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JSS 6810-133 : 2021 (Third Revision)



# श्रारत सरकार GOVERNMENT OF INDIA

रक्षा मंत्रालय MINISTRY OF DEFENCE

संयुक्त सेवा विनिर्देश JOINT SERVICES SPECIFICATION

ON

ZINC STEARATE

(DS Cat Part No. 6810-001 161) (NSN 6810720442739)

मानकीकरण निदेशालय रक्षा उत्पादन विभाग रक्षा मंत्रालय 'एव' ब्लॉक, निर्माण भवन डाकघर नई दिल्ली-११००११

DIRECTORATE OF STANDARDISATION
DEPARTMENT OF DEFENCE PRODUCTION
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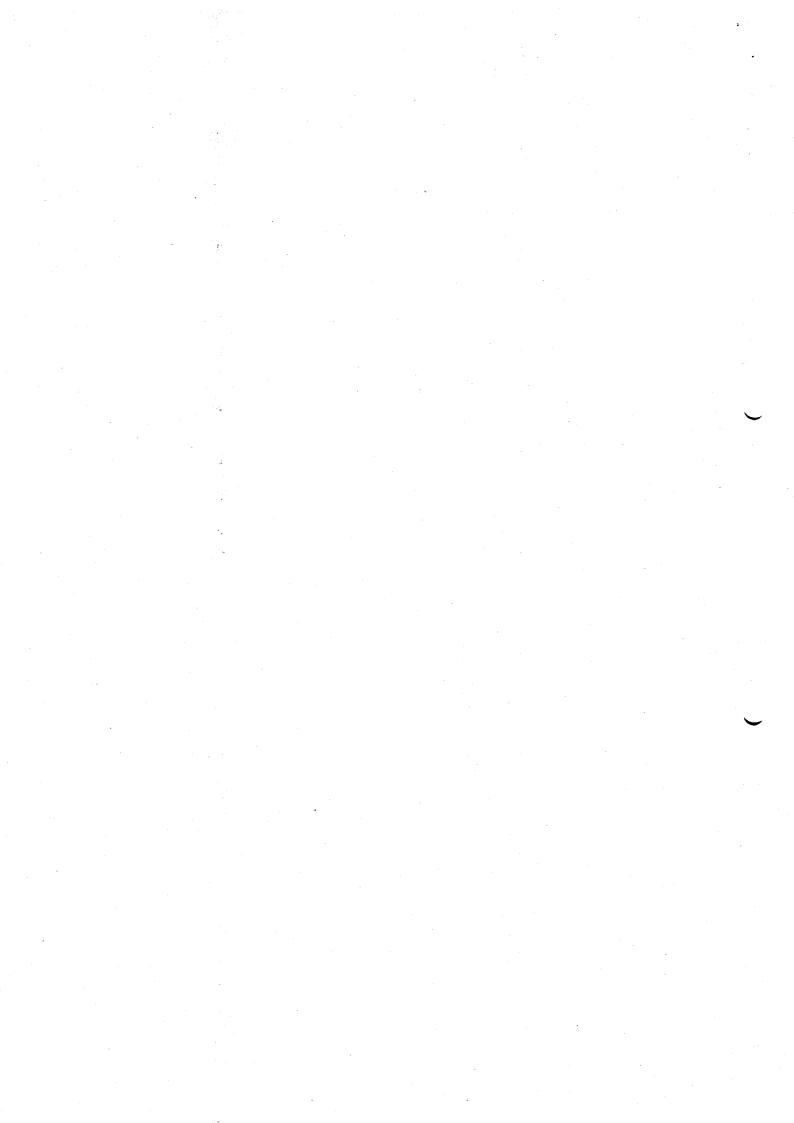
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No.	Date	S. No./Para No./ Column No.		Name & Appointment (In Block Letters)	& Date	
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#### 0. FOREWORD

- **0.1** This Joint Services Specification has been prepared by the 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** This JSS 6810-133 : 2021, (Third Revision):
  - a) was prepared in the year 2001.
  - b) was revised in the year 2010 & 2016.
  - c) is a revision of JSS 6810-133 : 2016 (Second Revision) and supersedes the same.
- **0.4** This specification is meant to govern Supply and Quality Assurance of Zinc Stearate.
- 0.5 Quality Assurance Authority for the item covered by this specification is the Controller, Controllerate of Quality Assurance (Military Explosives), Aundh Road, Pune-411020 (email id cqamear-dgqa@nic.in). 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-110011.
Secretary ASSC, e-mail id-assc.defstand@gov.in

- **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-110002

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-110011

**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 Directorate of Standardisation Website-**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 visit the Directorate of Standardisation website for registration obtaining user id/password to access the JSSs/JSGs.

#### 1. SCOPE

This specification is meant to govern Supply and Quality Assurance of Zinc Stearate suitable for use as a pressing aid in the pelleting of certain HE and Pyrotechnic materials.

#### 2. RELATED SPECIFICATIONS/DOCUMENTS

References are made in this specification to:

Table 1

S	Specification/	Nomenclature	
No.	Document No.		
a)	IS 138 : 2018	Ready Mixed Paint, Marking, for Packages and	
	(Fourth Revision)	Petrol Containers-Specification.	
b)	IS 460 (Part 1): 2020	Specification for Test Sieves	
ŕ	(Fourth Revision)	Part I Wire Cloth Test Sieves.	
c)	BS 593 : 1989	Specification for Laboratory Thermometers.	

#### 3. MATERIAL

The Zinc Stearate shall consist essentially of the Zinc Soap of Stearic acid, but the presence of a proportion of Zinc palminate will not be considered objectionable. It shall be in the form of a fine, white, free flowing powder, free from foreign matter and visible impurities.

## 4. TENDER SAMPLE

The manufacturer/supplier/contractor shall submit a tender sample of 1 kg, essentially from the same batch/lot of manufacture, free of all charges and conforming to this specification, when called for in the tender.

#### 5. PRE-INSPECTION OF STORES/CONSIGNMENT

- 5.1 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.
- 5.2 If the Quality Assurance Officer finds that the pre-inspection of the consignment as required above has not been carried out, the consignment is liable for rejection.

# 6 QUALITY ASSURANCE

#### 6.1 Inspection

- **6.1.1** The Zinc Stearate and the packages in which it is packed shall be subject to inspection by and to the approval of the Quality Assurance Officer/Quality Assurance Authority.
- **6.1.2** Sample of the material and of the packages may be taken from any portion of a consignment.

# 6.2 Sampling

A representative sample of 250 g shall be drawn from each container. Normally the number of containers to be selected at random from a batch/lot shall depend on the size of the batch/lot and shall be in accordance with the following table:

No. of Containers in a Batch/Lot	No. of Containers to be Sampled
Up to 25	3
, 26 to 50	4
51 to 100	5
101 to 150	6
151 to 300	7
301 to 500	8
501 and above	10

# 6.3 Criteria for Conformity

- **6.3.1** If, on examination, any sample is found not to conform to this specification, the whole batch/lot/consignment shall be rejected.
- **6.3.2** The foregoing provisions shall apply equally to prime contractors and to any subcontractor.

# 6.4 Test Requirements

Samples from any portion of batch/lot/consignment shall be in accordance with clause 3 above and shall comply with the following test requirements.

Table 2 Test Requirements of Zinc Stearate

S. No.	Characteristics	Passing Standard	Test Method
a)	Volatile Matter, % by mass	0.5 <i>Max</i>	Appx 'A'
b)	plI of Aqueous extract     or     Reaction of aqueous extract	5.0 Min 7.5 Max	Appx 'B'
	i) Alkalinity to Phenolphthalein ii) Acidity to Phenolphthalein, calculated as CH <sub>3</sub> COOH, % iii) Acidity to Methyl orange	Nil 0.05 Max Nil	

Table 2 Test Requirements of Zinc Stearate (Concluded)

S. No.	Characteristics	Passing Standard	Test Method
c)	Water Soluble matter, % by mass:		Appx 'C'
,	1) Total,	1.0 <i>Max</i>	· · · · · · · · · · · · · · · · · · ·
	2) Chlorides, calculated as Sodium	0.02 Max	
	chloride (NaCl)		
	3) Sulphates, calculated as Sodium	0.15 Max	
	sulphate (Na <sub>2</sub> SO <sub>4</sub> )	1	
d)	Total Zinc content, % by mass	10.15 Min	Appx 'D'
		11.00 <i>Max</i>	
e)	Melting Point	120°C Min	Appx 'E'
f)	Titre (solidification temperature of fatty acid	52°C Min	Appx 'F'
g)	Iodine value of fatty acid	8 Max	Appx 'G'
h)	Sieving Requirements, % by mass:		Appx 'H'
	1) Retained on 150 micrometre IS Sieve	Nil	
	2) Retained on 75 micrometre IS Sieve	4.0 <i>Max</i>	

#### 7. WARRANTY

The stores supplied against the contract shall be deemed to be warranted against the defective material and performance by the contractor for a period of 12 months from the date of receipt of the stores at the consignee's end and shall retain the properties described above. If during this period any of the stores supplied is found defective, the same shall be replaced by the manufacturer/supplier/contractor free of charges at the consignee's premises.

#### 8. PACKAGING

- 8.1 The material shall be packed in polythene bag of film thickness not less than 0.13 mm, heat sealed or bunch tied. The polythene bag is then packed inside close textured strong double walled gunny bag which is stitched in such a way that the polythene bag inside is not pricked or torned. The quantity per package shall be 25/50 kg or as agreed between purchaser and supplier.
- 8.2 For Rail Transit the material should be pre-packed in polythene bags and then steel drums suitable for Rail Transit.

# 9. MARKING

- 9.1 All packages containing the material shall be indelibly and legibly marked with the following details:
  - a) Nomenclature and Specification Number of the Material.
  - b) Name and Address of the Consignee.
  - c). A/T or SO Number and Date.

P,

- d) Consignment Number.
- e) Batch No. and Date of Manufacture.
- f) Gress and Net Mass.
- g) Consecutive Number of Package and Total Number of Packages in the Consignment.
- h) Date of Supply.
- j) Manufacturer's Initials or Recognised Trademark.
- 9.2 In addition to the above, the Quality Assurance Officer may suggest some more markings/identifications suitable at the time of inspection.
- 9.3 The paint used for marking should conform to IS 138 and to the satisfaction of the Quality Assurance Officer/Quality Assurance Authority.

# 10. DEFENCE STORES CATALOGUE NUMBER/NATO STOCK NUMBER

The Defence Stores Catalogue Number allotted to the item covered by this specification is 6810-001 161 and NATO Stock Number of the store is 6810720442739.

## 11. SAFETY OF OPERATIONS

Nothing in this specification shall relieve the supplier/contractor of his responsibility for the safety of operations in the manufacture, storage, transit or use of this store.

# 12. SUGGESTIONS FOR IMPROVEMENT

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-110011

APPX 'A' (Clause 6.4)

# **DETERMINATION OF VOLATILE MATTER**

A-1. Transfer 4 g  $\pm$  0.05 g of the material (M) to a glass dish (about 5 cm in diameter and 3 cm in height) fitted with a ground glass lid. Accurately weigh the dish and contents (M<sub>1</sub>). Place in an oven maintained at 100°C to 105°C for 2 hours, cool in a desicctor and weigh (M<sub>2</sub>). Calculate the loss in mass as volatile matter, % by mass.

Volatile Matter, % by mass 
$$=$$
  $M_1 - M_2$   $\dots \times 100$   $M$ 

**APPX 'B'** (Clause 6.4)

# DETERMINATION OF pH AND REACTION OF AQUEOUS EXTRACT

# B-1. AQUEOUS EXTRACT OF THE MATERIAL

- **B-1.1** Transfer 20 g  $\pm$  0.05 g of the material to a 500 ml beaker and add 40 ml of neutral Industrial Methylated spirit. Stir the mixture and break up any lumps in the material with a stirring rod. Add 200 ml of cold freshly boiled distilled water to the beaker and stir vigorously for about two minutes. Filter on a Buchner funnel and wash with two 50 ml portions of distilled water. Make up the combined filtrate and washing to 500 ml in a volumetric flask.
- **B-1.2** Prepare a blank similarly, omitting only the sample.

# B-2. pH

Find out pH of the aqueous extract electrometrically/potentiometrically by using standard electrodes.

# **B-3. REACTION OF AQUEOUS EXTRACT**

Take aqueous extract of 20 g of the material as described above, make up the combined filtrate and washing to 500 ml and divide into two equal portions.

### **B-3.1** Alkalinity to Phenolphthalein

To the first portion add five drops of Phenolphthalein indicator and note the colour of the solution. Consider the alkalinity to be NIL if the solution does not turn pink.

#### **B-3.2** Acidity to Phenolphthalein

If, the solution to which Phenolphthalein has been added as specified in clause (B-3.1) remains colourless; titrate with 0.1 N Sodium hydroxide solution to a pale pink end-point.

where,

V = ml of 0.1N Sodium hydroxide solution used

F = Factor of 0.1N Sodium hydroxide solution

W = Mass in g of sample represented by the aliquot taken

# **B-3.3** Acidity to Methyl Orange

To the second portion of the solution add a few drops of Methyl orange indicator. If the presence of acidity to Methyl orange is indicated by the solution turning pink or orange titrate the solution with 0.1 N Sodium hydroxide solution to a yellow end-point.

where,

V = ml of 0.1 N Sodium hydroxide solution used.

F = Factor of 0.1 N Sodium hydroxide solution used.

W = Mass in g of sample represented by the aliquot taken.

APPX 'C' (Clause 6.4)

# DETERMINATION OF WATER SOLUBLE MATTER (TOTAL, CHLORIDES AND SULPHATES)

C-1. Transfer 10  $g \pm 0.05$  g of the material to a 250 ml beaker. Thoroughly wet the sample with 10 ml of 95% Ethyl alcohol and break up any lumps with a stirring rod. Add about 150 ml of distilled water and stir until uniform suspension of the Zinc stearate is obtained. Cover the beaker with a clock glass and boil the solution for 15 minutes. Cool, filter on a No. 41 Whatman filter paper, wash with two 25 ml quantities of distilled water. Make up the filtrate to 250 ml is a volumetric flask.

# C-1.1 Water Soluble Matter (Total)

12

Transfer a 100 ml aliquot of the filtrate to a tared 6.25 cm x 3.75 cm glass basin. Evaporate the solution to dr ness. Dry at 100°C-105°C for ½ hour, cool in a desiccator and weigh. Calculate the increase in mass of the basin as per cent water soluble matter (Total).

#### C-1.2 Water Soluble Chlorides

Transfer a 50 ml aliquot of the filtrate to a 100 ml beaker, add 5 ml 2 N HNO<sub>3</sub> and 5 ml 0.1 N Silver nitrate solution. If a cloudiness develops, determine the chloride present by comparing with turbidities obtained in an identical manner from a standard chloride solution.

#### C-1.3 Water Soluble Sulphates

Transfer a 50 ml eliquot of the filtrate to a 250 ml beaker. Add 5 ml 2 N HCl and 100 ml distilled water, boil and add slowly 5 ml 10% Barium chloride solution. Allow to cool and stand overnight. If a precipitate forms determine the sulphate present by standard procedure, weighing as BaSO.

APPX 'D' (Clause 6.4)

# DETERMINATION OF TOTAL ZINC CONTENT

#### D-1. REAGENTS

# **D-1.1 Potassium Ferrocyanide Solution**

22 g Potassium ferrocyanide and 0.3 g Potassium ferrycyanide in 1 litre of solution.

#### **D-1.2** Diphenyl Benzidine Indicator

0.05 g Diphenyl benzidine dissolved in 5 ml concentrated Sulphuric acid.

NOTE - Only 5 ml of indicator should be made up at a time in no case it should be used after having been made for more than a week.

# D-1.3 Sulphuric Acid

2 N, Specific gravity 1.06-1.10

### D-1.4 Sulphuric Acid

4N

## D.1.5 Zinc oxide

Analar quality previously ignited and cooled.

#### D-2. PROCEDURE

**D-2.1** Weigh out accurately about 0.2 g of Analar quality Zinc oxide (M) and dissolve it in 150 ml of the 2 N Sulphuric acid in a 500 ml iodine flask. Add approximately 8 g of Ammonium sulphate, dissolve, and add 5 drops of the indicator. Run in from a burette 10 ml of Potassium ferrocyanide solution and shake the mixture until a blue colour develops. Continue the titration until the end point, a pale green coloration is reached. Shake vigorously throughout the titration and add the Potassium ferrocyanide solution very slowly during the last 2 ml of titration.

1 ml of Potassium ferrocyanide solution = 
$$\begin{array}{c} M & 65.4 \text{ g} \\ ---x & ----- = F \\ V & 81.4 \end{array}$$

where,

V = Volume in ml of potassium ferrocyanide solution used for titration.

M = Mass in g of ZnO taken.

M

**D-3.** Weigh out accurately about 1.5 g of the Zinc stearate (M), add 75 ml of 4 N Sulphuric acid and boil for 15 minutes, allow to cool until a cake of fatty acids solidifies. Then filter through a No. 41 Whatman paper into a 500 ml. Iodine flask. Boil the residue with two successive quantities of 30 ml of distilled water, cool and filter adding the washings to the original filtrate. Cool, add 8 g of Ammonium sulphate, dissolve, add 5 drops of indicator and titrate as above.

Zinc, % by mass 
$$=$$
  $\frac{V \times F \times 100}{M}$  where,  $V =$  Volume in ml of potassium ferrocyanide solution used for titration.

Mass in g of sample taken.

**APPX 'E'** (*Clause* 6.4)

# **DETERMINATION OF MELTING POINT**

- E-1. Grind a small portion (about 2 g) of the material so that it will pass a 75 micrometre IS Sieve and spread it over the bottom of a glass dish about 5 cm in diameter. Dry the material in a vacuum desiccator over Sulphuric acid for at least 16 hours. Use a thin-walled capillary tube, long enough to extend above the top of the bath, having an inside diameter of about 1 mm and sealed at one end. Fill the tube with the dried, ground sample to a depth of about 5 mm compacting the sample by tapping. Fasten the tube to the standardised thermometer so that the lower end of the tube is in contact with the bulb of the thermometer.
- E-2. Suspend the thermometer in a heating bath. Start the stirring and heat the bath rapidly to about 15°C, below the prescribed melting point, then adjust the heat source so that the rise in temperature does not exceed 1°C per minute. Observe and record the temperature at which the material in the tube first appears to become completely liquefied.
- E-3. When a total immersion thermometer is used and part of the liquid column is not immersed, add the following correction to the observed melting point.

Correction,  $^{\circ}C = N (T-t) \times 0.00016$ 

where,

N = Number of degree in the exposed mercury column

T = Uncorrected melting point.

t = Average temperature of the exposed column as indicated by the second thermometer.

APPX 'F' (Clause 6.4)

# DETERMINATION OF TITRE (SOLIDIFICATION TEMPERATURE OF FATTY ACIDS)

# F-1. EXTRACTION OF FATTY ACIDS

- F-1.1 Place 50 g of the sample in 600 ml beaker and moisten with Methylated spirit. Add 400 ml of hot 4 N Sulphuric acid and boil gently for 15 minutes with frequent stirring. Allow the fatty acids to separate and draw off the lower aqueous layer. Wash the molten fatty acids in the beaker by repeated addition of boiling distilled water, drawing off each wash as completely as possible, until the washings are no longer acid.
- F-1.2 Add a further quantity of hot water and allow the beaker to cool so that a solid cake of fatty acids forms. Remove the cake, absorb surface moisture by application of filter paper, cut or break the cake into pieces and place in a funnel fitted with a dry filter paper. Insert the funnel in a 25 mm diameter test tube and place together in a boiling water oven for 1-1/2 hours so that the cake is melted and the fatty acids are dried and filtered in one operation. Reserve a portion of this material for the determination of Iodine value, (Appx G).

#### F-2. PROCEDURE

- F-2.1 Support the test tube containing the molten fatty acids in the centre of an outer glass vessel 13 cm deep and 10 cm diameter (a 600 ml tall beaker is suitable). Suspend a thermometer reading to 70°C in 0.1 or 0.2 degree divisions (BS 593 Type A 70°C) centrally in the test tube so that the top of the bulb is about 1 cm below the surface of the molten acid.
- F-2.2 Allow to sool. The mercury column in the thermometer will fall rapidly at first and then more slowly. It will be found that, after a time, four observations can be made at intervals of about 5 seconds during which the temperature remains stationary. At this point, stir the fatty acids rapidly with the thermometer with a circular movement, three times to the right and three times to the left, taking care to break up the crystals which have formed in the tube. Return the thermometer to the centre of the tube. The mercury column, having fallen sharply during the stirring will rise and attain a maximum and fall again. The maximum temperature is the approximate titre.
- F-2.3 Remelt the fatty acids in the tube by warming on a water bath, stirring with the thermometer and heating the melted mass to about 3°C above the approximate titre. Return to the outer vessel and stir slowly until the temperature has fallen to the approximate titre, then stire rapidly as de cribed above until the temperature ceases to fall. Allow to stand without further stirring and note the maximum temperature reached. This is the accepted titre.

APPX 'G' (Clause 6.4)

# DETERMINATION OF IODINE VALUE OF FATTY ACIDS

#### G-1. APPARATUS

Iodine flask, 500 ml capacity, fitted with ground glass stoppers.

### G-2. REAGENT

Wijs Iodine Solution. Dissolve separately 7.9 g of pure Iodine trichloride and 8.7 g of pure Iodine in glacial Acetic acid (Analar quality) by heating on a water bath. Precautions should be taken to prevent absorption of moisture, pour the solutions into a 1 litre volumetric flask and make up to volume with glacial Acetic acid (AR).

#### G-3. PROCEDURE

Transfer an accurately weighed portion, or approximately 2.0 g of the fatty acids to a clean dry Iodine flask of 500 ml capacity. Add 20 ml of Carbon tetrachloride (Analar quality) and shake the flask until the sample has dissolved. Add 25 ml of the Wijs Iodine Solution by burette. Allow the flask to stand in the dark for 1 hour. Make a blank test in exactly the same way but omitting the sample and allow the flask to stand together with the test flask for the same length of time. After standing, add 20 ml of Potassium iodide solution (10% solution in water) to each flask, shake the flask well and add 200 ml of water. Titrate excess Iodine in each flask with 0.1 N Sodium thiosulphate solution using Starch as indicator towards the end of titration.

Iodine Value of Fatty Acids = 
$$\frac{127 \times F \times (V - V)}{W}$$

where,

F = Factor of 0.1N Sodium thiosulphate solution.

V = ml of 0.1N Sodium thiosulphate solution used to titrate the blank.

v = ml of 0.1N Sodium thiosulphate solution to titrate the sample solution.

W = Mass of sample in grams.

APPX 'H' (Clause 6.4)

# DETERMINATION OF SIEVING REQUIREMENTS

H-1. Take 5 g of the sample, place in a 600 ml beaker, add 50 ml of Acetone and stir vigorously. Pour the suspension on to a 150 micrometre IS Sieve of 10 cm diameter, which is nested into 75 micrometre I.S. Sieve of the same diameter and that, in turn, supported upon a Buchner funnel to collect and remove the Acetone and fines passing through. Wash any material remaining in the beaker on to the sieves with a jet of Acetone. Wash the material on the 150 micrometre IS Sieve with a jet of acetone and brush gently with a camel hair brush until no more material appears to pass through the sieve. Remove this sieve and wash the material retained 1 pon 75 micrometre IS Sieve in the same manner. Air dry the two sieves and brush the material retained into separate small glass dishes and weigh.

S No.	Characteristics	Passing Standard	Test Method
b)	i) pH of Aqueous extract	5.0 Min	Appendix 'B'
	or	7.5 <i>Max</i>	
	ii) Reaction of aqueous extract		
	aa) Alkalinity to Phenolphthalein	Nil	,
	ab) Acidity to Phenolphthalein,	0.05 Max	:-
	calculated as CH <sub>3</sub> COOH, %		
	ac) Acidity to Methyl orange	Nil	
c)	Water Soluble matter, % by mass:		Appendix 'C'
	i) Total,	1.0 <i>Max</i>	
	ii) Chlorides, calculated as Sodium	0.02 Max	
	chloride (NaCl)		
	iii) Sulphates, calculated as Sodium	0.15 Max	
	sulphate (Na <sub>2</sub> SO <sub>4</sub> )		
d)	Total Zinc content, % by mass	10.15 Min	Appendix 'D'
		11.00 <i>Max</i>	
e)	Melting Point	120°C Min	Appendix 'E'
f)	Titre (solidification temperature of fatty acid	52°C Min	Appendix 'F'
g)	Iodine value of fatty acid	8 Max	Appendix 'G'
h)	Sieving Requirements, % by mass:		Appendix 'H'
	i) Retained on 150 micrometre IS Sieve	Nil	
	ii) Retained on 75 micrometre IS Sieve	4.0 Max	

#### 7 WARRANTY

7.1 The stores supplied against the contract shall be deemed to be warranted against the defective material and performance by the contractor for a period of 12 months from the date of receipt of the stores at the consignee's end and shall retain the properties described above. If during this period any of the stores supplied is found defective, the same shall be replaced by the manufacturer/supplier/contractor free of charges at the consignee's premises.

# **8 PACKAGING**

- 8.1 The material shall be packed in polythene bag of film thickness not less than 0.13 mm, heat sealed or bunch tied. The polythene bag is then packed inside close textured strong double walled gunny bag which is stitched in such a way that the polythene bag inside is not pricked or torned. The quantity per package shall be 25/50 kg or as agreed between purchaser and supplier.
- 8.2 For Rail Transit the material should be prepacked in polythene bags and then steel drums suitable for Rail Transit.