# AMENDMENT NO. 2 JANUARY 1998

# TO

# IS 3962 : 1967 SPECIFICATION FOR WAXED PAPER FOR GENERAL PACKAGING

( *Page* 3, *clause* **0.2** ) — Insert the following after **0.2** and renumber the subsequent clause:

'**0.3** 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, 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, in addition to the compliance with the optional 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 waxed paper for general packaging.'

(*Page* 4, *clause* **4.5**) — Insert the following new clauses after **4.5:**

'**4.6 Additional Requirements for ECO Mark**

**4.6.1** *General Requirements*

**4.6.1.1** The product shall conform to the requirements for quality and performance prescribed under **4.1** to **4.5.**

**Price Group 1**

**4.6.1.2** The manufacturer shall produce to BIS, the environmental consent clearance from the concerned State Pollution Control Board as per the provisions of *Water (Prevention and Control of Pollution) Act,* 1974 and *Air (Prevention and Control of Pollution) Act,* 1981 along with the authorization, if required under the *Environment (Protection) Act,* 1986 and the Rules made thereunder, while applying for ECO Mark. Additionally the manufacturers shall also comply with the provisions under *Prevention of Food Adulteration Act,* 1954 and the Rules made thereunder wherever necessary.

**4.6.2** *Specific Requirements*

**4.6.2.1** The material 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.

**4.6.2.2** 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 2 when tested according to the methods given in Appendix C.

**Table 2 Limits of Contaminants in Paper**

| 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 ($Cr^{6+}$) | — | 0.1 | 0.1 |
| Lead (Pb) | — | 3.0 | 3.0 |
| Mercury (Hg) | — | 0.3 | 0.3 |
| Pentachlorophenot (PCP) | 0.05 | 0.05 | 0.05 |
| Polychlorinated biphenyls (PCBs) | 2.0 | 2.0 | 0.5 |

( *Page* 5, *clause* **5.2.1** **)** — Insert the following new clauses after **5.2.1:**

**'5.3 Additional Requirements for ECO Mark**

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

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

**5.3.3** The waxed paper may be sold along with instruction for proper use and mode of safe disposal so as to maximise its performance and minimise wastage.

**5.3.4** It shall be suitably marked on waxed 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* 7, *Appendix B*) — Insert the following after Appendix B :

# APPENDIX C

(*Clause* 4.6.2.2)

## 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 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 absorptiometer 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 millilitre 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 miliilitre 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 miliilitre 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*<br>ml | *Corresponding to* $Cr^{6+}$<br>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 *p*H to 1.0± 0.3 in each flask and dilute to 100-ml. Add 2.0-ml diphenylcarbazide solution, mix thoroughly and wait for 10 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 port 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.

**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 diphenylcaroazide solution, mix thoroughly, and wait for 10 minutes.

**C-1.4.2.4** *Photometric measurement*

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

**C-1.4.3** *Calculation*

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

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

where

m = mass of chromium determined in the aliquot of the sample solution, 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 continuously 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** *Potasium 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.

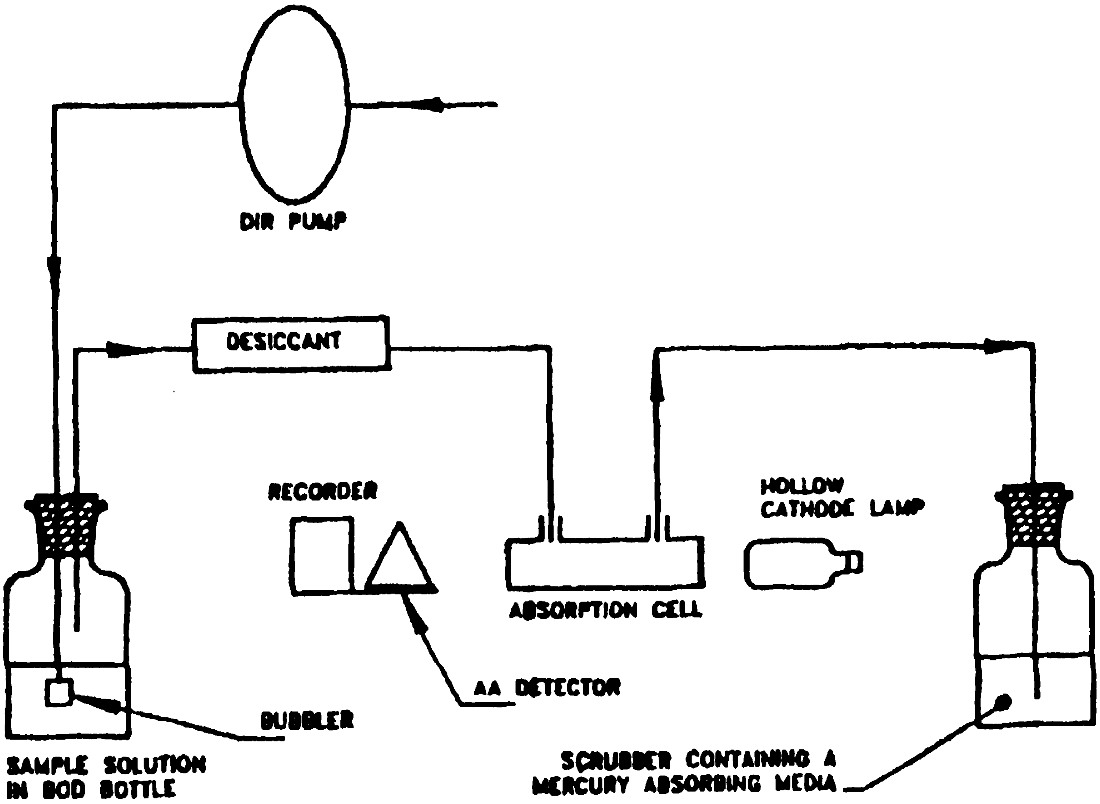

FIG. 1 SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR MEASUREMENT OF MERCURY BY COLD VAPOUR ATOMIC ABSORPTION TECHNIQUE

**C-2.4.2** *Standardization*

Transfer 100-ml of each of the 1.0, 2.0 and 5.0 µg/1 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 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 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/1 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:

$$\text{Mercury (as Hg), ppm} = \frac{C \times V}{M \times 1\,000}$$

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 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 $Pb(NO_3)_2$ in a mixture of 10-ml of concentrated $HNO_3$ and 100-ml of water and dilute to 1 litre. One millilitre 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 millilitre 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/1 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. Analyse 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 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** *Diluite 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 millilitre 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 millilitre 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. Analyse each standard solution and measure the absorbances.

**C-4.4.6** *Calculation*

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

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

where

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

## C-5 DETERMINATION OF PENTACHLOROPHENOL (PCP)

### C-5.1 Principle

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

### C-5.2 Apparatus

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

**C-5.2.2** *Soxhlet Extractor*

**A-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 11 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 **9** of IS 1060 (Part 1) : 1966.

**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 evaporte nearly to dryness.

**C-5.4.4** Treat the residue (**C-5.4.3**) with 1-ml of acetic anhydride, and heat on a water bath for about 30 min. Remove the flask from the water bath and cool the acetylated product. Transfer the content in a separating funnel and add 10-ml of hexane and 5-ml distilled water. Shake well for 2 minutes and let the layers be separated. Collect the hexane layer, dry over anhydrous sodium sulphate and evaporate nearly to dryness. Cool for at least 10 minutes, add 1-ml of internal standard solution and adjust the volume to 5.0-ml with *n*-hexane.

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

**C-5.4.6** *Calibration*

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

**C-5.5 Calculation**

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

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

where

$A$ = PCP content in µg from the graph;
$B$ = total volume of hexane extract before derivetization, 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 millilitre 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 millilitre 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 1l of *n*-hexane. One millilitrc 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 millilitre 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 **9** of IS 1060 (Part 1) : 1966.

**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 seperating 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 hexanc 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.

(CHD 16)

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# AMENDMENT NO. 3 DECEMBER 2003
# TO
# IS 3962 : 1967 SPECIFICATION FOR WAXED PAPER FOR GENERAL PACKAGING

( *Page* 5, *Table* 1, *Sl No.* (i), *col* 2 ) — Substitute 'Grammage' *for* 'Substance'.

[ *Page* 5, *Table* 1, *Sl No.*(iii) ] — Substitute the following for the existing:

| '(1) | (2) | (3) | (4) | (5) | (6) | (7) | (8) | (9) |
|---|---|---|---|---|---|---|---|---|
| iii) | Burst index, kPa.m$^2$/g, *Min* | 0.98 | 0 98 | 0.98 | 1.96 | — | 12.5 | — |

( CHD 15 )

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Reprography Unit, BIS, New Delhi, India

# AMENDMENT NO. 4 AUGUST 2004
# TO
# IS 3962 : 1967 SPECIFICATION FOR WAXED PAPER FOR GENERAL PACKAGING

( *Page* 5, *clause* **5.2** ) — Insert the following at the end:

'f) Grammage of paper.'

( CHD 15 )

Reprography Unit, BIS, New Delhi, India

IS : 3962 - 1967
( Reaffirmed 2009 )
(Reaffirmed 2014)
**(Reaffirmed 2019)**

# *Indian Standard*

# SPECIFICATION FOR WAXED PAPER FOR GENERAL PACKAGING

Third Reprint FEBRUARY 1984
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**INDIAN STANDARDS INSTITUTION**
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

**Gr 2** *January* 1968

# *Indian Standard*
# SPECIFICATION FOR WAXED PAPER FOR GENERAL PACKAGING

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# *Indian Standard*
# SPECIFICATION FOR WAXED PAPER FOR GENERAL PACKAGING

## 0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 10 August 1967, after the draft finalized by the Paper Products and Packaging Materials Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard has been formulated in order to define the quality and to assure the supply of proper quality of waxed papers for general packaging. The term general packaging, as usually accepted, includes carton overwraps, poster waxed paper for inside lining in wooden boxes for textile industry and waxed paper for many other miscellaneous general end uses. Another standard, namely, IS : 3263-1965*, has already been published giving requirements for waxed paper for confectionery.

**0.3** 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†. 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

**1.1** This standard prescribes the requirements and methods of sampling and test for waxed paper for general packaging.

## 2. TERMINOLOGY

**2.1** For the purpose of this standard, the definitions given in IS : 4261-1967‡ shall apply.

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*Specification for waxed paper for confectionery.

†Rules for rounding off numerical values ( *revised* ).

‡Glossary of terms relating to paper and pulp based packaging materials.

## 3. TYPES

**3.1** The waxed paper shall be of four types, depending on the nature of the paper as given below:

Type 1 : Waxed bleached poster paper,
Type 2 : Waxed greaseproof paper,
Type 3 : Waxed bleached or unbleached tissue paper, and
Type 4: Waxed kraft paper.

**3.1.1** All the four types may be coated on one or both sides or impregnated with paraffin wax, micro-crystalline wax and their blends.

## 4. REQUIREMENTS

**4.1 Description** — All the types of paper shall be uniform in thickness and shall not crack on folding, nor have any extraneous odour. The waxed paper shall be heat-sealed when heated and pressed together and shall have a reasonably low permeability to water vapour. When heat-sealed, the print, if any, shall not bleed.

**4.2 Base Paper** — Base paper for Types 1, 2 and 3 shall conform to the requirements of either Grade 1 or Grade 2 of IS : 2991-1965*, while the base paper for Type 4 shall conform to Grade 2 of IS : 1397-1967†.

**4.3 Wax** — The paraffin wax, microcrystalline wax or their blends shall have a minimum melting point of 60°C when tested according to the method given in Appendix A.

**4.4 Blocking Resistance** — The waxed paper shall not have any blocking when tested at a temperature of 38°C and 75 percent relative humidity for 24 hours when tested according to **10** of IS : 4006 ( Part I )-1966‡.

**4.5** All the types of waxed paper shall also comply with the requirements given in Table 1, when tested according to the test methods referred to in col 7, 8 and 9 of Table 1.

## 5. PACKING AND MARKING

**5.1** The waxed paper shall be packed securely and suitably as agreed to between the purchaser and the supplier.

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

a) Name of the material,
b) Weight of the package,

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*Specification for base paper for waxed paper.

†Specification for kraft paper ( *first revision* ).

‡Methods of test for paper and pulp based packaging materials: Part I.

c) Batch number,
d) Date of manufacture, and
e) Manufacturer's name or trade-mark or both.

**5.2.1** Each package may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

**TABLE 1 REQUIREMENTS FOR WAXED PAPER**

( *Clauses* 4.5 and 6 1.1 )

| SL No. | CHARACTERISTIC | REQUIREMENT FOR | | | | METHOD OF TEST, REF TO CL No. IN | | |
|---|---|---|---|---|---|---|---|---|
| | | Type 1 | Type 2 | Type 3 | Type 4 | Appendix | IS : 1060 ( Part 1 )- 1966* | IS : 1060 ( Part II ) 1960† |
| (1) | (2) | (3) | (4) | (5) | (6) | (7) | (8) | (9) |
| i) | Substance, g/m$^2$, *Min* | 40 | 40 | 25 | 50 | | 6 | – |
| ii) | Waxcoating content, percent by weight, *Min* | 20 | 20 | 30 | 30 | B | | |
| iii) | Burst factor, *Min* | 10 | 10 | 10 | 20 | — | 12.5 | — |
| iv) | Water vapour permeability, g/m$^2$ per 24 h at 38°C and 90±2 percent relative humidity, *Max:* | | | | | | | 14 |
| | Creased | 275 | 60 | 75 | 275 | | | |
| | Uncreased | 130 | 15 | 25 | 130 | | | |

*Methods of sampling and test for paper and allied products: Part I ( *revised* ).

†Methods of sampling and test for paper and allied products: Part II.

## 6. SAMPLING AND CRITERION FOR CONFORMITY

**6.1** Representative test samples shall be drawn as prescribed in 3 of IS : 1060 ( Part I )-1966*.

**6.1.1** *Number of Tests* — From each of the packets selected from the lot (**6.1**), one sheet shall be taken out at random. These sheets shall constitute the sample. From each of the sheets selected in the sample, one test piece shall be cut for each of the characteristics mentioned in Table 1, unless indicated otherwise, in the respective test methods. These test pieces shall be tested for blocking resistance and other requirements given in Table 1. A sheet not meeting the requirements for any one or more characteristics shall be considered as 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 docs not exceed the acceptance number. This acceptance number shall depend upon the size of the sample and shall be equal to 0 if the size is less than 13 and equal to 1 if it is greater than or equal to 13.

# APPENDIX A

( *Clause* 4.3 )

## DETERMINATION OF MELTING POINT OF WAX

### A-1. APPARATUS

**A-1.1 Thermometer** — of a suitable type, with an accuracy of 0.1°C and graduated at every 0.1°C.

**A-1.2 Test-Tube** — with a centrally bored cork for the insertion of the thermometer. The cork shall have a slit so as to permit circulation of air.

**A-1.3 Water-bath** — of a suitable type, with a thermometer.

### A-2. PROCEDURE

**A-2.1** Melt the sample of wax by warming it in a water-bath at a temperature just sufficient to melt it. Dip the thermometer and withdraw so as to get the bulb thinly coated with the wax. Let it cool. Insert this thermometer into the test-tube through the bored cork and then place the test-tube in the water-bath. Raise the temperature gradually, at the rate of 1°C, in three minutes. Note the temperature, accurately to 0.1°C, at which a transparent drop forms on the end of the thermometer bulb. Record this temperature as the melting point of the wax.

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*Methods of sampling and test for paper and allied products: Part I ( *revised* )

# APPENDIX B

[ *Table* 1, *Item* (ii) ]

## DETERMINATION OF WAX CONTENT

## B-1. APPARATUS

### B-1.1 Soxhlet Extractor

## B-2. REAGENTS

### B-2.1 Carbon Tetrachloride

**B-2.2 Alcoholic Potassium Hydroxide Solution** – approximately 0.5 N.

**B-2.3 Petroleum Ether** — boiling range 40-60°C.

## B-3. PROCEDURE

**B-3.1** Weigh accurately about 3 g of the paper, cut it into strips and fold into numerous small crosswise folds. Extract the strips in a Soxhlet extractor with carbon tetrachloride for 4 hours or until the wax is all removed.

**B-3.2** Evaporate the extract on a water-bath to dryness and add 25 ml of alcoholic potassium hydroxide solution and again evaporate to dryness. Cool the residue and take it up with petroleum ether and water and transfer to a separatory funnel. ( The volume of petroleum ether in the funnel should be about 25 ml and that of water 150 ml. The water should contain a small amount of sodium chloride to prevent emulsification. ) Shake the contents of the funnel thoroughly and then allow the two layers to separate completely. Draw off the water layer into a second separatory funnel.

**B-3.3** Re-extract the water layer one or more times, as may be found necessary, with a fresh 25-ml portion of the petroleum ether and wash with fresh 100-ml portions of water until the separated liquid is perfectly clear. An addition of a strong solution of salt may be found necessary.

**B-3.4** Transfer the petroleum ether extract to a weighed evaporating dish or flask and evaporate to dryness. Dry at 103 ± 2°C for one hour, cool and weigh.

## B-4. CALCULATION

**B-4.1** Wax, percent by weight $= \frac{100\,w}{W}$

where

$w$ = weight in g of the wax in the specimen, and
$W$ = weight in g of the paper taken for the test.

## INTERNATIONAL SYSTEM OF UNITS ( SI UNITS )

### Base Units

| *Quantity* | *Unit* | *Symbol* |
|---|---|---|
| Length | metre | m |
| Mass | kilogram | kg |
| Time | second | s |
| Electric current | ampere | A |
| Thermodynamic temperature | kelvin | K |
| Luminous intensity | candela | cd |
| Amount of substance | mole | mol |

### Supplementary Units

| *Quantity* | *Unit* | *Symbol* |
|---|---|---|
| Plane angle | radian | rad |
| Solid angle | steradian | sr |

### Derived Units

| *Quantity* | *Unit* | *Symbol* | *Definition* |
|---|---|---|---|
| Force | newton | N | 1 N = 1 kg.m/s$^2$ |
| Energy | joule | J | 1 J = 1 N.m |
| Power | watt | W | 1 W = 1 J/s |
| Flux | weber | Wb | 1 Wb = 1 V.s |
| Flux density | tesla | T | 1 T = 1 Wb/m$^2$ |
| Frequency | hertz | Hz | 1 Hz = 1 c/s ( s$^{-1}$ ) |
| Electric conductance | siemens | S | 1 S = 1 A/V |
| Electromotive force | volt | V | 1 V = 1 W/A |
| Pressure, stress | pascal | Pa | 1 Pa = 1 N/m$^2$ |

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