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BUREAU OF INDIAN STANDARDS

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भारतीय मानक मसौदा

जूट बैचिंग तेल — विशिष्टि

(IS 1758 *का तीसरा पुनरीक्षण*)

Draft Indian Standard

JUTE BATCHING OIL — SPECIFICATION

(*Third Revision of* IS 1758)

(ICS No. 75.100)

Lubricants and their related products Sectional Committee, PCD 25	Last date for receipt of comment is **4 February 2025**

FOREWORD

(*Formal clauses will be added later*)

Jute industry occupies a prominent place in the internal and export markets of India. It is therefore essential that the quality of jute goods manufactured in India should be of optimum quality.

Finished jute goods usually contain 2 percent or more by mass of oil, which is essential for jute batching.

Gunny bags are used for storing foodstuffs and therefore there is a chance of the kerosenic or other objectionable odour from the jute batching oil being transmitted to their contents. Freedom from objectionable odour is a characteristic which is considered very important by jute mills and users of jute goods.

This standard was originally issued in 1960. During subsequent years, it was noted that the various countries to which jute products were being exported had felt the need for upgrading the standard by way of prescribing absence from carcinogenic constituents, particularly when the jute bags are to be used for packaging of foodstuffs. Limits for pyrene content, ultraviolet absorbance (which would indicate polynuclear aromatic hydrocarbon), freedom from objectionable odour, and limits for distillation range were some of the additional requirements statutorily laid down in some countries for this purpose. Keeping this in view this standard was revised in 1975 incorporating these additional requirements, and classifying the material into two types to take care of the varied requirements of the user countries.

The second revision of the standard was taken up in 1986 on a request from Indian Jute Industries' Research Association who were facing some difficulties with the supplies of jute batching oil. It was observed by them that the samples of jute batching oil with density below 0.850 g/ml were unsuitable for use due to presence of higher amounts of low boiling fractions. In view of this the density limit was revised from 0.825 to 0.850. Further, it was decided to revise the pour point limit by specifying different limits for summer and winter months and the initial boiling point (IBP) for Type 1 was also specified.

This revision has been brought out to keep pace with the latest technological developments and international practices. In this third revision, the following major changes have been made:

a) To promote use of environment friendly products for jute batching application, two new categories of new generation, colorless, high flash point, poly-aromatic hydrocarbon (PAH) free, and non-toxic jute batching oils - Type 3 and Type 4 have been added;
b) Type 3 jute batching oil is based on fully formulated product with an emulsifier system for making oil in water emulsion instantaneously by mixing it with water, without the need for any additional emulsifier additives at user end;
c) For Type 4 jute batching oil, an emulsifier is to be added as per customer requirement at customer end before its use; and
d) For these two types of jute batching oil, limit for pyrene content has been made more stringent and newer requirement of overall PAH content has been included.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for jute batching oil.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 376: 2023	Sodium hydroxide, analytical reagent — Specification (*fourth revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1447 (Part 1) : 2021	Methods of Sampling of Petroleum and its Products Part 1 Manual Sampling (*second revision*)

IS 1448	Methods of test for petroleum and its products
(Part 10/Sec2): 2021/ISO 3016: 2019	Petroleum and related products from natural or synthetic sources Section 2 Determination of pour point (*third revision*)
(Part 12): 2013/ ISO 2049: 1996	Determination of colour (ASTM scale) (*second revision)*
(Part 18): 2020	Distillation of petroleum products (*third revision*)
(Part 21) : 2019 / ISO 2719 : 2016	Determination of flash point — Pensky-Martens closed cup method (*fourth revision*)
(Part 25/Sec 1) : 2018 / ISO 3104	Transparent and opaque liquids section 1 Determination of kinematic viscosity and calculation of dynamic viscosity (*second revision*)
(Part 32): 2019 / ISO 3838 : 2004	Crude petroleum and liquid or solid petroleum products — Determination of density or relative density - Capillary stoppered pyknometer and graduated bicapillary pyknometer methods (*third revision*)
IS 1541 : 2006	Glass filter funnels — Specification (*second revision*)
ASTM D6591	Standard Test Method for Determination of Aromatic Hydrocarbon Types in Middle Distillates—High Performance Liquid Chromatography Method with Refractive Index Detection

3 TYPE

3.1 The material shall be of the following four types:

a) Type 1,
b) Type 2,
c) Type 3, and
d) Type 4.

4 REQUIREMENTS

4.1 Description

The material shall consist wholly of a refined virgin distillate from petroleum and shall not contain any cracked product. It shall be free from sediment, water, and other visible impurities.

4.2 The material shall also comply with the requirements given in Table 1 when tested according to the appropriate test methods prescribed in col 7 of Table 1.

TABLE 1 REQUIREMENTS FOR JUTE BATCHING OIL
(*Clause* 4.2)

Sl No.	Characteristic	Requirement				Methods of Test
		Type 1	Type 2	Type 3	Type 4	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i.	Colour, ASTM, *Max*	L 7.0	L 7.0	L 0.5	L 0.5	IS 1448 (Part 12)
ii.	Flash point, Closed (Pensky Martens), ^{o}C *Min*	100	130	160	160	IS 1448 (Part 21)

iii.	Density at 15 °C, g/ml, *Min*	0.850	0.850	0.842 (T)	0.839 (T)	IS 1448 (Part 32)
iv.	Kinematic Viscosity at 40 °C, mm^2/s, *Max*	15				IS 1448 (Part 25/Sec 1)
v.	Pour point, °C, *Max*					IS 1448 (Part 10/Sec 2)
	Summer	21.0		-3		
	Winter (Nov to Feb)	9.0				
vi.	Emulsification Test	Shall pass the test / customer requirement[a]			*see* Note	Annex A for Types 1 and 2 Annex B for Type 3 Type 4
vii.	Iodine value	To Report				Annex C
viii.	Distillation					IS 1448 (Part 18)
	a) Initial Boiling Point, °C, *Min*	240	285	340	340	
	b) Final Boiling Point, at 760 mm Hg, °C, *Max*	371	-	410	410	
	c) Residue, percent by volume, *Max*	2	-	1	1	
ix.	UV absorbance test					Annex D
	a) Pyrene content, ppm, *Max*	25	-	10	10	
	b) Absorbance per cm optical length, in the range of UV wavelength, *Max*					
	280 nm – 299 nm	2.3	-	-	-	
	300 nm – 319 nm	1.2	-	-	-	
	320 nm – 359 nm	0.8	-	-	-	
	360 nm – 400 nm	0.3	-	-	-	
x.	PAH content, µg/gm (ppm), *Max*	-	-	10	10	ASTM D6591

[a] To be tested after the addition of emulsifier as per customer requirement.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed and supplied in suitable containers as agreed to between the purchaser and the supplier, and subject to the provisions of *Railways Red Tariff Rules* and *Red Tariff No. 20* issued by the Indian Railways Conference Association along with any future amendments.

5.2 Marking

5.2.1 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

5.2.2 Each container shall be marked with the following information:

a) Name and type of the material;
b) Manufacturer's name, address and recognized trade mark, if any;
c) Net mass/volume in the container;
d) Identification in code or otherwise to enable the lot to be traced back from records;
e) Date or year of manufacture/packing; and
f) Any other statutory requirements.

5.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

6. SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 1447 (Part 1).

6.2 Number of Tests

Tests for determining all the characteristics given in Table 1 shall be conducted on the composite samples.

6.3 Criteria for Conformity

The material shall be declared as conforming to the requirements of the specification if all the tests carried out on the composite samples meet the relevant specification requirements.

ANNEX A
[*Table* 1**,** *sl no. (vi)*]
EMULSIFICATION TEST FOR TYPE 1 AND TYPE 2

A-1 OUTLINE OF THE METHOD

A-1.1 The method consists of emulsifying jute batching oil with aqueous sodium oleate solution at room temperature at a specified ratio of oil and sodium oleate, and visually examining the stability of the 10 percent dilute emulsion after being left undisturbed at room temperature for 24 h.

A-2 APPARATUS

A-2.1 Emulsification Vessel

A tapered vessel, open on top, made of brass or enamelled metal and of dimensions shown in Fig. 1. It has an approximate capacity of 1500 ml.

A-2.2 Stirrer

Made of brass and of dimensions shown in Fig. 1. The three adjustable blades (A, B and C in Fig. 1) are set 25 mm apart in the vertical direction at an angle of 60º to one another by means of screws. When fitted to the vessel for emulsification tests, there shall be a clearance of not more than 3 mm between the tip of the top blade and the side of the vessel when the bottom blade is almost flush with the base of the vessel. The stirrer shall be located in this position during the emulsification test.

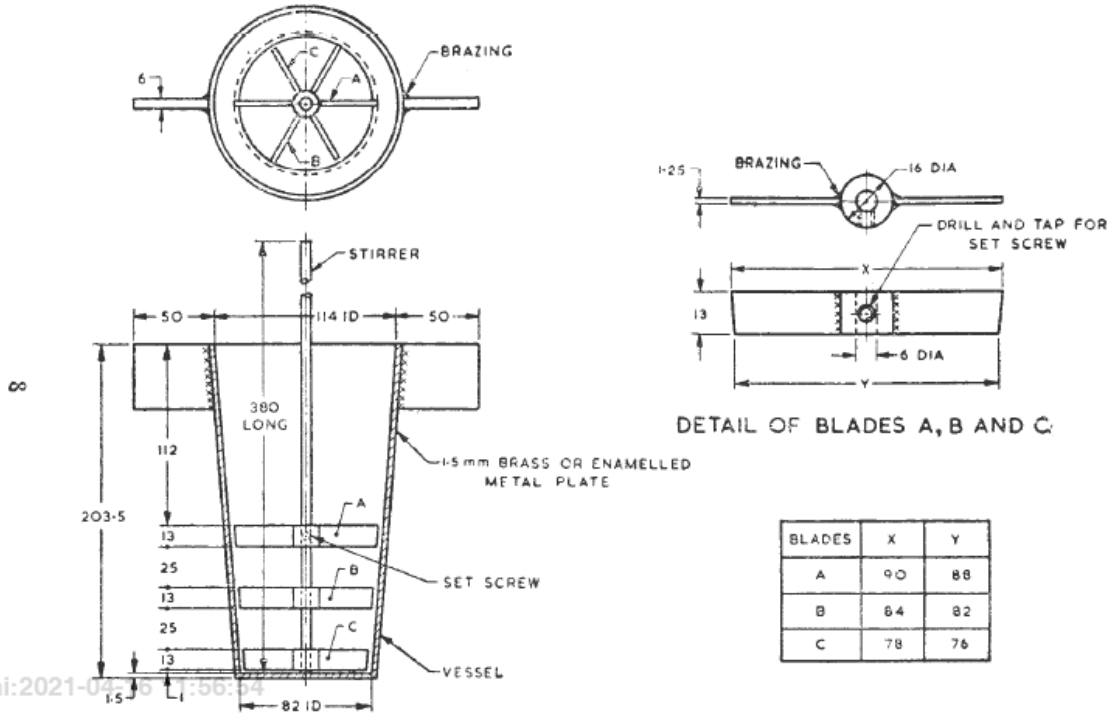
A-2.3 Electric Motor

A motor may be provided with a rheostat to control speed of stirring. There shall be a suitable arrangement to couple the stirrer to the spindle of the motor without excessive wobbling.

A-3 REAGENTS

A-3.1 Sodium Oleate Solution

Prepare a 4 percent (*m/v)* solution of analytical grade sodium hydroxide (*see* IS 376) in distilled water (*see* IS 1070) and to this solution add the equivalent quantity of pure oleic acid (equivalent mass 282) drop by drop with stirring. Dilute and mix the resulting gel-like substance with the requisite quantity of distilled water to yield a homogeneous solution containing 15 g of sodium oleate per 100 ml of solution. Prepare a stock solution and use it for a number of emulsification tests.



BLADES	X	Y
A	90	88
B	84	82
C	78	76

All dimensions in millimetres.

FIG. 1 ASSEMBLY OF APPARATUS FOR EMULSIFICATION TEST

A-4 PROCEDURE

A-4.1 Carry out the test at the prevailing room temperature.

A-4.2 Connect the motor to the stirrer and adjust the latter inside the tapered vessel so that the three blades are 25 mm apart with the bottom blade almost flush with the bottom of the vessel. Place 10 ml of sodium oleate solution **(A-3.1)** in the vessel and set the stirrer in motion at about 400 rev/min by adjusting the rheostat. Take 300 ml of sample oil in a separating funnel and add to the sodium oleate solution drop by drop until a gel is formed. Increase the rate of addition thereafter and add the entire quantity of the oil in the course of an hour. Add about 2 ml of distilled water every 5 min to ensure the presence of an optimum quantity of water in the gel. Keep an account of the volume of water thus added. Periodically, note the stirring efficiency and adjust the rheostat to give efficient stirring. The speed of stirring shall not be less than 150 rev/min when the gel has reached the maximum consistency.

A-4.3 Continue mixing the gel for 5 min after the addition of the sample oil and add 290 ml (including the volume added during the gel formation) of distilled water to the gel taking about 15 min for this addition. Maintain the stirring between 150 rev/min and 400 rev/min during this period. The resulting emulsion is nearly 50 percent in oil content.

A-4.4 Filter the 50 percent emulsion through a thin pad of cotton wool placed inside a 150 mm diameter glass filter funnel (*see* IS 1541) and wait until the whole of the emulsion is filtered. Stir the filtered emulsion with a glass rod or by rotating the container by hand. Transfer quickly 100 ml of this emulsion into a 500 ml graduated cylinder provided with a stopper, and dilute with 400 ml of distilled water. Place the stopper in position and mix the contents uniformly by inverting the stoppered cylinder about ten times. Allow the resulting

10 percent emulsion to stand on the laboratory bench undisturbed for 24 h and examine it at the end of this period.

A-4.5 The oil shall be regarded as having passed the emulsification test if no free oil has separated on top when examined visually.

A-4.5.1 The stability of emulsion shall be determined visually. For the purpose of this test, the emulsion shall be regarded as stable if there is no separation of free oil from the emulsion. Creaming of the emulsion, that is, separation into a thick emulsion layer on top and a thin white coloured aqueous layer at the bottom shall not be confused with separation of free oil, A creamed emulsion becomes homogeneous on shaking or agitation whereas free oil, if present, separates from the emulsion after a short period.

A-4.5.2 In the above emulsification test, the volumetric ratio of sample oil to 15 percent sodium oleate soap solution **(A-3.1)** is 30 : 1, and when calculated on the basis of 30 percent strength, this ratio becomes 60 : 1. Soap of the latter strength is used in the commercial preparation of emulsions in the jute industry and the jute industry require jute batching oils which give satisfactory emulsion with a minimum of 60 parts by volume of oil to every one part by volume of a 30 percent soap solution.

ANNEX B
[*Table* 1**,** *sl no. (vi)*]
EMULSIFICATION TEST FOR TYPE 3

B-1 OUTLINE OF THE METHOD

B-1.1 The emulsion is kept at 20 °C to 25 °C in a 100 ml capacity cylinder and the separation of oil in the emulsion is visually observed after being left undisturbed at room temperature for 24 h.

B-2. APPARATUS

B-2.1 Graduated cylinder(s) with stopper (1000 ml)

B-2.2 Graduated cylinder(s) with stopper (100 ml)

B-2.3 Pipette (5 ml)

B-2.4 *p*H meter

B-2.5 Conical flask (150 ml)

B-3 REAGENTS

B-3.1 Distilled water

B-3.2 Calcium chloride (anhydrous), powder

B-3.3 Magnesium sulphate, powder

B-3.4 Sodium bicarbonate, powder

B-3.5 Dilute hydrochloric acid, liquid

B-4 PREPARATION OF SYNTHETIC HARD WATER (200 ppm)

For carrying out this test, first, synthetic hard water of 200 ppm hardness shall be prepared by dissolving 133.2 mg $CaCl_2$ (anhydrous), 197.1 mg of $MgSO_4.7H_2O$ and 40.0 mg of $NaHCO_3$ in 1 l distilled water. After adding the aforesaid salts in required amounts to distilled water, shake the cylinder (1000 ml) until they dissolve. Adjust *p*H of synthetic hard water to 7.0 ± 0.2 by using dilute HCl (if required).

B-5 PREPARATION OF SAMPLE

Take 95 ml of synthetic hard water (200 ppm hardness) in 150 ml conical flask. Add 5 ml of oil to water with stirring. Stir the solution for 2 min after addition of oil in water in order to prepare 5 percent by volume oil-in-water emulsion.

B-6 PROCEDURE

B-6.1 Conduct the test in duplicate. Transfer the prepared oil-in-water emulsion **(B-5)** in 100 ml graduated cylinder with stopper.

B-6.2 Keep the solution for observation at ambient temperature without disturbing for 24 h. Thereafter, observe for oil separation.

B-6.3 If no more than 1.0 ml oil is present at the top of the emulsion, the sample passes the test.

ANNEX C
[*Table* 1, *Item* (vii)]
DETERMINATION OF IODINE VALUE (WIJS)

C-1 OUTLINE OF THE METHOD

C-2 APPARATUS

C-2.1 Iodine Value Flask, glass stoppered – 500 ml

C-2.2 An engraved stem thermometer calibrated between 10 °C and 65 °C in 0.l ºC intervals and with the 0 ºC point marked on the stem is recommended. The thermometer shall have an auxiliary reservoir at the upper end, a length of about 370 mm and a diameter of about 6 mm.

C-3 REAGENTS

C-3.1 Potassium Dichromate — Crystals

C-3.2 Concentrated Hydrochloric Acid — Relative density 1.16

C-3.3 Potassium Iodide Solution

Prepare a fresh solution by dissolving 10 g of potassium iodide, free from potassium iodate, in 100 ml of water.

C-3.4 Starch Solution

Make a paste of 0.5 g of soluble starch in cold water, pour in 100 ml of boiling water, boil for 5 min, cool and bottle. The solution should be prepared afresh every two or three days.

C-3.5 Standard Sodium Thiosulphate Solution, 0.1 N

C-3.5.1 Weigh accurately about 25 g of sodium thiosulphate crystals ($Na_2S_2O_3.5H_2O$) and dissolve in water which has been boiled to free it from carbon dioxide and make to 1000 ml. Store the solution in a cool place in a dark-coloured stock bottle with a guard tube filled with

soda lime. After storing the solution for about one week, filter and standardize it as prescribed in **C-3.5.2.**

C-3.5.2 Weigh accurately about 0.2 g of finely ground potassium dichromate which has been previously dried to constant mass at (105 ± 2) ºC, in a clean and dry conical flask, and dissolve in a small quantity of water. Add 60 ml of water, 4 ml of concentrated hydrochloric acid and 20 ml of potassium iodide solution. Titrate the mixture with sodium thiosulphate solution, using starch solution as an indicator towards the end. The end-point is taken when the blue colour changes to green. Calculate the normality (*N*) of sodium thiosulphate solution as follows:

$$N = \frac{1000 \times m}{49.03 \times V}$$

where

m = mass in g of the potassium dichromate, and
V = volume in ml of sodium thiosulphate solution required for titration.

C-3.6 Iodine Crystals — Re-sublimed.

C-3.7 Acetic Acid

Glacial, 99 percent, having a melting point of 14.8 ºC and free from reducing impurities.

C-3.7.1 The melting point of the acetic acid and presence of reducing impurities can be tested as follows:

C-3.7.1.1 *Melting Point Determination* **—** Take a 15 cm long test tube and fill it to about two-thirds with the acetic acid. Insert into the acid a thermometer satisfying the requirements specified in **C-2.1** through a cork stopper fitting the test tube. The amount of acid should be at least double the quantity required to cover the bulb of the thermometer when the bottom of the latter is 12 mm from the bottom of the test tube. Suspend this tube within a larger test tube through a cork. Cool the acid by immersing the assembly in ice water until the temperature is 10 ºC, then withdraw the assembly from the ice water and stir the acid rather vigorously for a few moments, thus causing the supercooled liquid to crystallize partially and give a mixture of liquid and solid acid. Take thermometer readings every 15 s and consider as the true melting point that temperature at which the reading remains constant for at least 2 min.

C-3.7.1.2 *Test for Reducing Impurities (Potassium Permanganate Test)* **—** Dilute 2 ml of acetic acid with 10 ml of water and add 0.1 ml of 0.1 N potassium permangante solution and maintain at (27 ± 2) °C. The test shall be taken as having been satisfied if the pink colour is not discharged at the end of 2 h.

C-3.8 Chlorine Gas — Dry.

C-3.9 Iodine Trichloride (ICl_3)

C-3.10 WIJS Iodine Monochloride Solution

Prepare this solution by one of the following two methods, and store in a glass-stoppered bottle in a cool place, protected from light:

a) Dissolve 13 g of iodine **(C-3.6)** in 1 l of acetic acid **(C-3.7)**, using gentle heat, if necessary, and determine the strength by titration with standard sodium thiosulphate solution **(C-3.5)**. Set aside 50 ml to 100 ml of the solution and introduce chlorine gas into the remainder until a characteristic colour change occurs (a sudden disappearance of the colour) and the halogen content is nearly doubled, as ascertained again by titration. If the halogen content has been more than doubled, reduce it by adding the requisite quantity of the iodine-acetic acid solution, which was set aside earlier. A slight excess of iodine does not harm but avoid an excess of chlorine.

NOTE — If the titration of 20 ml of original iodine-acetic acid solution requires 22 ml of standard sodium thiosulphate solution, 20 ml of the finished WIJS solution should require between 43 ml to 44 ml (and not more than 44 ml) of the same sodium thiosulpate solution.

b) As an alternative method for preparing WIJS solution, dissolve 8 g of iodine trichloride **(C-3.9)** in approximately 450 ml of acetic acid **(C-3.7)**. Dissolve separately 9 g of iodine **(C-3.6)** in 450 ml of acetic acid **(C-3.7)**, using gentle hear, if necessary, and then cool. Add gradually the iodine solution to the iodine trichloride solution until the colour has changed to reddish-brown. Add 50 ml more of the iodine solution and dilute the mixture with acetic acid till 10 ml of the mixture is equivalent to 20 ml of standard thiosulphate solution when the halogen content is estimated by titration in the presence of an excess of potassium iodide and water. Heat the solution to 100 ºC for 20 min and cool. Take precaution to restrict the access of water vapour while preparing the solution.

C-3.11 Carbon Tetrachloride or Chloroform

Inert to WIJS solution giving a homogeneous mixture with it.

C-4 PROCEDURE

C-4.1 Make sure that the glass apparatus used is absolutely clean and dry. Weigh accurately by difference 0.45 g to 0.55 g of the oil into a clean, dry 500 ml glass-stoppered iodine value flask. Add 25 ml of carbon tetrachloride or chloroform **(C-3.11)**, agitate to dissolve the contents. Add 25 ml of WIJS solution **(C-3.10)** and replace the glass stopper after wetting with potassium iodide solution **(C-3.3)**; swirl for intimate mixing and allow to stand in the dark at room temperature for 2 h with occasional swirling. Carry out a blank test simultaneously under similar experimental conditions. After a period of 2 h, add 15 ml of potassium iodide solution **(C-3.3)** and 100 ml of water and titrate the liberated iodine with standard sodium thiosulphate solution **(C-3.5)**, swirling the contents of the flask continuously to avoid any local excess until the colour of the solution is straw-yellow. Add 0.5 ml of starch solution and continue the titration until the blue colour disappears.

C-5 CALCULATION

Calculate iodine value as follows:

$$\text{Iodine value} = \frac{1269 \times (B - S) \times N}{M}$$

where

B = volume in ml of standard sodium thiosulphate solution required for the blank,
S = volume in ml of standard sodium thiosulphate solution required in the test with the material,
N = normality of standard sodium thiosulphate solution, and
M = mass in g of the material taken for the test.

ANNEX D
[*Table* 1, *item* (ix)]
ULTRAVIOLET (UV) ABSORBANCE TEST

D-1 OUTLINE OF THE METHOD

Mineral oil is extracted in a solution of n-hexadecane

D-2 GENERAL INSTRUCTIONS

Because of the sensitivity of the test, the possibility of errors arising from contamination is great. It is of the greatest importance that all glassware be scrupulously cleaned to remove all organic matter, such as oil, grease, detergent residues, etc. Examine all glassware, including stoppers and stopcocks under ultraviolet light to detect any residual fluorescent contamination. As a precautionary measure it is recommended practice to rinse all glassware with purified iso-octane immediately before use. No grease is to be used on stopcocks or joints. Great care to avoid contamination of jute batching oil samples in handling and to assure absence of any extraneous material arising from inadequate packaging is essential. As some of the polynuclear aromatic hydrocarbons sought in this test are very susceptible to photo-oxidation, the entire procedure is to be carried out under subdued light.

NOTE — Rubber stoppers and stopcock grease on ground glass joints shall not be used for this method.

D-3 APPARATUS

D-3.1 Glassware

Assorted beakers, separatory funnels fitted with tetra fluoroethylene polymer stopcocks, graduated cylinders and volumetric flasks of 200 ml capacity.

D-3.2 Reservoir

A 500 ml capacity reservoir having a 24/40 standard taper male fitting at bottom and a suitable ball joint at the top for connecting to the nitrogen supply. The female fitting of the chromatographic columns described in **D-3.3** and the male fitting of the reservoir should both be equipped with glass hooks.

D-3.3 Chromatographic Columns

D-3.3.1 A chromatographic column made from nominal 1.3 cm outside diameter × 75 cm glass tubing tapered at one end and joined to a 2 mm bore tetrafluoroethylene polymer stopcock. The opposite end is flanged and joined to a female 24/40 standard taper fitting. This provides for accommodating the 500 ml reservior described above.

D-3.3.2 A chromatographic column made from nominal 1.7 cm outside diameter × 115 cm glass tubing tapered at one end and joined to a 2 mm bore tetrafluoroethylene polymer stopcock. The opposite end is flanged and joined to a 2 cm to 5 cm outside diameter × 9.0 cm

glass tube having a female 24/40 standard taper fitting. This provides for accommodating the 500 ml reservoir described above.

D-3.4 Spectrophotometer

A spectrophotometer equipped to automatically record absorbance of liquid samples in 1 cm path length cells in the spectral region of 280 nm to 400 nm with a spectral slit width of 2 nm or less. At an absorbance level of about 0.4, absorbance measurements shall be repeatable within ± 0.01 and accurate within ± 0.05. Wavelength measurements shall be repeatable within ± 0.2 nm and accurate within ± 1.0 nm. Instrument operating conditions are selected to realize this performance under dynamic (automatic) recording operations. Accuracy of absorbance measurements is determined at 290 nm, 345 nm, and 400 nm, using potassium chromate as the reference standard.

D-3.5 Spectrophotometer Cells

Two fused quartz cells having path length of (1.00 ± 0.005) cm or better.

D-4 REAGENTS AND MATERIALS

D-4.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used.

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

D-4.2 Organic Solvents

All solvents used throughout the procedure shall meet the requirements and test described. The solvents iso-octane, benzene, cyclo-hexane, nitromethane, and *n*-hexadecane shall pass the following test:

To the specified quantity of solvent in a 150 ml beaker, add 1 ml of purified *n*-hexadecane and evaporate on the steam-bath under a stream of nitrogen. Discontinue evaporation when 1 ml or less of residue remains.

To the residue from benzene and nitromethane, add a 10 ml portion of purified iso-octane and re-evaporate and repeat the same process once more to ensure complete removal of solvent. To the residue from *n*-hexadecane, add iso-octane to make up to 10 ml volume.

Determine the absorbance in 1.0 cm path length cells compared to water as reference. The absorbance of the solution of solvent residue shall not exceed 0.05 between 280 nm and 400 nm.

D-4.2.1 *Iso-octane* (2,2,4 *trimethylpentane*)

Use 240 ml for the above test. Purify, if necessary, by passing through a column of activated silica gel.

D-4.2.2 *Benzene*

Use 200 ml for the above test. Purify, if necessary, by distillation or otherwise.

D-4.2.3 *Cyclohexane*

Use 10 ml for the above test. Purify, if necessary, by distillation, silica gel percolation or otherwise.

D-4.2.4 *Nitromethane*

Use 125 ml for the above test. Purify, if necessary, by distillation or otherwise.

D-4.2.5 *n-Hexadecane*

Determine the absorbance of this solvent directly. Purify, if necessary, by silica gel percolation or otherwise.

D-4.2.6 *Standard Reference Pyrene (melting point 150 ºC to 152ºC*)

The standard reference absorbance is the absorbance at 334 millimicrons of a standard reference solution of pyrene containing a concentration of 1.0 mg/l in purified iso-octane measured against iso-octane of the same spectral purity in 1.0 cm cells. (This absorbance will be approximately 0.28)

D-4.2.7 *Chrysene Solution*

Prepare a solution of concentration of 5.0 mg/l by dissolving 5.0 mg of chrysene in purified iso-octane in a 1 litre volumetric flask. Adjust the volume with iso-octane.

D-4.2.8 *Nitrogen Gas*

Water-pumped or equivalent purity, cylinder with regulator, and valve control flow at 0.3515 kgf/cm^2.

D-4.2.9 *Silica Gel* (150-75 *micron*) — Purified and activated by the following procedure:

Place about 1 kg of silica gel in a large column and wash with contaminant-free benzene until a 200 ml sample of the benzene coming off the column will pass the ultraviolet absorption test for benzene. This test is performed as stipulated under **D-4.2**. When the silica gel has been sufficiently cleaned, activate the gel before use by placing the 1 kg batch in a shallow container in a layer not greater than 2.5 cm in depth and heating in an oven (*Caution:* Explosion Hazard) at 130 ºC for 16 h and store in a vacuum desiccator. Reheating about once a week is necessary if the silica gel is repeatedly removed from the desiccator.

D-4.2.10 *Aluminum Oxide* (180-75 *micron*) — Purified and activated by the following procedure:

Place about 1 kg of aluminium oxide in a large column and wash with contaminant-free benzene until a 200 ml sample of the benzene coming off the column will pass the ultraviolet absorption test for benzene. This test is performed as stipulated under **D-4.2**. (*Caution*:

Remove benzene from adsorbent under vacuum to minimize explosion hazard in subsequent heating. When the aluminium oxide has been sufficiently cleaned and freed of solvent, activate it before use by placing the 1 kg batch in a shallow container in a layer not greater than 2.5 cm in depth. Heat in an oven at 130 °C for 16 h. Upon removal from heat, store at atmospheric pressure over 80 percent (by mass) sulphuric acid in a desiccator for at least 36 h before use. This gives aluminium oxide with between 6 percent and 9.5 percent volatiles. This is determined by heating a weighed sample of the prepared aluminium oxide at 1 100ºC for 2 h and then quickly reweighing. To ensure the proper adsorptive properties of the aluminium oxide, perform the following test:

a) Weigh (50 ± 1) g of the activated aluminium oxide and pack into the chromatographic column (1.3 cm × 75 cm) described under **D-3.3.1**. Use glass wool at the column exit to prevent the aluminium oxide from passing through the column.

b) Place a 250 ml graduated cylinder under the column to measure the amount of eluate coming from the column.

c) Pre-wet the aluminium oxide by passing 40 ml of iso-octane through the column. Adjust the nitrogen pressure so that the rate of descent of the iso-octane coming off the column is between l.5 ml/min to 2.5 ml/min.

d) Just prior to the last of the iso-octane reaching the top of the aluminium oxide bed, add 10 ml of the iso-octane solution containing 5.0 mg of chrysene per litre **(D-4.2.7)**.

e) Continue percolation until the iso-octane is just above the aluminium oxide. Then add 200 ml of a mixture of benzene and iso-octane (33.33 percent benzene and 66.67 percent iso-octane, by volume) to the column and continue percolation.

f) Continue percolation, collecting the elutes (40 ml of the pre-wet solution, 10 ml of the sample solution, and 200 ml of the gradient solution) in the 250 ml graduated cylinder until the level of the gradient solution is just above the aluminium oxide. Add 200 ml of eluting solution of benzene and iso-octane (90 percent benzene and 10 percent iso-octane, by volume) to the column and continue collecting until a total of 250 ml of solution has been obtained. This may be discarded. Now begin to collect the final elute.

g) Place a 100 ml graduated cylinder under the column and continue the percolation until a 100 ml elute has been obtained.

h) Measure the amount of chrysene in this 100 ml fraction by ultraviolet analysis. If the aluminmm oxide is satisfactory, more than 80 percent of the original amount of chrysene should be found in this fraction.

NOTE — If the amount of chrysene recovered is less than 80 percent, the original batch of aluminium oxide should be sieved between 150 microns and 90 microns. Activation and testing of this sieved batch should indicate satisfactory aluminium oxide for use.

D-5 PREPARATION OF THE SAMPLE

Precautions shall be taken to ensure that an uncontaminated sample of the mineral oil is obtained since ultraviolet absorption is very sensitive to small amounts of extraneous material contaminating the sample through careless handling.

D-6 PROCEDURE

D-6.1 Blank Determination

Before proceeding with the analysis of sample, determine the absorbance of the solvent residues by carrying out the procedure without a sample.

D-6.2 Weigh out (20.0 ± 0.l) g of the jute batching oil sample into a beaker and transfer to a 250 ml separatory funnel fitted with a tetrafluoroethylene polymer stopcock, using enough cyclohexane to give a final total volume of 50 ml (jute batching oil plus cyclohexane).

D-6.3 Add 25 ml of nitromethane saturated with cyclohexane and shake vigorously by hand for 3 min. Recover the lower nitromethane layer in a 150 ml beaker containing 1 ml of *n*-hexadecane. Repeat the extraction four more times, recovering each extract in 150 ml beaker containing 1 ml of *n*-hexadecane. Care should be taken not to fill the beaker to such a capacity that solvent losses may occur. Evaporate the combined nitromethane extracts to l ml of *n*-hexadecane residue containing the nitromethane soluble mineral oil extractives. Remove the beaker from the steam-bath and allow to cool.

> NOTE — Complete removal of the nitromethane is essential. This can be assured by two successive additions of 5 ml of iso-octane and re-evaporation.

D-6.4 Weigh (50 ± 1) g of activated aluminium oxide and pack into the chromatographic column (l ·3 × 75 cm) described under **D-3.3.1** (*see* Note).

> NOTE — A small plug of glass wool is placed at the column exit to prevent the aluminium oxide from passing through the column. After adding aluminium oxide, tap the column lightly to remove air voids. All percolations using aluminium oxide are performed under nitrogen pressure. The 500 ml reservoir described under **D-3.2** is to be used to hold the elution solvents.

D-6.5 Pre-wet the column by adding 40 ml of iso-octane to the column. Adjust nitrogen pressure so that rate of descent of the iso-octane coming off the column is 2.0 ml/min to 3.0 ml/min. Be careful to maintain the level of solvent in the reservoir to prevent air from entering the aluminium oxide bed. New or additional solvent is added just before the last portion of the previous solvent enters the bed. To minimize possible photo-oxidation effects, the following procedures shall be carried out in subdued light.

D-6.6 Before the last of the iso-octane reaches the top of the aluminium oxide bed, release the nitrogen pressure and turn off the stopcock on the column. Transfer the *n*-hexadecane residue from the 150 ml beaker (*see* **D-6.3**) on to the column, using several washes of iso-octane (total volume of washes should not be greater than 10 ml to 15 ml).

D-6.7 Open the stopcock and continue percolation until the iso-octane is about 1 cm above the top of the aluminium oxide bed. Add 200 ml of iso-octane to the reservoir, and continue the percolation at the specified rate.

D-6.8 Just before the iso-octane surface reaches the top of the aluminium oxide bed, add 200 ml of a mixture of benzene and iso-octane (33.33 percent benzene and 66.67 percent iso-octane, by volume) to the reservoir, and continue the percolation.

D-6.9 Just before the last of this mixture reaches the top of the aluminium oxide bed, release the nitrogen pressure, turn off the stopcock, and discard all the elution solvents collected up to this point.

D-6.10 Add to the reservoir 300 ml of a mixture of benzene and iso-octane (90 percent benzene and 10 percent iso-octane, by volume), place a 25 ml graduated cylinder under the column, continue the percolation until 20 ml of eluate has been collected, and then discard the elute.

D-6.11 At this point, place a clean 250 ml conical flask under the column. Continue the percolation and collect all the remaining elute.

NOTE — Allow the column to drain completely. An increase in the nitrogen pressure may be necessary as the last of the solvent comes off the column.

D-6.12 Place 1 ml of *n*-hexadecane into a 150 ml beaker. Place this onto a steam-bath under nitrogen stream and transfer in small portions of the elute from **D-6.11**. Wash out the conical flask with a small amount of benzene and transfer to the evaporation beaker. Evaporate until only 1 ml of hexadecane residue remains. Remove the beaker from the steam-bath and cool.

NOTE — Complete removal of the benzene is essential. This can be assured by two successive additions of 5 ml of iso-octane and re-evaporation.

D-6.13 Place a sample of 113.5 g activated silica gel in a 500 ml glass stoppered conical flask Add to the silica gel 46.2 g (41 ml) of nitromethane. Stopper and shake the flask vigorously until no lumps of silica gel are observed and then shake occasionally during a period of 1 h. The resultant nitromethane treated silica gel is 29 percent by mass of nitromethane and 71 percent by mass of silica gel.

D-6.14 Place a small plug of glass wool in the tapered end of the 1·7 cm outside diameter × 115 cm column, described under **D-3.3.2**, adjacent to the stopcock to prevent silica gel from passing through the stopcock. Pack the nitromethane-treated silica gel into the column, tapping lightly. The resulting silica gel bed should be about 95 cm in depth. Place into a flask 170 ml of iso-octane saturated with nitromethane.

D-6.15 Place a 100 ml graduated cylinder under the column and transfer the residue from the beaker in **D-6.12** with several washes of the 170 ml of iso-octane saturated with nitromethane, onto the top of the column (total volume of washes should not be greater than 10 ml to 15 ml). Permit iso-octane solution to enter the silica gel bed until the liquid level is at the top level. Place the remaining amount of 170 ml of iso-octane saturated with nitromethane in the reservoir above the bed for percolation through the silica gel. Apply nitrogen pressure to the top of the column, adjusting the pressure so that the iso-octane is collected at the rate of 2.5 ml/min to 3.0 ml/min, and percolate iso-octane through the bed until a quantity of 75.0 ml of elute is collected. Discard the 75 ml of elute. Turn off the

stopcock and add 250 ml of benzene to the reservoir above the bed. Use a 400 ml beaker to collect the remaining elute.

D-6.16 Open the stopcock, renew the pressure, and percolate the remaining iso-octane and benzene through the column eluting the remaining aromatics. Transfer the elute in small portions from the 400 ml beaker to 150 ml beakers containing 1 ml of *n*-hexadecane and evaporate on the steam bath under nitrogen. Rinse the 400 ml beaker well with small portions of iso-octane to obtain a complete transfer.

NOTE — Complete removal of the nitromethane and benzene is essential. This can be assured by successive additions of 5 ml of iso-octane and re-evaporation.

D-6.17 Transfer the residue with several washes of iso-octane into a 200 ml volumetric flask. Add iso-octane up to the mark.

D-6.18 Record the spectrum of the sample solution in a 1 cm cell compared to iso-octane from 270 nm to 400 nm. After making necessary corrections in the spectrum for cell differences and for the blank absorbance, record the maximum absorbance in each of the wavelength intervals, that is 280 nm to 299 nm, 300 nm to 319 nm, 320 nm to 359 nm and 360 nm to 400 nm.

D-6.19 If the spectrum shows no discernible peak corresponding to the absorbance maximum of the pyrene reference standard solution at 334 nm, the maximum absorbances in the respective wavelength intervals recorded shall not exceed those prescribed in Table 1.

D-6.20 If such a peak is evident in the spectrum of the sample solution, and the spectrum as a whole is not incompatible with that of pyrene contaminant yielding such a peak of the observed absorbance, calculate the concentration of pyrene that would yield this peak (334 nm) by the baseline technique described in Indian Standard Recommended practice for general technique of ultra violet (UV) quantitative analysis. Correct each of the maximum absorbances in the respective specified wavelength intervals by subtracting the absorbance due to pyrene, determined as follows:

$$\text{Absorbance due to pyrene} = \frac{C_p \times S_a}{S_p}$$

where

C_p = calculated concentration of pyrene in sample solution,
S_a = absorbance of pyrene reference standard solution at wavelength of maximum absorbance of sample solution in the respective specified wavelength intervals, and
S_p = concentration of pyrene reference standard solution in same units of concentration.

Also calculate the pyrene content of the oil sample in parts per million as follows:

$$\text{Pyrene content (ppm)} = \frac{200/1000 \times C}{20/1000} = 10\ C$$

where

C = calculated concentration of pyrene in mg/l of sample solution.

D-6.21 The pyrene content so determined shall not exceed 25 ppm. The maximum absorbances corrected for pyrene content as described in **D-6.18** for each of the specified wavelength intervals shall not exceed the limits prescribed in Table 1.

D-6.22 If the spectrum as a whole of the sample solution is in any respect clearly incompatible with the presence of pyrene as the source of the peak at 334 nm, then the maximum absorbances in the respective wavelength intervals without correction for any assumed pyrene content shall not exceed the limits prescribed in Table 1.