked that ECO Mark label is applicable only to the greaseproof 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.

6 Sampling

6.1 Representative samples of packets for the test shall be drawn from each lot as prescribed in **4** of IS 1060 (Part 1).

6.1.1 *Number of tests*

Each of the packets selected from the lot (*see* **6.1**) shall first be examined for the requirements agreed under **5.1**. Then from each of these packets one sheet shall be taken out at random.

These sheets shall constitute sample. Each of these sheets shall first be examined and tested for the general requirements given under **4.1** and then one test piece shall be cut for various requirements mentioned in **4.2**. 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 characteristics shall be considered as a defective.

6.1.2 *Criterion for conformity*

A lot shall be declared as conforming to all the requirements of this specification if the number of defective sheets found does not exceed the acceptance number. This acceptance number shall depend upon the size of the sample (*see* **6.1**) 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.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
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 4 / Sec 7) : 2018	Methods of sampling and test for paper and allied products: Part 4 methods of test for paper, board and pulp: Sec 7 determination of pH of aqueous extracts - Hot extraction method
IS 1060 (Part 5/Sec 2):2021	Methods of Sampling and Test for Paper and Allied Products Part 5 Methods of Test for Paper and Board Sec 2 Determination of moisture content of a lot Oven drying method
IS 1060 (Part 5/Sec 6) : 2014	Methods of sampling and test for paper and allied products: Part 5 methods of test for paper and board: Sec 6 determination of tensile properties - Constant rate of elongation method (20 Mm/min)
IS 1060 (Part 6/Sec 1) : 2014	Methods of sampling and test for paper and allied products: Part 6 methods of test for paper: Sec 1 determination of tearing resistance - Elmendorf method
IS 1060 (Part 6 / Sec 2) : 2014	Methods of sampling and test for paper and allied products: Part 6 methods of test for paper: Sec 2 determination of bursting strength of paper
IS 4661 (Part 1) : 2022	Paper Board Pulps and Related Terms Vocabulary Part 1: Alphabetical Index
IS 4661 (Part 2) : 2022	Paper Board Pulps and Related Terms Vocabulary Part 2: Pulping Terminology
IS 4661 (Part 3) : 2022	Paper Board Pulps and Related Terms Vocabulary Part 3 Paper Making Terminology
IS 4661 (Part 4) : 2022	Paper Board Pulps and Related Terms Vocabulary Part 4 Paper and Board Grades and Converted Products
IS 4661 (Part 5) : 2022	Paper Board Pulps and Related Terms Vocabulary Part 5 Properties of Pulp Paper and Board
ISO 8784-1:2014	Pulp, paper and board — Microbiological examination — Part 1: Enumeration of bacteria and bacterial spores based on disintegration

ANNEX B

(*Clause* 4.2)

OIL TRANSUDATION TRST FOR DETERMINATION OF GREASR RESISTANCE OF PAPER

B-1 GENERAL

This method gives an accelerated comparison of the relative rates at which oils or greases, such as commonly found in foodstuffs, may be expected to penetrate papers, such as uncoated or unimpregnated greaseproof, glassine and vegetable parchment.

B-2 APPARATUS

B-2.1 Tubs — Of any rigid material, 25 mm ID and at least 25 mm in height, the ends of which have been smoothened.

B-2.2 Pipette or Medicine Dropper — Calibrated to deliver 1.1 ml.

B-2.3 Timing Device — Stopwatch or laboratory timer.

B-3 Reagent

B-3.1 Turpentine, Water-Free and Coloured

To 100 ml of pure gum spirit turpentine, relative density 0.851 to 0.866 at 27 °C, add 5 grams of anhydrous calcium chloride and 1.0 gram of an oil-soluble red dye. The container, shake well, and let stand for at least 10 h, shaking occasionally. Stopper the container, shake well, and let stand for at least 10 h, shaking occasionally. Then filter through a dry filter paper at a temperature of approximately 27°C and. store in an airtight bottle.

B-3.2 Standard Sand

Standard sand IS 650 screened to pass 850 micron sieve but to be retained on 600 micron sieve.

B-4 Test Specimens

B-4.1 Prepare at least ten 10 cm square specimens from representative samples taken in accordance with **6.1**.

B-5 Procedure

B-5.1 Condition and test the specimens as specified in **5** of IS 1060 (Part 1)

B.5.2 Make an equal number of tests on each side of the sample. If possible note those made on the felt side and on the wire side separately.

B-5.3 place each specimen on a sheet of white coated and calendered book paper of grammage 100 to 105 g/m^2 resting on a smooth flat surface. Place an end of the tube on the specimen and put 5 g of sand in the tube. Since the purpose of the tube is solely to ensure a uniform area of the sand pile, remove it immediately after the addition of the sand. Using the pipette or medicine dropper, add 1.1 ml of the coloured turpentine to the sand, and start the timing device.

B-5.4 Move the test specimens undergoing test to unsoiled positions on the coated paper and examine the uncovered areas for staining every 30 s for the first 2 min, every minute for the next 8 min and every 3 min thereafter. As soon as the first red stain appears on the coated paper, note the time. Record the time elapsed, in seconds between the application of the turpentine and the appearance of the first definitely red stain as transudation time record it merely as 1200+.

Note — It is advisable to make a few preliminary tests if the approximate period is not known.

B-6 Report

Report the average, maximum and minimum test results in seconds to three significant figures, and if possible, for both wire side up and felt side up. When a test exceeding 1200 seconds is included in an average, report the calculated average followed by a plus sign. The following is an example of the recommended form:

Oil Transudation Time Seconds	*Top Side Up*	*Wire Side Up*
Maximum	1200+	1150
Minimum	900	800
Average of 15 tests	1150+	1000
Grand average	1080+	

ANNEX C

(*Clause* 4.3.2, 4.4.2.2 *Table 3* & *Table* 4)

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

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

C-1.1 Principle ─ The hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution at a wave length of 550 nm.

C-1.2 Apparatus

C-1.2.1 *Spectrophotometer* – Any spectrophotometer suitable for measurement at a wavelength of about 550 nm or photoelectric absorption meter fitted with filters giving maximum transmission near 550 nm.

C-1.2.2 *Shaker* – Any shaker suitable for rotating/moving at 30 ± 2 revolution/min.

C-1.3 Reagents

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

C-1.3.2 *Nitric Acid* — Concentrated.

C-1.3.3 *Sulphuric Acid*— Approximately 0.2 N.

C-1.3.4 *Diphenylcarbazide Solution* – Dissolve 0.25 g of diphenylcarbazide in 50 ml acetone. Store in a brown bottle. Discard when solution becomes discoloured.

C-1.3.5 *Stock Chromium Solution* – Dissolve 0.141 g $K_2Cr_2O_7$ in distilled water and dilute to 100 ml. One ml of this solution contains 500 µg of chromium (as Cr^{6+}).

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

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

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

C-1.4 Procedure

C-1.4.1 *Preparation of Calibration Curve* ─ Into each of a series of ten 250-ml volumetric flasks, place the quantities of standard chromium solution as indicated below:

Standard Chromium Solution ml	*Corresponding to* Cr^{6+} µg
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 ± 0.3 in each flask and dilute to 100 ml. Add 2.0 ml diphenylcarbazide solution, mix thoroughly and wait for 10 minutes.

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

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

C-1.4.2 *Determination*

C-1.4.2.1 *Sample preparation* ─ Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-1.4.2.2 *Preparation of test solution* ─ Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for 18 ± 2 h using shaker rotating/moving at 30 ± 2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size. Add sufficient amount of aluminium sulphate and filter if any precipitate appears. Transfer the solution quantitatively to a volumetric flask of suitable capacity, dilute to the mark and mix well.

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

C-1.4.2.4 *Photometric measuremen* ─ Carry out the photometric measurements of the test solution according to the methods given in C-**1.4.1.1.**

C-1.4.3 *Calculation* ─ By means of the calibration curve (*see* C**-1.4.1.3)** determine the quantity of chromium present:

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

where

m = mass of chromium determined in the aliquot of the sample solution, µg;
M = mass of paper sample taken for testing, g; and
D = ratio of the volume of test solution to the volume of aliquot portion taken for the colour development.

C-2 DETERMINATION OF MERCURY (as Hg)

C-2.1 Principle ─ The flameless atomic absorption procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapour. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapour passes through a cell positioned in the light path of mercury hallow cathode lamp of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and record.

C-2.2 Apparatus

C-2.2.1 *Atomic Absorption Spectrometer* (AAS) *and Associated Equipment* ─ Instrument settings recommended by the manufacturer shall be followed. Instruments designed specifically for the measurement of mercury using the cold vapour technique may be substituted for the AAS.

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

C-2.2.3 *Mercury Hollow Cathode Lamp*

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

C-2.3 Reagents

C-2.3.1 *Sulphuric Acid* — Concentrated.
C-2.3.2 *Nitric Acid* — Concentrated.

C-2.3.3 *Stannous Chloride Solution* ─ Dissolve 25 g of stannous chloride ($SnCl_2$) in water containing 50-ml of concentrated hydrochloric acid and dilute to 250-ml. If a suspension forms, stir reagent ontinuously during use.

C-2.3.4 *Sodium Chloride - Hydroxylamine Sulphate Solution* — Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulphate $(NH_2OH)_2$ H_2SO_4 in distilled water and dilute to 100 ml.

C-2.3.5 *Potassium Permanganate Solution* ─ Dissolve 5 g of potassium permanganate in distilled water and dilute to 100 ml.

C-2.3.6 *Potassium Persulphate Solution* ─ Dissolve 5 g of potassium persulphate in distilled water and dilute to 100 ml.

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

C-2.3.8 *Standard Mercury Solution* ─ Prepare a series of standard mercury solutions containing 0 to 5 µg/l by appropriate dilution of stock mercury solution (C-**2.3.7**) with water containing 10 ml of concentrated nitric acid per litre. Prepare standards daily.

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

C-2.4 Procedure

C-2.4.1 *Instrument Operation* — Follow the procedure of the manufacturer's operating manual. Connect the mercury vapour generating assembly as shown in Fig. **1**.

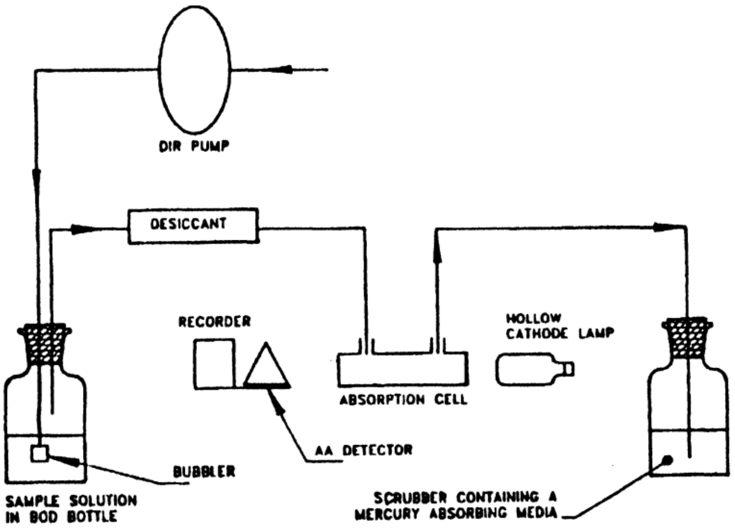

FIG.1 SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR MEASUREMENT OF MERCURY BY COLD VAPOUR ATOMIC ABSORPTION TECHNIQUE

C-2.4.2 *Standardization* ─ Transfer 100-ml of each of the 1.0, 2.0 and 5.0 µg/l standard mercury solution and a blank of 100 ml water to 300 ml BOD bottles. Add 5 ml of concentrated sulphuric acid and 2.5 ml of concentrated nitric acid to each bottle. Add 15 ml of potassium permanganate solution to each bottle and let stand for at least 15 minutes. Add 8-ml of potassium persulphate ($K_2S_2O_8$) solution to each bottle and heat for 2 h in a water bath at 95°C. Cool and add 6 ml of sodium chloride-hydroxylamine sulphate solution to reduce the excess permanganate. After decolourization add 5 ml of stannous chloride solution and attach the bottle immediately to the aeration apparatus forming a closed system. As mercury is volatilised and carried into the absorption cell, absorbance will increase to a maximum within a few seconds. As soon as recorder returns approximately to the base line, remove stopper holding the aeration frit from the reaction bottle and replace with a bottle containing distilled water. Flush the system for a few seconds and run the next standard in the same manner. Construct a standard calibration curve by plotting absorbance (peak height) versus mercury concentration in µg.

C-2.4.3 *Determination*

C-2.4.3.1 *Sample preparation* ─ Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-2.4.3.2 *Preparation of test solution* ─ Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for 18 ± 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 1.599 9 g of $Pb(NO_3)_2$ in a mixture of 10 ml of concentrated NO_3 and 100 ml of water and dilute to 1 litre. One ml of this solution contains 1.0 mg of lead (as Pb).

C-3.3.5 *Intermediate Lead Solution*

C-3.3.6 *Standard Lead Solution* ─ Dilute 100-ml of intermediate lead solution to 1 litre with dilute nitric acid (1:499). One milliliter of this solution contains 0.1 mg of lead (as Pb).

C-3.4 Procedure

C-3.4.1 *Sample Preparation* ─ Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-3.4.2 *Preparation of Test Solution* ─ Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200-ml of extraction fluid for 18 ± 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 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 ± 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 to 20 ml. Continue heating and adding concentrated nitric acid until digestion is complete. Wash down with distilled water and then filter if necessary. Quantitatively transfer filtrate to a 100 ml volumetric flask, dilute to the mark and mix thoroughly.

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

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

C-4.4.6 *Calculation* ─ Construct a standard calibration graph by plotting the absorbance versus mg of cadmium concentration of each standard. Read the concentration of the sample from the graph and determine the cadmium content of the sample from the calibration graph using the following formula:

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

where

C = concentration of cadmium from the calibration curve;
F = dilution factor; and
M = mass of paper sample taken for testing, g.

C-5 DETERMINATION OF PENTACHLOROPHENOL (PCP)

C-5.1 Principle ─ PCP is extracted with acetone by Soxhlet extraction. Acetone extract is evaporated to dryness and subjected to acetylation. The acetylated PCP is determined quantitatively by Gas Chromatograph-Electron Capture Detector (GC-ECD).

C-5.2 Apparatus

C-5.2.1 *Round Bottom Flask* — 250 ml.

C-5.2.2 *Soxhlet Extractor*

C-5.2.3 *Water Bath*

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

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

C-5.2.6 *Gas Chromatograph with ECD* — Capillary columns.

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

C-5.3 Reagents

C-5.3.1 *Acetone*

C-5.3.2 n-*Hexane*

C-5.3.3 *Acetic Anhydride*

C -5.3.4 *Sodium Sulphate Anhydrous*

C-5.3.5 *PCP Stock Solution* ─ Dissolve 10 mg of pentachlorophenol in 100-ml of acetone. One millilitre of this solution contains 0.1 mg of pentachlorophenol.

C-5.3.6 *PCP Standard Solution* ─ Dilute 10 ml of stock solution with acetone to 100-ml. One millilitre of this solution contains 0.01 mg of pentachlorophenol.

C-5.3.7 *Internal Standard Stock Solution* ─ Dissolve 1 g of 2, 4 dibromophenol in 1 l of acetone. One millilitre of this solution contains 1 mg of dibromophenol.

C-5.3.8 *Internal Standard Solution* ─ Dilute 1 ml of the stock solution (C-**5.3.7**) with acetone to 100 ml. One millilitre of this solution contains 10 µg of dibromophenol.

C-5.4 Procedure

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

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

C-5.4.3 *Clean Up* ─ Transfer the extract (C-**5.4.2**) to the silica gel packed coloumn and elute with about 25 ml of *n*-hexane at the rate of 2 ml/min. Collect the eluent in a flask. Dry over anhydrous sodium sulphate and evaporate nearly to dryness.

C-5.4.4 Treat the residue (C-**5.4.3**) with 1 ml of acetic anhydride, and heat on a water bath for about 30 min. Remove the flask from the water bath and cool the acetylated product. Transfer the content in a separating funnel and add 10-ml of hexane and 5 ml distilled water. Shake well for 2 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:

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

where

A = PCP content in µg from the graph;
B = total volume of hexane extract before derivatization, ml;
V_i = volume of eluent injected, ml;
M = mass of the paper sample taken for testing, g;
C = volume of hexane extract carried through derivatization, ml;
V_t = volume of total eluent, ml; and
X = moisture content, percent by mass.

C-6 DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)

C-6.1 Principle ─ PCB is extracted with boiling ethanolic potassium hydroxide solution. An aliquot of the extract is mixed with distilled water and subjected to extraction with hexane. The PCB content is determined quantitatively by GC-ECD by comparing the pattern of the peaks with the pattern of a suitable technical PCB.

C-6.2 Apparatus

C-6.2.1 *Erlenmeyer Flask* — 200-ml.

C-6.2.2 *Water Bath*

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

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

C-6.2.5 *Gas Chromatograph with ECD* — Capillary columns.

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

C-6.3 Reagents

C-6.3.1 *Ethanolic Potassium Hydroxide Solution* ─ Prepare 1 N ethanolic potassium hydroxide solution by dissolving the required amount of potassium hydroxide in absolute ethanol that has been purified as follows:

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

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

C-6.3.2 n-*Hexane*

C-6.3.3 *Sodium Sulphate Anhydrous*

C-6.3.4 *PCB Stock Solution* ─ Dissolve 10 mg of any PCB technical in 100 ml of hexane. One ml of this solution contains 0.1 mg of PCB.

C-6.3.5 *PCB Standard Solution* ─ Dilute 10 ml of stock solution with hexane to 100 ml. One ml of this solution contains 0.01 mg of PCB.

C-6.3.6 *Internal Standard Stock Solution* — Dissolve 1 g of 2, 4 dibromophenol in 1 litre of *n*-hexane. One ml of this solution contains 1 mg of dibromophenol.

C-6.3.7 *Internal Standard Solution* ─ Dilute 1 ml of the stock solution (C**-6.3.6**) with hexane to 100 ml. One ml of this solution contains 10 µg of dibromophenol.

C-6.4 Procedure

C-6.4.1 Determine the moisture content of the sample as given in 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 µg from the graph;

V_i = volume of eluent injected, ml;
M = mass of the paper sample taken for testing, g;
V_t = volume of total eluent, ml; and
X = moisture content, percent by mass.