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भारत सरकार GOVERNMENT OF INDIA रक्षा मंत्रालय MINISTRY OF DEFENCE

संयुक्त सेवा विनिर्देश JOINT SERVICES SPECIFICATION

ON

BASIC LEAD STEARATE DCAN 6810-001 012

मनकी करण 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. ThisSecond Revision of the Joint Services Specification 6810-145 has been approved by Rear Admiral, Sanjay Misra,DGNAI, ViceChairman,Offg Chairman,Armament Standardisation Sub-Committee by circulation.

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

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1.	ADGWE/GS (WE-2/3), New Delhi
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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	Authority	Amended by	Signature
No.	Date	S.No./Para No./		Name & Appointment	&
		Column No.		(in block letters)	Date
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0. FOREWORD

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

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

0.3 This JSS 6810-145 : 2017 (Second Revision):

- a) was first prepared in the year 2008.
- b) was revised in the year 2013and supersedes the same.

0.4 This Joint Services Specification would be used for Manufacture, Supply and Quality Assurance of Basic Lead Stearate.

0.5 Quality Assurance Authority for the item covered in the specification is The Controller, Controllerate of Quality Assurance (Military Explosives), Aundh Road, Pune-411020. (email id <u>cqamear-dgqa@nic.in)</u>. Enquiries regarding the specification relating to any 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, ManakBhawan, 9, Bahadur Shah ZafarMarg, 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 supply and quality assurance of Basic Lead Stearate suitable for use in the manufacture of propellants.

2. RELATED SPECIFICATIONS/DOCUMENTS

References re made in this specification to:

Table 1 Related	Specifications
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S. No.	Specification No. & Year	Nomenclature
a)	IS 138 : 1992	Ready Mixed Paint, Marking, for Packages
	(Third Revision)	and Petrol Containers - Certification.
	Amd 1	
	Reaffirmed 2014	
b)	IS 460 (Part - I) : 1985	Specification for Test sieve, Part I Wire cloth
	(Third revision)	test sieves
	Amd 1	
	Reaffirmed 2013	
c)	JSG 0112 : 2015	General method of tests and assessment of
	(Second Revision)	impurities in chemical/material used in the
		manufacture of explosives and ammunition.

3. MATERIAL

The material shall consist essentially of Basic Lead Stearate, 2PbO.Pb $(C_{18}H_{35}O_2)_2$, in the form of fine, dry white powder free from foreign matter and any visible and mechanical impurities.

4. MANUFACTURE

The Basic Lead Stearate shall be manufactured by a process which will produce the product conforming to this specification.

5. TENDER SAMPLE

The manufacture/supplier shall submit two tender sample of each 250 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 as the tender.

6. PRE-INSPECTION OF STORES/CONSIGNMENT

6.1 The manufacturer/supplier/contractor 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 the inspection to the Quality Assurance Officer nominated under the terms of the contract. A declaration by the contractor that the 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 as required above has not been carried out, the consignment is liable for rejection.

7. QUALITY ASSURANCE

7.1 Inspection

The Basic Lead Stearate 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

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

No. of containers in a batch/lot	No. of containers to be samples
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

7.2.2 Samples of the material and of the packages may be taken from any portion of the consignment.

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

7.3 Criteria for Conformity

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

7.4 Test Requirements

The sample taken from any portion of the supply shall comply with clause **3.1** above and shall also conform to the following requirements:

S. No.	Characteristics	Passing Standard	Test Method
a)	Moisture content, percent by mass	0.5 <i>Max</i>	Appx 'A'
b)	Lead content, percent by mass	49.0 <i>Min</i>	Appx 'B'
		53.0 <i>Max</i>	

 Table 2 - Test Requirements of Basic Lead Stearate

S. No.	Characteristics	Passing Standard	Test Method
c)	Free stearic acid content, percent by	2.0 Max	Appx 'C'
	mass		
d)	Water soluble acids Calculated as	0.02 Max	Appx 'D'
	Acetic acid, percent by mass		
e)	Solubility in boiling Acetic acid	Completely	Appx 'E'
		Soluble	
f)*	Sieving requirement(Granulation)	All should	Appx 'F'
	Material passing through 37 µ m IS	Pass	
	sieve		
g)	Melting point of fatty acid in °C	52 Min	Appx 'G'
h)	Iodine Value of Fatty acid	5.0 <i>Max</i>	Method No 17 of
			JSG 0112

Table 2 Test Requirements of Basic Lead Stearate (Concluded)

* Details of IS sieve mentioned at S. No. 6 will be found in IS 460 (Part I).

NOTE -The material must also satisfy the end use requirements which will be assessed by carrying out suitable practical trials whenever so called for in the contract.

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

The Basic Lead Stearate shall be packed in a polythene bag made of 0.13 mm thick polythene film and capacity 50kg. This bag shall be hermetically sealed by double heat sealing and packed in a wooden case/box. Alternatively the material shall be packed in an internal polythene bag of 0.13 mm film thickness sealed hermetically and packed in external fibre cans or multi-walled paper sacks. In case of imports the packing shall be fit for overseas supply.

10. MARKING

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 package and total number of packages in consignment.
- h) Date of supply.
- j) Manufacturer's initials or recognized trademark.

(Not applicable for S. No. (d) & (g) 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.

11. DEFENCE STORES CATALOGUE NUMBER/NATO STOCK NUMBER

Defence Stores Catalogue Number for Basic Lead Stearate is 6810-001 012.

12. SAFETY OF OPERATIONS

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

13. SUGGESTIONS FOR IMPROVEMENT

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

The Director, Directorate of Standardisation, Ministry of Defence, 'H' Block, NirmanBhawan PO, New Delhi - 110011.

APPX 'A' (*Clause* 7.4)

DETERMINATION OF MOISTURE CONTENT

Transfer about 3.0 g of the material in to a previously dried and tared glass moisture dish (M_1) and weigh accurately (M_2) . Carefully remove the stopper and place the dish alongwith the stopper in an oven at 100°C to 105°C, for 3 hours. Remove from the oven and cool in a desiccator and weigh (M_3) :

Moisture content, percent by mass = $(M_2) - (M_3)$ $(M_2) - (M_1)$

APPX 'B' (*Clause* 7.4)

DETERMINATION OF LEAD CONTENT

Weigh accurately about 0.5 g of the sample (M_1). Transfer it to a 40 ml beaker, add 15 ml of glacial acetic acid and heat on a hot plate to dissolve the Lead Stearate. Then add 100ml to 150 ml of distilled water and cool under running water to crystallise out the stearic acid. Remove the separated stearic acid by filtering through a Whatman filter paper No 41. Collect the filtrate in a 40 ml beaker. Wash the residue on filter paper with a jet of cold distilled water. Heat the filtrate to boiling and precipitate Lead as Lead chromate by addition of boiling 1N solution of Potassium dichromate (49.035 g of Potassium dichromate dried at 130°C dissolved in distilled water and volume made to 1000 ml), until colour of the precipitate changes from lemon yellow to orange. Place the beaker on boiling water bath for one hour, then cover and keep overnight. Filter the precipitate through a previously dried and tared sintered glass crucible G4 (M_2) and wash with distilled water. Finally wash with 5 ml of 5% dilute acetic acid. Dry at 100 to 105°C to constant mass and weigh (M_3):

Lead content, percent by mass = $\frac{(M_3 - M_2) \times 0.6411 \times 100}{M_1}$

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APPX 'C' (*Clause* 7.4)

DETERMINATION OF FREE STEARIC ACID

Weigh accurately about 10g of sample (M_1) and place in a paper thimble. Extract the material with Ethyl Ether in soxhlet extractor for 3 hours. Evaporate the ether from the extraction flask (M_2) on a water bath. Dry the flask in an oven at about 150°C for one hour, cool in desiccator and weigh (M_3) :

Free stearic acid content, percent by mass = $(M_3) - (M_2)$ (M₁)

APPX 'D' (*Clause* 7.4)

DETERMINATION OF WATER SOLUBLE ACIDS

Transfer accurately about 10 g of the sample (M_1) in to an iodine flask. Add 100 ml of distilled water and shake for 15 minutes. Filter through Whatman No 54 filter paper. Wash the residue with a small portion of distilled water. Collect the filtrate and washings in a 500 ml conical flask. Add phenolphthalein indicator and titrate with N/10 Sodium hydroxide solution (Titre A). Run a blank determination side by side for the same quantity of distilled water (Titre B).

		(A - B) x F x 0.0060 x 100	
Water soluble acids	=		
(as CH ₃ COOH), percent by mass		\mathbf{M}_1	
(as CH_3 COOH), percent by mass	_	\mathbf{M}_1	

where,

F = Factor of N/10 Sodium hydroxide solutionused for titration.

APPX 'E' (*Clause* 7.4)

DETERMINATION OF SOLUBILITY IN BOILING ACETIC ACID

Take about one g sample in a test tube, add glacial acetic acid and heat to boiling. Observe the solubility. The whole of the material shall be soluble.

APPX 'F' (*Clause* 7.4)

SIEVING

Take accurately about 5.0 g sample in a 50 ml beaker. Add 400 ml of Acetone and stir vigorously. Pour the suspension into a 37 μ m IS sieve. Wash the beaker with a jet of Acetone in to the sieve till no more material is left in the beaker. Brush gently with a camel hair brush until no more material appears to pass through the sieve. Air dry the sieve with the material retained on it and brush off the material ion to a small, tared glass dish (M₁) and weigh (M₂).

Alternatively

Transfer 5 ml of Teepol solution into a 50 ml beaker, add 10 ml of distilled water into it. Weigh accurately about 10 g of the sample and transfer to the beaker containing Teepol solution so that material gets wetted and a paste is formed. Stir well with a glass rod to break all the lumps. Add about 250 ml of distilled water and stir well. Pour the mixture over a37 μ m IS sieve. Transfer all the contents of the beaker to the sieve with a jet of distilled water. Using camel hair brush wash the material on the sieve with distilled water till no more material passes through the sieve and is free from Teepol. Collect the retained portion of the sample in a 250 ml beaker using a jet of distilled water. Filter through a tared sintered glass crucible (M₁). Dry the crucible at 105°C for 2 hours. Cool in a desiccator and weigh (M₂):

Material retained on the sieve, percent by mass = $\begin{array}{ccc} M_2 & - & M_1 \\ ----- & x & 100 \\ M \end{array}$

where,

M = Mass of the material taken for the test.

APPX 'G' (*Clause* 7.4)

DETERMINATION OF MELTING POINT

G-1. SEPARATION OF FATTY ACID

G-1.1 Weigh about 2g of sample. Transfer it to a 400 ml beaker. Add 50 ml of glacial acetic acid and heat on a hot plate to dissolve the material completely. Then add 200 ml to 250 ml of distilled water and cool it under running water to crystallise out the stearic acid Filter through a Whatman No.41 filter paper and wash it thoroughly with distilled water. Wipe out any water drops remaining on the cake of fatty acid with a filter paper. Use this fatty acid for the determination of Melting point and Iodine value.

G-1.2 Grind a small portion of the above fatty acid. Take a thin walled capillary tube long enough to extend above the top of the bath, having an inside diameter of 1 mm and sealed at one end. Fill the tube with the dried, ground material to a depth of about 5 mm compacting the material 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. Suspend the thermometer in a heating bath. Start the stirrer 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 liquified. 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 degrees in the exposed mercury column;

T = Uncorrected melting point; and

T = Average temp of the exposed column as indicated by a second thermometer.