

IJO STANDARD 98/01

[Revised 2005]



INTERNATIONAL JUTE STUDY GROUP (IJSG)

A. BACKGROUND OF IJO STANDARD 98/01

Use of mineral-oil based batching oil in the process of manufacturing jute bags used for packaging selected food materials has been a matter of concern for the final consumers due to alleged and possible hydro-carbon contamination of such bags. In order to resolve the issue to the satisfaction of all concerned, IJO organised an international workshop in February 1998 in Calcutta which was attended by the producers and buyers of the jute bags and also by end-user industries of food grade materials like Cocoa Beans, Coffee Beans and Shelled Nuts. The workshop resulted in the elaboration of a draft standard specification for jute bags used in the packaging of such food grade materials, including the establishment of maximum tolerance levels for hydrocarbon residues. The standard arrived at stipulates that the bags should not contain unsaponifiables exceeding 1,250 mg/kg. The International Jute Council (IJC) accepted the recommendation of this workshop in March 1998 and adopted the specifications as IJO Standard 98/01. The standard entered into force on 01 October 1998. The standard has been accepted by the International Cocoa Organisation in May 1998.

IJO STANDARD 98/01

[Special Criteria for the Manufacture of Jute Bags used in the Packing of Selected Food Materials (Cocoa Beans, Coffee Beans and Shelled Nuts).]

1.0 INTRODUCTION AND GENERAL PROVISIONS

These IJO criteria have been established in response to the perceived need to provide for a universally acceptable specifications relating to the manufacture of jute bags used in the packing of food materials with particular reference to Cocoa Beans, Coffee Beans and Shelled Nuts. The criteria are intended to facilitate the minimization of mineral hydrocarbon contamination arising from the use of jute bags. Good manufacturing practice shall be followed in the production of jute bags which should meet the defined chemical and organoleptic criteria described in paragraphs 2.1 and 2.2 below. Jute bags shall also conform to the legal requirements for food packaging materials and waste management of the respective countries.

2.0 ANALYTICAL CRITERIA

Ingredients of batching oils shall be non-toxic and approved for use in packaging materials that will come into contact with the selected food materials. Batching oils shall not contain compounds that could produce off-flavours or off-tastes in food material packed in jute bags.

2.1 Chemical Criteria

Unsaponifiables	<1250 mg/kg
Recommended Methods	as in section B

2.2 Organoleptic Criteria

Jute bags shall be analysed for their olfactory qualities. No undesirable odours or odours untypical of jute shall be present. No unacceptable odours shall develop after artificial ageing of the bags.

3.0 CERTIFYING AUTHORITIES

The Governments concerned have authorized specified institutions/agencies for collection and testing of samples and for issue of necessary certificates for jute bags and cloth conforming to the IJO Standard 98/01 specifications. The names of the authorized certifying agencies are at **Appendix-IV**.

4.0 CERTIFICATE

The certificate stating that the samples drawn from the lot conforms to IJO Standard 98/01 specifications will be issued by the certifying authorities (mentioned at **Appendix-IV**) in the prescribed form which is at **Appendix-III**. The certificate shall be issued by the authorities based on the test report of the designated testing agencies indicating that samples conform to the specifications of IJO Standard 98/01.

5.0 APPROVED MANUFACTURERS

The Governments concerned have authorised Government agencies to issue permits/ licenses to jute mills/manufacturers of jute cloth/bags conforming to the specifications of IJO Standard 98/01.

6.0 GOVERNMENT AGENCIES CONCERNED WITH IJO STANDARD 98/01

- India**
- Ministry of Textiles,
Government of India
Udyog Bhavan
New Delhi-110 011
www.texmin.nic.in

 - Jute Commissioner's Office
Ministry of Textile
Government of India
C.G.O. Complex, 3rd MSO Building
4th Floor, DF Block, Salt Lake City
Kolkata - 700 064
www.jutecomm.com

 - Jute Manufactures Development Council (JMDC)
3A Park Plaza, 71 Park Street
Kolkata -700 016
www.jmdcindia.com
- Bangladesh**
- Ministry of Textiles and Jute
Government of the People's Republic of Bangladesh
Bangladesh Secretariat
Dhaka -1000
www.bangladesh.gov.bd/jute/jute_information.htm

 - Directorate of Jute
Ministry of Textiles and Jute
Government of the People's Republic of Bangladesh
Karim Chamber
99 Motijheel C/A, Dhaka-1000.

B. DETERMINATION OF UNSAPONIFIABLES IN JUTE BAGS/ CLOTH USED FOR PACKAGING SELECTED FOOD MATERIALS: RECOMMENDED METHOD

This document prescribes the method of sampling and test for unsaponifiables for food grade jute packaging materials (bags/cloth).

Quality criteria: Unsaponifiable matter

1.0 SCOPE AND FIELD OF APPLICATION

Description of a simple method to control the compliance with the chemical criteria mentioned in Standard 98/01 of the International Jute Organisation (IJO) now known as International Jute Study Group (IJSJG): *Special Criteria for the Manufacture of Jute Bags used in the packaging of selected Food Materials (Cocoa Beans, Coffee Beans and Shelled Nuts)*, i.e. if the bags contain less than 1,250 mg/kg unsaponifiable matter based on the mineral hydrocarbon fraction. Moreover, the method allows the determination of the extractable oil from a jute bag.

This validated method can be used for jute bags as well as other vegetable fibre based fabrics (e.g. Sisal, Hessian).

The method cannot be used for determining unsaponifiable matters other than mineral hydrocarbons. It may not give precise result in case of very high contamination (approx. 10,000 mg/kg) due to the diminished solubility in the solvent, especially for compounds other than mineral hydrocarbons. Additionally, this method gives no results for the composition of the hydrocarbon fraction.

2.0 DEFINITIONS AND ABBREVIATIONS

2.1. Definitions

- Unsaponifiable matter in jute bag/cloth : mineral oils/waxes, unsaponifiable matter from edible oils
- Unsaponifiable matter in edible oils : mainly sterols, higher alcohols and other non-volatile organic compounds, in general 0.2-1.5% of pure fat, rarely up to 3%
- Batching oil : either mineral oils and waxes, edible oils or mixtures of both, added to soften fibres for spinning

3.0 PRINCIPLES OF THE METHOD

Extraction of the fabrics with organic solvent. Saponification of the extract with an ethanolic alkaline solution. Dilution with distilled water and extraction of the unsaponifiable matter by an organic solvent. Solvent evaporation. Conditioning the residue in a desiccator. Gravimetric determination.

4.0 CHEMICALS AND MATERIALS

4.1 Chemicals

Before using chemicals reference to the Sigma /Aldrich / Merck Guide to Chemical Safety and / or other adequate manuals or safety data sheets approved by local authorities are to be made. The safety instructions given in **Appendix-I** are to be noted carefully.

- Ethanol, p.a
- n - Hexane, LiChrosolv /equivalent grade
(Quality - parameters indicated in **Appendix - V**)
- Potassium hydroxide A.R
- Sodium sulphate, anhydrous A.R
- Self indicating silica gel.

4.2 Materials

- Desiccator
- 500 ml separation funnels, PTFE stopcock and stoppers are recommended
- Round bottom flasks 250 and 500 ml capacity -Borosil
- Condenser 'Twisselman'/or equivalent quality
- Soxhlet apparatus -Borosil
- Glass wool
- Extraction thimble

5.0 PREPARATION AND CHECK OF REAGENTS

All glassware must be cleaned prior to use with hexane and then dried to remove any potential contamination.

5.1 Ethanolic Potassium Hydroxide, Approximately 1 N Ethanolic Solution

Sixty grams (60g) potassium hydroxide is dissolved in fifty millilitre (50 ml) water and made up to one litre (1 litre) with ethanol. Freshly prepared solution should be colourless.

5.2 1 N Potassium Hydroxide

Fifty six grams (56 g) potassium hydroxide is dissolved in one hundred millilitre (100 ml) water and made up to one litre (1 litre) with water.

6.0 APPARATUS

- Water bath linear with support
- Analytical balance, capable of weighing to the nearest 0.1 mg (e.g. Mettler, AT200)
- Rotary vacuum evaporator (e.g. Buchi Rotavap)
- Thermostatically Controlled, Hot Air Oven ($100 \pm 2^{\circ}\text{C}$)

7.0 SAMPLING AND SAMPLE PREPARATION

7.1 Lot

All bag/cloth produced under similar condition of production and delivered to a buyer against one dispatch note shall constitute a lot.

7.1.1 Sampling Procedure

Random Sampling Techniques will be followed for collecting samples and preparation of specimens for carrying out the required tests.

Lot	Sample		
Up to 100 bales	2 bales	1 bag/bale	- 10 specimens (min)/bag
101 - 150 bales	3 bales	do	- do
151 - 200 bales	4 bales	do	- do

For each additional 50 bales or below - 1 bale is to be selected randomly.

One bag from each bale is to be randomly selected. Small samples (3 cm x 3 cm)/specimens are to be taken from at least 10 different places/parts of the bag. Small samples/specimens from each bag will be mixed to prepare composite sample for each bag and tested. Same system of drawing samples from each bag collected from different bales should be followed for preparing composite samples for each bag for testing.

In the case of cloth, the sample size will be 2 metres length of the fabric per roll and 2 rolls for a lot of 100 rolls and one additional roll for every 50 rolls thereafter.

In case even one sample fails the test, the whole lot will be rejected.

7.2 Sample Preparation

Similar squares (e.g. 3 by 3 cm) are cut out from bag/ cloth material avoiding ink markings, dirt, stitching seams as far as possible.

Bulked sample of (at least 30g) squares drawn from 10 to 15 different places of each bag/cloth sample will represent one sample.

7.3 Extraction

A previously dried 500 ml round bottom flask is weighed to the nearest 0.01 g (m_e). For extraction each received bulk sample is weighed accurately to the nearest 0.01g (m_s) and placed into a soxhlet thimble and positioned in a soxhlet extractor fitted to a 500 ml round bottom flask filled with 300 ml hexane. The extractor will siphon at least 10 cycles.

The extract is evaporated to dryness under reduced pressure (335 mbar) at 40°C (water bath). The open flask is cooled in a desiccator for 3 hours (with silica bluegel as drying agent) and the flask is weighed to the nearest 0.01g (m_{e+R}). The drying process is to be repeated successively for 15 min until constant weight is obtained. Alternately the drying can be done in Hot Air Oven at 75°C \pm 3°C for 30 minutes to attain constant weight.

7.4 Determination of the Mass of Extracted Oil

The mass of extracted oil (m_R) is determined according to calculation under 8.3.1. The mass has to exceed 0.3g but not 5g to proceed with the saponification as described in 7.5. In case the mass of extracted residue is less than 0.3g more sample should be taken for extraction. In this case the mass of extracted oil (m_R) is equal to the mass used for saponification (m_{SP}).

In case of lower weight of residue, the oil content extracted from the bulked sample is too low to gain sufficient precise value for the unsaponifiable matter. If the extracted oil exceeds 5g, an aliquot of maximum 5g to the nearest 0.01g (m_{SP}) in a round bottom flask is weighed and proceeded for the saponification.

7.5 Saponification and Extraction

50 ml of the ethanolic 1 N potassium hydroxide solution is added to the residue, the reflux condenser is attached and the mass is boiled gently for one hour. Heating is stopped. 100 ml of distilled water is added through the top of the condenser and swirled.

The lukewarm matter content is transferred into a 500 ml separation funnel A. The flask is rinsed several times with hexane (100 ml in all) and then poured the mass into the separation funnel. The funnel is stoppered and shaken vigorously for two minutes, periodically releasing pressure by inverting the separation funnel and opening the stopcock.

The layers are allowed to separate. The lower aqueous soap solution is then removed into a second separation funnel B and the aqueous solution is extracted again with 100 ml hexane in funnel B. The soap solution is discharged. The hexane phase from funnel B is combined with the hexane phase in funnel A. 100 ml of 1 N aqueous potassium hydroxide solution is added to funnel A, shaken vigorously and the aqueous solution is discharged.

The hexane extract is washed cautiously two times with 100 ml 50% v/v ethanol and once with 100 ml distilled water to neutral reaction on pH paper.

To avoid emulsions the separator funnel is rotated gently.

All alkaline solutions have to be neutralized prior to disposal.

A previously dried 200 ml round bottom flask is weighed to the nearest 0.001g (m_b). One funnel is plugged with glasswool and put on a bed of 20g anhydrous sodium sulphate. Then the hexane solution is filtered into the flask. The solvent is evaporated under vacuum to dryness (320 mbar) at 40°C (water bath). The open flask is cooled in a desiccator for 3 hours (with silica bluegel as drying agent). The flask is weighed to the nearest 0.001g (m_b+R). The drying is repeated successively for 15 min until constant weight.

8.0 PROCEDURE

8.1 Instrument Check

The analytical balance is conditioned, calibrated and ensured.

8.2 Reagent Blank and Reference Compounds

The blank run without sample must gain no gravimetrically measurable residue.

8.3 Calculations

8.3.1 Determination of the Mass of Extracted Oil m_R from Jute Bags/Cloth

$$m_R = m_{e+R} - m_e \text{ [g]}$$

m_R : mass of extracted oil [g]

m_e : mass of 500 ml round bottom flask [g]

m_{e+R} : mass of 500 ml round bottom flask with residue (extracted oil) [g]

8.3.2 Determination of the Extractable Oil Content C_o in Jute Bags/Cloth

$$C_o = \frac{m_R}{m_s} \times 100 \text{ (\%)}$$

m_s : mass of sample * [g]

C_o : extractable oil content [%]

8.3.3 Determination of the Unsaponifiable Matter C_u in Jute Bags/Cloth

$$C_u = \frac{m_{b+R} - m_b}{m_s} \times \frac{m_R}{m_{SP}} \times 10^6 \quad \left[\frac{\text{mg}}{\text{kg}} \right]$$

m_b : mass of the 250 ml round bottom flask [g]

m_{b+R} : mass of 250 ml round bottom flask with residue [g]

m_{SP} : mass of oil used for saponification [g] (= m_R , if $m_R < 5$ g)

8.4 Repeatability

The difference between two single test results obtained with the same method on identical test material, under the same conditions (same operator, same apparatus, same laboratory and short intervals of time) should not exceed 20%.

* Under ordinary laboratory conditions.

9.0 CALCULATION

9.1 Expression of Results

Extractable oil content* C_o in %, calculated per mass jute bags/cloth.

Unsaponifiable matter* C_u in mg per kg jute bags/cloth [ppm] as received.

Retention of samples

Samples are to be retained by the respective Institutions responsible to conduct testing and certifying for a period not less than 120 days.

Note: If an apparatus for drying under vacuum is not used, dry in an oven regulated at $75^{\circ}\text{C} \pm 2^{\circ}\text{C}$ placing the flask in an almost horizontal position and cooling it in a desiccator till constant weight.

10.0 ANALYTICAL FLOW SHEET

Appendix - II

11.0 INTERNAL CONTROL PLAN

As long as there is no batching oil free jute bags/cloth available, once a year weigh light mineral oil (bp $>270^{\circ}\text{C}$) to the nearest 50.0 mg and carry out the procedure in (7.2 - 7.5). The recovery should be between 90% and 110%.

12.0 REFERENCES

1. IUPAC 2.401, Standard methods for the analysis of oils, fats and derivatives: Determination of the unsaponifiable matter.
2. Sacks for the Transport of food aid, European Standard EN 766.
3. British Standard Institute. Method for the determination of added oil content of jute yarn, rove and fabric. British Standard 3845:1990 (Now withdrawn).

13.0 APPENDICES

Appendix-I : Safety Instructions

Appendix-II : Analytical Flow Sheet

Appendix-III : Certificate for Food Grade Jute Bags/Cloth [IJO Standard 98/01]

Appendix-VI : Inspection and Certification Agencies/Authorities

Appendix-V : LiChrosolv

* Under ordinary laboratory conditions.

Appendix - I

Unsaponifiables
in Jute bags/cloth

SAFETY INSTRUCTIONS

(This information refers to occupational exposure)

CAS N°	COMPOUND	HAZARDS	PRECAUTIONS	DISPOSAL
64-17-5	Ethanol	Flammable. Contains methanol: ingestion or contact with eyes may cause blindness	Use with adequate ventilation	Solvent waste
110-54-3	Hexane (-n)	May cause dizziness, headache, nausea. Extremely flammable	Use with adequate ventilation	Solvent waste
1310-58-3	Potassium hydroxide	Toxic corrosive	Avoid contact	Neutralise
7757-82-6	Sodium sulphate	Irritant	Avoid contact: Avoid inhalation	Chemical waste, no special precautions

Appendix - II

Unsaponifiables
in Jute bags/cloth

ANALYTICAL FLOW SHEET

<u>Steps</u>	<u>Critical Points</u>
Preparation of the sample	Sample from jute bag/cloth lot at random
↓	
Soxhlet extraction with hexane	Minimum 10 cycles
↓	
Solvent evaporation	
↓	
Saponification with ethanolic KOH	1 N KOH
↓	
Separation with hexane	Avoid emulsion
↓	
Washing with KOH and dist. Water	Avoid emulsion
↓	
Neutralize with dist. Water & Ethanol: water	Check pH, avoid emulsion
↓	
Evaporation	
↓	
Drying	Regenerated silica-bluegel
↓	
Weighing	Constant weight
↓	
Calculation	

INSPECTION AND CERTIFICATION AGENCIES/AUTHORITIES

Bangladesh	India
<p>1. Bangladesh Council of Scientific and Industrial Research (BCSIR) Dr. Kudrat-i-Khuda Road Dhanmondi, Dhaka-1205 Tel: (880-2) 8621741 Fax: (880-2) 8613022 E-mail: bcsir@bangla.net</p>	<p>1. Indian Jute Industries' Research Association (IJIRA) 17, Taratala Road Kolkata -700 088 Tel: (+911-033) 2401 4615 Fax: (+911-033) 2401 4621 E-mail: ijira@vsnl.com</p>
<p>2. Bangladesh Jute Research Institute (BJRI) Manik Mia Avenue Dhaka-1207 Tel: (880-2) 9110953, 8121931-2 Fax: (880-2) 9118415 E-mail: bjri@bracbd.net</p>	<p>2. M/s. Surveillance Generale Superintendente (SGS) India Ltd. 4, Govt. Place (North) Delta House Kolkata-700 001 Tel: (+911-033) 2248 2461/6955 Fax: (+911-033) 2248 1745/ 2242-0745</p>
<p>3. Bangladesh Standards & Testing Institution (BSTI) 116/Kha, Tejgaon Industrial Area Dhaka-1208 Tel: (880-2) 8821462, 9897888, 9131581-82, 9880007, 9898115 Fax: ((880-2) 9131581 E-mail: bsti@bangla.net Website: www.bstibd.org</p>	<p>3. Textile Committee Govt. of India, Ministry of Textiles Block GN, Plot No. 38/3, Sector-V Salt Lake City Kolkata- 700 091 Fax: (+911-033) 2357 5202 E-mail: tccal2@vsnl.net</p>
<p>4. SGS (Bangladesh) Ltd. Noor Tower (7th & 8th floor) 73, Sonargaon Road Dhaka-1205 Tel: (880-2) 9676500 Fax: (880-2) 9676491, 9676495 E-mail: sgs@bangla.net Website: www.sgs.com</p>	

Appendix - V

Unsaponifiables
in Jute bag/cloth

n - Hexane LiChrosolv

LiChrosolv - Equivalent
Grade HPLC
N-Hexane for Chromatography
M = 86.18 g/mol

Assay (GC)	=	Min. 97%
Non Volatile Substances	=	Max. 0.001%
Water	=	Max. 0.01%
Acidity	=	Max. 0.0005 meq/g
Alkalinity	=	Max. 0.0002 meq/g
UV Transmittance	=	At 210 nm - min. 50% At 220 nm - min. 80% From 245 nm - min. 98%

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