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JOINT SERVICES SPECIFICATION

ON

GLYCERINE FOR NITRATION

(DS Cat. No. 6810 - 001 037)

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<u>RECORD OF AMENDMENTS</u>

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

Sl. No.	CONTENTS	
0.	FOREWORD	
1.	SCOPE	
2.	RELATED SPECIFICATIONS / DOCUMENTS	3
3.	MATERIAL	3
4.	MANUFACTURE	3
5.	DESCRIPTION	
6.	TENDER SAMPLE	
7.	PRE - INSPECTION OF STORES AND CONSIGNMENT	
8.	QUALITY ASSURANCE	
9.	SUITABILITY FOR MANUFACTURE OF NITROGLYCERINE	
10.	PACKAGING	
11.	MARKING	
12.	SAFETY OF OPERATIONS	
13.	DEFENCE STORES CATALOGUE NUMBER	
14.	SUGGESTIONS FOR IMPROVEMENT	7
	APPENDICES	8 - 9

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 document is the revision of JSS 6810 - 109 : 2007 (Revision No. 2) and supersedes the same.

0.4 This specification would be used for manufacture, supply and quality assurance of Glycerin, for Nitration.

0.5 Quality Assurance Authority for the item covered by 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 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.

Their regional / Branch offices.

(b) Copies of JSS / JSG from :-

or

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

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

Directorate of Standardisation Website <u>www.defstand.gov.in</u> 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 JSS / JSGs are available on the Directorate of Standardisation Website <u>www.defstand.gov.in</u> 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>

1.1 This specification is meant to govern manufacture, supply and quality assurance of Glycerin, for Nitration suitable for use in the manufacture of Nitroglycerine.

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 1783 (Part 2) : 1988 (Third Revision) Reaffirmed 2005 AMD 1	Drums, Large, Fixed Ends, Part 2 Grade B Drums.
(c)	IS 1784 : 1998 (Third Revision) Reaffirmed 2005	Screwed Closures for Drums.
(d)	IS 1796 : 1986 (Second Revision) Reaffirmed 2005	Specification for Glycerine.
(e)	JSG 0112 : 1997	Methods of Tests and Assessment of Impurities in Chemicals, Materials, used in the Manufacture of Explosives and Ammunition.

3. <u>MATERIAL</u>

3.1 The material shall consist of Dynamite grade refined Glycerin of minimum 98.7 % purity conforming to IS 1796.

4. <u>MANUFACTURE</u>

4.1 Glycerin, for Nitration shall be manufactured by a process which will produce the product conforming to this specification.

5. <u>DESCRIPTION</u>

5.1 The Glycerin shall consist essentially of Glycerol $\{C_3 H_5 (OH)_3\}$. It shall be clear transparent, syrupy liquid not more deeply coloured than yellow brown and shall be free from foreign matter and visible impurities.

6. <u>TENDER SAMPLE</u>

6.1 The manufacturer / supplier / contractor shall submit two tender samples each of 500 g essentially from the same batch / lot of the manufacture free of all charges and conforming to this specification, to the Quality Assurance Officer / Quality Assurance Authority as stated in the tender.

7. <u>PRE - INSPECTION OF STORES / CONSIGNMENT</u>

7.1 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.

7.2 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.

8. <u>QUALITY ASSURANCE</u>

8.1 **Inspection.**

8.1.1 The glycerin 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.

8.2 Sampling.

8.2.1 Two representative samples of 500 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
26to 50	4
51 to 100	5
101 to 150	6
151 to 300	7
301 to 500	8
501 and above	10

8.3 <u>Criteria for Conformity</u>.

8.3.1 If on examination, any sample is found not conforming to this specification, the whole batch / lot / consignment may be rejected.

8.4 **Test Requirements.**

8.4.1 Samples taken from any portion of the batch / lot / consignment of material shall conform to clause 5.1 and in addition shall conform to the test of Glycerin Dynamite Grade given in IS 1796. It shall also comply with the following table of requirements :-

Sl. No.	Characteristic	Passing Standard	Test Method
(a)	Sulphuric acid test	Shall not be darker or any colour other than standard	Appendix 'A'
(b)	Nitrogen content, % by mass	Max. 0.03	Appendix 'B'
(c)	Water content, % by mass	Max. 1.0	Appendix 'C'
(d)	Sulphates calculated as Sodium sulphate, % by mass	Max. 0.005	Method No 8 of JSG 0112

Table : Requirements of Glycerine, for Nitration

9. <u>SUITABILITY FOR MANUFACTURE OF NITROGLYCERINE</u>

9.1 Glycerin, for Nitration shall be satisfactory in all respects in each stage of manufacture of Nitroglycerine. When nitrated, the Nitroglycerine produced must separate quickly and completely from acids and there must be practically no flocculent matter present. To check this trial nitration shall be carried by the respective Ordnance Factory and for this purpose requisite quantity shall be supplied to the manufacturing factory by the contractor. The decision of the manufacturing factory as to the suitability or otherwise, for nitration of the sample, shall be final.

10. <u>PACKAGING</u>

10.1 Glycerin, for Nitration, shall be supplied in sound, clean and dry galvanized drums / stainless steel tankers or other approved packages, each containing an approved quantity and suitably packed to avoid damage either in transit or deterioration in storage.

10.2 Glycerin, for Nitration shall be supplied in sound, clean and dry galvanised drums of nominal capacity 200 liters. The drum should conform to IS 1783 (Part 2) Grade B but galvanised by hot dipping. The closure should be as per IS : 1784.

10.3 14 BG (1.994 mm thick) heavy duty hot dipped Galvanised Iron drums (200 liters nominal capacity) with hoops will be used unless otherwise specified in the contract. These drums shall not be painted internally or at seam sealing / joints with any other paint or any other protective compound.

10.4 The stainless steel tankers for transportation of the glycerin shall be duly inspected and cleared before filling by the Quality Assurance Officer.

10.5 Any other form of packages shall have the prior approval of the Quality Assurance Officer / Quality Assurance Authority.

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

11. MARKING

11.1 All packages containing the material shall be indelibly and legibly marked with the following details :-

- (i) Nomenclature and Specification No. of the Material.
- (ii) Name and Address of the Consignee.

- (iii) A.T. or S.O. No. and Date.
- (iv) Consignment No.
- (v) Lot / Batch No. and Date of Manufacture.
- (vi) Gross and Net Mass.

(vii) Consecutive No. of Package and Total Number of Packages in the Consignment.

- (viii) Date of Supply.
- (ix) Manufacturers / Supplier / Contractor initial or his Recognised Trademark.

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

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

12. <u>SAFETY OF OPERATIONS</u>

12.1 Nothing in this specification shall relieve the manufacturer / contractor / user of his responsibility for the safety of his operation during manufacture, storage transit or use of this store.

13. <u>DEFENCE STORES CATALOGUE</u>

13.1 Defence Stores Catalogue Number allotted to this store is 6810 - 001 037.

14. <u>SUGGESTIONS FOR IMPROVEMENT</u>

14.1 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.

APPENDIX 'A'

A. <u>SULPHURIC ACID TEST</u>

A.1 Cool 2 ml of the Glycerin in a test tube and 2 ml of 96 % Sulphuric acid in a measuring cylinder in ice water for at least 15 minutes. Add the acid to the Glycerin and mix the contents of the tube rapidly by rolling the test tube horizontally in the fingers. Return the tube forthwith to the ice water bath and after 15 minutes compare it with a standard colour prepared as follows :-

Mix (a) 10 ml of Copper sulphate solution (200 g of $CuSO_4$ 5H₂O crystal per liter)

- (b) One ml of Potassium Dichromate solution (80g of $K_2 Cr_2 O_7$ per liter)
- (c) One ml of N/10 Potassium permanganate solution.

Make the mixture up to 250 ml mix and take 4 ml for the test.

A.2 The colour shall not be darker or any colour other than standard colour solution prepared as above.

APPENDIX 'B'

B. <u>NITROGEN CONTENT</u>

B.1 Treat 2 g of the Glycerin by the Kjeldahl method with 20 ml of fuming Sulphuric acid, 10 g of Potassium sulphate, 2g of Sucrose and 0.05 g of Selenium/Aluminium powder and estimate the Nitrogen content from the amount of Ammonia evolved.

APPENDIX 'C'

C. DETERMINATION OF WATER CONTENT BY KARL FISHER'S REAGENT

C.1 Standardisation of Fisher's Reagent.

C.1.1 As Fisher's Reagent is not stable and continuously changes its water equivalent, the reagent must be standardized on the same day the titration of water is to be run.

C.1.2 Pipette exactly 10 ml of Methanol into a dry filtration flask and titrate with Fisher's reagent to change of colour (V_1 ml). Then transfer accurately weighed quantity (w), about 50 mg, of standard water to the flask and titrate again to the end point. Note the total volume of Fisher's Reagent used (v_2 ml)

Calculation

The water equivalence of Fisher's reagent in mg/ml

$$= \frac{W}{V_2 - V_1}$$

C.2 Procedure.

C.2.1 Transfer 50 ml of Methanol (anhydrous) to the titration flask and titrate to the end point with the Fisher's Reagent (V_3 ml).

C2.2 Then transfer an accurately weighed quantity of the sample, containing 0.01 to 0.10 g of water, to the flask, Titrate again to the end point (V_4 ml)

Calculation

Where,

E = Water equivalent of Fisher's Reagent in mg of H₂O/ m1 as determined above.

W = Mass of the sample, in g.

 $V_4 - V_3 =$ ml of Fisher's Reagent used for the sample.