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क्रम सं० 11/50/09 /समन्वय मानकी
दिनांक ..... के अनुसार रक्षा
संयुक्त सेवा अनुरोधित ASSL
Ministry of Defence
Dated ..... dated .....

सहायक निदेशक (तक. सम.)
Asst. Director (T.C.)
मानकीकरण निदेशालय
Directorate of Standardisation
रक्षा मंत्रालय, नई दिल्ली
Ministry of Defence, New Delhi

रक्षा मंत्रालय
MINISTRY OF DEFENCE

De no: 6155-ME
Dt 10-08-21

संयुक्त सेवा स्पेसिफिकेशन
JOINT SERVICES SPECIFICATION

ON

LEAD THIOCYANATE

(DS Cat. No. 6810 - 000 649)
NSN 00 6810 000 19 0001

मानकीकरण निदेशालय
रक्षा उत्पादन विभाग
रक्षा मंत्रालय
'एच' ब्लाक, निर्माण भवन डाकघर
नई दिल्ली – 110 011

DIRECTORATE OF STANDARDISATION
DEPARTMENT OF DEFENCE PRODUCTION
MINISTRY OF DEFENCE
'H' BLOCK, NIRMAN BHAWAN PO
NEW DELHI - 110 011

TC-232

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क्रम सं० 11/50/09 /समन्वय मानकों
दिनांक ..... के अनुसार रक्षा
संयुक्त सेवा अनुरोध पत्र ASSL
Approved by Ministry of Defence
Vide letter no. .....
Coord. HQ dated 18/12/2015

सहायक निदेशक (तक. सम.)
Asst. Director (T.C.)
मानकीकरण निदेशालय
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TC.232

LIST OF MEMBERS ASSOCIATED WITH FORMULATION OF THIS STANDARD

1. This Joint Services Specification has been approved by Dr. DK Kharat, Sc 'G' Director, Tech (Armaments), TCG, DRDO, Chairman, Armament Standardisation Sub - Committee.

2. The following members have been present / consulted in preparing the document :-

Sl. No.	Name & Designation	Organisation
1.	Shri S.K Rastogi, Sc 'F' Additional Director	Dte of Armaments, DRDO Orgn, New Delhi
2.	Col SK Mohan	ADGWE / GS (WE - 2 / 3), New Delhi
3.	Col RN Nambiar	Dte of Arty (GS / Artillary - 5), New Delhi
4.	Shri B.P. Singh, DONA	Dte Gen of Naval Armt, Naval HQ, New Delhi
5.	Air Cmde R Kumar, PDA	Dte of Armt & Safety Eqpt, Air HQ, New Delhi
6.	Lt. Col K.S Hundal, JD, EME, Armt	DGEME, Army HQ, New Delhi
7.	Commander Shrinivas	DGNAI, Naval HQ, New Delhi
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9.	Dr. R.K Upadhyay, PScO	CQA (ME), Pune
10.	Col G.K Natu	CQA (Amn), Pune
11.	H Saphui, JAG (SG)	CQA (SA), Ichapur, West Bengal
12.	Col V.K Rana	CQA (W), Jabalpur
13.	Dr.[Mrs.] N. Sikder, Sc 'F'	HEMRL, DRDO, Pune
14.	Shri I.S Kalashetti, Sc 'F'	ARDE / DRDO Orgn, Pune
15.	Shri Rajeev Puri, Addl GM	Ammunition Factory, Pune
16.	Shri Satvinder Singh SSO - II	Secretary ASSC

RECORD OF AMENDMENTS

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

0. FOREWORD

0.1 This Joint Services Specification has been prepared by the Armament 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 specification shall be used to Guide Design, Manufacture, Quality Assurance and Procurement of Lead Thiocyanate.

0.4 (a) First revision was done in the year 1997.

(b) This specification is the revision of JSS 6810 - 47 : 2008 (Revision No.2) and supersedes the same.

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 JSS / JSG 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.

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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 JSS / 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 Lead thiocyanate. This composition is suitable for use in sensiting agent in cap composition of Small Arms Ammunition (SAA) and Gun Ammunition (GA). It is also used in the preparation of squib composition and QF Composition.

2. **RELATED SPECIFICATIONS / DOCUMENTS**

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 460 (Pt - I) : 1985 Reaffirmed 2008 AMD 1	Test Sieves : Part 1, Wire Cloth Test Sieves.
(c)	IS 695 : 1986 (Third Revision) Reaffirmed 2008	Specification for Acetic Acid.
(d)	IS 5670 : 1984 (First Revision) Reaffirmed 2011 AMD 1	Lead Thiocyanate for Explosive Pyrotechnic Compositions.
(e)	JSG 0112 : 1997 (Revision No.1)	General Methods of Tests And Assessment of Impurities in Chemicals / Materials used in the Manufacture of Explosives and Ammunition.

3. **MATERIAL / FINISH.** Lead thiocyanate shall consist of uniform crystalline powder white or light yellow in colour, free flowing and free from foreign matter and any visible impurities.

4. **MANUFACTURE**

4.1 Lead thiocyanate shall be manufactured by a process, which will produce the product conforming to this specification.

4.2 Nothing in this specification shall relieve the manufacturer of his responsibility for the safety of his operations.

5. **TENDER SAMPLE.** The contractor shall submit a tender sample of 250 g free of all charges, conforming to this specification when called for in the tender.

6. **PRE - INSPECTION OF THE STORES / CONSIGNMENT**

6.1 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.

6.2 If the Quality Assurance Officer finds that 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 Lead thiocyanate and the packages in which it is packed shall be subject to the inspection and approval of the Quality Assurance Officer / Quality Assurance Authority.

7.1.2 Samples of the material and the packages, in which it is packed, may be taken from any portion of the batch.

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

7.1.4 The foregoing provisions shall equally apply to the prime contractor and sub contractor, if any.

7.2 **Sampling.** Normally two representative samples each of 250 g supplied free of all charges, shall be drawn from each batch. However the number of samples to be drawn shall be at the discretion of the Quality Assurance Officer / Quality Assurance Authority.

7.3 **Test Requirements.** Samples taken from any portion of the supply shall comply with the clause 3 and in addition shall conform to the following requirements :-

Sl. No.	Test Characteristic	Passing Standard	Test Method
(a)	Moisture, % by mass	Max. 0.05	Method 1(b) of JSG 0112 (Desiccant) (Conc. H_2SO_4)
(b)	Matter insoluble in Ammonium acetate (20 % m/v) solution, % by mass	Max. 0.20	Appendix 'A'

Sl. No.	Test Characteristic	Passing Standard	Test Method
(c)	Grit, % by mass	Max. 0.05	Appendix 'B'
(d)	Chlorides as $PbCl_2$, % by mass	Max. 0.20	Appendix 'C'
(e)	Thiocyanate (Calculated as Lead thiocyanate after correcting for Sodium), % by mass	Min. 98.0	Appendix 'D'
(f)	pH of suspension in aqueous medium	Min. 4.5 Max. 5.5	Method 5 (b) of JSG 0112
(g)	Lead (Calculated as Lead thiocyanate after correcting for Chloride), % by mass	Min. 98.0	Appendix 'E'
(h)	Sieving Requirement		
	(i) For GA and SAA Cap Composition		Method 18 of JSG 0112
	(aa) Passing through 90 micrometre IS Sieve	All	
	(ab) Passing through 63 micrometre IS Sieve	Not less than 80 %	
	(ii) For Squib composition		-do-
	(aa) Passing through 53 micrometre IS Sieve	All	
	(iii) For QF composition		-do-
	(aa) Passing through 150 micrometre IS Sieve	All	
(i)	Bulk density g/ml	Min. 0.8 Max. 1.1	IS 5670

8. **WARRANTY.** The stores supplied against the contract shall be deemed to have been warranted against the defective material and performance by the contractor / manufacturer for a period of two year from the date of receipt of the stores at the consignee's end and if during this period any of the stores supplied is found defective, the same shall be replaced by the contractor / manufacturer free of charges at the consignee's premises.

9. PACKAGING

9.1 Lead thiocyanate shall be packed in in clear, sound, dry and air - tight containers of 500 g in amber coloured glass bottles or 500 g in polythene bags having film thickness 0.13 mm hermetically sealed and packed in paper containers.

9.2 The material packed in any other package shall receive a prior approval of the Quality Assurance Officer / Quality Assurance Authority.

9.3 The inclusion of any foreign matter/ impurities in any package shall render whole consignment liable for rejection.

10. MARKING

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

(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) 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) Manufacturer's Name, Initials or his Recognised Trade Mark.

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

10.3 The paint used for marking shall be of good quality and conforming to IS 138.

11. SAFETY OF OPERATIONS. Nothing in this specification shall relieve the manufacturer / contractor / user of his responsibility for the safety of his operations during manufacture, storage and transit or use of the store.

12. DEFENCE STORES CATALOGUE NUMBER

12.1 The Defence Stores Catalogue Number allotted to this store is 6810 - 000 649.

13. SUGGESTIONS FOR IMPROVEMENT

13.1 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. MATTER INSOLUBLE IN AMMONIUM ACETATE

A.1 Place 2 ± 0.01 g of the sample into a tared G4 sintered glass crucible. Wash the sintered glass crucible and contents with 200 ml of 20 % (m/v) Ammonium acetate solution at 35 °C to 40 °C, then with hot water and finally with 10 ml of ractified spirit. Dry the sintered glass crucible at 100 °C to 110 °C for 2 hours. Cool and weigh. Calculate the increase in mass of the sintered glass crucible as percentage of matter insoluble in Ammonium acetate.

Calculation.

$$\text{Matter insoluble in Ammonium acetate, \% by mass} = \frac{M_3 - M_1}{M_2 - M_1} \times 100$$

Where,

M_1 = Mass of tared G4 sintered glass crucible

M_2 = Mass of tared G4 crucible with sample under test

M_3 = Mass of G4 crucible with residue remaining after Ammonium acetate wash.

APPENDIX 'B'

B. GRIT

B.1 Brush the contents of the sintered glass crucible after the above determination into a small beaker and treat with aqua regia. Boil for 5 minutes on a hot plate Filter again through the sintered glass crucible taking care to quantitatively transfer all the contents of the beaker into the crucible. Wash and dry in an electric oven at 105 °C ± 5 °C to constant mass. Cool in a desiccator and weigh. Calculate as follows :-

Calculation.

$$\text{Grit, \% by mass} = \frac{M_4 - M_1}{M_2 - M_1} \times 100$$

Where,

M_4 = Mass of G4 crucible + matter insoluble in aqua - regia

M_2 = Mass of tared G4 crucible with sample.

M_1 = Mass of tared G4 crucible

APPENDIX 'C'

C. ESTIMATION OF CHLORIDES

C.1 Cover 5.0 ± 0.1 g of sample with 100 ml of distilled water and allow standing for one hour with occasional stirring. Filter and wash twice by decantation. To the combined filtrate and washings, add 10 ml of Bismuth nitrate reagent stir well and stand for 15 minutes. Carry out a black determination. If sample under test indicates precipitate when seen visually, filter the precipitate on a Whatman No. 42 filter paper. Wash the precipitate with water and transfer the precipitate by means of 10 ml of 0.1 N Nitric acids to a conical flask. Then, add about 20 ml of 0.05 Silver nitrate solution, add 5 ml of concentrated Nitric acid. Boil for 2 to 3 minutes to ensure full coagulation of Silver chloride and then titrate with 0.05 N Potassium thiocyanate / Ammonium thiocyanate, using 5 ml of 10 % Ferric alum solution as an internal indicator. Rur blank using same quantities of all the reagents.

Calculation.

$$\text{Chlorices as } PbCl_2 \text{ \% by mass} = \frac{(X-Y) \times f \times 0.00695}{\text{Mass of sample}} \times 100$$

Where

X = Volume in ml of standard Thiocyanate solution required for blank.

Y = Volume in ml of standard Thiocyanate solution required for sample.

f = Factor of 0.05 N of Thiocyanate solution.

APPENDIX 'D'

D. DETERMINATION OF THIOCYANATE (AS LEAD THIOCYANATE)

D.1 **Reagents.**

(a) Dilute Nitric acid - one % (v/v)

(b) Standard Silver nitrate solution - 0.1 N

(c) Ferric alum indicator - saturated solution

(d) Standard Potassium thiocyanate solution - 0.1 N

D.2 **Procedure.**

D.2.1 Weigh about 0.7 g of the prepared sample accurately. Dissolve it in about 150 ml of dilute Nitric acid. Solution shall be affected by very gentle warming at about 50 °C. (Hot water decomposes lead thiocyanate and hence a higher temperature should be avoided).

D.2.2 Add 50 ml of standard Silver nitrate solution from a pipette. Then add 2 ml of ferric alum indicator. Determine the excess silver nitrate by titrating with standard potassium thiocyanate solution. The end point is reached when the first permanent appearance of a faint reddish colouration due to the formation of ferric thiocyanate complex is observed. Carry out a blank determination at the same time.

Calculation.

$$\text{Lead thiocyanate, \% by mass} = \frac{(X-Y) \times f \times 1.617}{M} (2.0 \times P + 1..6 \times L)$$

Where

X = Volume in ml of Potassium thiocyanate solution required for the blank.

Y = Volume in ml of Potassium thiocyanate required for the sample

f = Factor of Potassium thiocyanate solution

P = Percentage of Sodium thiocyanate (See clause D.3)

L = Percentage of Lead chloride

M = Mass in g of the material taken for the test

D.3 **Determination of Sodium Thiocyanate.** For applying the correction for Sodium thiocyanate, determine the percentage of Sodium thiocyanate as in D.3.1 to D.4.

D.4 **Magnesium Uranyl Acetate Reagent.** Prepare the reagent by mixing the following :-

Solution A.

Crystallized uranyl acetate	:	85 g
Glacial acetic acid (to IS 695)	:	60 g
Water	:	1000 ml

Solution B.

Crystallized magnesium acetate	:	500 g
Glacial acetic acid (IS 695)	:	60 g
Water	:	1000 ml

Heat the solution A and B separately to about 70 °C and maintain at this temperature until the ingredients have dissolved. Mix the two solutions and cool to 20 °C place the vessel containing the mixed reagent in water at 20°C and keep at this temperature for 2 hours.

D.3.2 **Wash Liquid.** Shake one milli litre of 1 per cent Sodium chloride solution with 25 ml of magnesium uranyl acetate reagent. Wash the resulting precipitate thoroughly with 95 percent alcohols and then suspend in 1000 ml of 95 % alcohol. After standing for an hour with frequent shaking, filter the mixture and use the filtrate as wash liquid.

D3.3 **Procedure.** Take accurately weighed 5 g of the sample in a 300 ml beaker. Add 100 ml of water and then 10 ml concentrated Nitric acid. The reaction between thiocyanate and nitric acid, once begun is extremely vigorous and shall be carried out under a hood.

After the vigorous reaction has subsided, evaporate the solution to one half of its original volume and filter. Wash the residue, the beaker and the filter paper with about 50 ml of distilled water. In a 150 ml beaker, carefully evaporate the filtrate and washings nearly to dryness. Dissolve this residue in 25 ml of water and netralise with Ammonium hydroxide. Warm to hasten solution of soluble salts and filter into a 100 ml beaker using small amount of water for washing. Evaporate to a volume of 5 ml or less. Add 25 ml of Magnesium uranyl acetate reagent to the beaker and place in 1000 ml beaker containing water to a depth of 10 mm to maintain a temperature of 20°C. Stir the contents of the small beaker for 30 minutes at fairly rapid rate with a mechanical stirrer. Transfer the precipitate of Sodium magnesium uranyl acetate to a tared sintered glass crucible No. G4 using a total of 25 to 30 ml of the wash liquid for transferring and washing. Dry the washed precipitate and crucible in an oven at 100 °C to 110 °C. Cool in a desiccator and weigh. Carry out a blank determination at the same time.

Calculation.

$$\text{Sodium thiocyanate, \% by mass} = \frac{5.4 \times (A - B)}{M}$$

Where

A = Mass in g of Sodium magnesium uranyl acetate obtained from the sample.

B = Mass in g of the Sodium magnesium uranyl acetate from the blank.

M = Mass in g of the material taken for the test.

D3.4 Flame photometer may be used alternatively for the determination of Sodium wherever such facilities exist.

APPENDIX 'E'

E. DETERMINATION OF LEAD
(As Lead thiocyanate corrected for chloride as Lead chloride)

E.1 Weigh about 0.5 g of Lead thiocyanate accurately into a 400 ml beaker and add 25 ml of water followed by 3 ml of concentrated Nitric acid and cover with a watch glass. Warm carefully and when the reaction ceases, add 10 ml of 1 : 1 Sulphuric acid and evaporate Sulphur trioxide fumes. Cool and cautiously add 75 ml of water and warm to dissolve soluble salts. Add 75 ml of cold water and allow standing for 30 minutes. Filter the lead sulphate on a weighed filtering crucible. Wash 5 times with 3 % sulphuric acid and then 5 times with 50 % alcohol. Dry the crucible at 105 °C to 110 °C for about 1/2 hour. Heat in an air bath for about 20 minutes. Cool in desiccators and weigh.

Calculation.

$$\text{Lead thiocyanate, \% by mass} = \frac{106.6\ A}{M} - (1.16 \times L)$$

Where,

A = Mass of Lead Sulphate.

M = Mass of Sample.

L = % Lead Chloride.