JSS 6810-80 : 2016
(Third Revision)

भारत सरकार
GOVERNMENT OF INDIA
रक्षा मंत्रालय
MINISTRY OF DEFENCE

संयुक्त सेवा विनिर्देश
JOINT SERVICES SPECIFICATION

ON

LEAD NITRATE (NORMAL)
DCAN 6810-000 952

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-110 011

LIST OF MEMBERS ASSOCIATED WITH FORMULATION OF THIS STANDARD

1. This Joint Services Specification has been approved by Shri RS Gauba, Sc 'G', Associate Director, PO-II, DRDO, Chairman, Armament Standardisation Sub-committee.

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

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

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 Service 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 6810-80 : 2016, (Third Revision):

a) was prepared in the year 1984.
b) was revised in the year 2003 & 2011 and supersedes the same.

0.4 This specification would be used for Manufacture, Supply and Quality Assurance of Lead Nitrate, Normal.

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, 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.

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 Inspection of Lead Nitrate (Normal).The material is suitable for use in the manufacture of Lead Styphnate.

2. RELATED SPECIFICATIONS/DOCUMENTS

Reference is made in this specification to:

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 460 (Part 1) : 1985 (Third Revision) AMD 1 Reaffirmed 2013	Specification for Test Sieves Part 1 Wire Cloth Test Sieves
c)	JSG 0112 : 2015 (Second Revision)	General Methods of Tests and Assessment of Impurities in Chemical/Materials used in the Manufacture of Explosives and Ammunition

3. MATERIAL/FINISH

Lead Nitrate, normal shall consist essentially of normal lead nitrate and should be in the form of colorless crystalline powder free from foreign matter, grit and visible impurities.

4. MANUFACTURE

Lead Nitrate (Normal) shall be manufactured by a process which will produce the product conforming to this specification.

5. TENDER SAMPLE

The manufacturer/supplier shall submit two tender samples each of 100 g essentially from the same batch/lot of the manufacture free of charges and conforming to this specification, to the Inspecting Officer/Inspection Authority as stated in the tender.

6. PRE-INSPECTION OF STORES/CONSIGNMENT

6.1 Manufactures/contractors must satisfy themselves that the stores are in accordance with the terms of contract and fully conform to the required specification, by carrying out a through 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

7.1.1 The Lead Nitrate Normal and the packages in which it is packed shall be subject to inspection by and to the approval of the Inspecting Officer/Inspection Authority.

7.1.2 Samples of the material and of the packages may be drawn from any portion of the batch/lot/consignment.

7.2 Sampling

A 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 shall be rejected.

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

7.4 Test Requirements

Samples taken fom any portion of the batch/lot/consignment of the material shall conform to **3** and shall also conform to the test requirements shown in the following table:

S. No.	*Characteristics*	*Passing Standard*	*Test Method*
a)	Volatile matter at 95ºC to 100ºC for two hours, percent by mass	1.25 *Max*	JSG 0112 Method 1
b)	Lead Nitrate content, percent by mass	98.00 *Min*	APPX ‘A’

S. No.	*Characteristics*	*Passing Standard*	*Test Method*
c)	Matter insoluble in Nitric acid, percent by mass 1) Total 2) Gritty matter retained on 63 micrometer IS Sieve	 0.1 *Max* Nil	APPX 'B'
d)	pH of water extract	4.8 *Max*	JSG 0112 Method 5 (b)
e)	Chlorides calculated as PbCl2, percent by mass	0.02 *Max*	APPX 'C'
f)	Copper compounds calculated as Cu, percent by mass	0.01 *Max*	APPX 'D'
g)	Iron compounds calculated as Fe, percent by mass	0.02 *Max*	APPX 'E'
h)	Silver and Mercury Compounds	Nil	APPX 'F'

8. WARRANTY

The stored supplied against the contract shall be deemed to bear warranted for 12 months from the date 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 users premises within a period of three months from date of intimation of defect.

9. PACKAGING

9.1 Lead Nitrate (normal) shall be supplied in double polyethylene bags each of film thickness 0.13 mm. The mouth of the bags shall be bunch tied, and the bags placed in hessian or poly woven bags. The quantity per package shall not exceed 50 kg. The packages shall be stored in a dry place.

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

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

10. MARKING

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

a) Nomenclature and Specification Number of the Material.

b) Name and Address of the Consignee.

c) AT or SO Number and Date.

d) Consignment Number.

e) Batch Number and Date of Manufacture.

f) Gross and Net mass.

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

h) Date of Supply.

j) Manufacturer's initials or recognized trademark.

10.2 In addition to the above the Quality Assurance Officer may suggest some more marking/identification suitable at the time of Inspection.

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

11. SAFETY OF OPERATIONS

Nothing in this specification shall relieve the supplier/user/manufacturer of his responsibility for the safety of his operations in handling, transit or storage of this store.

12. DEFENCE CATALOGUE NUMBER

The Defence Catalogue Number allotted to this store is 6810-000 952.

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, Nirman Bhawan PO,
New Delhi-110 011.

APPX 'A'
(*Clause* 7.4)

DETERMINATION OF LEAD NITRATE CONTENT

A-1. Transfer approximately 0.3 g of the sample, accurately weighed in a 250 ml beaker, and dissolve it in about 100 ml of distilled water (heat if necessary). Filter the insolubles if present, wash the filter paper thoroughly with distilled water. Add dilute acetic acid until it is distinctly acidic. Add 2 g of Sodium acetate or Ammonium acetate as buffer. Heat to boil, add from a pipette 4% Potassium chromate solution in slight excess (10 ml will usually suffice). Boil gently for 5-10 minutes (or until the precipitate settles). The supernatant liquid must be coloured slightly orange. Filter through a sintered glass crucible G4, wash thoroughly with hot dilute solution of Sodium acetate then with hot water and finally with alcohol till it is free from K_2CrO_4. Dry the crucible at 120°C to constant mass. Weigh as $PbCrO_4$.

A-2. Lead Nitrate content, percent by mass = $\frac{102.47 \times \text{mass of precipitate}}{\text{Mass of sample taken}}$

Alternative method

A-3. Transfer approximately 1.3 g of sample accurately weighed to a conical flask. Dissolve it in 100 ml of CO_2 free distilled water. Add 4 g of Ammonium acetate and titrate with N/10 EDTA solution using Methyl thymol blue as indicator, until the blue solution becomes pale yellow.

1 ml of N/10 EDTA = 0.03312 g of $Pb(NO_3)_2$

APPX 'B'
(*Clause* 7.4)

DETERMINATION OF MATTER INSOLUBLE IN NITRIC ACID

B-1. Total Insoluble Matter - 10 g of the sample is dissolved in 100 ml of 10% Nitric acid (mass/mass) heat if necessary, insolubles are filtered, washed with distilled water, dried, weighed and percent calculated.

Calculation

$$\text{Total insoluble Matter, percent by mass} = \frac{\text{Mass of insoluble matter}}{\text{Mass of sample taken}} \times 100$$

B-2. Grit Matter Retained on 63 micrometre IS Sieve - The insoluble matter from **B-1** above is boiled with 20 ml of Sodium hydroxide solution (50 g NaOH dissolved in 100 ml of distilled water) and is immediately diluted with 200 ml to 300 ml of distilled water. The insoluble matter (grit) is then washed three times with distilled water by decantation and then once with 2% Hydrochloric acid (mass/mass) and finally with distilled water and Acetone. The insoluble matter is dried and sieved through 63 micrometre IS Sieve and the portion retained on the sieve is weighed and the percentage is calculated.

Calculation

$$\text{Gritty matter retained on 63 micrometre IS Seive percent by mass} = \frac{\text{Mass of insoluble matter (Grit) retained on 63 micrometer IS Sieve}}{\text{Mass of sample taken}} \times 100$$

APPX 'C'
(*Clause* 7.4)

DETERMINATION OF CHLORIDES

C-1. Reagents

a) Nitric acid, dilute.
b) N/20 standard Silver nitrate solution.
c) *Standard Sodium Chloride Solution* - Dissolve 1.649 g of NaCl AR quality in water and make up the volume to 1000 ml. Pipette out 10 ml of the solution, dilute with water, make up the volume to 1000 ml. One millilitre of this solution is equivalent to 0.01 mg of chloride as (Cl) or 0.039 mg of $PbCl_2$.

C-2. Procedure - In a Nessler's tube take exactly 2 g sample. Add 20 ml distilled water, l ml concentrated HNO_3 and l ml of N/20 $AgNO_3$ solution. The resulting turbidity is then compared by adding standard Sodium chloride solution from burette in the blank till turbidity is matched.

Alternatively

C-3. Chlorides can be estimated in the Chloride Titrator.

Instrument - Standard Analytic Chloride Titrator

Reagents - Use chloride free distilled water for all solutions and rinsing the electrodes.

a) *Acid reagents* - Add 100 ml of glacial Acetic acid and 7.1 ml of 60% Nitric acid (mass/mass) to 500 ml water, mix thoroughly and dilute to 1 litre.

b) *Gelatin reagent* - Dissolve 6.2 g of Gelatin reagent (mixture of Gelatin, Thymol blue and Thymol in proportion of 60:1:1 by mass) in little hot water and neck 1 litre.

C-4. Procedure - Weight about 5 g sample accurately, dissolve in distilled water dilute to 100 ml in a standard volumetric flask. Take 1 ml of this solution in a titration vial, add 4 ml of acid reagent and 0.2 ml of gelatin reagent and titrate according to the instruction of the instrument. Record timer reading (R_2). Similarly titrate 0.1 ml of about 0.9% (C_1) Sodium chloride (AR quality) and record reading (R_1).

C-4.1 Titrate blank without sample, record timer reading (b).

C-5. Chlorides as $PbCl_2$, percent by mass $= \dfrac{C_1 (R_2 - b) \times 10 \times 4.759}{(R_1 - b) \times \text{mass of sample}}$

APPX 'D'
(*Clause* 7.4)

DETERMINATION OF COPPER COMPOUNDS

D-1. Dissolve 2 g sample in 25 ml of distilled water, add 10 ml Ammonium acetate solution, 2 ml glacial Acetic acid, 0.3 ml of Pyridine and 2 ml Ammonium thiocyanate. Then add 10 ml of chloroform and shake vigorously. Any blue or green color in the Chloroform shows the presence of Cu.

D-1.1 Compare the colour by carrying a blank determination using standard $CuSO_4$ solution.

Alternatively

D-2. Preparation of Standard Copper Solution (l ml = 0.01 mg Cu) - Dissolve 3.93 Cupric sulphate ($CuSO_4 . 5H_2O$), 50 ml Sulphuric acid (concentrate) in 1000 ml water.

D-2.1 Dilute 1 ml of the above solution (l ml = l mg Cu) to 100 ml with water immediately before use.

D-3. Dissolve 1 g sample in 20 ml water and extract by vigorously shaking with 10 ml of 0.01 percent mass/volume solution of Zinc dibenzyl dithiocarbamate (or any other suitable colorimetric reagent) in carbon tetrachloride. A yellow colour denotes presence of Cu. It is estimated by adding standard Copper solution (l ml = 0.01 mg Cu) from a dropping pipette to the reagents in place of sample till the colour matches.

Alternatively

D.4. Copper compounds can be estimated by any other standard colorimetric method.

APPX 'E'
(*Clause* 7.4)

DETERMINATION OF IRON COMPOUNDS

E-1. Preparation of Standard Iron Solution (l ml = 0.1 mg Fe) - Dissolve Ammonium ferric sulphate ($FeNH_4 (SO_4)_2$). 12 H_2O = 8.65 g and 50 ml concentrate Nitric acid in 1000 ml water.

E-1.1 Dilute 10 ml of this solution (l ml = l mg Fe) to 100 ml with water, immediately before use.

E-2. Method - Dissolve 2 g in 100 ml distilled water, precipitate the Lead with H_2S and filter. Wash the precipitate with distilled water saturated with H_2S. Make up the filtrate to 200 ml with distilled water. Take 100 ml of the filtrate add 2-3 drops concentrate H_2SO_4 evaporate to dryness. To the residue add 2 ml of concentrate HCl and 2 ml to 5 ml of water now slowly evaporate to dryness on the steam-bath. Take up the residue with 2 ml of 1:1 HCl and dilute to 50 ml in Nessler's tube. Add 30 mg of Ammonium persulphate and 3 ml of 10% Ammonium thiocyanate mix.

E-2.1 Carry out a blank determination similarly by using same quantities of reagents except the sample.

E-2.2 If the red colour produced in the sample is darker than the blank, then the standard iron solution is run from a burette till colour matches. Note the volume of standard iron solution added. Comparison of adding and unknown should be made immediately after adding the reagents as the colour fades on standing.

Alternatively

E-3. Iron Compounds can be Estimated as Follows - Dissolve 1 g in 5 ml distilled water, add 3 ml dilute Sulphuric acid and filter. Take 4 ml of the filtrate add 5 ml distilled water, l ml dilute Hydrochloric acid and one drop of N/10/$KMnO_4$ mix.

E-3.1 Add 5 ml Ammonium thiocyanate solution (approximately 7.5 M) and 10 ml of a mixture of equal volume of Amyl alcohol and Amyl acetate, shake vigorously and allow to separate.

E-3.2 Carry out of a blank determination by using same quantity of except the sample.

E-3.3 Compare the colour produced in the upper layer of the sample with that of the blank.

E-3.4 Run standard Iron solution from a burette in the blank (if colour produced in the sample is darker) till colour of the same matches with that of the sample. Note the volume of standard Iron solution added.

E-3.5 Iron compound as Fe, percent by mass = V x 0.02

where,

V = Volume of standard Iron solution added.

Standard Iron solution : same as **E-1**.

APPX 'F'
(*Clause* 7.4)

QUALITATIVE TEST OF SILVER AND MERCURY

Shake 1 ml of 10% sample solution in 10% (mass/mass) HNO_3 with 10 drops of 0.002% solution of dithiozone in chloroform. A yellow or orange yellow colour indicates presence of silver and or mercury.