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मानक

IS 1397 (1990): Kraft Paper [CHD 15: Paper and its products]

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भारतीय मानक

काफ्ट कागज — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard KRAFT PAPER — SPECIFICATION (Second Revision)

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BUREAU OF INDIAN STANDARDS

MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002 Paper and Pulp Based Packaging Sectional Committee, CHD 16

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 31 May 1990, after the draft finalized by the Paper and Pulp Based Packaging Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1960. It was later felt that certain additional requirements were necessary for some special end uses. As such, Grade 1 was introduced which satisfied the optional requirements of pH, ash, alkalinity, etc, and the standard revised in 1967. In this second revision, the kraft paper has been classified into three grades, depending upon the use of the raw materials and the end uses.

This standard has been formulated in order to define the quality of kraft paper and to assure the availability of proper quality of such paper to the consumers. The kraft paper is meant for wrapping and general packing purposes and is not meant for use in corrugated board box manufacture. The requirements of the kraft liner is covered in a separate standard (*see* IS 9588 : 1980 'Specification for kraft liner').

In preparing this standard, considerable assistance has been derived from the data made available by the Stationery Office, Government of India, Calcutta and the Forest Research Institute, Dehradun.

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 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 JULY 1997 TO IS 1397 : 1990 KRAFT PAPER — SPECIFICATION

(Second Revision)

(*Second cover page*, *Foreword*) — Insert the following as fifth paragraph in the FOREWORD:

'A scheme for labelling environment friendly products to be known as ECO Mark has been introduced at the instance of the Ministry of Environment and Forests (MEF). The ECO Mark shall be administered by the Bureau of Indian Standards (BIS) under the *BIS Act*, 1986 as per the Resolution No. 71 dated 20 February 1991 and No. 425 dated 28 October 1992 published in the Gazette of the Government of India. For a product to be eligible for ECO Mark it shall also carry Standard Mark of BIS for quality, besides meeting additional environment friendly (EF) requirements. For this purpose, the Standard Mark of BIS would be a single mark being a combination of the ISI Mark and the ECO logo. Requirements to be satisfied for a product to qualify for the BIS Standard Mark for ECO friendliness, will be included in the relevant published Indian Standard through an amendment. These requirements will be optional; manufacturing units will be free to opt for the ISI Mark alone also.

This amendment is based on the Gazette Notification No. 364 dated 7 September 1995 for packaging material/package (Part I Paper Board and Plastics excluding laminates) as environment friendly products, published in the Gazette of India. This amendment is, therefore, being issued to this standard to include EF requirements for kraft paper.'

(*Page 2, clause* **5.6**) — Insert the following new clauses after **5.6**:

'5.7 Additional Requirements for ECO Mark

5.7.1 General Requirements

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

5.7.1.2 The manufacturer shall produce to BIS, the environmental consent clearance from the concerned State Pollution Control Board as per the provisions of *Water (Prevention and Control of Pollution) Act*, 1974 and *Air (Prevention and Control of Pollution) Act*, 1981 along with the authorization, if required under the *Environment (Protection) Act*, 1986 and the Rules made thereunder, while applying for ECO Mark.

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Price Group 1

Additionally the manufacturers shall also comply with the provisions under *Prevention of Food Adulteration Act*, 1954 and the Rules made thereunder wherever necessary.

5.7.2 Specific Requirements

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

- a) *Type* 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) *Type* B Manufactured from pulp made from 100 percent waste paper or agricultural/industrial waste.

5.7.2.2 Paper 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 B.

Table 3 Limits of Contaminants in Paper	
(<i>Clause</i> 5.7.2.2)	

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)	
Cadmium (Cd)	_	0.5	0.5	
Chromium (Cr6+)		0.1	0.1	
Lead(Pb)		3.0	3.0	
Mercury (Hg)		0.3	0.3	
Pentachlorophenol	l (PCP) 0.05	0.05	0.05	
Polychlorinated (PCB ₅)	biphenyls 2.0	2.0	0.5	

(*Page 2, clause* **6.3**) — Insert the following new clauses after **6.3**:

'6.4 Additional Requirements for ECO Mark

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

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

6.4.3 The kraft paper may be sold along with instructions for proper use and mode of safe disposal so as to maximise its performance and minimise wastage.

6.4.4 It shall be suitably marked on kraft paper that ECO Mark label is applicable only to the packaging material/package 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.'

(*Page* 3, *Annex* A) — Insert the following after Annex A:

'ANNEX B (*Clause* 5.7.2.2)

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

B-1 DETERMINATION OF CHROMIUM (as Cr⁶⁺)

B-1.1 Principle

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

B-1.2 Apparatus

B-1.2.1 Spectrophotometer

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

B-1.2.2 Shaker

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

B-1.3 Reagents

B-1.3.1 Extraction Fluid — Mix 5.7 ml of acetic add in distilled water.

B-1.3.2 *Nitric Acid* — Concentrated.

B-1.3.3 Sulphuric Acid — Approximately 0.2 N.

B-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.

B-1.3.5 Stock Chromium Solution

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

B-1.3.6 Intermediate Chromium Solution

Take 10 ml of stock chromium solution and dilute to 1 000 ml with distilled water. One millilitre of this solution contains 5.00 μ g of chromium (as Cr⁶⁺).

B-1.3.7 Standard Chromium Solution

Take 10 ml of intermediate chromium solution and dilute to 1 000 ml with distilled water. One millilitre of this solution contains 0.05 μ g of chromium (as Cr⁶⁺).

B-1.3.8 Indicator Paper — Covering the pH range 0.5 to 1.5.

B-1.4 Procedure

B-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	Corresponding to Cr ⁶⁺
ml	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

B-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.

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.

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

B-1.4.2 *Determination*

B-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.

B-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.

B-1.4.2.3 According to the expected chromium content, take an aliquot portion of the test solution containing 10 to 100 μ g 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.

B-1.4.2.4 Photometric measurement

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

B-1.4.3 Calculation

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

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

where

- $m = \text{mass in } \mu g$ 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 the test sample.

B-2 DETERMINATION OF MERCURY (as Hg)

B-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.

B-2.2 Apparatus

B-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.

B-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.

B-2.2.3 Mercury Hollow Cathode Lamp

B-2.2.4 Recorder/Printer/Display Meter

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

B-2.3 Reagent

B-2.3.1 Sulphuric Acid — Concentrated.

B-2.3.2 Nitric Acid — Concentrated.

B-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.

B-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.

B-2.3.5 Potassium Permanganate Solution

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

B-2.3.6 Potassium Persulphate Solution

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

B-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.

B-2.3.8 Standard Mercury Solution

Prepare a series of standard mercury solutions containing 0 to 5 μ g/l by appropriate dilution of stock mercury solution (**B-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.

B-2.4 Procedure

B-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.

B-2.4.2 Standardization

Transfer 100 ml of each of the 1.0, 2.0 and 5.0 μ g/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

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

'minutes. Add 8 ml of potassium persulphate ($K_2S_2O_8$) solution to each bottle and heat for 2 hours 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.

B-2.4.3 Determination

B-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.

B-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.

B-2.4.3.3 According to the expected mercury content, take an aliquot portion of the test solution containing not more than 5 μ g/1 of mercury to a 300 ml BOD bottle and treat as in **B-2.4.2**.

B-2.5 Calculation

Determine peak height of sample from recorder chart and read mercury value from standard curve:

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

where

C = concentration of mercury from the calibration curve in $\mu g/1$,

V = volume of test solution prepared in ml, and

M = mass of sample paper taken in g.

B-3 DETERMINATION OF LEAD (as Pb)

B-3.1 Principle

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

B-3.2 Apparatus

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

B-3.2.2 *Lead Hollow-Cathode Lamp or Multielement Hollow-Cathode Lamp* — For use at 283.3 nm.

B-3.2.3 Hot Plate

B-3.3 Reagents

B-3.3.1 Nitric Acid — concentrated.

B-3.3.2 *Nitric Acid* — 1:1.

B-3.3.3 *Dilute Nitric Acid* — 1:499.

B-3.3.4 Stock Lead Solution

Dissolve $1.599 \ 9 \ g$ of Pb(NO₃)₂ in a mixture of 10 ml of concentrated HNO₃ and 100 ml of water and dilute to 1 litre. One millilitre of this solution contains 1.0 mg of lead (as Pb).

B-3.3.5 Intermediate Lead Solution

B-3.3.6 Standard Lead Solution

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

B-3.4 Procedure

B-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.

B-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.

B-3.4.3 Transfer the extract to a 250-ml conical flask. Add 5 ml concentrated 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.

B-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.

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

B-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:

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

where

C = concentration of lead from the calibration curve in mg/1,

F = dilution factor, and

M = mass of paper sample taken in g.

B-4 DETERMINATION OF CADMIUM (as Cd)

B-4.1 Principle

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

B-4.2 Apparatus

B-4.2.1 *Atomic Absorption Spectrometer* — With graphite oven technique in place of conventional burner assembly.

B-4.2.2 *Cadmium Hollow* — *Cathode Lamp or Multielement Hollow Cathode Lamp* — For use at 228.8 nm.

B-4.2.3 Hot Plate

B-4.3 Reagents

B-4.3.1 *Nitric Acid* — Concentrated.

B-4.3.2 *Nitric Acid* — 1:1.

B-4.3.3 *Dilute Nitric Acid* — 1:499.

B-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).

B-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 millilitre of this solution contains 50 μ g of cadmium (as Cd).

B-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 millilitre of this solution contains $0.5 \mu g$ of cadmium (as Cd).

B-4.4 Procedure

B-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.

B-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.

B-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.

B-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.

B-4.4.5 Prepare a reagent blank and sufficient standards containing 5.0, 7.5 and 10.0 mg/1 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. Analyse each standard solution and measure the absorbances.

B-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:

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

where

C = concentration of cadmium from the calibration curve in mg/1,

F = dilution factor, and

M = mass of paper sample taken in g.

B-5 DETERMINATION OF PENTACHLOROPHENOL (PCP)

B-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).

B-5.2 Apparatus

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

B-5.2.2 Soxhlet Extractor

B-5.2.3 Water Bath

B-5.2.4 Separating Funnels — 60 ml and 100 ml.

B-5.2.5 Injection Syringes $-1 \mu l$, 5 μl and 10 μl .

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

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

Amend No. 1 to IS 1397 : 1990 B-5.3 Reagents

B-5.3.1 *Acetone*

B-5.3.2 n-Hexane

B-5.3.3 Acetic Anhydride

B-5.3.4 Sodium Sulphate Anhydrous

B-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.

B-5.3.6 PCP Standard Solution

Dilute 10 ml of stock solution with acetone to 100 ml. One millilitie of this solution contains 0.01 mg of pentachlorophenol.

B-5.3.7 Internal Standard Stock Solution

Dissolve 1 g of 2, 4 dibromophenol in 1 1 of acetone. One millilitre of this solution contains 1 mg of dibromophenol.

B-5.3.8 Internal Standard Solution

Dilute 1 ml of the stock solution (**B-5.3.7**) with acetone to 100 ml. One millilitre of this solution contains $10 \ \mu g$ of dibromophenol.

B-5.4 Procedure

B-5.4.1 Determine the moisture content of the sample as given in **9** of IS 1060 (Part 1) : 1966.

B-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.

B-5.4.3 Clean Up

Transfer the extract (**B-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 evaporte nearly to dryness.

B-5.4.4 Treat the residue (**B-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.

B-5.4.5 Inject 2 μ l 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.

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

B-5.5 Calculation

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

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

where

A = PCP content in µg from the graph;

- B =total volume of hexane extract before derivatization, ml;
- V_i = volume of eluent injected, ml;
- M = mass of the paper sample taken in g;
- C = volume of hexane extract carried through derivatization, ml;
- $V_{\rm t}$ = volume of total eluent, ml; and
- X = moisture content, percent by mass.

B-6 DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)

B-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.

B-6.2 Apparatus

B-6.2.1 *Erlenmeyer Flask* — 200 ml.

B-6.2.2 Water Bath

B-6.2.3 Separating Funnels — 60 ml and 100 ml.

B-6.2.4 Injection Syringes $-1 \mu l$, 5 μl and 10 μl .

B-6.2.5 Gas Chromatograph with ECD — Capillary columns.

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

B-6.3 Reagents

B-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 $% \operatorname{Max}$ may also be used.

B-6.3.2 n-Hexane

B-6.3.3 Sodium Sulphate Anhydrous

B-6.3.4 PCB Stock Solution

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

B-6.3.5 PCB Standard Solution

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

B-6.3.6 Internal Standard Stock Solution

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

B-6.3.7 Internal Standard Solution

Dilute 1 ml of the stock solution (**B-6.3.6**) with hexane to 100 ml. One millilitre of this solution contains 10 μ g of dibromophenol.

B-6.4 Procedure

B-6.4.1 Determine the moisture content of the sample as given in 9 of IS 1060 (Part 1) : 1966.

B-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.

B-6.4.3 The hexane extract is filtered, dried over anhydrous sodium sulphate, and evaporated under vacuum to approximately 5 ml and the residue is taken for clean up.

B-6.4.4 Clean up

Transfer the hexane extract (**B-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.

B-6.4.5 Inject 2 μ l 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.

B-6.4.6 Calibration

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

Amend No.1 to IS 1397 : 1990 **B-6.5** Calculation

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

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

where

 $A = PCB \text{ content in } \mu \text{g from the graph;}$ $V_i = \text{volume of eluent injected, ml;}$ M = mass of the paper sample taken in g; X = moisture content, percent by mass; $V_t = \text{volume of total eluent, ml; and}$ X = moisture content, percent by mass.

(CHD 16)

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Indian Standard

KRAFT PAPER – SPECIFICATION (Second Revision)

1 SCOPE

1.1 This Indian Standard prescribes the requirements and methods of sampling and test for kraft paper for wrapping and general packing purposes.

2 REFERENCES

2.1 The Indian Standards listed below are the necessary adjuncts to this standard:

IS No. Title

1060 (Part 1): Methods of sampling and test for paper and allied products: 1966 Part 1 (revised) 1060 (Part 2): Methods of sampling and test for paper and allied products: 1960 Part 2 1064 : 1980 Paper sizes (second revision) 1070 : 1977 Water for general laboratory use (second revision) 4661 : 1986 Glossary of terms used in paper trade and industry (first

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 4661 : 1986 and the following shall apply.

revision)

3.1.1 Kraft Paper

It is a paper made from the unbleached pulp and complying with the requirements as laid down in this standard.

4 GRADES

Kraft paper shall be of three grades, namely, Grade 1, Grade 2, and Grade 3.

5 REQUIREMENTS

5.1 Materials

5.1.1 Grade 1

Generally it is made from 100 percent unbleached sulphate pulp, or from a mixture of bamboo pulp and wood pulp, or from any other equivalent pulp that will ensure compliance with the requirements given for this grade of kraft paper in Table 1. Grade 1 kraft is normally called virgin kraft by the paper trade and industry.

5.1.2 Grade 2

It may be made from bagasse, rice/wheat straw, grass, jute, a mixture of these along with sulphate pulp or any other equivalent materials that will ensure compliance with the requirements given for this grade of kraft paper in Table 1. Grade 2 kraft is normally called semivirgin kraft by the paper trade and industry. It is designated as agricultural residue kraft (AR kraft).

5.1.3 Grade 3

It may be made from 100 percent waste paper or a mixture of waste paper and agricultural waste or any other material that will ensure compliance with the requirements given for this grade of kraft paper in Table 1. Grade 3 kraft is normally called non-virgin kraft by the paper trade and industry.

5.2 Finish

5.2.1 Grade 1

The surface shall be machine glazed, ribbed or plain or machine finished. The paper shall be of uniform formation, thickness and substance. It will be free from specks, shives, foreign matter, holes and other blemishes. The surface should also be receptive to printing.

5.2.2 Grade 2

The surface shall be machine finished or machine glazed with reasonably good formation, thickness and substance. It shall generally be free of specks, shives, foreign matter, holes and other blemishes. The surface should be receptive to printing.

5.2.3 Grade 3

The surface shall be machine glazed with reasonably good formation and thickness and moderately uniform substance. Specks, shives and foreign matter, although expected to be present, shall be within acceptable limits.

5.3 Size

The size of the rolls shall be as agreed to between the purchaser and the supplier. When in the sheet form, it shall be either of Al or any other size of A-series as given in Table 1 of IS 1064 : 1980. The permissible tolerance on the size shall be in accordance with 4 of IS 1064 : 1980.

5.4 Substance

The substance of kraft paper shall be as agreed to between the purchaser and the supplier. A tolerance of \pm 5 percent shall be permitted on the nominal substance when tested in accordance with **6** of IS 1060 (Part 1): 1966.

5.5 The paper shall also comply with the requirements as given in Table 1.

5.6 Optional Requirements

When agreed to between the purchaser and the supplier, Grade 1 of the kraft paper shall also comply with the requirements given in Table 2.

6 PACKING AND MARKING

6.1 A ream of 500 sheets shall be the measure of quantity for kraft paper in sheets. Packages shall contain 500 or 250 sheets according to the size and the weight of the paper and packed as agreed to between the purchaser and the vendor.

6.2 Kraft paper in rolls shall be rolled on a core of 70-75 mm inside diameter and in length corresponding to the width of the paper, with a wooden plug at each end extending to a minimum of 75 mm into the core.

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

Package

- a) Description, substance and grade of the paper;
- b) Contents of the package (number of sheets);
- c) Weight in kg per ream of 500 sheets including wrapping paper;
- d) Size in millimetres;
- e) Machine direction;
- f) Lot number;
- g) Month and year of manufacture; and
- h) Trade-mark, if any.

Roll

- a) Description, substance and grade of the paper;
- b) Length and width of the roll;
- c) Weight in kg of the roll including the weight of the core and the plugs;
- d) Lot number;
- e) Month and year of manufacture; and
- f) Trade-mark, if any.

Table 1 Requirements for Kraft Paper

(*Clause* 5.5)

SI No.	Characteristic	Requirement			Method of Test, Ref
(1)	(2)	Grade 1 (3)	Grade 2 (4)	Grade 3 (5)	(Part 1) : 1966 (6)
i)	Moisture content, percent <i>Max</i> (as received without condi- tioning)	9	9	9	9
ii)	$\rho H, Min$	5.5	5.5	5.5	10
iii)	Burst index KPam ² /g, Min	2.45	1.95	1.50	12.5
iv)	Tear index : each direction mN.m/g <i>Min</i>	8.80	6.85	4.90	12.7
v)	Tensile index N. m/g <i>Min</i> MD	64.0	44.0	29.5	12.3 & 12.4
	C D	34.5	24.5	15.5	
vi)	Folding endurance : CD <i>Min</i> (No. of double folds) 60 g/m ²	30	18	10	12.6
	80 g/m^2	40	25	16	
vii)	Cobb value, 60 secs 27°C				
	Front Max	20	21	25	13.2.2
	Back Max	20	21	25	
]	NOTE : Burst factor = E Tear factor = T Breaking length Km = T	Burst index \times 10.2 The rear index \times 10.2 The rear index \times 0.1	02		

		X	,			
SI No.	Characteristic	Requirement	Method of Test, Ref to			
			Annex	Cl No.in IS 1060 (Part 1): 1966	Cl No. in IS 1060 (Part 2): 1960	
(1)	(2)	(3)	(4)	(5)	(6)	
i)	pН	5.5 to 7.5		10	—	
ii)	Ash, percent, Max	7.5		11	—	
iii)	Elongation at break, percent, each direction, <i>Min</i>	1.5		12.3		
iv)	Chlorides (as NaCl), per- by weight, <i>Max</i>	0.02		—	17	
v)	Sulphates (as Na_2SO_4), per- cent by weight, <i>Max</i>	0.12		—	18	
vi)	Fatty and/or similar acids (as $C_{17}H_{33}COOH$), percent by weight, Max	0.25	_	_	19	
vii)	Alkalinity (as $CaCO_3$), percent by weight, Max	2.0	А			

Table 2Optional Requirements for Kraft Paper, Grade 1

(Clause 5.6)

7 SAMPLING

7.1 Representative samples for the test shall be drawn as prescribed in 3 of IS 1060 (Part 1): 1966.

7.2 Number of Tests

Each of the rolls/packages selected from the lot (*see* 7.1) shall first be examined for the requirements given in 5.3. Then from each of these rolls/packages, a sheet of suitable size shall be cut out after removing at least the top three layers. Test pieces shall then be cut from these sheets for testing the various requirements mentioned in 5.2, 5.4, 5.5 and 5.6. A roll or sheet not meeting the requirements for any one or more of these characteristics shall be considered as defective. Test for *p*H shall be conducted on a composite sample.

7.3 Criteria for Conformity

A lot shall be declared as conforming to the requirements of this specification if the

requirement for pH is satisfied and if the number of defective rolls and sheets does not exceed the acceptance number. This acceptance number shall depend on the size of the sample (*see* **8.1**) 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.

8 TESTS

8.1 Tests shall be carried out as prescribed in the method referred to in col 6 of Table 1 col 4, 5 and 6 of Table 2.

8.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table 2, Item* (vii)]

DETERMINATION OF ALKALINITY

A-1 REAGENTS

A-1.1 Hydrochloric Acid

0.02 *N*.

A-1.2 Standard Sodium Hydroxide Solution 0.1 N.

A-2 PROCEDURE

Place about 5 g, accurately weighed sample of paper (cut into small pieces) in a stoppered bottle containing 250 ml of 0.02 N hydrochloric acid. Allow the mixture to stand for about one hour with occasional shaking. Decant a portion of this solution and titrate a measured

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quantity against 0.1 N sodium hydroxide solution using methyl orange as indicator. Carry out a blank titration taking the same volume of hydrochloric acid as of the solution taken in the previous titration.

A-3 CALCULATIONS

Alkalinity (as CaCo₃), percent by mass

$$=\frac{1\,250\,(A-B)\,N}{V\,W}$$

where

- A = volume of 0.1 N sodium hydroxide required for the blank titration.
- B = volume of 0.1 N sodium hydroxide required for the extract,
- N =normality of sodium hydroxide,
- V = volume of the extract taken for the titration, and
- W = weight of sample taken.

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