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# भारत सरकार GOVERNMENT OF INDIA रक्षा मंत्रालय MINISTRY OF DEFENCE

# संयुक्त सेवा विनिर्देश JOINT SERVICES SPECIFICATION

**ON** 

LITHARGE (LEAD MONOXIDE) (DCAN 6810-001 022)

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

# <u>LIST OF MEMBERS ASSOCIATED WITH REVISION OF THIS STANDARD</u>

- 1. This Third Revision of the Joint Services Specification 6810-111 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
9.	CQA (Amn), Pune
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, New Delhi
16.	DG(ACE), Pune

# **RECORD OF AMENDMENTS**

Amendment		<b>Amendment pertains to</b>	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 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 6810-111 : 2017 (Third Revision).
  - a) was prepared in the year 2001.
  - b) was revised in the year, 2007 & 2013.
  - c) is revision of JSS 6810-111 : 2013 (Second Revision) and supersedes the same.
- **0.4** This JSS would be used to govern supply and quality assurance of Litharge (Lead monoxide).
- **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 <a href="mailto:cqamear-dgqa@nic.in">cqamear-dgqa@nic.in</a>). 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 supply and quality assurance of Litharge (lead monoxide) suitable for use in Ammunition sealing compositions and in the manufacture of Casting Powders  $A_{10}$  and  $A_{11}$  for Milan Missile.

## 2. RELATED SPECIFICATIONS/DOCUMENTS

References are made in this specification to:

**Table 1 Related Specifications/Documents** 

S. No.	Specification No. & Year	Nomenclature
a)	IS 460 (Part 1): 1985	Test Sieves
	(Third Revision)	Part 1 : Wire Cloth Test Sieves.
	Amd 1	
	Reaffirmed 2013	
b)	IS 12600 : 1989	Specification for Low Heat Portland Cement.
	Amd 5	
	Reaffirmed 2014	
c)	JSG 0112 : 2015	Methods of Tests and Assessment of Impurities in
	(Revision No.2)	Chemicals/Materials used in the Manufacture of
		Explosives and Ammunition.

## 3. MATERIAL

Litharge (Lead monoxide) shall be in the form of a fine yellow powder which shall be homogeneous and free from grit, foreign material and visible impurities.

#### 4. TENDER SAMPLE

The manufacturer/supplier/contractor shall submit a tender sample of 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 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 alongwith 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. 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.

## 7. QUALITY ASSURANCE

## 7.1 Inspection

- **7.1.1** Litharge (Lead Monoxide) and its constituents may be inspected during manufacture. After manufacture Litharge (Lead monoxide) and the packages in which it is contained shall be subject to inspection by and to the final approval of the Quality Assurance Officer/ Quality Assurance Authority.
- **7.1.2** The samples of the material and of the ingredients may be taken at any stage of manufacture. Samples of the finished material, and of the packages in which it is contained, may be taken from any portion of the batch/lot/consignment.

## 7.2 Sampling

Two representative samples each 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

## 7.3 Criteria for Conformity

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

<sup>\*</sup>Not applicable when the store is manufactured by Ordnance Factories.

## 7.4 Testing

The Samples taken from any portion of the batch/lot/consignment of material shall conform to clause 3 and in addition shall conform to the following test requirements:

**Table 2 : Test Requirements of Litharge (Lead Monoxide)** 

S.	Characteristic	Passing	Test Method
No.		Standard	
a)	Volatile matter at 100°, % by mass	1.0 <i>Max</i>	Method 1 (a) of
			JSG 0112
b)	Insoluble matter in Acetic acid, % by mass	1.0 <i>Max</i>	Appx 'A'
c )	Lead oxide content, calculated as lead	95.0 Min	Appx 'D'
	monoxide (PbO), % by mass		
d)	Carbonates, calculated as Lead carbonate,	3.0 <i>Max</i>	Appx 'E'
	% by mass		
e)	Relative density g/ml	8.9 <i>Min</i>	Appx 'B'
f)	Specific surface cm <sup>2</sup> /cm <sup>3</sup>	12000 Min	Appx 'C'
g)	Sieving requirement	All to pass	Method 18 of
		through 90	JSG 0112
		micrometre IS	
		Sieve	

**NOTE** - The details of IS Sieves referred to shall be found in IS 460(Part 1).

## 8. PACKAGING

- **8.1** Litharge (Lead Monoxide) must be supplied in sound, clean, dry, airtight moisture-proof packages.
- **8.2** Any other form of packages shall have the prior approval of the Quality Assurance Officer/Quality Assurance Authority.
- **8.3** The inclusion of any foreign matter or impurities in any of the packages shall render the whole batch/lot/consignment liable to rejection.

## 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.
  - 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 by Ordnance Factories.
- **9.2** In addition to the above, the Quality Assurance Officer may suggest some more markings/identifications suitable at the time of inspection.

## 10. SAFETY OF OPERATIONS

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 the store.

## 11. DEFENCE CATALOGUE NUMBER/NATO STOCK NUMBER

Defence Catalogue Number allotted to this store is 6810-001 022.

## 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'

## DETERMINATION OF INSOLUBLE MATTER IN ACETIC ACID

**A-1**. Mix 5 g of the sample with 5 ml of water and add 20 ml of 36% Acetic acid. Boil the mixture for several minutes. Cool the solution and filter through a G4 sintered glass crucible (M1). Wash the undissolved residue with dilute Acetic acid, dry at 110°C and weigh (M2).

## A-2. CALCULATION

Insoluble matter in Acetic acid % by mass 
$$=$$
  $(M2 - M1)$   $(M2 - M1)$   $(M3 -$ 

M1 = Mass of the empty crucible; and

M2 = Mass of the crucible + sintered residue.

APPX 'B'

## **DETERMINATION OF DENSITY**

**B-1**. Weigh accurately 20 g to 25 g of the sample into a tared 100 ml density bottle. Add a little n- Butanol and mix thoroughly to ensure complete wetting of the sample. Evacuate for 30 minutes to remove trapped air. Fill the density bottle with n-Butanol and stand in a water bath at  $20^{\circ}\text{C} \pm 0.05^{\circ}\text{C}$  for 30 minutes. Insert the ground glass stopper, remove from the bath, dry the outside rapidly with filter paper and weigh quickly. Determine the mass of the bottle when filled with distilled water and also when filled with n-Butanol at  $20^{\circ}\text{C} \pm 0.05^{\circ}\text{C}$  in each case.

## **B-2. CALCULATION**

Density of the sample of Lead oxide 
$$=$$
 -----  $g/ml$   $M3 + M5 - M4$  where,

M1 = mass in g of the density bottle;

M2 = mass in g of the density bottle filled with distilled water;

M3 = mass in g of the density bottle filled with n – Butanol;

M4 = mass in g of the density bottle containing the sample and filled with n- Butanol; and

M5 = mass in g of the sample.

APPX 'C'

## **DETERMINATION OF SPECIFIC SURFACE**

- **C-1** Determine the Specific surface by the method described in Appx 'A' of BS 12600 modified as indicated below:
- **C-2.** Transfer 24.0 g of the well mixed sample to the permeability cell in 4 to 5 increments, tapping the cell smartly on a firm wooden bench after each addition in order to consolidate the material.
- **C-3.** Insert the steel plunger and again tap several times, applying hand pressure if necessary until the collar of the plunger comes into contact with the top of the permeability cell. Remove the plunger carefully, as described in BS 12600, inspect the bed for cracks etc, then proceed as indicated in BS 12600.

## C-4. CALCULATION

Insert the value of E so found in place of the figure of 0.475 when calculating the specific surface by the formula given in BS 12600.

APPX 'D'

## LEAD OXIDE CONTENT

Dissolve 0.75 g of sample in 20 ml of Acetic acid. Filter the undissolved portion. Add 20 ml of 20 % Ammonium acetate solution to the filtrate. Dilute to 200 ml. Add 4 % Potassium dichromate ( $K_2$   $Cr_2$   $O_7$ ) solution in excess. Boil the solution for 15 minutes. Allow the precipitate of lead chromate to settle. Filter over a G4 sintered glass crucible. Wash with hot water and finally with Alcohol. Dry at 120 °C and weigh. Calculate the percentage of lead oxide from the mass of the lead chromate obtained:

 $1 \text{ g of PbCrO}_4 = 0.6905 \text{ g of PbO}.$ 

APPX 'E'

#### **CARBONATE CONTENT**

**E-1.** Weigh accurately about 10 g of sample (M) in the bottom of the flask of Schroetter's or Mohr's alkalimeter (See Annex 'A'). Place 1:1 Hydrochloric Acid and concentrated Sulphuric acid in the bulbs as indicated in figure and weigh (M1). Now allow the Hydrochloric acid to flow down on sample. Close the stopper. The gas evolved passes through concentrated Sulphuric acid which absorbs the moisture. Heat the apparatus on low flame till the contents begin to boil. Continue boiling for about three minutes. Aspirate  $CO_2$  free air through the apparatus to remove last traces of  $CO_2$ . Cool to room temperature and weigh (M2).

## E-2. CALCULATION

Carbonate as Lead carbonate	$(M1-M2) \times 6.0731 \times 100$
$(PbCO_3)$ , % by mass =	
	M

## ANNEX 'A'

