



रक्षा मंत्रालय
MINISTRY OF DEFENCE

संयुक्त सेवा विनिर्देश
JOINT SERVICES SPECIFICATION

ON

CARBAMITE
(DS Cat. No. 6810 - 000 940)

मानकीकरण निदेशालय
रक्षा उत्पादन विभाग
रक्षा मंत्रालय
‘एच’ - ब्लॉक, निर्माण भवन डाकघर
नई दिल्ली - 110 011

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DEPARTMENT OF DEFENCE PRODUCTION
MINISTRY OF DEFENCE
‘H’ - BLOCK, NIRMAN BHAWAN PO
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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 the 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 (a) First revision was done in the year 1999.

(b) This specification is a revision of JSS 6810 - 125 : 2008 (Revision No. 2) and supersedes the same.

0.4 This specification is meant to govern Manufacture, Supply and Quality Assurance of Carbamite.

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 / 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.

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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 Quality Assurance of Carbamite suitable for use as stabiliser and plasticiser in propellant explosives.

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) AMD 1 Reaffirmed 2009	Ready Mixed Paint, Marking, for Packages and Petrol Containers - Specification.
(b)	IS 460 (Part 1) : 1985 (Third Revision) AMD 1 Reaffirmed 2008	Specification for Test Sieves : Part 1, Wire Cloth Test Sieves.
(c)	JSG 0112 : 1997 (Revision No. 1)	General Methods of Tests and Assessment of Impurities in Chemicals / Materials used in the Manufacture of Explosives end Ammunition.

3. **MATERIAL**

3.1 The material shall consist essentially of Diethyl diphenyl Urea, $\text{CO}(\text{N C}_2\text{H}_5\text{C}_6\text{H}_5)_2$, and shall not be darker than pale buff in colour. It may be supplied in the form of lumps, powder or flakes as specified in the contract. It shall be free from grit, visible impurities and foreign matter.

3.2 The material may be dyed if required by the contract.

4. **MANUFACTURE.** Carbamite shall be manufactured by a process which will produce the product conforming to this specification.

5. **TENDER SAMPLE.** The manufacturer / supplier / contractor shall submit a tender sample of 250 g essentially from the same batch / lot of manufacture, free of all charges and conforming to this specification, when called for in the tender.

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 the pre - inspection of the consignment as required above had not been carried out, the consignment is liable for rejection.

7. QUALITY ASSURANCE

7.1 INSPECTION. The Carbamite and packages in which it is contained shall be subject to inspection by and to the approval of the Quality Assurance Officer / Quality Assurance Authority.

7.2 SAMPLING. A representative sample 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 during examination, any sample is found not to conform to this specification, the whole batch / lot / consignment 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 from any portion of batch / lot / consignment shall conform to clause 3 and in addition shall conform to the following test requirements :

Test Requirements of Carbanite

Sl. No.	Characteristic	Passing Standard	Test Method
(a)	Melting Test	Bright clear liquid free from scum and deposit.	Appendix 'A'
(b)	Setting, point	Min. 71.7 °C Max. 72.5 °C	Appendix 'A'
(c)	Volatile matter, at 60 °C for 2 hours, % by mass	Max. 0.1	JSG 0112 Method 1 (a)
(d)	Ash, % by mass	Max. 0.1	JSG 0112 Method 2 (a)
(e)	Primary amines calculated as Aniline, % by mass	Max. 0.03	Appendix 'B'
(f)	Secondary amines calculated as Ethyl aniline, % by mass	Max. 0.10	Appendix 'B'
(g)	Tertiary amines, calculated as Diethyl aniline, % by mass	Max. 0.05	Appendix 'C'
(h)	Acidity calculated as Hydrochloric acid, % by mass	Max. 0.004	JSG 0112 Method 5 (a)
(j)	Hydrolysable chlorine compounds calculated as Chlorine, % by mass	Max. 0.01	Appendix 'D'
(k)	Size requirements :		JSG 0112 Method No. 18
	(i) Powder	All to pass (600 micrometer IS Sieve)	
	(ii) Flakes	All to pass 6.3 mm IS Sieve and thickness of the flakes shall be 0.63 mm Max.	

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Note. The Indian Standard (IS) Sieve (s) mentioned above are the fine mesh normal test sieve (s) governed by Indian Standard Specification No. 460 of latest issue respectively.

8. WARRANTY. The stores supplied against the contract shall be deemed to be warranted against the defective material and performance by the contractor for a period of six months from the date of receipt of the stores at the consignee's end and shall retain the properties described above. If during this period any of the stores supplied is found defective, the same shall be replaced by the manufacturer / supplier / contractor free of charges at the consignee's premises.

9. PACKAGING

9.1 The carbamite, shall be supplied in sound, clean, and dry wooden cases, lined with Paper Kraft, paraffin waxed to approved specification or other approved packages, containing an approved quantity.

9.2 The inclusion of any foreign matter or impurities in any of the packages shall 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 :

- (a) Nomenclature and Specification Number of the Material.
- (b) Name and Address of the Consignee.
- (c) A.T. or S.O. Number and Date.
- (d) Consignment Number.
- (e) Batch No. and Date of Manufacture.
- (f) Gross and Net Mass.
- (g) Consecutive Number of Package 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 markings / identifications 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. DEFENCE STORES CATALOGUE NUMBER

11.1 The Defence Stores Catalogue Number allotted to this store is 6810 - 000 940.

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

13. SUGGESTIONS FOR IMPROVEMENT

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

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

APPENDIX 'A'

A. SETTING POINT

A.1 Fill a test tube 2.5 cm x 15 cm to within 4 cm of the top with the melted carbamite and fit it with a wire stirrer and a cork carrying a thermometer so arranged that its bulb is situated centrally in the tube and its lower extremity is 2.5 cm from the bottom. Support the tube centrally by means of a cork in a larger test tube 4 cm x 18 cm and support the latter inside a litre beaker by means of the cover of the beaker. Fill the beaker to within 2.5 cm of the top, with water at 60 °C to 65 °C. Stir the melt vigorously as the temperature falls until as crystals form, the temperature begins to rise. As soon as this occurs, discontinue the stirring and take the highest temperature then recorded as the setting point.

APPENDIX 'B'

B. ESTIMATION OF PRIMARY AND SECONDARY AMINES

B.1 REAGENT.

B.1.1 70 % Acetic Acid Solution. 70 ml AR Acetic acid are diluted to 100 ml with distilled water.

B.1.2 Standard Aniline Stock Solution. 0.15 g of AR Aniline are dissolved in 5 ml of concentrated pure Hydrochloric acid and made up to 100 ml.

B.1.3 Standard Ethylaniline Stock Solution. 0.3 g of pure Ethylaniline are dissolved in 5 ml pure concentrated Hydrochloric acid and made up to 100 ml.

B.1.4 Alcoholic Hydrochloric Acid. 23 ml of pure concentrated Hydrochloric acid are dissolved in rectified Spirit and the mixture made up to 250 ml with rectified Spirit.

B.1.5 Alcoholic Naphthylamine. 1 g of AR Naphthylamine is weighed out and dissolved in 100 ml of rectified Spirit not more than 30 minutes before the performance of the test. The top layer of naphthylamine in the bottle which has been exposed to the air should not be used.

Note. A series of re - standardisation tests is necessary when a new supply of Naphthylamine is taken into use.

B.2 EXTRACTION.

B.2.1 Weigh 3 g Carbamite and place in a clean and dry 15 cm x 2.5 cm test tube with a well - fitting stopper (preferably ground - in). Add 5 ml of 70 % (v/v) aqueous Acetic acid solution and place the tube in a bath of water at 70 °C to 75 °C for 10 minutes. The tube may need occasional gentle agitation to ensure that Carbamite is completely dissolved. After withdrawal, cool the contents of the tube under running water, gently shaking and when the solution has reached room temperature, add about 10 ml of cold distilled water. Stopper the tube and shake vigorously up and down for at least 2 minutes to bring Carbamite out of solution in the form of fine granular crystals. Decant off as much liquid as possible through a previously prepared filter (No. 41 Whatman paper) into a 100 ml standard flask.

B.2.2 The precipitated Carbamite left in the tube should now be redissolved by adding 10 ml of the 70 % Acetic acid solution and heating for a further 10 minutes at 70 °C - 75 °C with occasional agitation. The cooling is repeated and about 15 ml of water added to ensure complete precipitation of Carbamite on shaking. The solution is poured on to the same filter and Carbamite residue is washed three times by decantation with cold water and fourth time on the filter paper.

B.3. NITROSATION. Add 4 ml of a normal Sodium nitrite solution to the contents of the flask and make up to the 100 ml mark with distilled water. Ensure complete mixing of the solutions and stand the flask in a cool, dark place for 30 minutes.

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B.4 STANDARDISATION.

B.4.1 A blank test is prepared by diluting 15 ml of 70 % Acetic acid in a 100 ml flask and, after adding Sodium nitrite as before, making up to the mark.

B.4.2 Standardisation tests are prepared by adding graded amounts of standard Aniline and Ethylaniline solutions to 15 ml of 70 % Acetic acid in 100 ml flasks. Sodium nitrite is added to these and each is made up to the mark and treated exactly as Carbamite extracts. The standard Amine stock solutions contain about 0.5 g of the pure Amine (Aniline and Ethylaniline), accurately weighed and dissolved in dilute Hydrochloric acid and made up to a suitable volume, for example 100 ml. As a guide in making standard solutions, it may be noted that 0.001 g of Aniline and 0.002 g of Ethylaniline have each been found to give strong colorations under the conditions of test and these quantities should not be greatly exceeded in any one standard test solution.

B.5 COLOUR TESTS.

B.5.1 Provide a clean, dry 15 cm x 2.5 cm test tube for each colour test and weigh approximately 0.07 g of crushed Sulphuric acid into each.

B.5.2 Pipette 2 ml of each of the mixed nitrated solutions into each test tube, directing the liquid slowly on to the Sulphamic acid. It is important to ensure that the whole quantity of liquid reaches the Sulphuric acid and that none is left on the walls of the tube. Gently swirl the effervescing mixture obtained and allow the reaction to proceed for 20 minutes, repeating the swirling movement at intervals.

B.5.3 Measure out 50 ml of rectified Spirit cooled below 10 °C into each tube. Add 2 ml of normal alcoholic Hydrochloric acid and place the tube in an ice water mixture. When the temperature of the rectified Spirit is about 5 °C add 2 ml of freshly prepared 1 % alcoholic Naphthylamine solution. Mix the contents of the tubes and stand the latter in ice water mixture for about 20 minutes in order to develop colour.

B.5.4 The resulting solutions are examined in comparison with rectified Spirit in Spekker Absorptiometer (using yellow - green filters, Ilford No.605, ink 4 cm glass cells). The blank and the Ethylaniline standards should yield a very low reading at this stage. Incomplete reaction with Sulphamic acid makes itself apparent as a violet coloration in the blank.

B.5.5 After measurement, return each solution to its respective tube and make a mark on the outside to indicate the volume of liquid in the tube. Place the tubes in a bath of water at 60 °C for 30 minutes in order to further develop the colour. Allow to cool in the dark for 60 minutes and make good the loss of evaporated alcohol by adding alcohol up to the mark. Mix the solutions and measure the colorations once more in the Spekker Absorptiometer exactly as before.

B.6 CALCULATION OF RESULT. The coloration which develops in the cold corresponds with the quantity of Primary amines presents, the coloration which develops at 60 °C corresponds with the quantity of Primary and Secondary Amines present and a calibration curve is drawn un showing Spekker drum readings (less the blank) plotted against the known amounts. From the curves the percentage of Amine in Carbamite sample is calculated.

C. THE DETERMINATION OF TERTIARY AMINES

C.1. PREPARATION OF SOLUTIONS.

C.1.1 Standard Diethyl Aniline. Dissolve an accurately weighed, quantity about 0.1 g of pure Amine in dilute Hydrochloric acid and dilute to 250 ml. 1 ml contains 0.0005 g of Amine. Dilute 25 ml of this solution to 250 ml, 1 ml of the solution now contains 0.00005 g of Amine.

C.1.2 Acetic Acid, 70 % (for extraction). Take 350 ml of the pure glacial Acetic acid and dilute to 500 ml.

C.1.3 Acetic Acid, 14 %. Dilute 50 ml of the 70 % glacial Acetic acid to 250 ml.

C.1.4 Acetate Reagent. Dissolve 50 g of Sodium acetate and 10 ml of pure glacial Acetic acid in water and dilute to 250 ml.

C.1.5 Dilute Sodium Nitrite. Approximately N / 5 - Dissolve 2.76 g of the pure salt in water and make up to 250 ml.

C.2 EXTRACTION.

C.2.1 Weigh 2 g Carbamite and place in a clean and dry 15 cm x 2.5 cm test tube with a well-fitting stopper (preferably ground-in). Add 5 ml of 70% (v/v) aqueous Acetic acid and place the tube in a bath of water at 70 °C to 75 °C for 10 minutes. The tube may need occasional gentle agitation to ensure that Carbamite is completely dissolved. After withdrawal, cool the contents of the tube under running water, gently shaking, and when the solution has reached room temperature, add about 10 ml of cold distilled water. Stopper the tube and shake it up and down vigorously for at least 2 minutes to bring Carbamite out of solution. Decant off as much liquid as possible through a previously prepared filter into a 50 ml standard flask.

C.2.2 Redissolve Carbamite left in the tube by adding a further 5 ml of 70 % Acetic acid and heating again for 10 minutes as before. Repeat the precipitation process and add the extract to that previously filtered. Wash Carbamite residue with cold water twice by decantation and again on the filter. Add distilled water to the extract and washings and make up to 50 ml mark and mix.

C.3. TEST.

C.3.1 Pipette "N" ml Carbamate extract into a 25 ml flask (5 ml will usually be found to give a conveniently strong coloration, but a larger amount could be used).

C.3.2 Prepare the standard and blank tests as follows. Measure accurately 1 ml of the diluted Diethylaniline solution into a 25 ml flask and by adding N / 10 Sodium hydroxide solution make the solution neutral to a fragment of litmus paper suspended in it. Add "N" ml of 14 % Acetic acid to this and place the same amount in a second 25 ml flask as a blank.

C.3.3 To each solution (which include the Carbamate tests and the standard and blank) add 5 ml of the Acetate reagent and 0.5 ml of diluted Sodium nitrite solution. Make each solution upto 25 ml with distilled water. Mix the solutions and transfer each into a clean, dry 15 cm x 2.5 cm test tube. Mark the tubes on the outside to show the volume of liquid inside. Place all the tubes together into a beaker of suitable size half filled with cold water.

C.3.4 Heat the beaker and contents slowly so that the water reaches about 95 °C in 20 - 25 minutes and maintain it at that temperature for a further 20 minutes. The dye stuff should then have disappeared and the test solution is yellow.

C.3.5 When cold, add distilled water to restore the level of solution in each tube to the original. Examine the solutions in 4 cm cell against distilled water in a Spekker Absorptiometer using violet filters. Ilford No. 601.

C.3.6 Calculate a factor relating the drum readings (less the blank) with the amount of Diethylaniline and by means of it calculate the percentage of Tertiary Amines in Carbamate.

D. DETERMINATION OF HYDROLYSABLE CHLORINE COMPOUNDS

D.1. Warm 5 g Carbamite on a water bath for 15 minutes with 10 ml of 5 % solution of Sodium ethylate in alcohol. Dilute the liquid with 100 ml of distilled water and allow Carbamite to crystallise. Filter and test the filtrate for chlorides and calculate as Chlorine.