



**GOVERNMENT OF INDIA
MINISTRY OF DEFENCE**

JOINT SERVICES SPECIFICATION

ON

CALCIUM SILICIDE

(DS Cat Nos.)

- (a) Calcium Silicide, Size 150 micrometre 6810-000 930**
- (b) Calcium Silicide, Size 75 micrometre 6810-000 931**
- (c) Calcium Silicide, Size 63 micrometre 6810-000 932**

**JSS 6810-107: 2007
(Revision No. 2)**

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

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LIST OF MEMBER ASSOCIATED WITH PREPARATION OF THIS DOCUMENT

1. This Joint Services Specification has been approved by Dr. BR Gandhe, Director, Directorate of Armaments (R&D), Chairman Armament Standardisation Sub Committee by circulation.

2. The following members were present/consulted in approving the document: -

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17.	Lt Col Sanjay Singh	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)	

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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 The specification JSS 6810-107: 1994 (Revision No. 1) was issued in Mar 1994. The present document JSS 6810-107: 2007 (Revision No. 2) is revision of JSS 6810-107: 1994 (Revision No. 1) and supersedes the same.

0.4 This specification would be used for supply and quality assurance of Calcium Silicide sizes 150 micrometre, 75 micrometre and 63 micrometre.

0.5 Quality Assurance Authority for the item covered in this specification is Controllerate of Quality Assurance (Military Explosives), Aundh Road, Kirkee, Pune - 411 020. Enquiries regarding this specification relating to any contractual conditions should be addressed to the Quality Assurance Authority named in the tender or contract. Other enquiries should be referred to: -

The Director,
Directorate of Standardisation,
Ministry of Defence,
'H' Block, Nirman Bhawan PO,
New Delhi 110 011.

0.6 Copies of this specification can be obtained on payment from: -

The Director,
Directorate of Standardisation
Ministry of Defence
Standardisation Document Centre
Room No -5, 'J' Block, Nirman Bhawan PO
New Delhi - 110 011

0.7 This specification holds good only for the supply order for which it is issued.

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1. SCOPE

1.1 This specification is meant to govern supply and quality assurance of Calcium Silicide sizes 150 micrometre, 75 micrometre and 63 micrometre suitable for use in Explosives, Pyrotechnic and smoke compositions.

2. RELATED SPECIFICATIONS

2.1 Reference is made in this specification to: -

- (i) IS 138: 1981 - Ready Mixed Paint, Marking, for Packages and Petrol Containers – Specification (Third Revision)
Reaffirmed 2004 Amds 1
- (ii) IS 460 (Part 1): 1985 - Specification for test sieves: Part I Wire cloth test sieves
(Third Revision)
Reaffirmed 2004
- (iii) JSG 0112: 1997 (Revision No. 1) - General Methods of test and assessment of impurities in Chemicals/ Materials used in the manufacture of Explosives and Ammunition

2.2 Copies of Indian Standards are obtainable on payment from :-

Bureau of Indian Standards
Manak Bhawan,
9 Bahadur Shah Zafar Marg,
New Delhi - 110 002

or

their regional/branch offices.

2.3 Copies of Joint Services Guide are obtainable on payment from: -

The Director
Directorate of Standardisation,
Ministry of Defence,
'H' Block, Nirman Bhawan PO,
New Delhi - 110 011.

3. DESCRIPTION

3.1 Calcium Silicide shall be in the form of a fine powder which shall comply with the appropriate sieving requirement given in clause 6. It shall be free from grit and visible impurities.

4. MANUFACTURE

4.1 Calcium Silicide shall be manufactured by a process which will produce the product conforming to this specification.

5. TENDER SAMPLE

5.1 The manufacturer/supplier/contractor shall submit a tender sample of 500 g essentially from the same batch/lot of manufacture free of all charges and conforming to this specification, to the Quality Assurance Authority/Quality Assurance Officer as stated in the contract.

6. PRE-INSPECTION OF 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 Calcium Silicide and the packages in which it is packed shall be subject to inspection by and to the approval of the Quality Assurance Officer/Quality Assurance Authority.

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

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7.2 Sampling

7.2.1 Two representative samples 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

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

7.4 Test Requirements

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

Table: TEST REQUIREMENTS OF CALCIUM SILICIDE

Sl.No.	Characteristics	Passing Standard	Test method
1	2	3	4
(a)	Moisture (at 105°C ± 2deg C for 3 h)		Method 1 of JSG 0112
	(i) Size 150 micrometre, percent by mass Max	0.1	
	(ii) Size 75 micrometre, per cent by mass Max	0.2	
	(iii) Size 63 micrometre, per cent by mass Max	0.2	

(b)	Silicon, per cent by mass	Min	60.0	Appendix `A'
(c)	Iron			
	(i) Total Iron and Iron compounds calculated as the metal, per cent by mass	Max	10.0	Appendix `B'
	(ii) Free metallic Iron calculated as the metal, per cent by mass	Max	0.2	Appendix `D'
(d)	Calcium, per cent by mass	Min	20.0	Appendix `C'
(e)	Total Silicon, Calcium and Iron, per cent by mass	Min	92.0	Appendix `F'
(f)	Alkalinity calculated as Calcium oxide (CaO), per cent by mass	Max	2.0	Appendix `E'
(g)	Carbides and Phosphides		To pass test	Appendix `F'
(h)	Free carbon, per cent by mass	Max	3.0	Appendix `G'
(j)	Apparent density	Min	1.20	Appendix `H'
		Max	1.50	
(k)	Sieving requirement			Method 18 of JSG 0112
	(i) Size 150 micrometre, Retained on 150 micrometre IS Sieve		Nil	
	(ii) Size 75 micrometre, Retained on 125 micrometre IS Sieve,		Nil	
	Retained on 75 micrometre IS Sieve per cent by mass	Max	2.0	
	(iii) Size 63 micrometre			
a) Retained on 150 micrometre IS Sieve,		Nil		
b) Retained on 63 micrometre IS Sieve, per cent by mass	Max	35		

Note:- Particulars of Sieves referred to shall be found in IS 460(Part 1).

8. WARRANTY

8.1 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 The Calcium Silicide shall be supplied in sound, clean and dry, hermetically sealed iron drums or other approved packages containing an approved quantity. The packages constituting a consignment shall comply in every respect with current regulations for transport of dangerous goods.

9.2 The inclusion of any foreign matter or impurities in any of the packages will render the whole consignment liable to 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) Lot/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) Contractor's initial or recognised trade mark.

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. SAFETY OF OPERATIONS

11.1 Nothing in this specification shall relieve the contractor of his responsibility for the safety of his operations during manufacture, handling and transit of this store.

12. DEFENCE STORES CATALOGUE NUMBERS

12.1 Defence Stores Catalogue Numbers allotted to this store are: -

- (i) Calcium Silicide, 6810-000 930
Size 150 micrometre
- (ii) Calcium Silicide, 6810-000 931
Size 75 micrometre
- (iii) Calcium Silicide, 6810-000 932
Size 63 micrometre

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.

DETERMINATION OF TOTAL SILICON

1. 0.5 g of the sample is fused in a Nickel crucible with 8 g of Sodium Carbonate containing 1 percent of Potassium Nitrate. After cooling, the melt is extracted with water and any fused residue is submitted to a further fusion with caustic soda. After cooling, the product is dissolved in water and added to the solution previously obtained.

2. The solution is acidified with Hydrochloric acid, evaporated to dryness, and the residue baked at 120 °C for two hours. It is then moistened with 5 ml concentrated Hydrochloric acid, 100 ml of cold distilled water added and warmed on a boiling water bath for 15 minutes. The liquid is then filtered, the residue of Silica being transferred completely to the paper, washed with warm water and dried. The filtrate is evaporated to dryness, and the solid residue baked at 120 °C and extracted as before. After filtration, (reserve the filtrate for determination of Iron) the second residue of Silica is washed with cold water, dried and ignited together with the first residue at a bright red heat. After weighing, the Silica is volatilised by treatment with Hydrofluoric acid and Sulphuric acid and the loss of mass (due to SiO₂) is calculated as percentage of Silicon on the sample.

2.1. Calculation

$$\% \text{ Silicon} = \frac{\text{Loss in mass due to SiO}_2 \times 0.46744 \times 100}{\text{Mass of the sample taken}}$$

APPENDIX B'

DETERMINATION OF IRON

1. The filtrate reserved from Appendix `A' is acidified with a few drops of Nitric acid, boiled, and Ammonia is added to precipitate Iron. After standing on the water-bath to coagulate the precipitate, it is filtered hot through a fluted No. 54 Whatman filter paper and the filtrate laid aside for the determination of Calcium. The Iron is dissolved from the filter paper with a few drops of concentrated Hydrochloric acid and re-precipitated. This is again filtered and the filtrate is combined with the previous one. The Iron precipitate is ashed in a porcelain crucible and weighed as Fe₂ O and calculated as percentage of Iron.

1.1 Calculation

$$\% \text{ Iron} = \frac{\text{Mass of ash} \times 0.7 \times 100}{\text{Mass of the sample taken}}$$

DETERMINATION OF CALCIUM

1. The filtrate reserved from `Appendix B' is acidified with glacial Acetic acid, brought to boil and the Calcium is precipitated with Ammonium oxalate. After standing overnight, it is filtered into a Gooch crucible, washed free from oxalate, dissolved in dilute Sulphuric acid, heated to 80 °C and titrated against N/10 Potassium permanganate.

1.1 Calculation

$$\% \text{ Calcium} = \frac{\text{Titre} \times \text{factor of KMnO}_4 \times 0.002005 \times 100}{\text{Mass of the sample taken}}$$

APPENDIX 'D'

DETERMINATION OF METALLIC IRON

1. 30 ml of an aqueous solution of Copper Sulphate (25 g of crystals per litre) are placed in a flask fitted with a stopper carrying gas inlet and exit tubes. A stream of Carbon dioxide is passed through the flask and is continued throughout the subsequent operations. After the air has been displaced from the flask, 2 g of the finely divided sample are rapidly introduced, the flask is stoppered and the solution is boiled for exactly 10 minutes. The solution is then allowed to cool and filtered rapidly with the aid of suction. The filtrate is transferred to a flask and 10 ml of dilute Sulphuric acid is added. A stream of Carbon dioxide is passed through the flask and the solution is titrated against N/10 Potassium permanganate solution.

1.1 Calculation

1 1ml of N/10 Potassium permanganate solution = 0.0056 g of metallic Iron

$$\% \text{ Metallic Iron} = \frac{\text{ml of KMnO}_4 \times 0.0056 \times \text{factor of KMnO}_4 \times 100}{\text{Mass of the sample taken}}$$

2. The presence of metallic Iron may be confirmed by treatment of the finely divided material with a magnet.

DETERMINATION OF ALKALINITY

1. 2 g of the finely powdered sample are treated with 200 ml of water, containing 0.5 g of Ammonium Nitrate, for 2 hours at a temperature of 20 °C. The solution is filtered and 100

ml of the filtrate are titrated against N/10 acid, Methyl orange being used as indicator, and calculated as Calcium Oxide (CaO).

1.1. Calculation

1 ml of N/10 acid = 0.0028 g CaO

% Alkalinity as CaO = $\frac{\text{Titre} \times 0.0028 \times \text{factor of N/10 acid} \times 100 \times 2}{\text{Mass of the sample taken}}$

APPENDIX `F'

DETERMINATION OF CARBIDES AND PHOSPHIDES

1. 10 g of the sample are heated in a boiling waterbath with 50 ml of distilled water for two hours with constant agitation while a stream of Nitrogen is passed through the mixture. The ensuing Nitrogen is passed first through a small reflux condenser to remove excess water vapour, then through an absorption tube packed successively with (a) cotton wool impregnated with lead acetate, and (b) moist Potassium hydrogen sulphate dispersed on asbestos fibre and finally into a series of gas absorption vessels each containing 25 ml of decinormal Silver Nitrate.
2. At the end of the two hours the Silver Nitrate solution is filtered and the combined filtrate is titrated to turbidity point against decinormal Caustic soda solution.
3. If more than 2.0 ml of 0.1 N Sodium Hydroxide is required to a 10 g sample, the specimen fails to meet the specification requirements.

DETERMINATION OF FREE CARBON

1. About 0.5 g of the sample is weighed in a porcelain boat and introduced into a combustion tube. Air is displaced by a rapid stream of dry chlorine in the cold. Heat is then applied gently until Calcium Silicide ignites. When the combustion is complete as indicated by the material ceasing to glow, the tube is allowed to cool and the boat is withdrawn. The residue in the boat is extracted with dilute Hydrochloric acid to remove Calcium and iron compounds and the insoluble matter consisting of Carbon, Silicon Silica and possibly Carborundum, is dried in the boiling water oven and weighed. It is then gently ignited at a low red/heat to remove Carbon, and weighed again. The difference is recorded as mass of free carbon.

APPENDIX `H'

DETERMINATION OF APPARENT DENSITY

1. 20 g of the sample are placed in a glass cylinder graduated in half ml, the cylinder being approximately 15 cm high 2 cm in diameter. The cylinder is dropped vertically from a height of 6 cm on to a piece of hard leather thirty times. The surface of the column of powder is then leveled off by the minimum amount of side tapping and the total volume is read.
2. The above procedure is conveniently arranged by sliding the cylinder through two wooden filter-stand rings, clamped one above the other on the same support, the lower ring being so arranged as to limit the travel of the cylinder to 6 cm.