भारत सरकार
GOVERNMENT OF INDIA
रक्षा मंत्रालय
MINISTRY OF DEFENCE

संयुक्त सेवा विनिर्देश
JOINT SERVICES SPECIFICATION

ON

PENTA-ERYTHRITOL DI-OLEATE, LEAD FREE

मानकीकरण निदेशालय
रक्षा उत्पादन विभाग
रक्षा मंत्रालय
'एच'-ब्लाक, निर्माण भवन डाकघर
नई दिल्ली-११००११

DIRECTORATE OF STANDARDISATION
DEPARTMENT OF DEFENCE PRODUCTION
MINISTRY OF DEFENCE
'H' BLOCK, NIRMAN BHAVAN POST OFFICE
NEW DELHI-110011

LIST OF MEMBERS ASSOCIATED WITH FORMULATION OF THIS STANDARD

1. This Second Revision of the Joint Services Specification 6810-118 has been approved by RS Gauba, Sc 'G' Member Secretary, ARMREB; Chairman, Armament Standardisation Sub-Committee by circulation.

2. The representatives of following organisation have been present/consulted in preparing the document:

S. No.	Organisation
1.	ADGWE/GS (WE-2/3), New Delhi
2.	Dte of Arty (GS/Artillary-5), New Delhi
3.	Dte Gen of Naval Armt, Naval HQ, New Delhi
4.	Dte of Armt & Safety Eqpt, Air HQ, New Delhi
5.	DGEME, Army HQ, New Delhi
6.	DGNAI, Naval HQ, New Delhi
7.	DGAQA, JD (Armt) Gp, New Delhi
8.	CQA (ME), Pune
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10.	CQA (SA), Ichapur, West Bengal
11.	CQA (W), Jabalpur
12.	HEMRL, DRDO, Pune
13.	ARDE/DRDO Orgn, Pune
14.	Ammunition Factory, Pune
15.	Secretary ASSC
16.	DG(ACE) Pune

RECORD OF AMENDMENTS

Amendment		Amendment pertains to S. No./Para No./ Column No.	Authority	Amended by	Signature & Date
No.	Date			Name & Appointment (in block letters)	

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0. FOREWORD

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 This JSS 6810-118 : 2017 (Second Revision).

a) was prepared in the year 1998.

b) was revised in the year 2013 and supersedes the same.

0.4 This JSS would be used for Manufacture, Supply and Quality Assurance of Panta-Erythritol Di-oleate, Lead free.

0.5 Quality Assurance Authority for the item covered in 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 approach the Directorate of Standardisation for obtaining user id/password to access the website.

1. SCOPE

This specification is meant to govern manufacture supply and quality assurance of Penta-Erythritol Di-oleate, Lead free suitable for use in the manufacture of plastic propellant and plastic explosive and also used in RDX/Wax special No. 8 Aluminum composition as Surface active agent for coating of wax on RDX.

2. RELATED SPECIFICATIONS/DOCUMENTS

References are made in this specification to:

Table 1

S. No.	Specification No. & Year	Nomenclature
a)	IS 138 : 1992 (Third Revision) AMD 1 Reaffirmed 2014	Ready Mixed Paint, Marking, for Packages and Petrol Containers - Specification
b)	IS 9591 : 1996 (First Revision) Reaffirmed 2013	Plasticizer Esters - Methods of sampling and tests

3. MATERIAL

The Penta-Erythritol Di-oleate, Lead free shall consist essentially of the partial oleic esters of Penta- Erythritol. It shall be in the form of a brown liquid, free from visible impurities and foreign matter.

4. MANUFACTURE

Penta-Erythritol Di-oleate $(C_{17}\ H_{33}\ COOCH_2)_2.\ C(CH_2\ OH)_2$ shall be manufactured by coalification of Penta-Erythritol which will produce the product conforming to this specification.

5. TENDER SAMPLE

The manufacturer/supplier/contractor shall submit two tender samples each of 100 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.

6. PRE-INSPECTION OF STORES/CONSIGMENT

The 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 the pre-inspection of the consignment as required above has not been carried out, the consignment is liable for rejection.

7. QUALITY ASSURANCE

7.1 Inspection

The Penta-Erythritol Di-oleate 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 Sampling

Two representative samples of 100 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

7.3 Criteria for Conformity

7.3.1 If on examination, any sample is found not to conform to this specification, the whole batch/lot/consignment shall be rejected.

7.3.2 The foregoing provisions shall apply equally to prime contractors and to any sub-contractors, if any.

7.4 Test Requirements

Samples taken from any portion of batch/lot/consignment of material shall conform to Clause **3** above and in addition shall conform to the test requirements in the following table:

Table 2 Test Requirements

S. No.	Characteristics	Passing Standard	Test method
1	**2**	**3**	**4**
a)	Relative Density 20/20°C	0.945 to 0.953	Method No. 6 of IS 9591
b)	Refractive index, 20 °C Sodium D line	1.475 to 1.480	Methods No. 7 of IS 9591
c)	Acidity, Calculated as oleic acid, percent by mass	1.5 *Max*	Appx 'A'
d)	Ester content, calculated as Penta- Erythritol Di - oleate, percent by mass	96.0 to 103.0	Appx 'B'
e)	Lead and lead compounds, together calculated as Pb, percent by mass	0.03 *Max*	Appx 'D'
f)	Acetone insoluble matter	Nil	Appx 'C'

8. WARRANTY

The stores supplied against this specification shall be deemed to bear warranty for 12 months from the date of receipt of store at consignee's end and against defective design/ material/workmanship/performance. If during this period any of the stores supplied is found defective, the same shall be rectified/replaced by the contractor, free of charge, at the user's premises within a period of three months from date of intimation of defect.

9. PACKAGING

9.1 Penta-Erythritol Di-oleate, Lead Free, shall be supplied in sound, clean, dry and leak proof plastic container/glass bottle. After filling, the containers shall hold and agree quality and shall be securely closed.

9.2 Any other form of package shall have the prior approval of the Quality Assurance Officer/Quality Assurance Authority.

10. MARKING

10.1 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.

d) * Consignment No.

e) Lot/Batch No. and Date of Manufacture.

f) Gross and net mass.

g) * Consecutive number of packages and total number of packages in consignment.

h) Date of supply.

j) Manufacturer's initials or recognized trademark.

*****(Not applicable when the store is manufactured in Ordnance Factories).

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

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

10.4 Any paint used on the containers and the paint or other materials used for marking shall not contain lead or lead compounds, calculated as metallic lead, together exceeding 0.2 percent.

11. DEFENCE CATALOGUE NUMBER

Defence Catalogue Number for Penta Erythritol, Di-oleate Lead free will be included after allotment.

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* 7.4**)**

METHOD FOR DETERMINATION OF ACIDITY

PROCEDURE

Dissolve 10 g of the material in 50 ml of Ethyl Alcohol, nutralise to Phenolphthalein. Titrate the mixture with 0.1 N Sodium Hydroxide, using 0.5 ml of a 0.5 percent alcoholic solution of Phenolphthalein as indicator. Calculate the acidity thus found as Oleic acid $C_{17}H_{33}COOCH$ percent by mass of the sample.

1 ml 0.1N NaOH = 0.02825g $C_{17}H_{33}COOCH$

APPX 'B'
(*Clause* 7.4**)**

METHOD FOR DETERMINATION OF ESTER CONTENT

B-1. APPARATUS

a) Erlen-Meyer flask.

b) Reflux condenser.

c) Boiling water bath.

B-2. REAGENTS

a) 0.5 N Alcoholic Potash

b) 0.5 N Standard Hydrochloric acid

B-3. PROCEDURE

B-3.1 Weigh accurately between 2.5 g and 3.0 g of the material in a flask and add 50 ml of approximately 0.5 N alcoholic Potassium Hydroxide with the help of pipette. Boil for one hour under reflux condenser. Withdraw the flasks still carrying the condenser and immerse them in cold water. When cool, wash down the inside of each condenser with two 20 ml portions of distilled water. Disconnect the flask and wash each joint with a further 20 ml of water.

B-3.2 Carry out a blank determination at the same time and under the same condition.

B-3.3. Calculate the Ester content and express as Penta-Erythritol Di-oleate, using the following formula:

$$\text{Penta-Erythritol Di-oleate, = percent by mass} = \frac{16.63\,(Y - X)}{(C_{17}H_{33}COOCH_2)_2\,C(CH_2OH)_2 M}$$

where,

X = ml of 0.5 N Hydrochloric acid required for test;

Y = ml of 0.5 N Hydrochloric acid required by blank; and

M = Mass (in grams) of the sample taken for test.

NOTE - The distilled water shall be well boiled and cooled before use.

APPX 'C'
(*Clause* 7.4**)**

C. METHOD FOR DETERMINATION OF ACETONE INSOLUBLE MATTERS

PROCEDURE

Take 10 ml of the material in a test tube and add 10 ml of Acetone. Shake to mix and allow to stand for 10 minutes. No precipitate or appreciable opalescence shall be produced.

APPX 'D'
(*Clause* 7.4**)**

METHOD FOR DETERMINATION OF LEAD AND LEAD COMPOUNDS TOGETHER CALCULATED AS Pb

D-1. OUTLINE OF THE METHODS

Determination of Lead in Lead free Penta-Erythritol Di-oleate is carried out by treating the ash of the Penta-Erythritol Di-oleate with Nitric acid and then with Ammonium Acetate solution, and precipitating lead as Lead Sulphide and finally oxidizing to Lead Sulphate.

D-2. REAGENTS

D-2.1 Dilute Nitric Acid-lead-free, of strength approximately 4 N.

D-2.2 Ammonium Acetate solution-lead-free, 10 percent w/v.

D-2.3 Concentrated Sulphuric Acid-lead-free Sp. Gr. 1.84.

D-2.4 Dilute Sulphuric acid-2 percent, obtained by dissolving 11 ml of concentrated Sulphuric acid in one litre of water.

D-2.5 Ammonium Acetate-solid, lead-free.

D-2.6 Hydrogen Sulphide-gas, from Kipp's apparatus.

D-3 PROCEDURE

D-3.1 Weigh 4.5 g to 5.5 g of the material in a Silica basin and ash at low heat not exceeding 500°C until free form carbon, taking care to avoid loss of the light ash. Treat the ash so obtained with dilute Nitric acid. The quantity of acid is immaterial provided it is sufficient to extract the soluble matter, but avoid too great an excess since it has to be evaporated off. Allow the basin to stand on boiling water-bath for at least three hours. In case a large quantity of insoluble residue is left, heat the basin on the water - bath overnight. Decant off the supernatant liquid through a filter paper and extract the insoluble residue again on a boiling water-bath for one hour with dilute Nitric acid. Filter through the same filter paper and wash the residue thoroughly on the filter paper with hot water. Treat the residue on the filter paper with 10 ml of Ammonium Acetate solution. Filter and wash again. Mix the filtrate and washings in a 500 ml evaporating basin, add 2 ml of concentrated Sulphuric acid and evaporate the contents of the basin on sand-bath till fumes appear. Add 100 ml of water to the basin and allow to stand on the boiling water bath for 15 minutes. Then dilute the contents to about 150 ml and allow to stand overnight at room temperature. Filter the insoluble matter through whatman filter paper No. 42 (9 cm) and wash thoroughly with dilute Sulphuric acid. Transfer the filter paper and residue to a small beaker, cover with 20 ml of water and add 1 g to 2 g of Ammonium Acetate. Heat the beaker on the water-bath for not less than half an hour, stirring the contents occasionally. Decant the liquid through No. 42 Whatman filter paper (9 cm). Repeat the extraction with water and Ammonium Acetate.

Transfer all the insoluble matter including the filter pulp to the filter and wash thoroughly with warm water collecting the filtrate and washing in a 150 ml beaker. Pass Hydrogen Sulphide through the liquid for 10 minutes to 15 minutes and filter the precipitated Lead Sulphide at once through a Whatman filter paper No. 40 (9 cm) wash thoroughly but quickly with Hydrogen Sulphide water keeping the residue on the filter paper, if any, covered with liquid till washing is completed. Transfer the precipitate and filter paper to a tared silica crucible. Dry, carefully ignite to sulphate, cool and weigh.

D-3.2 Calculation

Calculate as Lead (Pb) and express the result as percentage of the material taken for test.

$$\% \text{ Pb} = \frac{0.6832 \times W_2}{W_1}$$

where,

W_2 = Mass of $PbSO_4$; and
W_1 = Mass of sample taken.