

इंटरनेट

मानक

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Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 12999 (1990): Folding box board, coated [CHD 15: Paper and its products]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”



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IS 12999 : 1990  
Reaffirmed 2008

भारतीय मानक

मुड़ाऊ बाक्स बोर्ड, लेपित – विशिष्ट

*Indian Standard*

**FOLDING BOX BOARD, COATED –  
SPECIFICATION**

UDC 676.272

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

February 1991

Price Group 2

## FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Paper and Pulp Based Packaging Sectional Committee had been approved by the Chemical Division Council

This standard has been formulated to define the quality of folding box board, coated.

Boards for such purposes are generally coated on one side and are termed as chromo boards.

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. 2 MARCH 2006**  
**TO**  
**IS 12999 : 1990 FOLDING BOX BOARD, COATED —**  
**SPECIFICATION**

( *Page 2, Table 1, col 2, Sl No.9* ) — Substitute 'Burst index\*, *Min*' for 'Burst index\*'

( *Page 2, Table 1, col 2, Sl No. 10* ) — Substitute 'Moisture ( as received ), percent by weight' for 'Moisture (as received), percent by weight, *Min*'

( CHD 15 )

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Reprography Unit, BIS, New Delhi, India

**AMENDMENT NO. 1 JANUARY 1998**  
**TO**  
**IS 12999 : 1990 FOLDING BOX BOARD, COATED —**  
**SPECIFICATION**

*(Second cover page, Foreword)* — Insert the following as fourth 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, 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 199S 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 folding box board, coated.'

*(Page 1, clause 4.4)* — Insert the following new clauses after **4.4**:

**4.5 Additional Requirements for ECO Mark**

**4.5.1 General Requirements**

**4.5.1.1** The product shall conform to the requirements for quality and performance prescribed under **4.1** to **4.4**.

#### Amend No. 1 to IS 12999 : 1990

**4.5.1.2** 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.

#### 4.5.2 Specific Requirements

**4.5.2.1** 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.

**4.5.2.2** 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 Annex E.

**Table 2 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 <sup>6+</sup> )	—	0.1	0.1
Lead(Pb)	—	3.0	3.0
Mercury (Hg)	—	0.3	0.3
Pentachlorophenol (PCP)	0.05	0.05	0.05
Potychlorinated biphenyls (PCBs)	2.0	2.0	0.5



**Amend No. 1 to IS 12999 : 1990**

(Page 1, clause 5.2) — Insert the following new clauses after 5.2:

**5.3 Additional Requirements for ECO Mark**

**5.3.1** For ECO Mark, folding box board shall be packed in such packages which shall be recyclable/reusable or biodegradable.

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

**5.3.3** The folding box board shall 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 that ECO Mark label is applicable only to the folding box board 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 4, Annex D) — Insert the following after Annex D:

**ANNEX E**

(Clause 4.5.2.2)

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

**E-1 DETERMINATION OF CHROMIUM (as Cr<sup>6+</sup>)**

**E-1.1 Principle**

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

**E-1.2 Apparatus**

**E-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.

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**E-1.2.2 Shaker**

Any shaker suitable for rotating/moving at  $30 \pm 2$  rev/min.

**E-1.3 Reagents**

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

**E-1.3.2 Nitric Acid** — Concentrated.

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

**E-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.

**E-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  $\mu\text{g}$  of chromium (as  $Cr^{6+}$ ).

**E-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  $\mu\text{g}$  of chromium (as  $Cr^{6+}$ ).

**E-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  $\mu\text{g}$  of chromium (as  $Cr^{6+}$ ).

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

**E-1.4 Procedure**

**E-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:

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<i>Standard Chromium Solution</i>	<i>Corresponding to Cr<sup>5+</sup></i>
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

**E-1.4.1.1** Add sulphuric acid to adjust the solution pH to  $1.0 \pm 0.3$  in each flask and dilute to 100 ml. Add 2.0 ml diphenylcarbazide solution, mix thoroughly and wait for 10 minutes.

**E-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.

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

**E-1.4.2** *Determination*

**E-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.

**E-1.4.2.2** *Preparation of test solution*

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

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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.

**E-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 pH of the solution to  $1.0 \pm 0.3$  by adding 0.2 N sulphuric acid. Dilute to 100 ml. Add 2 ml diphenylcarbazide solution, mix thoroughly, and wait for 10 minutes.

#### **E-1.4.2.4 Photometric measurement**

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

#### **E-1.4.3 Calculation**

By means of the calibration curve (see **E-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.

## **E-2 DETERMINATION OF MERCURY (as Hg)**

### **E-2.1 Principle**

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

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**E-2.2 Apparatus**

**E-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.

**E-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.

**E-2.2.3 Mercury Hollow Cathode Lamp**

**E-2.2.4 Recorder/Printer/Display Meter**

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

**E-2.3 Reagents**

**E-2.3.1 Sulphuric Acid** — Concentrated.

**E-2.3.2 Nitric Acid** — Concentrated.

**E-2.3.3 Stannous Chloride Solution**

Dissolve 25 g of stannous chloride ( $\text{SnCl}_2$ ) in water containing 50 ml of concentrated hydrochloric acid and dilute to 250 ml. If a suspension forms, stir reagent continuously during use.

**E-2.3.4 Sodium Chloride** — *Hydroxylamine Sulphate Solution*

Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulphate ( $\text{NH}_2\text{OH}$ )<sub>2</sub>  $\text{H}_2\text{SO}_4$  in distilled water and dilute to 100 ml.

**E-2.3.5 Potassium Permanganate Solution**

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

**E-2.3.6 Potassium Persulphate Solution**

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

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**E-13.7 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.

**E-2.3.8 Standard Mercury Solution**

Prepare a series of standard mercury solutions containing 0 to 5  $\mu\text{g}/\text{l}$  by appropriate dilution of stock mercury solution (E-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.

**E-2.4 Procedure**

**E-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.

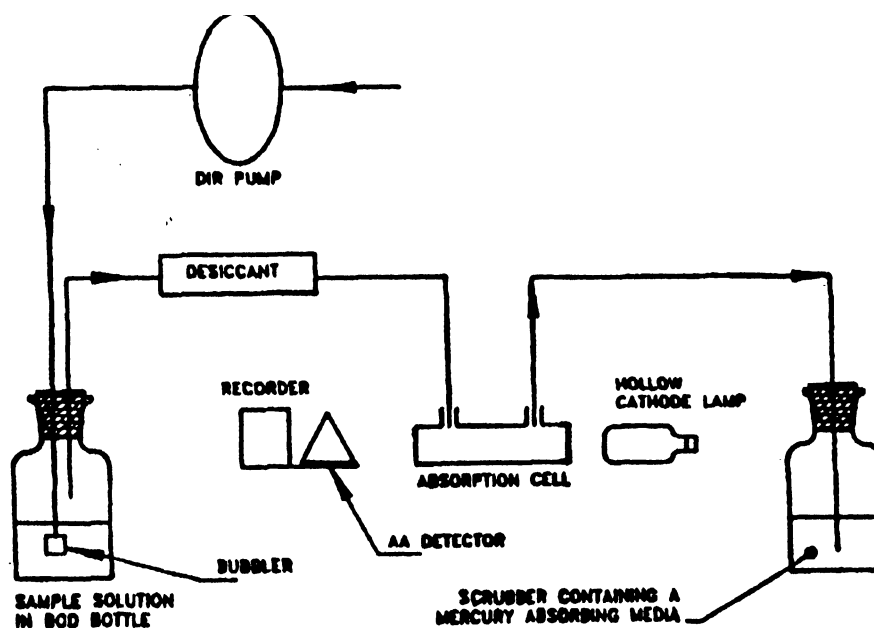


FIG. 1 SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR MEASUREMENT OF MERCURY BY COLD VAPOUR ATOMIC ABSORPTION TECHNIQUE

**E-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 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 decolorization 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.

**E-2.4.3** *Determination*

**E-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.

**E-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  $18 \pm 2$  h using shaker rotating/moving at  $30 \pm 2$  rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size. Transfer the solution quantitatively to a volumetric flask of suitable capacity, dilute to the mark and mix.

**E-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 **E-2.4.2**.

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### E-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.

### E-3 DETERMINATION OF LEAD (as Pb)

#### E-3.1 Principle

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

#### E-3.2 Apparatus

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

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

#### E-3.2.3 Hot Plate

#### E-3.3 Reagents

**E-3.3.1 Nitric Acid** — concentrated.

**E-3.3.2 Nitric Acid**— 1: 1.

**E-3.3.3 Dilute Nitric Acid** — 1 : 499.



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### **E-3.3.4** *Stock Lead Solution*

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

### **E-3.3.5** *Intermediate Lead Solution*

### **E-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).

## **E-3.4 Procedure**

### **E-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.

### **E-3.4.2** *Preparation of Test Solution*

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

**E-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.

**E-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.

**E-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 (**E-3.4.3**). Inject a suitable portion of each standard

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solution in order of increasing concentration. Analyse each standard solution and measure the absorbances.

#### **E-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.

#### **E-4 DETERMINATION OF CADMIUM (as Cd)**

##### **E-4.1 Principle**

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

##### **E-4.2 Apparatus**

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

**E-4.2.2 Cadmium Hollow-Cathode Lamp or Multielement Hollow-Cathode Lamp**— for use at 228.8 nm.

**E-4.2.3 Hot Plate**

##### **E-4.3 Reagents**

**E-4.3.1 Nitric Acid**— Concentrated.

**E-4.3.2 Nitric Acid**— 1 : 1.

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**E-4.3.3** *Dilute Nitric Acid — 1 : 499.*

**E-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).

**E-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)

**E-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).

**E-4.4 Procedure**

**E-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.

**E-4.4.2** *Preparation of Test Solution*

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

**E-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.

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**E-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.

**E-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.

#### **E-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.

### **E-5 DETERMINATION OF PENTACHLOROPHENOL (PCP)**

#### **E-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).

#### **E-5.2 Apparatus**

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

**E-5.2.2** *Soxhlet Extractor*

**E-5.2.3** *Water Bath*

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

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

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

**E-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.

**E-5.3 Reagents**

**E-5.3.1** *Acetone*

**E-5.3.2** *n-Hexane*

**E-5.3.3** *Acetic Anhydride*

**E-5.3.4** *Sodium Sulphate Anhydrous*

**E-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.

**E-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.

**E-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.

**E-5.3.8** *Internal Standard Solution*

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

**E-5.4 Procedure**

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

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**E-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.

#### **E-5.4.3 Clean Up**

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

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

**E-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.

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

#### **E-5.5 Calculation**

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

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

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where

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

**E-6 DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)**

**E-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.

**E-6.2 Apparatus**

**E-6.2.1** *Erlenmeyer Flask* — 200 ml.

**E-6.2.2** *Water Bath*

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

**E-6.2.4** *Injection Syringes* — 1  $\mu\text{l}$ , 5  $\mu\text{l}$  and 10  $\mu\text{l}$ .

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

**E-6.2.6** *Glass Columns* — Length 20 cm, internal diameter 12 mm filled with 5 cm of silica gel having particle size  $63 \mu\text{m} \times 200 \mu\text{m}$ .

**E-6.3 Reagents**

**E-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:

## **Amend No. 1 to IS 12999 : 1990**

Dissolve 15 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.

### **E-6.3.2** *n-Hexane*

### **E-6.3.3** *Sodium Sulphate Anhydrous*

### **E-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.

### **E-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.

### **E-6.3.6** *Internal Standard Stock Solution*

Dissolve 1 g of 2,4 dibromophenol in 11 of *n*-hexane. One millilitre of this solution contains 1 mg of dibromophenol.

### **E-6.3.7** *Internal Standard Solution*

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

## **E-6.4 Procedure**

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

**E-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.



**Amend No. 1 to IS 12999 : 1990**

**E-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.

**E-6.4.4** *Clean Up*

Transfer the hexane extract (**E-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.

**E-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.

**E-6.4.6** *Calibration*

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

**E-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)

Dimensions in millimetres

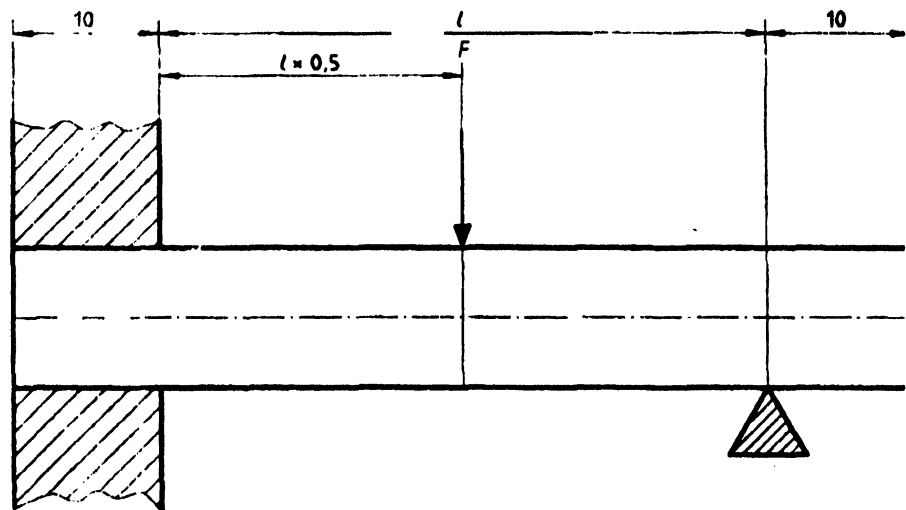


Figure 6 — Testing of residual permanent deflection of the longest straight part

**Page 12**

Insert the following new subclause:

**7.13 Dimensional stability of tents in synthetic fabrics**

Erect the tent after it has been immersed in water for 10 min. In cases where it is possible to change the tension and thus have different positions, the alternative giving the most tautness shall be chosen. Reduce the relative humidity to (0 to 5) %.

Since the temperature does not play a role in these tests, the temperature can be increased in order to achieve the lower relative humidity, if necessary, by directing a heat source at the tent.

If a heat source is used, then the surface temperature of the tent shall not exceed 60 °C.

**Page 13**

Delete figure 7.

Page 13, *subclause* 9.2

Replace "table B.1 and B.2" with "table A.1 and A.2".

**Page 14**

Delete annex A.

*Pages* 15 to 18

Annex B becomes annex A.

Therefore replace "B" with "A" in all relevant places of this annex.

# *Indian Standard*

## **FOLDING BOX BOARD, COATED – SPECIFICATION**

### **1 SCOPE**

**1.1** This standard prescribes the requirements and the methods of sampling and test for folding box board, coated, white and coloured, used for multicolour and bronze printing in line and half tone screen and subsequent conversion into folding box cartons.

### **2 REFERENCES**

**2.1** The following Indian Standards are the necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
1060 ( Part 1 ) : 1966	Methods of sampling and test for paper and allied products, Part 1 ( <i>revised</i> )
1060 ( Part 2 ) : 1960	Methods of sampling and test for paper and allied products, Part 2
4006 ( Part 1 ) : 1985	Methods of test for paper and pulp based packaging materials, Part 1 ( <i>first revision</i> )
4661 : 1986	Glossary of terms used in paper trade and industry ( <i>first revision</i> )
9894 : 1981	Methods of test of smoothness/ roughness of paper

### **3 TERMINOLOGY**

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

### **4 REQUIREMENTS**

#### **4.1 General**

The board shall be uniform in thickness, generally free from holes, hard spots and lumps, shall lie flat and be dimensionally stable. The printing surface shall be smooth, of even finish, formation, absorbency, colour and shall be clean and free from loosely bound fibres and blisters.

**4.1.1** The coating shall adhere firmly to the substrate and should not come off when rubbed with a wet cotton swab.

#### **4.2 Strength**

The board shall be stiff and after being properly creased shall fold neatly through an angle of 180° without cracking.

#### **4.3 Slitting and Cutting**

Sheets shall be cut cleanly and square to the specified dimensions. Reels shall be evenly wound. All cut edges shall be entirely free from loose fibres and dust.

**4.4** The board shall also comply with the requirements given in Table 1.

### **5 PACKING AND MARKING**

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

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

- a) Description of the material;
- b) Thickness and substance;
- c) Net mass of contents;
- d) Month and year of manufacture; and
- e) Indication of the source of manufacture.

### **6 SAMPLING AND CRITERIA FOR CONFORMITY**

**6.1** The boards shall be sampled in accordance with 3 of IS 1060 ( Part 1 ) : 1966.

#### **6.2 Tests**

From each of the packets, selected from the lot ( *see 6.1* ), the board shall be taken out at random. These boards shall constitute the sample. The boards selected shall first be tested for general requirements given in **4.1**. One test piece shall be cut, from each board selected and tested for each of the characteristics mentioned in **4.2** and Table 1. A board not meeting the requirements for any one or more characteristics shall be considered as defective.

#### **6.3 Criterion for Conformity**

A lot shall be declared as conforming to all the requirements of this specification if the number of defective boards found does not exceed the acceptance number. This acceptance number shall depend upon the size of the sample and shall be zero if the size is less than 13 and one if it is greater than or equal to 13.

**Table 1 Requirements for Folding Box Board, Coated**  
( Clauses 4.4 and 6.2 )

Sl No.	Characteristic	Requirement	Annex	Method of Test to			
				CI No. (IS 1060 (Part 1) : 1966)	CI No. (IS 1060 (Part 2) : 1966)	CI No. (IS 9894: 1981 1960)	CI No. (IS 4006 (Part 1): 1985)
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
1	Bulk	1.2	—	8	—	—	—
2	Coating g/m <sup>2</sup>	10-15	A				
3	Wax pick No.						
	Front	No Pick 6A					
	Back	No Pick 4A	B				
4	Surface pH	6.0 - 8.0	C				
5	Gloss percent, <i>Min</i>	40		15			
6	Brightness percent, <i>Min</i>	75	—	—	13		
7	Stiffness factor <i>Min</i> ( Taber instrument )	170	—	—			12.2
8	Sizing ( C 088 ) ( 60 sec 27°C )						
	Front	25		13.2.2			
	Back	Not to differ more than 25% of front					
9	Burst index*	1.4		12.5			
10	Moisture ( as received ) percent by weight, <i>Min</i>	7-8		9			
11	Ash, percent by weight, <i>Mix</i>	15		11			
12	Stain length, mm, <i>Min</i>	100	D				
13	Smoothness, ml / <i>Min</i>	60	—	—	—	4	—
	Front, Ag 60 — 100						
	Back, Ag 500 — 1 000						

$$*Burst\ index = \frac{Burst\ Strength\ (kPa)}{g/m^2}$$

The Burst Strength is read in either lbs/in<sup>2</sup> or kg/cm<sup>2</sup>. It shall be multiplied by 6.894 8 or 98.066 respectively to get the value in kPa.

## ANNEX A

( Table 1, Sl No. 2 )

### METHOD FOR DETERMINATION OF COATING

#### A-0 GENERAL

**A-0.1** This method is suitable for the quantitative determination of the coating on the ordinary types of mineral coated boards. It is not intended for special types of coated boards in which lacquers, varnishes or similar materials are used.

#### A-1 TEST SPECIMEN AND CONDITIONING

**A-1.1** The test specimen shall be cut from the sample in such a way as to be thoroughly representative of the board. The specimen shall

consist of a sheet of the board not less than 150 cm<sup>2</sup> in area.

**A-1.1.1** Condition all test specimens as prescribed in 5 of IS 1060 ( Part 1 ) : 1966.

#### A-2 REAGENTS

##### A-2.1 Enzyme Solution

Prepare an aqueous solution containing 1.5 g of a suitable enzyme and 25 ml of 0.1 N caustic soda solution per litre.

**NOTES**

**1** The enzyme recommended is trypsin, but some of the mixtures of enzymes used commercially for desizing cotton and degumming silk have been found to be more rapid in action, less expensive and more stable.

**2** While an enzyme may not be essential if starch is the only adhesive used, it will not interfere with the removal of the coating in such cases, and it is essential where casein is the binder.

**A-3 PROCEDURE**

**A-3.1** Measure the area of the test specimen and weigh it with an accuracy of  $\pm 0.5$  percent. From this data, calculate the substance of the paper according to **6** of IS 1060 ( Part 1 ) : 1966.

**A-3.2** Lay the specimen in a flat-bottomed tray, preferably of glass, with the coated side of the specimen up. Cover the specimen completely with the enzyme solution and allow to stand for at least 1 h at 50°C. Remove the test specimen from the enzyme solution, lay it on a plane glass surface, and brush off the coating, using a camel hair brush and taking care not to dislodge paper

fibres. Additional treatment with the enzyme solution and heating may be required for very resistant coatings.

**A-3.3** After the coating is entirely removed, stand the glass pane with the test specimen on it at a slight angle and, using a wash bottle, wash each side of the specimen with water while it is held against the glass by one corner. After it is thoroughly washed, dry it at room conditions, allow it to come to equilibrium with the standard atmosphere, and again weigh with an accuracy of  $\pm 0.5$  percent and calculate the substance according to **6** of IS 1060 ( Part 1 ) : 1966.

**A-4 CALCULATION**

**A-4.1** Coating in  $\text{g/m}^2 = p - q$

where

$p$  = substance, in  $\text{g/m}^2$ , of the coated board; and

$q$  = substance, in  $\text{g/m}^2$ , of the board after coating has been removed.

**ANNEX B**

( Table 1, Sl No. 3 )

**METHOD FOR DETERMINATION OF WAX PICK NUMBER****B-0 GENERAL****B-0.1 Waxes**

The Dennison standard paper testing waxes are available in a series with graded adhesive powers. The complete series consists of 18 waxes from 2A to 26A, the adhesive strength increasing with the number.

**B-1 PROCEDURE**

**B-1.1** Condition the test specimen as prescribed in **5** of IS 1060 ( Part 1 ) : 1966.

**B-1.2** Place the test specimen on a smooth surface, like a hardwood block or table glass or metal surfaces; artificially cooled surfaces shall not be used. The sample sheet should be separated from the block or table by 8 or 10 sheets of paper.

**B-1.3** Select a wax stick, with clean and flat end. Heat the end over an alcohol or low gas flame, rotating slowly until several drops of melted wax

have fallen. Take care that the wax does not catch fire.

**B-1.4** Quickly place the molten wax end on the surface of the specimen, with firm but not undue pressure, and withdraw the fingers immediately. Allow 15 minutes for the wax to cool.

**B-1.5** Place a wooden block with a hole over the wax stick, with the stick protruding through the hole in the block; press the block down firmly with one hand and with the other pull the wax stick from the sheet with a quick jerk at right angles to the board surface.

**B-1.6** Examine both the end surface of the wax stick and the specimen. Repeat the test using waxes in advancing numerical order, until the sample surface blisters, breaks, picks or lifts.

**B-1.7** Record as wax pick number, the highest numbered wax which does not disturb the surface of the board.

## ANNEX C

( Table 1, Sl No. 4 )

### METHOD FOR DETERMINATION OF SURFACE pH

#### C-1 APPARATUS

##### C-1.1 pH Meter

Any standard pH meter with glass electrode and a single combination electrode calibrated against standard buffer solution at two pH values ( see C-1.2 ).

##### C-1.2 Buffer Solutions

Two standard solutions, one with pH 4 and the other with pH 9.

##### C-1.3 Distilled Water

pH 6.0 to 7.2 and carbon dioxide free.

#### C-2 PROCEDURE

**C-2.1** Cut a test specimen approximately 50 × 50 mm from the sample sheet drawn as prescribed in 4.1. Place one large drop of distilled water on the test piece and place electrode in drop, also touching the paper. Take the reading on pH meter after about two minutes.

## ANNEX D

( Table 1, Sl No. 12 )

### METHOD FOR DETERMINATION OF STAIN LENGTH

#### D-1 APPARATUS

##### D-1.1 IGT Printability Tester

**D-1.2 Inking Disc** — 2 cm wide.

##### D-1.3 Di-n-butyl phthalate

Chemically pure stained with Sudan red IV ( 1 g per 100 ml ) and filtered through cotton wool.

**D-1.4** Syringe fitted with injection needle cut straight across very carefully to length of 25 mm.

#### D-2 SAMPLE

**D-2.1** Cut sample strips of width 25 mm and of length 25 cm in both machine and cross directions. Minimum of 5 strips from each direction are required. These strips shall be conditioned at  $65 \pm 5$  percent relative humidity and  $27 \pm 2^\circ\text{C}$  for a period of 24 h.

#### D-3 PROCEDURE

**D-3.1** The test shall be carried out at a temperature of  $27 \pm 2^\circ\text{C}$ .

**D-3.2** Mount the sample on IGT instrument in the normal printing position.

**D-3.3** Fit the inking disc on to the shaft making

sure that it snaps into position, then by means of the control provided bring the disc into contact with the segment. The spring tension should be adjusted to 70 kgf.

**D-3.4** Fill the syringe with the stained di-n-butyl phthalate and remove the piston so that the di-n-butyl phthalate may flow by its own weight from the syringe. Allow a drop of di-n-butyl phthalate to fall at top dead centre from a height of 1.5 cm on to the surface of the clean inking disc already in position on the IGT instrument. The weight of the drop shall be  $5.80 \pm 0.24$  mg.

**D-3.5** Immediately release the pendulum, letting it fall freely so that the drop of di-n-butyl phthalate is squeezed out along the surface of the sample. The resulting stain should be roughly elliptical in shape. Any other shape, for example multitailed shall be disregarded but noted in the test report.

**D-3.6** Measure the length of the stain in mm.

#### D-4 REPORTING

**D-4.1** Five tests are carried out from each lot and the result is expressed to the nearest millimetre from the average of five testings.

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