<u>JSS 8305 - 14 : 2015</u> (Revision No. 3)



# रक्षा मंत्रालय MINISTRY OF DEFENCE

# संयुक्त सेवा विनिर्देश JOINT SERVICES SPECIFICATION

ON

BLEACHED COTTON LINTERS (DS Cat. No. 8305 - 000 497)

मानकीकरण निदेशालय रक्षा उत्पादन विभाग रक्षा मंत्रालय 'एच' - ब्लाक, निर्माण भवन डाकघर नई दिल्ली - 110 011

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# <u>JSS 8305 - 14 : 2015</u> (Revision No. 4)

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Amendment		Amendment pertains to :	Authority	Amended by	Signature
No.	Date	Sl. No. / Para No. / Column No.		Name & Appointment (IN BLOCK LETTERS)	& Date

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## 0. <u>FOREWORD</u>

0.1 This Joint Services specification has been prepared by Armament Standardisation Sub Committee on the authority of the Standardisation Committee, Ministry of Defence.

0.2 This specification has been approved by the Ministry of Defence and is mandatory for use by the Defence Services.

0.3 (a) Second revision was done in the year 1998.

(b) This specification is a revision of JSS 8305 - 14 : 2009, (Revision No. 3) and supersedes the same.

0.4 This Specification would be used for Manufacture, Supply and Quality Assurance of Bleached Cotton Linters.

0.5 Quality Assurance Authority for the item covered in this specification is The Controller, Controllerate of Quality Assurance (Military Explosives), Aundh Road, Pune - 411 020. Enquiries regarding technical parameters shall be addressed to the Quality Assurance Authority, while other enquiries shall be referred to :

The Director, Directorate of Standardisation, Ministry of Defence, 'H' - Block, Nirman Bhawan PO, New Delhi - 110 011.

0.6 Non registered users can obtain the following on payment :

#### (a) Copies of IS from :

Bureau of Indian standards, Manak Bhawan, 9, Bahadur Shah Zafar Marg, New Delhi - 110 002.

or

Their regional / Branch offices.

## (b) Copies of JSSs / JSGs from :

The Director, Directorate of Standardisation Standardisation Documents Centre, Ministry of Defence, Room No. 05, 'J' - Block, Nirman Bhawan PO, New Delhi - 110 011.

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0.7 Indian Standard (IS) specifications are available free of cost for registered users on :

Directorate of Standardisation Website www.ddpdos.gov.in For registration visit our website.

#### 0.8 This specification holds good only for the supply order for which it is issued.

0.9 <u>DIRECTORATE OF STANDARDISATION WEBSITE</u>: All the approved JSSs / JSGs are available on the Directorate of Standardisation Website **www.ddpdos.gov.in**. Defence Organisations desirous of accessing a copy of this document are requested to approach the Directorate of Standardisation for obtaining user id / password to access the website.

**1.** <u>SCOPE</u>: This specification is meant to govern Manufacture, Supply and Quality Assurance of Bleached Cotton Linters suitable for use in the manufacture of Nitrocellulose for explosives.

## 2. <u>RELATED SPECIFICATIONS / DOCUMENTS</u>

2.1 Reference is made in this specification to :-

Sl. No.	Specification No. & Year	Nomenclature
(a)	IS 138 : 1992 (Third Revision) Reaffirmed 2009 AMD 1	Ready Mixed Paint, Marking, for Packages and Petrol Containers - Specification.
(b)	IS 200 : 1989 (Second Revision) Reaffirmed 2010	Textiles - Determination of Copper Number of Cotton Textile Materials.

**3.** <u>MATERIAL / FINISH</u>: Bleached cotton linters shall essentially consist of cotton linters, obtained during second cut of delinting operation, which have been bleached or semi bleached and shall be free from mechanical and gross impurities.

## <u>Note</u>.

1. <u>Second Cut</u>: Cotton linters that are removed in the second stage of delinting operation. They are generally 2 mm to 6 mm long.

2. <u>Mechanical Impurities</u> : Freedom from contamination by dirt, husk, woody matter, leaf stalk, seed coat, sand, metallic and other contaminations.

3. <u>Gross Impurities</u> : The impurities which result from faulty delinting/ginning such as presence of naps / neps etc.

(a) <u>Naps</u>: These are matted portions of fibre that are stringy or ropy.

(b) <u>Neps</u>: These are small loose entaglements of fibres, which are ropy in appearance.

4. <u>MANUFACTURE</u> : Cotton Linters shall be subjected to suitable mechanical treatment like kiering, washing, bleaching etc to produce a product conforming to this specification.

**5.** <u>**TENDER SAMPLE</u></u> : The manufacturer / supplier / contractor shall submit a tender sample of 2 kg free of all charges and conforming to this specification, to the Quality Assurance Authority / Quality Assurance Officer as stated in the tender / contract.</u>** 

## 6. <u>PRE - INSPECTION OF STORES / CONSIGNMENT</u>

6.1 Manufacturers / contractors must satisfy themselves that the stores are in accordance with the terms of the contract and fully conform to the required specification, by carrying out a thorough pre - inspection of each lot before actually tendering the same for inspection to the Quality Assurance Officer nominated under the terms of the contract. A declaration by the contractor that a necessary pre - inspection has been carried out on the stores tendered, will be submitted along with the challan. The declaration will also indicate the method followed in carrying out pre - inspection showing the features checked / tested and will have the test certificate attached to the challan / declaration.

6.2 If the Quality Assurance Officer finds that pre - inspection of the consignment as required above has not been carried out, the consignment is liable for rejection.

## 7. <u>QUALITY ASSURANCE</u>

7.1 <u>INSPECTION</u>: The material and the packages in which it is packed shall be subjected to inspection by and to the approval of the Quality Assurance Officer / Quality Assurance Authority.

## 7.2 <u>SAMPLING</u>:

7.2.1 A representative sample of 2 kg shall be drawn from each bale and No. of bales to be inspected / Sampled from a lot / Consignment / manufacture shall be as per table given below :-

No. of Bales in the Lot (N)	No. of Bales to be Selected (n)
Up to 5	All
6 to 50	5
51 to 100	8
101 to 300	13
301 to 500	20
501 to 1000	32
1001 and above	50

## TABLE : No. OF BALES TO BE SELECTED AT RANDOM FROM A LOT

7.2.2 From each consignment a minimum of 3 bales shall be opened and the contents examined visually for the presence of any foreign matter. If foreign matter is found to be present, the firm should remove foreign matter from all the bales and reoffer the consignment for inspection.

7.3 <u>CRITERIA FOR CONFORMITY</u> : If on examination, any sample is found not to conform to this specification, the whole bale / lot / consignment may be rejected.

## 7.4 <u>TEST REQUIREMENTS</u>

7.4.1 Samples taken from any portion of the bale/lot/consignment of the material shall conform to clauses 3 and 4 above and shall also conform to the test requirements shown in the following table :-

Sl. No.	Characteristic	Passing Standard	d Test Method	
(a)	Moisture content, % by mass	7.0 Max.	Appendix 'A'	
(b)	Acidity	Nil	Appendix 'B'	
(c)	Mineral matter, % by mass	0.25 Max.	Appendix 'C'	
(d)	Ether soluble matter, % by mass	0.25 Max.	Appendix 'D'	
(e)	Matter soluble in 3 % boiling alkali, % by mass	3.0 Max.	Appendix 'E'	
(f)	Matter soluble in 1 % boiling alkali, % by mass	2.0 Max.	Appendix 'F'	
(g)	Malachite Green dye test	Shall pass	Appendix 'G'	
(h)	Copper number	1.0 Max.	Appendix 'H'	
(j)	Alpha Cellulose content, % by mass	99.0 Min.	Appendix 'J'	
(k)	Dynamic viscosity in Ns/m <sup>2</sup>	0.5 Min. 5.0 Max.	Appendix 'K'	
(1)	Fibre length		Appendix `L'	
	(i) Less than 1.0 mm	Not more than 12 %.		

## TABLE : TEST REQUIREMENTS OF BLEACHED COTTON LINTERS

Sl. No.	Characteristic		Passing Standard	Test Method
	(ii)	More than 6.0 mm	Not more than 12 %	
	(iii)	Average fibre length in mm	Between 2 to 6	

<u>Note</u>. The exact limit of viscosity required may be spelt out in the supply orders / indents placed by the concerned user factory.

7.4.2 "The dynamic viscosity of each sampled bale from a lot shall be within specified limit of dynamic viscosity as spelt out by concerned user factory in supply order. Each lot of BCL shall be in strict conformance with supply order requirement for dynamic viscosity by user factory, otherwise the lot stands rejected."

7.4.3 In addition to the above, the Quality Assurance Officer shall draw a sample of 10 kg from each bale / lot / consignment for carrying out practical trials at the discretion of the Quality Assurance Authority or the consignee.

7.4.4 The samples for practical trials shall perform to the satisfaction of the consignee / user, when trial nitration is carried out at the user's end in the presence of the Quality Assurance Authority or his authorised representative.

8. <u>WARRANTY</u>: The stores supplied against the contract shall be deemed to be warranted against defective material and performance by the contractor for a period of 12 months from the date of receipt of the store at the consignee's end and if during this period any of the stores supplied is found defective, the same shall be replaced by the manufacturer / supplier / contractor free of all charges at the consignee's premises.

## 9. <u>PACKAGING</u>

9.1 The material shall be packed in the form of a suitable bale size having net mass as agreed to between the purchaser and supplier. The wrapping of the bale will be with polythene lined closed textured hessian cloth. The polythene used shall be colourless having film thickness minimum 0.13 mm. The bales will be secured with galvanised steel wire in such a way that it will withstand hazards of transport and handling.

9.2 The inclusion of any foreign matter or impurities in any of the containers / packages shall render the whole consignment liable to rejection.

## 10. <u>MARKING</u>

10.1 All packages containing the material shall be indelibly and legibly marked with the following details (as applicable) :-

- (a) Nomenclature and Specification No. of the Material.
- (b) Name and Address of the Consignee.

- (c) A/T or S.O Number and Date.
- (d) Consignment Number.
- (e) Lot / Bale Number and Date of Manufacture.
- (f) Gross and Net Mass.

(g) Consecutive Number of Package and Total Number of Packages in the Consignment.

- (h) Date of Supply.
- (j) Contractors Initials or Recognised Trademark.

10.2 In addition to the above, the Quality Assurance Officer may suggest some more markings and identifications considered suitable at the time of inspection.

10.3 The paint used for marking shall conform to IS 138 and to the satisfaction of the Quality Assurance Officer / Quality Assurance Authority.

**11.** <u>SAFETY OF OPERATIONS</u>: Nothing in this specification shall relieve the manufacturer / user of his responsibility for the safety of operations in manufacture, handling, storage and use of this store.

## 12. <u>DEFENCE STORES CATALOGUE NUMBER</u>

12.1 Defence Stores Catalogue Number allotted to this store is 8305 - 000 497.

## 13. <u>SUGGESTIONS FOR IMPROVEMENT</u>

13.1 Any suggestion for improvement in this document may be forwarded to :-

The Director, Directorate of Standardisation, Ministry of Defence, 'H' - Block, Nirman Bhawan PO, New Delhi - 110 011. <u>JSS 8305 - 14 : 2015</u> (Revision No. 4)

## APPENDIX 'A'

#### A. <u>DETERMINATION OF MOISTURE</u>

A.1 Weigh a flat bottomed dish with a good fitting press lid  $(M_1)$ . Place about 10 g to 20 g of the sample in it and weigh again  $(M_2)$ . Expose the sample in an oven at 100 °C to 105 °C for three hours. Replace the lid and cool the dish in a desiccator to room temperature and weigh again  $(M_3)$ . Calculate the moisture percentage.

#### A.2 CALCULATION

		$(M_2 - M_3) \ge 100$
Moisture, % by mass	=	
		$(M_2 - M_1)$

## **APPENDIX 'B'**

## B. <u>DETERMINATION OF ACIDITY</u>

B.1 Open out with fingers about 2 g of the sample and place in a beaker. Pour 100 ml of a solution containing 0.025 g of Congo Red per 100 ml of solvent, being a mixture of 10 ml redistilled methylated spirit and 90 ml distilled water. Stir well, remove the linters and squeeze dry. Open out the linters with fingers. The sample shall be uniformly coloured a bright cerise. Traces of acidity are shown by blueing and dulling of a tint. Carry out the test on a further five 2 g samples and record your observations. The colour shall be uniformly bright cerise.

## **APPENDIX 'C'**

## C. <u>DETERMINATION OF MINERAL MATTER</u>

C.1 Heat a Silica or porcelain crucible or dish in a muffle furnace for 15 minutes, cool in a desiccator and weigh  $(M_1)$ . Place about 10 g of the sample in it and weigh again  $(M_2)$ . Carefully burn off the sample, taking care that the sample is not blown off. Transfer the dish to a muffle furnace and ignite at 600 °C to 625 °C for half an hour. Cool the dish in air till safe to place in a desiccator. Cool to room temperature and weigh again  $(M_3)$ .

## C.2 <u>CALCULATION</u>

		$(M_3 - M_1) \ge 100$
Mineral matter,	=	
% by mass		$(M_2 - M_1)$

## APPENDIX 'D'

## D. <u>DETERMINATION OF ETHER SOLUBLE MATTER</u>

D.1 Extract about 5 g of the sample in a soxhlet apparatus with Ethyl ether for three hours at the rate of 12 extractions per hour. Increase the time of extraction if the rate is slower. Finally boil off the Ether and dry the flask in an oven for one hour at 105 °C. Cool the flask in a desiccator and weigh.

## D.2 CALCULATION

		(M <sub>2</sub> - M <sub>1</sub> ) x 100
Ether soluble,	=	
% by mass		Μ

Where :

$M_2$	=	Mass	in g	of the	dry	flask	with	residue.
4			0		· .			

- $M_1$  = Mass in g of the dry empty flask.
- M = Mass in g of the sample taken on dry basis.

## **APPENDIX 'E'**

## E. <u>DETERMINATION OF MATTER SOLUBLE IN 3 % BOILING ALKALI</u>

E.1 Weigh accurately about 2 g of the dry sample into a 500 ml conical flask and add 200 ml of a 3 per cent boiling solution of Sodium hydroxide. Fit an air condenser and immerse the flask upto its neck into a vigorously boiling water bath and let stand for one hour. Decant off the solution through a previously weighed G1 sintered glass crucible and wash the linters down till free from alkali with portions of hot distilled water before transferring to the crucible wash well. Extract dry, first with distilled water and then with Alcohol or Acetone. Dry at 105 °C for three hours in an oven and weigh.

#### E.2 <u>CALCULATION</u>

Matter soluble in 3 %		(M <sub>1</sub> - M <sub>2</sub> ) x 100
boiling alkali, % by mass	=	
		$M_1$

Where :

- $M_1$  = Mass in g of the sample on dry basis.
- $M_2$  = Mass in g of the residue.

## **APPENDIX 'F'**

## F. <u>DETERMINATION OF MATTER SOLUBLE IN 1 PERCENT BOILING</u> <u>ALKALI</u>

F.1 The matter which goes into solution on treatment with hot 1 % Sodium hydroxide is a measure of low molecular mass portion of cellulose made up of beta and gamma and other degraded celluloses.

F.2 Extract as above a 2 g sample with 1 % Sodium hydroxide solution for one hour, filter and dry the residue as stated above and weigh.

## F.3 CALCULATION

Matter soluble in 1 %		(M <sub>1</sub> - M <sub>2</sub> ) x 100
boiling alkali, % by mass	=	
		$M_1$

Where :

 $M_1$  = Mass in g of the sample on dry basis.

 $M_2$  = Mass in g of the residue.

## APPENDIX 'G'

## G. <u>MALACHITE GREEN DYE TEST</u>

G.1 <u>PRINCIPLE</u> : The portion of sample which does not get bleached by Chlorine after dyeing fast with acid dye is a measure of presence of ligneous, woody or husky matter in the sample of cotton or pure cellulose.

#### G.2 <u>REAGENT</u>

(i) Dissolve 15 g of Malachite green in 30 ml hot Ethyl alcohol. Filter the solution into 100 ml Ethyl ether. Filter the precipitate, wash well with Ether and dry in vacuum over Sulphuric acid. Dissolve 5 g of this dye in 1 ml of Glacial Acetic acid and 100 ml of distilled water and make up to one litre.

(ii) Mix equal volumes of 40 % aqueous Formaldehyde solution and distilled water.

(iii) Shake 20 g of Bleaching powder, containing about 37 % available Chlorine, with 1 litre of distilled water and allow to settle. Stopper the solution well and do not use for the test, if more than a week old.

G.3 <u>PROCEDURE</u>: Immerse 5 g of the sample in a mixture of 10 ml of the dye as prepared in G. 2 (1), 5 ml of Formaldehyde solution and 250 ml water. Heat for one hour at 80 °C. Add 25 ml of 0.1 % Acetic acid followed by 25 ml of Bleaching powder solution dropwise through a pipette. Stir vigorously with each addition of Bleaching solution. Heat for further 5 minutes. Make a pad of the linters on a buchner funnel and filter the liquor through the pad, rinse well under the tap and then with distilled water. Sqeeze dry and examine for green specks. Repeat on six more portions of the sample. Reject the lot if more than 5 specks of any size are observed in each of the portions after the treatment.

## **APPENDIX 'H'**

## H. <u>DETERMINATION OF COPPER NUMBER</u>

H.1 Copper number is the mass in g of copper reduced by 100 g of bone - dry cellulose from cupric to cuprous state in alkaline solution.

#### H.2 <u>REAGENTS</u>

(i) Dissolve 130 g of Anhydrous Sodium carbonate and 50 g of Sodium bicarbonate in water and make up the volume to one litre.

(ii) Dissolve 100 g of Crystalline Copper sulphate in water and make up the volume to one litre.

(iii) Dissolve 100 g of Ferric alum in water, add 140 ml concentrated Sulphuric acid and make up the volume to one litre.

(iv) Potassium permanganate solution 0.01 N.

(v) Dissolve 1.49 g of Ortho - phenanther line Monohydrate in 100 ml of water containing 0.7 g of Hydrated ferrous sulphate.

(vi) 2 N Sulphuric acid solution.

H.3 <u>PROCEDURE</u>: Heat quickly to boiling a mixture of 95 ml of reagent (i) and 5 ml of reagent (ii) and pour it over a weighed 2.5 g portion of the sample placed in a 250 ml conical flask. Fix an air condenser and immerse in a vigorously boiling water bath upto its neck. Stir gently to distribute the sample evenly after every 30 minutes.

H.4 Remove the flask after exactly three hours and cool in water. Filter through a G2 sintered crucible and wash three times with water. Discard the filtrate and clean the flask. Without applying suction, flood the same with 15 ml of reagent (iii) and allow to react for two minutes. Apply suction to filter. Repeat with a further 10 ml of reagent (iii). Wash the conical flask with 15 ml of reagent (vi) (Sulphuric acid) twice to dissolve all the oxide particles, pouring the solution each time into the crucible and filtering. Finally wash well with distilled water and filter the sample dry.

H.5 Titrate the filtrate and washing in the filter flask with standard 0.01 N Potassium permanganate solution using 5 ml of reagent (v) as an internal indicator, to a sharp end point indicated by a change in colour from red to blue.

H.6 Carry out a blank with 25 ml of reagent (iii) and as much water as was used in the sample.

## H.7 CALCULATION

	V x 0.06354 x F
=	
	М
	=

(1 ml of 0.01 N Potassium permanganate = 0.0006354)

Where :

V = Volume of standard Potassium permanganate (0.01 N) required by sample (after deducting blank)

M = Dry mass in g of the sample.

F = Factor of standard Potassium permanganate (Reference may be made to IS 200).

## **APPENDIX 'J'**

## J. <u>DETERMINATION OF ALPHA CELLULOSE</u>

J.1 <u>PRINCIPLE</u>: The fraction of Cellulose, which remains insoluble in 8.3 % Sodium hydroxide solution after previously swelling in 17.5 % solution of Sodium Hydroxide at  $20 \degree$ C is taken as Alpha Cellulose.

#### J.2 <u>REAGENTS</u>

(i) Dissolve a quantity of Sodium hydroxide pellets in distilled water and let it stand for several days. Decant off the clear portion and dilute with distilled water to a specific gravity of  $1.192 \pm 0.001$  at 20 °C. Titrate an aliquot with standard acid and adjust the strength to  $17.5 \pm 0.1$  g per 100 g of the solution.

(ii) Dilute another portion with Carbon dioxide free distilled water to a specific gravity of  $1.090 \pm 0.001$  at 20 °C and adjust the strength to 8.3 g per 100 g of solution.

## (iii) Acetic acid (2N).

J.3 PROCEDURE : Place all the solutions on a water bath at 20 °C  $\pm$  0.2 °C. Weigh accurately about 3 g of the sample and place in a beaker. Determine the moisture content of the sample side by side. Accurately measure 75 ml of 17.5 % Sodium hydroxide in a measuring cylinder. Add 15 ml of this to the sample and macerate it with a glass rod for one minute, add 10 ml more and macerate for 45 seconds, add 10 ml more and mix for 15 seconds. Thus after 2 minutes a slurry of the sample in 35 ml of the 17.5 per cent solution will be formed. Stir and allow to settle for 3 minutes. Add 10 ml more and mix for 2.5 minutes. Add 10 ml again and mix well for 2.5 minutes. Repeat with the two remaining portions of 10 ml. Cover the beaker alongwith the glass rod with a watch glass. All these operations shall be carried out with the beaker in the water bath at 20 °C. Let it stand for a further 30 minutes. Add 100 ml of distilled water at 20 °C quickly and mix well. Leave the diluted mixture in the bath for a further 30 minutes. Filter through a tarred G1 sintered crucible. Rinse the beaker and residue with 25 ml of 8.3 % Sodium hydroxide solution at 20 °C and quantitatively transfer all the fibres to the crucible. During filtration always keep the pad well covered with solution to prevent drawing of air through the pad. Wash the residue with 50 ml portions of distilled water at 20 °C five times. Wash with 400 ml of distilled water, disconnect the suction and add 25 ml of 2N Acetic acid at 20 °C and allow to soak for five minutes. Draw off by suction and wash with water until free from acid as indicated by a drop of 0.1 % Phenolphthalein solution. Transfer the crucible to an oven and dry for six hours at 100 °C to 105 °C. Cool in a desiccator and weigh quickly.

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# J.4 <u>CALCULATION</u>

		M <sub>1</sub> x 100
Alpha Cellulose,	=	
% by mass		$M_2$

Where :

- $M_1$  = Mass in g of the residue.
- $M_2 \quad = \quad Mass \ of \ the \ sample \ on \ dry \ basis.$

## **APPENDIX 'K'**

#### K. <u>DETERMINATION OF DYNAMIC VISCOSITY</u>

#### K.1 <u>APPARATUS</u>

(i) <u>Viscometer Tube</u> : It is essential that tube should comply with the following requirements. The distance between adjacent graduation marks must be exactly 5 cm and the volume of the tube between graduation marks m and n must be 11.8 ml  $\pm$  1.25 ml. The internal diameter of the tube must be uniformly 1.0 cm  $\pm$  0.05 cm.

(ii) Polished steel balls of 1.6 mm  $\pm$  0.02 mm diameter and mass of the steel ball may be 0.0165 g  $\pm$  0.005 g.

- (iii) A stop watch reading 1/5th second.
- (iv) A small plumb line with mass to swing freely in the viscometer tube.
- (v) A water bath maintained at a constant temperature of 20 °C  $\pm$  0.1 °C.

#### K.2 <u>CHEMICALS</u>

K.2.1 <u>Cuprammonium Hydroxide Solution</u> : The Cuprammonium Hydroxide solution shall have the following composition :-

- (i)
   Copper
    $15 \pm 0.1 \text{ g/l}$  

   (ii)
   Ammonia
    $200 \pm 5 \text{ g/l}$
- (iii) Nitrous Acid less than 0.5 g/l

The solution shall be prepared and tested before use as given in clause K.2.2 and K.2.3.

#### K.2.2 Preparation of Cuprammonium Solution

K.2.2.1 Take a suitable bottle of 5 litre capacity, fitted with cork carrying a centrifugal stirrer and air inlet iron tube. The other end of the iron tube is connected to the wash bottle, containing a solution of Ammonia (0.88 specific gravity). Place 2.6 litres of liquor Ammonia (0.88 specific gravity), 0.4 litre of water, 3 g of cane sugar and 180 g of precipitated reduced copper (passing through 250 micron IS Sieve in the reaction bottle, well lagged externally with felt, keeping the space between the bottom and the vessel filled with ice to prevent loss of Ammonia and excessive formation of Nitrous Acid. Rotate the stirrer at a speed of approximately 400 rev/min.

K.2.2.2 Blow air through the wash bottle into the reaction bottle at the rate of 10 1/h. Compare calorimetrically the solution with a standard solution containing 15 g/l of copper. When the comparison shows that the concentration of copper in the solution under preparation exceeds that of the standard (which is reached usually in about 5 or 6 hours of acration), allow the solution to settle for 30 minutes and syphon off the clear liquid into a stoppered bottle. Again allow the solution to settle and syphon off. Analyse the clear liquid for copper and Ammonia, and adjust the concentration of copper to  $15 \pm 0.1$  g/l of copper and to  $200 \pm 5$  g/l of Ammonia. Check the Nitrous acid by analysis and reject the solution if the value obtained exceeds 0.5 g/l.

K.2.2.3 The cuprammonium solution shall be stored in a blackened bottle fitted with a tap at the bottom and connected at the top through a vessel containing alkaline pyrogallol to a glass vessel (such as a Kipp's apparatus) filled with Nitrogen. Keep it preferably at temperatures below 10 °C, the stability of the solution being greater at such temperatures. Ensure that the solution is standardised before use with respect to copper content, Ammonia content and Nitrous Acid content.

## K.2.3 <u>Analysis of Cuprammonium Solution</u>

K.2.3.1 <u>Copper Content</u> : Boil off the Ammonia from 25 ml of Cuprammonium solution, acidify with Nitric acid (1 : 1) and boil again. Remove all Nitrous acid by adding a trace of Bromine and re - boiling. Cool and then add Ammonia solution dropwise until blue colour is formed. Then add 10 ml glacial Acetic acid followed by 3 g to 5 g of Potassium iodide and titrate in the usual way the liberated iodine against standard Sodium thiosulphate solution. Calculate the copper content from the volume of standard Sodium thiosulphate solution consumed.

Copper content, g/l = ml of 0. 1 N Sodium thiosulphate x 0.2544.

K.2.3.2 <u>Ammonia</u>: Add 2 ml of Cuprammonium solution to 25 ml of 2 N Sulphuric acid and titrate the excess acid with normal Sodium hydroxide, using Methyl red as an indicator. Run a blank, using the same reagents except Cuprammonium solution. Calculate the Ammonia content from the difference between the readings for the blank and the solution under test. Correct the value so obtained by subtracting from it, the Ammonia equivalent to Copper which is  $0.536 \times C \text{ g/l}$  of Ammonia, C being the Copper concentration (g/l).

Ammonia content, g/l =  $(1 \text{ N Sodium hydroxide (ml) for blank - 1 N Sodium hydroxide (ml) for sample) x 8.5-0.536 x C$ 

K.2.3.3 <u>Nitrous Acid</u>: Determine the volume of Cuprammonium solution necessary to decolorize 10 ml of 0.1 N Potassium permanganate in the presence of excess of dilute Sulphuric acid at 50  $^{\circ}$ C and calculate the Nitrite in the usual way.

Nitrous acid content g/l = 23.51 Cuprammonium solution (ml) required to decolorize 10 ml of 0.1 N Potassium permanganate.

## K.3 <u>PREPARATION OF SAMPLE SOLUTION</u>

K.3.1 Place a pair of small glass rods in a specific gravity bottle and weigh accurately with lid. Fill the bottle with distilled water at 20 °C and reweigh. Determine the mass of the specific gravity bottle in this way. Calculate the mass of the dry sample required for making exactly 2 % solution by applying moisture correction to the weighed sample. Pre drying of the BCL sample is not advisable. Weigh accurately the sample required into the dried specific gravity bottle and fill with Cuprammonium solution, replace the lid and seal off with paraffin wax to render airtight. Place in an opaque container, fix to a rotary device, so that solution will be completed within 24 hours. Pour the solution into the viscometer using a small funnel and glass rod. The viscometer shall remain in the water jacket at 20 °C throughout.

## K.4 <u>METHOD</u>

K.4.1 The water jacket of the viscometer is filled with water at 20 °C  $\pm$  0.1 °C. The viscometer tube containing the sample is placed in position and the three piece diaphragm tightened centrally. The rubber tube and screw clip are removed leaving the short glass tube protruding through the stopper to act as a releasing tube for the steel ball. The steel ball is placed in the solution at the top of this short glass tube, from which it is allowed to slide into the main bulb of solution in the viscometer tube. The time taken for the steel ball to fall through a distance of 15 cm is noted. The mean of two determinations is taken and the dynamic viscosity 'Z' is calculated from the expression.

$$Z = 0.042 t$$

Where :

t = Observed time in seconds of fall of steel ball.

## APPENDIX 'L'

## L. <u>METHOD FOR DETERMINATION OF FIBRE LENGTH</u>

#### L.1 <u>REAGENTS</u>

- (i) Sodium hydroxide 4 N
- (ii) Glycerol water mixture, 90 per cent v/v

#### L.2 <u>APPARATUS</u>

- (i) Soxhlet
- (ii) Projection Microscope
- (iii) Stage Micrometer
- (iv) Screen

#### L.3 <u>PROCEDURE</u>

L.3.1 Open the bulk sample of bleached cotton linters and reduce the bulk by method of quartering to 25 g.

L.3.2 Transfer 0.5 g sample to 100 ml glass stoppered conical flask. Add 80 ml of 4 N Sodium hydroxide solution. Shake vigorously for one minute. Filter through sintered glass crucible G - 3. Wash with distilled water till free from alkali, as tested by Phenolphthalein. Then wash with Acetone and Petroleum ether and dry in air.

L.3.3 Wet the pad with 2 ml to 3 ml Glycerol solution (90 %), remove the pad and place on a watch glass. Transfer small portions to 100 ml glass stoppered conical flask and swirl the contents with 25 ml to 50 ml of Glycerol solution (90 per cent) till even dispersion is attained.

L.3.4 Using suitable glass tube, transfer a large drop of dispersion to the centre of 5.0 cm glass slide. Cover with second slide and squeeze together till the drop covers the whole area.

L.3.5 Trace the length of individual fibres on the screen by means of coloured crayon pencils. Measure the length of fibres using map measuring device.

or

Measure the length of the fibre by means of thread which is afterwards measured by measuring scale.

L.3.6 Prepare number of slides as above, so as to obtain minimum 50 readings.

## L.4 <u>CALCULATION</u>

L.4.1 Calculate number of fibres as percentage of the whole, which fall into the following distribution :-

Less than 1.0 mm, 1.01 mm to 2.0 mm, 2.01 mm to 3.0 mm, 3.01 mm to 4.0 mm, 4.01 mm to 5.0 mm, 5.01 mm to 6.0 mm and more than 6.01 mm.

L.4.2 From the above values, calculate percentage by mass of the fibres falling in different groups assuming that the cross section area of all the fibres is constant.