रक्षा मंत्रालय
MINISTRY OF DEFENCE

संयुक्त सेवा विनिर्देश
JOINT SERVICES SPECIFICATION

ON

DIPHENYLAMINE
(DS Cat. No. 6810 - 001 049)

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

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), 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 Sushil Chander	ADGWE / GS (WE - 2 / 3), New Delhi
3.	Col RN Nambiar	Dte of Arty (GS / Artillary - 5), New Delhi
4.	Capt. Rakesh Verma, DNAI	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.	Shri. P Upadhyay, Director	DGNAI, Naval HQ, New Delhi
8.	Shri A.K. Parashar, PScO	DGAQA, JD (Armt) Gp, New Delhi
9.	Dr. R.K Upadhyay, PScO	CQA (ME), Pune
10.	Shri. A.V. Shinde, JAG (SG)	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 P. W. Sonawane, Jt. Director	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 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 2000.

(b) This specification is a revision of JSS 6810 - 129 : 2009, (Revision No. 2) and supersedes the same.

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

0.5 Quality Assurance Authority for the item covered by this specification is The Controller, Controllerate of Quality Assurance (Military Explosive), 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 Quality Assurance of Diphenylamine suitable for use in the manufacture of propellants.

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)	JSG 0112 : 2015 (Revision No. 2)	General Methods of Tests and Assessment of Impurities in Chemicals / Materials used in the Manufacture of Explosives and Ammunition.

3. MATERIAL : The Diphenylamine is to consist of the crystalline compound $(C_6H_5)_2$ NH. It shall be in the form of small crystals or lumps, free from foreign matter and visible impurities. It shall be white or at the most slightly yellow in colour and when molten shall give a clear light yellow light liquid. It shall possess the characteristic odour of Diphenylamine.

4. MANUFACTURE : The Diphenylamine 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 500 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 Manufacturer / contractor 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 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 has not been carried out, the consignment is liable for rejection.

7. QUALITY ASSURANCE

7.1 INSPECTION : The Diphenylamine and the packages in which it is packed shall be subject to inspection by and to the final 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 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 to 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 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 be in accordance with the clause 3 and shall comply with the following test requirements :-

TEST REQUIREMENTS OF DIPHENYLAMINE

Sl. No.	Characteristics	Passing Standard	Test Method
(a)	Moisture (over Caustic potash in a vacuum desiccator, % by Mass	0.5 Max.	JSG 0112 Method 1 (b)

Sl. No.	Characteristics	Passing Standard	Test Method
(b)	Reaction :-		Appendix 'A'
	(i) Alkalinity	Nil	
	(ii) Acidity, Calculated as Sulphuric acid, % by mass	0.005 Max.	
(c)	Organic matter insoluble in Ether alcohol, % by mass	0.02 Max.	Appendix 'B'
(d)	Mineral Matter (i.e. residue on ignition), % by mass	0.05 Max.	JSG 0112 Method No. 2 (a)
(e)	Primary Amines, calculated as Aniline, % by mass	0.20 Max.	Appendix 'C'
(f)	Organic bases alkaline to Methylorange, calculated as Aniline, % by mass	0.25 Max.	Appendix 'D'
(g)	Setting point ºC	51.5 Min. 53.0 Max.	Appendix 'E'
(h)	Sulphuric acid test	Colourless or pale green fluorescent solution	Appendix 'F'

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 12 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 Diphenylamine shall be supplied in sound, clean and dry wooden cases, lined with non - absorbent paper or other approved packages, containing an approved quantity.

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 :-

(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 Packages in Consignment.

(h) Date of Supply.

(j) Manufacturer's Initials or Recognised Trademark.

10.2 The white or black paint used for marking should conform to IS 138 and to the satisfaction is the Quality Assurance Officer / Quality Assurance Authority.

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

11. DEFENCE STORES CATALOGUE NUMBER

11.1 The Defence Stores Catalogue Number allotted to this store is 6810- 001 049.

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 specification shall be forwarded to:-

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

APPENDIX 'A'

A. REACTION

A.1 Dissolve 20 g of the sample in 50 ml of Ether in a separating funnel. Shake the solution four times with successive 30 ml. quantities of distilled water. Collect the washing in a round bottomed flask of resistance glass. If the extract is acidic to Phenolphthalein the procedure is as follows :

A.1.1 Warm the solution on the steam bath to expel Ether, then boil over a bunsen to drive off Carbon dioxide. Tightly stopper the flask and cool. Then titrate with N / 100 Barium hydroxide with phenolphthalein as indicator. Calculate the acidity as Sulphuric acid.

A.1.2 If the extract is alkaline, add excess of N / 100 Hydrochloric acid and titrate as above. Calculate the alkalinity as Sodium carbonate ($Na_2 CO_3$).

APPENDIX 'B'

B. DETERMINATION OF ORGANIC MATTER INSOLUBLE IN ETHER - ALCOHOL

B.1 Dissolve 20 g of the material in about 150 ml of Ether - alcohol mixture (2 parts by volume of Ethyl ether and one part of Ethyl alcohol) in a beaker, at room temperature. Filter off the insoluble matter on to a prepared asbestos Gooch or similar filter, thoroughly wash with ether - alcohol dry and weigh.

APPENDIX 'C'

C. DETERMINATION OF PRIMARY AMINES

C.1 The method of determining primary amines in Diphenylamine depends on extraction of these bases from an ethereal solution of the Diphenylamine by means of Hydrochloric acid, diazotisation and coupling in alkaline solution with standard solution of R - Salt, the quantity of R - salt required being a measure of the primary Amines present.

C.2 Dissolve 50 g of the sample in 100 ml of Ether and extract solution three times with 30 ml of dilute Hydrochloric acid in Separating funnel. Return the aqueous extract to the Separating funnel, wash once with Ether, separate and expel excess of Ether by heating on the water bath. Make up the volume of the solution to 200 ml with distilled water.

C.3 For an approximate determination of the percentage of Primary amines present, place 50 ml. of prepared solution in a conical flask, cool to 0 °C and add an excess of 2 % solution of Sodium nitrate at 0 °C. After standing for 5 minutes to allow diazotisation to complete itself, make the solution alkaline by the addition of Sodium carbonate in moderate excess, and saturate with Sodium chloride, the temperature not being allowed to rise above 0 °C. Add a solution of R-salt, standardised against Aniline as described below, from a burette in small quantities at a time. After each addition, allow the solution to stand in ice for 5 minute - 10 minute to permit complete coupling of the R - salt added. Filter small test portions (1 ml - 2 ml) into the two test tubes and add one drop of R -salt solution to one and one drop of freshly prepared ice - cold diazo solution to the other. Prepare the diazo solution by diazotising a small quantity of a standard solution of Aniline hydrochloride containing 0.0005 g - 0.001 g of Aniline per ml.

C.4 If the Primary amines are in large excess, a deep red colouration will develop rapidly in the portion to which R-salt has been added, while the other portion will remain practically colourless. When the end point is nearly reached a red or yellowish red colour may develop slowly in each portion, but more deeply in the R - salt portion than the other. At the end point, which may be indefinite to the extent of 2 ml - 3 ml. of R - salt, the depth of tint developed in each portion is practically equal, and when the end point is passed the diazo test portion shows a deeper colour than the R - salt portion. In this way an approximate value is obtained for the primary base content and a more accurate determination is carried out with a fresh portion of the prepared solution as follows :

C.5 Diazotise the solution as before and after diazotisation is complete make alkaline and run in the quantity of R - salt solution found necessary for the approximate determination, Saturate the solution with Sodium chloride and allow the mixture to stand in ice for 15 minutes with occasional vigorous shaking. Filter small test portions (5 ml - 10 ml) into test tubes. These test portions should be almost colourless. Add to one, two drops of R - salt solution and, to the other, two drops of diazo solution. If the tints developed are practically equal to the titration is complete. If, however, a deeper tint is developed in the R - salt test portion, insufficient R - salt has been added and the whole operation must be repeated with the addition of a larger volume of R - salt.

C.6 From the volume of R - salt finally found equivalent to the Primary amine present, calculate the percentage of Aniline on the weight of sample taken.

C.7 PREPARATION OF REAGENTS

C.7.1 R-salt solution:-

Dissolve 2 g - 4 g of R-salt in a litre of water and filter.

Note. Carry out the Standardisation of the R-salt Solution in the same way as described above.

C.7.2 Aniline hydrochloride Solution :-

Dissolve 0.5 g - 1 g of freshly distilled Aniline in a slight excess of dilute Hydrochloric acid and dilute with water to one litre.

APPENDIX 'D'

D. DETERMINATION OF ORGANIC BASES ALKALINE TO METHYL ORANGE

D.1 Dissolve 20 g of the sample in 50 ml of Ether and extract with four successive 30 ml portions of dilute Hydrochloric acid. Transfer the extracts to a steam distillation flask and boil off the Ether. Make the contents of the flask alkaline by the addition of Caustic soda solution and steam distil into 10 ml of dilute Hydrochloric acid until the small quantity of Diphenylamine which has passed into solution during extraction of the Ether solution has distilled over. The more volatile bases will also have passed over completely. Make the solution neutral to Phenolphthalein by the addition of carbonate free Caustic soda. Add Methyl orange and titrate the bases with N/10 Hydrochloric acid, and calculate as Aniline.

APPENDIX 'E'

E. DETERMINATION OF SETTING POINT

E.1 Fill a test tube 2.5 cm x 15 cm to within 3.8 cm of the top with the Melted Diphenylamine 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 3.8 cm x 18 cm and support of 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 40 °C - 45 °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 and recorded as the setting point.

APPENDIX 'F'

F. SULPHURIC ACID TEST

F.1 Take 0.2 g of the material. Dissolve it in 2 ml of Sulphuric acid (Sp. Gr. 1.5) and then add it to 20 ml of concentrated Sulphuric acid. This shall be a colourless or pale green fluorescent solution.