Foreword

This Bangladesh Standard was adopted by the Bangladesh Standards and Testing Institution on .... after the draft finalized by the Paper, Pulp, board and Stationery Products Sectional Committee and approved by the Chemical Divisional Committee.

Kraft liner are strong papers containing sulphate/soda pulps and other pulps. Kraft liner is attached to the corrugating medium suitably to give the corrugated board of desired quality. Kraft liner and corrugating medium used for making corrugated board boxes are normally of a substance not less than 140 g/m^2 and 80 g/m^2 respectively.

Due to its growing demand the sectional committee decided to formulate this standard. While formulating this standard the sectional committee gave due consideration to the views of the producers, consumers and technologists and felt that it should be related to the prevailing trade and manufacturing practices followed in this field in the country.

In the preparation of this standard, assistance derived from the following publications is acknowledged with thanks:

IS 9588 : 2025 Kraft Liner and Corrugating Medium — Specification (Second Revision);
Bureau of Indian Standards.

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 BDS 103.

Bangladesh Standard

Specification for Kraft Liner and Corrugating Medium

1. Scope

1.1 This standard prescribes the requirements and the methods of sampling and test for kraft liner and corrugating medium meant for making corrugated board boxes.

2. Normative references

2.1 The following Bangladesh Standards are necessary adjuncts to this Standard. For undated references the latest edition of the publication referred to applies.

BDS 103	Rules for rounding off numerical values.
BDS ISO 186	Paper and board - Sampling to determine average quality.
BDS ISO 216	Writing paper and certain classes of printed matter - Trimmed sizes - A and B series, and indication of machine direction.
BDS ISO 287	Paper and board — Determination of moisture content of a lot — Oven-drying method.
BDS ISO 535	Paper and board - Determination of water absorptiveness - Cobb method.
BDS ISO 536	Paper and board - Determination of grammage.
BDS ISO 2758	Paper — Determination of bursting strength.
BDS ISO 4046	Paper, board, pulps and related terms - Vocabulary
Part 1	Alphabetical index.
Part 2	Pulping terminology.
Part 3	Paper making terminology.
Part 4	Paper and board grades and converted products.
Part 5	Properties of pulp, paper and board.
BDS ISO 6588-2	Paper, board and pulps - Determination of pH of aqueous extracts – Part 2: Hot extraction.
ISO 12192	Paper and board — Determination of compressive strength — Ring crush method.

3. Terms and Definitions

For the purposes of this standard, then definitions given in BDS ISO 4046 part 1-5 and the following shall apply.

3.1 Liner — Kraft liner is a paper having a substance between 120 g/m^2 and 220 g/m^2 made from bleached/unbleached sulphate pulp/brown waste paper and having the required properties. It is used for outside and inside facings.

3.2 Corrugating Medium — Corrugating medium is a paper having a substance between 70 g/m^2 and 180 g/m^2 and is used for fabrication of corrugated boards.

4. Grades

4.1 Kraft liner - Kraft liner shall be of three grades, namely, Grade 1, Grade 2 and Grade 3, based on the raw materials.

4.2 Corrugating Medium – Corrugating medium shall be of three grades, namely, Grade 1, Grade 2 and Grade 3.

5. Requirements

5.1 Kraft liner

5.1.1 Manufacture

5.1.1.1 Grade 1

Kraft liner of Grade 1 is generally made from old corrugated boxes and bleached/unbleached sulphate pulp or from a mixture of bamboo pulp or wood pulp, or from any other equivalent pulp that will ensure compliance with the requirements given in Table 1. Grade 1 kraft is normally called virgin kraft by the paper trade and industry.

5.1.1.2 Grade 2

Kraft liner of Grade 2 may be made from old corrugated boxes and 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 in Table 1. Grade 2 kraft is normally called semi-virgin kraft by the paper trade and industry. It is designated as agricultural residue kraft.

5.1.1.3 Grade 3

Kraft liner of Grade 3 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 in Table 1. Grade 3 kraft is normally called non-virgin kraft by the paper-trade and industry.

5.1.2 Finish and Workmanship

5.1.2.1 The surface of kraft liner shall be machine glazed or machine finished. It shall be free from specks, shives, foreign matter, holes and other blemishes.

5.1.2.2 The paper may be of uniform formation, thickness and substance. The surface should also be receptive to printing.

5.1.3 Grammage

The grammage (substance) of kraft liner shall be 120 g/m^2 and 220 g/m^2 when tested according to the method prescribed in BDS ISO 536. A tolerance of ± 5 percent shall be permitted on the grammage.

5.1.4 The kraft liner shall also comply with the requirements given in Table 1.

5.2 Corrugating Medium

5.2.1 Grammage

5.2.1.1 Grade 1

The grammage (substance) of Grade 1 corrugating medium shall be 180 g/m^2 or 140 g/m^2 or 120 g/m^2 when tested according to the method prescribed in BDS ISO 536.

5.2.1.2 Grade 2

The grammage (substance) of Grade 2 corrugating medium shall be 180 g/m^2 or 140 g/m^2 or 120 g/m^2 or 100 g/m^2 when tested according to the method prescribed in BDS ISO 536.

5.2.1.3 Grade 3

The grammage (substance) of Grade 3 corrugating medium shall be 70 g/m^2 and 180 g/m^2 when tested according to the method prescribed in BDS ISO 536.

5.2.2 Tolerance - A tolerance of ± 5 percent shall be permitted on the grammage.

5.2.3 Corrugating medium shall also comply with the requirements given in Table 2.

Table 1 Requirements for Kraft liner

Sl. No. (1)	**Characteristic** (2)	**Requirement**			**Method of Test** (6)
		Grade 1 (3)	**Grade 2** (4)	**Grade 3** (5)	
i.	Burst index, Kpa.m^2/g, Min.	3.42	2.74	1.5	BDS ISO 2758
ii.	pH	5.5 – 9.0			BDS ISO 6588-2
iii.	Moisture content, percent, Max. (as received without conditioning)	9.0			BDS ISO 287
iv.	Cobb (60 seconds at 27°C), g/m^2, Max.				BDS ISO 535
	a) Top Side	25	30	40	
	b) Bottom Side	30	35	60	
v.	Ring crush, CD, kgf/152 mm, Min.				ISO 12192
	a) 220 g/m^2	33	32	20.0	
	b) 180 g/m^2	32	30	17.0	
	c) 170 g/m^2	32	30	15.0	
	d) 150 g/m^2	32	30	12.0	
	e) 140 g/m^2	23	22	11.5	
	f) 120 g/m^2	23	22	11.0	
NOTE – If grammage are found between the value mentioned above, nearest grammage value shall be considered for the determination of ring crush. For equal distance, lowest value shall be considered.					

Table 2 Requirements for Corrugating Medium

Sl. No. (1)	**Characteristic** (2)	**Requirement**			**Method of Test** (6)
		Grade 1 (3)	**Grade 2** (4)	**Grade 3** (5)	
i.	Burst index, Kpa.m^2/g, Min.	1.95	1.76	1.2	BDS ISO 2758
ii.	pH	5.5 – 9.5			BDS ISO 6588-2
iii.	Moisture content, percent, Max. (as received without conditioning)	9.0			BDS ISO 287
iv.	Cobb (60 seconds at 27°C), g/m^2, Max.				BDS ISO 535
	a) Top Side	40	45	50	
	b) Bottom Side	45	50	55	

Table 2 Continue

Sl. No. (1)	Characteristic (2)	Requirement			Method of Test (6)
		Grade 1 (3)	Grade 2 (4)	Grade 3 (5)	
v.	Ring crush, CD, kgf/152 mm, Min.				ISO 12192
	a) 180 g/m^2	26	24	15.0	
	b) 170 g/m^2	26	24	14.0	
	c) 150 g/m^2	19	16	12.0	
	d) 140 g/m^2	19	16	9.0	
	e) 120 g/m^2	14	10	6.5	
	f) 115 g/m^2	14	10	6.5	
	g) 110 g/m^2	-	8.5	6.0	
	h) 100 g/m^2	-	8.5	6.0	
	i) 80 g/m^2	-	-	5.5	
NOTE – If grammage are found between the value mentioned above, nearest grammage value shall be considered for the determination of ring crush. For equal distance, lowest value shall be considered.					

5.3 Size and Tolerance on Size

The size of the roll shall be as agreed to between the purchaser and the supplier. The permissible tolerance on the size shall be in accordance with BDS ISO 216.

5.4 Joints - There shall not be more than three joints in a roll. All joints shall be joined properly using a suitable adhesive tape.

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

Table 3 Maximum Permissible Limits of Contaminants in Paper

Sl. No.	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)
i.	Cadmium (Cd)	-	0.5	0.5
ii.	Chromium (Cr^{6+})	-	0.1	0.1
iii.	Lead (Pb)	-	3.0	3.0
iv.	Mercury (Hg)	-	0.3	0.3
v.	Pentachlorophenol (PCP)	0.05	0.05	0.05
vi.	Polychlorinated biphenyls (PCBs)	2.0	2.0	0.5

6. Packing and Marking

6.1 Packing

Liner paper and corrugating medium shall be packed in rolls on a core of 70 mm to 77 mm inside diameter and in length corresponding to the width of the paper, with a wooden/plastic plug at each end extending to a minimum of 75 mm into the core.

6.2 Marking

6.2.1 Each package shall be marked with the following information:

a) Description, substance and grade of material;
b) Diameter and width of roll;
c) Mass in kg of the roll including the mass of the core and plugs;
d) Lot number;
e) Month and year of manufacture;
f) Manufacturer's name and trade-mark, if any; and
g) Linear meterage (Optional).

6.2.2 The containers may also be marked with the BSTI Certification Mark.

NOTE - The use of the BSTI Certification Mark is governed by the provisions of the Bangladesh Standards and Testing Institution Act 2018 and the Rules and Regulations made there under. Details of conditions be under which a license for the use of the BSTI Certification Mark may granted to manufacturers or processors, may be obtained from the Bangladesh Standards and Testing Institution.

7. Sampling and Criteria for Conformity

7.1 Sampling

Representative samples for the test shall be drawn as prescribed in BDS ISO 186.

7.2 Number of Tests

Each of the rolls selected from the lot (see 7.1) shall first be examined for the requirements given in 5.3 and 5.4. Then from each of these rolls, a sheet of suitable size shall be cut, removing at least the top three layers. Test pieces of kraft liner shall then be cut from these sheets for testing various requirements mentioned in 5.1.1, 5.1.2, 5.1.3 and 5.1.4 and test pieces of corrugating medium shall be cut for testing various requirements mentioned in 5.2.1, 5.2.2 and 5.2.3. Tests for these characteristics except pH shall be conducted individually on each of the sample sheets. A roll or sheet not meeting the requirements for any one or more of these characteristics shall be considered as defective. Test for pH shall be conducted on 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 7.1) and shall be equal to 0 if the sample size is less than 13. It shall be equal to 1 if the sample size is greater than or equal to 13.

Annex A

(Clause 5.5; Table 3)

Determination of Chromium, Lead, Mercury, Cadmium, Pentachlorophenol and Polychlorinated Biphenyl

A-1 Determination of Chromium (as Cr^{6+})

A-1.1 Principle

The hexavalent chromium is determined calorimetrically by reaction with diphenylcarbazide in acid solution at a wave length of 550 nm.

A-1.2 Apparatus

A-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.

A-1.2.2 Shaker

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

A-1.3 Reagents

A-1.3.1 Extraction Fluid - Mix 5.7 ml of acetic acid in distilled water.

A-1.3.2 Nitric Acid - Concentrated.

A-1.3.3 Sulphuric Acid - Approximately 0.2 N.

A-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.

A-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}).

A-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 µg of chromium (as Cr^{+6}).

A-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 pg of chromium (as Cr^{+6}).

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

A-1.4 Procedure

A-1.4.1 Preparation of Calibration Curve

Into each of a series of ten 250 ml volumetric flasks, place the quantities of standard chromium solution as indicated below:

Standard Chromium Solution (ml)	Corresponding to Cr^{6+} (µg)
1.0	0.2
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

A-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.

A-1.4.1.2 Carry out the measurement on the spectrophotometer or on a photoelectric calorimeter 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.

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

A-1.4.2 Determination

A-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.

A-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 hours 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.

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

A-1.4.2.4 Photometric measurement

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

A-1.4.3 Calculation

By means of the calibration curve (see A-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.

A-2 Determination of Mercury (as Hg)

A-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.

A-2.2 Apparatus

A-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.

A-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.

A-2.2.3 Mercury Hollow Cathode Lamp

A-2.2.4 Recorder/Printer/Display Meter

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

A-2.3 Reagents

A-2.3.1 Sulphuric Acid - Concentrated.

A--2.3.2 Nitric Acid - Concentrated.

A-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.

A-2.3.4 Sodium Chloride - Hydroxylamine Sulphate Solution.

Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulphate $(NH_2OH)_2$ H_2SO4 in distilled water and dilute to 100 ml.

A-2.3.5 Potassium Permanganate Solution

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

A-2.3.6 Potassium Persulphate Solution

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

A-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 1000 ml. One millilitre of the solution contains 1 mg of mercury as Hg.

A-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 (A-2.3.7) with water containing 10 ml of concentrated nitric acid per litre. Prepare standards daily.

NOTE - Use mercury free distill water for the preparation of reagents and standards.

A-2.4 Procedure

A-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.

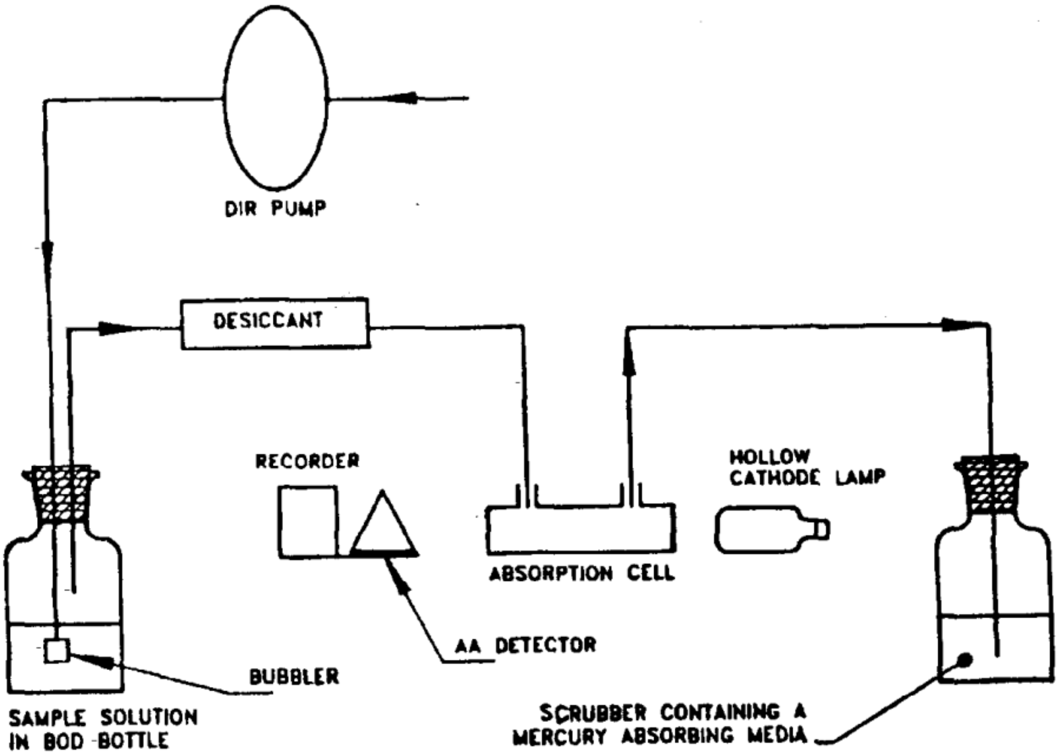

FIG. 1 Schematic Arrangement for Measurement of Mercury by
Cold Vapor Atomic Absorption Technique

A-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 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.

A-2.4.3 Determination

A-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.

A-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 hours 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.

A-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 A-2.4.2.

A-2.5 Calculation

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

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

where

C = concentration of mercury from the calibration curve;

V = volume of test solution prepared, ml; and

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

A-3 Determination of Lead (as Pb)

A-3.1 Principle

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

Spectrometric method.

A-3.2 Apparatus

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

A-3.2.2 Lead Hollow-Cathode Lamp or multielement Hollow-Cathode Lamp - for use at 283.3 nm.

A-3.2.3 Hot Plate

A-3.3 Reagents

A-3.3.1 Nitric Acid - concentrated.

A-3.3.2 Nitric Acid - 1 : 1.

A-3.3.3 Dilute Nitric Acid - 1 : 499.

A-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).

A-3.3.5 Intermediate Lead Solution

A-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).

A-3.4 Procedure

A-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.

A-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 hours using shaker rotating/moving at 30 ± 2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size.

A-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.

A-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.

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

A-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.

A-4 Determination of Cadmium (as Cd)

A-4.1 Principle

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

A-4.2 Apparatus

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

A-4.2.2 Cadmium Hollow-Cathode Lamp or Multielement Hollow-Cathode Lamp - for use at 228.8 nm.

A-4.2.3 Hot Plate

A-4.3 Reagents

A-4.3.1 Nitric Acid - Concentrated.

A-4.3.2 Nitric Acid- 1 : 1.

A-4.3.3 Dilute Nitric Acid – 1 : 499.

A-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).

A-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 miliilitre of this solution contains 50 µg of cadmium (as Cd).

A-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).

A-4.4 Procedure

A-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.

A-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 hours using shaker rotating moving at 30±2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size.

A-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.

A-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.

A-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.

A-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.

A-5 Determination of Pentachlorophenol (PCP)

A-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).

A-5.2 Apparatus

A-5.2.1 Round Bottom Flask - 250 ml.

A-5.2.2 Soxhlet Extractor

A-5.2.3 Water Bath

A-5.2.4 Separating Funnels - 60 ml and 100 ml.

A-5.2.5 Injection Syringes - 1 µg, 5 µg and 10 µg.

A-5.2.6 Gas Chromatography with ECD - Capillary columns.

A-5.2.7 Glass Columns-Length 20 cm, internal diameter 12 mm filled with 5 cm of silica gel having particle size 63 km x 200 pm.

A-5.3 Reagents

A-5.3.1 Acetone

A-5.3.2 n-Hexane

A-5.3.3 Acetic Anhydride

A-5.3.4 Sodium Sulphate Anhydrous

A-5.3.5 PCP Stock Solution

Dissolve 10 mg of pentachiorophenol in 100 ml of acetone. One millilitre of this solution contains 0.1 mg of pentachlorophenol.

A-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.

A-5.3.7 Internal Standard Stock Solution

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

A-5.3.8 Internal Standard Solution

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

A-5.4 Procedure

A-5.4.1 Determine the moisture content of the sample as given in BDS ISO 287.

A-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 hours. Filter the acetone extract, dry over anhydrous sodium sulphate, and evaporate under vacuum to a small volume approximately to 5 ml and cool.

A-5.4.3 Clean Up

Transfer the extract (A-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.

A-5.4.4 Treat the residue (A-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 acetylate dproduct. Transfer the content in a separating funnel and add 10 ml of hexane and 5ml 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.

A-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.

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

A-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 pg 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.

A-6 Determination of Polychlorinated Biphenyls (PCBs)

A-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.

A-6.2 Apparatus

A-6.2.1 Erlenmeyer Flask - 200 ml.

A-6.2.2 Water Bath.

A-6.2.3 Separating Funnels - 60 ml and 100 ml.

A-6.2.4 Injection Syringes - 1 µg, 5 µg and 10 µg.

A-6.2.5 Gas Chromatograph with ECD - Capillary columns.

A-6.2.6 Glass Columns - Length 20 cm, internal diameter 12 mm filled with 5 cm of silica gel having particle size 63 µg x 200 µg.

A-6.3 Reagents

A-6.3.1 Ethanolic Potassium Hydroxide Solution

Prepares 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 hours, filter and distill.

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

A-6.3.2 n-Hexane

A-6.3.3 Sodium Sulphate Anhydrous

A-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.

A-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.

A-6.3.6 Internal Standard Stock Solution

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

A-6.3.7 Internal Standard Solution

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

A-6.4 Procedure

A-6.4.1 Determine the moisture content of the sample as given in BDS ISO 287.

A-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 hours, 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.

A-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.

A-6.4.4 Clean Up

Transfer the hexane extract (A-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.

A-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.

A-6.4.6 Calibration

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

A-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 pg from the graph;

V_i = volume of eluent injected, ml; and

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

V_t = volume of total eluent, ml; and

X = moisture content, percent by mass.