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2 - NITRO DIPHENYL AMINE
(DS Cat No. 6810 - 001 008)

सत्यमेव जयते

CONTROLLERATE OF QUALITY ASSURANCE (MILITARY EXPLOSIVES)

AUNDH ROAD, PUNE - 411 020.

DEPARTMENT OF DEFENCE PRODUCTION

MINISTRY OF DEFENCE

AMENDMENT RECORD

Amendment		Authority	Clauses Affected	Remarks
D.C.	DATE			

2- NITRO - DIPHENYL AMINE

CONTENTS

THIS SPECIFICATION OR ANY OTHER PATTERN, DRAWINGS OR ANY OTHER INFORMATION ISSUED IN CONNECTION THEREWITH MAY ONLY BE USED FOR A SPECIFIC ORDER PLACED BY THE COMPETENT AUTHORITY. IT IS NOT TO BE USED FOR ANY OTHER PURPOSE WHATSOEVER WITHOUT THE EXPRESS WRITTEN SANCTION OF THE DIRECTOR GENERAL OF QUALITY ASSURANCE, MINISTRY OF DEFENCE, NEW DELHI - 110 011 .

0. FOREWORD

0.1 This specification has been prepared by the CONTROLLERATE OF QUALITY ASSURANCE (MILITARY EXPLOSIVES) AUNDH ROAD, PUNE -411 020.

0.2 This specification is a revision of IND/ME/877 (Prov) and supersedes the same.

0.3 For additional copies or any other enquiry regarding this specification reference should be made to the Quality Assurance Authority (i.e. CQA(ME) Aundh road, Pune-411 020).

1. SCOPE

1.1 This specification is meant to govern supply and inspection of 2 - nitro diphenyl amine.

1.2 The material is suitable for use in the manufacture of double base propellants.

2. RELATED SPECIFICATIONS AND DOCUMENTS

2.1 The related documents mentioned at clause 2.2 are those applicable at the date of publication of this specification. It is contractor' s/manufacturer's responsibility to confirm their current applicability and to obtain from the Authority Holding Sealed Particulars (i.e. CQA(ME) Aundh road, Pune-411 020) information concerning any change that may be necessary due to cancellation, replacement or supersession of any of these documents.

2.2 The following related specifications have been referred to in the preparation of this specification:-

(i) IS 460 (Part 1) : 1985, AMD-1 (Reaffirmed 2008)	Test Sieves : Part 1 wire cloth Test Sieves
(ii) IS 2552 : 1989, AMD-1 (Reaffirmed 2011)	Steel Drums (Galvanised and ungalvanised)

2.3 Copies of this specification and other related specifications are obtainable on payment basis as follows:-

SPECIFICATION		SOURCE OF SUPPLY
IS Specification	:	Bureau of Indian Standards, Manak Bhawan 9, Bahadur Shah Zafar Marg, NEW DELHI – 110 002 or Their regional / Branch offices
IND/ME/ Specification	:	C. Q. A. (ME), AUNDH ROAD, PUNE - 411 020.
JSS	:	The Director Directorate of Standardization Standardization Documents Centre Ministry of Defence Room no 05, 'J' Block Nirman Bhawan PO New Delhi – 110 011

3. MATERIAL

3.1 The material shall consist essentially of 2.nitrodiphenyl amine, (O_2N) $C_6H_4.NH.C_6H_5$, in the form of red crystals, free from grit, foreign matter, any visible and mechanical impurities.

4. TENDER SAMPLE

4.1 The contractor shall submit two tender samples each of 250 g essentially from the same batch/lot of manufacture, free of charge and conforming to this specification.

5. PRE-INSPECTION OF STORES / CONSIGNMENT

5.1 Before tendering the store to the Quality Assurance Officer, the contractor shall carry out a thorough inspection of each delivery to satisfy himself that the store fully conforms to this specification and shall render a certificate to that effect to the Quality Assurance Officer.

6. QUALITY ASSURANCE

6.1 Inspection

6.1.1 The 2 -nitrodiphenyl amine 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.

6.1.2 Samples of the material and of the packages may be taken from any portion of a consignment.

6.1.3 If, on examination, any sample be found not to conform to this specification, the whole consignment may be rejected.

6.1.4 The foregoing provisions shall apply equally to prime contractors and to sub-contractors, if any.

6.2 Sampling

6.2.1 Normally two representative samples each of 500 g shall be drawn from each batch/lot. However, the number of samples to be drawn will be at the discretion of the Quality Assurance Officer.

6.3 Test Requirement

6.3.1 Samples taken from any portion of the supply shall comply with clause 3.1 above and shall also conform to the following test requirements:-

Sl. No.	Characteristics	Passing standard	Test Method
1	2	3	4
1.	Volatile natter, Percent by mass, Max.	0.6	Appendix 'A'
2.	Reaction of water extract (a) Alkalinity to phenol- phthalein, as NaOH, percent by mass, Max.	0.005	Appendix 'B'
	(b) Acidity to phenol- phthalein, as HCl, percent by mass, Max.	0.005	
3.	Free aniline and salts of aniline as aniline, percent by mass, Max.	0.05	Appendix 'C'
4.	Matter insoluble in ether or benzene percent by mass, Max.	0.03	Appendix 'D'
5.	Ash, percent by mass, Max.	0.05	Appendix 'E'
6.	Solidification point, Min. Max.	 73°C 75°C	Appendix 'F'
7.	2 -nitro diphenyl amine content percent by mass, Min.	98.0	Appendix 'G'
8.	Sulphuric acid test	A clear red coloured solution must be obtained.	Appendix 'H'

7. PACKAGING AND MARKING

7.1 Packaging

7.1.1 The 2-nitrodiphenylamine shall be packed in a polythene bag made of 0.13 mm thick polythene film and capacity 50 kg. This bag will be hermetically sealed and packed in a wooden case/box. Alternatively the material may be packed in an internal polythene bag of 0.13 mm film thickness, sealed hermetically and packed in external fibre drums or sound, dry, clean, rust free mild steel drums (conforming to IS : 2552 latest issue).

7.1.2 Material packed in any other containers/packages shall have the prior approval of the Quality Assurance Officer/ Quality Assurance Authority.

7.1.3 The inclusion of any foreign matter or impurities in any of the containers/packages shall render the whole consignment liable to rejection.

7.2 Marking

7.2.1 All packages constituting a consignment shall be legibly and durably marked with the following details as applicable:-

(i) Nomenclature and specification number of the material.
(ii) Name and address of the consignee.
(iii) A/T /S. O. No. and date
(iv) Consignment No.
(v) Lot No. / Batch No. and date of manufacture
(vi) Consecutive No. of package and total No. of packages.
(vii) Gross and net mass.
(viii) Date of supply
(ix) Contractor's initials or recognized trade mark.

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

7.2.3 The paint or other materials used for marking and also the paint used on the packages shall be of good quality and to the satisfaction of the Quality Assurance Officer.

8. SAFETY OF OPERATIONS

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

9.0 DEFENCE STORE CATALOGUE NUMBER

9.1 Defence Store Catalogue number allotted to this store is 6810 – 001 008.

Date: - 28/01/2016

(Mrs. MGP DHANRAJ)
Controller
CQA [ME], PUNE.

10. APPENDICES

APPENDIX 'A'

VOLATILE MATTER

Weigh a dry glass stoppered 60 mm x 40 mm weighing bottle (M_1). Introduce 5 g 0.01 g of the sample and re-weigh accurately (M_2). Carefully remove the stopper and place the bottle and stopper side by side in an oven at 100°C ± 5 deg C for 3 hours. Replace the stopper in the bottle, remove from the oven, cool in a desiccator for 20 minutes and reweigh (M_3).

$$\text{Volatile matter, Percent by mass} = \frac{M_2 - M_3}{M_2 - M_1} \times 100$$

APPENDIX 'B'

DETERMINATION OF ACIDITY OR ALKALINITY

Take about 20 g of sample accurately weighed in a 250 ml iodine flask. Add 100 ml boiling neutral distilled water to melt the material. Close the flask and shake it vigorously for 10 minutes or until the sample solidifies. Cool to room temperature, decant and filter through a No. 41 Whatman filter paper. Repeat the extraction with three more portions of 50 ml boiling distilled water. Collect whole of the filtrate in another iodine flask.

To the filtrate so collected above, add 5 drops of phenolphthalein indicator and titrate with 0.1 N sodium hydroxide solutions or 0.1 N hydrochloric acid as appropriate. Carry out a blank determination, and apply correction, as necessary to the titre.

(i) $$\text{Acidity as HCl Percent by mass} = \frac{0.00365 \times t_1 \times f_1 \times 100}{M}$$

Where, t_1 = titre of 0.1 N NaOH solution

f_1 = factor of NaOH solution

M = Mass of sample taken.

(ii) $$\text{Alkalinity as NaOH Percent by mass} = \frac{0.0040 \times t_2 \times f_2 \times 100}{M}$$

Where, t_2 = titre of 0.1 N HCl

f_2 = factor of HCl

M = Mass of sample taken.

APPENDIX 'C'

DETERMINATION OF FREE ANILINE AND SALTS OF ANILINE AS ANILINE

To the filtrate obtained as in appendix B, after determination of acidity or alkalinity, add 25 ml of N/10 bromate -bromide solution (prepared by dissolving 2.784 g $KBrO_3$ and 15 g KBr in sufficient amount of distilled water and making the volume to one litre in a standard volumetric flask) by pipette, cool the mixture to 15°C and add 5 ml conc. hydrochloric acid. After one minute add 10 ml of 10 % potassium iodide solution and titrate with N/10 sodium thiosulphate solution using freshly prepared 1 % starch solution as indicator (titre A). Carry out a blank determination simultaneously using 25 ml of bromate-bromide solution and titrate in the manner described above (titre B).

$$\text{Aniline (Free aniline and aniline salts calculated as \% aniline),} = \frac{(B - A) \times 0.1551 \times F}{M}$$

Where M = Mass of sample taken for test as in Appendix 'B'.

F = Factor for N/10 $Na_2S_2O_3$ solution.

APPENDIX 'D'

DETERMINATION OF MATTER INSOLUBLE IN ETHER OR BENZENE

Weigh accurately about 25 g of sample (M) into a 400 ml beaker and dissolve by warming on a water bath in 100 ml pure diethylether or 100 ml of pure benzene. Filter the solution through a tared sintered glass crucible G-3(M_1) and wash the residue thoroughly three to four times with more portions of the same solvent. Dry the crucible containing the residue in an oven at 100°C ± 5 deg C for 1 hour, cool in a desiccator and weigh (M_2)

$$\text{Matter insoluble in ether/benzene, Percent by mass} = \frac{M_2 - M_1}{M} \times 100$$

APPENDIX 'E'

DETERMINATION OF ASH CONTENT

Weigh accurately about 5 g of sample (M) into a previously dried, cooled and weighed porcelain/silica dish (M_1) (Platinum dish can also be used). Heat gently over a small flame until the material ignites, then allow it to continue burning. Finally ignite the dish by placing it in a muffle furnace maintained at 800°C ± 25 degC, until all the carbon has burnt off. Remove the dish from the furnace, cool, transfer it to a desiccator, cool for further half an hour, and weigh (M_2).

$$\text{Ash content, Percent by mass} = \frac{M_2 - M_1}{M} \times 100$$

APPENDIX 'F'

DETERMINATION OF SOLIDIFICATION POINT

Take a clean and dry test tube 25 mm x 150 mm and fill with the sample and melt by placing in hot water bath/oven. Then add more sample to the tube until the melted liquid reaches to within 38 mm from the top of the tube. Fit it with an aluminium wire stirrer and a cork carrying a thermometer, conforming to the following essential specifications shall be used with range 38 to 82 degrees celcius, subdivisions with 0.1 degree celcius graduation and a hole, so arranged that its bulb is situated centrally in the tube and its lower extremity is 25 mm from the bottom. Fit in an auxiliary thermometer into the cork in such a way as to record the temperature of the middle of the emergent stem of the thermometer inserted in the melt of the sample. Support the tube centrally by means of a cork in a larger test tube 40 mm x 200 mm and support the latter inside a one litre beaker by means of a cover which closes the mouth of the beaker. Fill the beaker to within 25 mm of the top with water at 60°C to 65°C. Stir the sample melt vigorously as the temperature falls, until, as crystals form, the temperature begins to rise. As soon as this occurs, discontinue the stirring and note the highest temperature recorded (t_1).

Correct the above temperature for expansion of mercury by adding a correction obtained as follows :-

Correction for

mercury expansion = $n (t_1 - t_2) \times 0.000159$

Where, n = number of degrees from the level of the melt of the sample to the temp, for observed solidification point. (t_1).

t_1 = observed solidification point, °C

t2 = temp. °C in the auxiliary thermometer (that of the middle of the emergent stem of the thermometer in the sample)

0.000159 = coefficient of expansion of mercury in the bulb.

Solidification point = t_1 + correction in deg °C.

APPENDIX 'G'

DETERMINATION OF 2 – NITRODIPHENYLAMINE

Reagents required

(1) Denatured spirit

(2) Bromine

Procedure :-

Transfer an accurately weighed sample of approximately 0.5 g ± 0.01 g (M) of the material to a 600 ml squat beaker. Add 30 ml of rectified spirit and heat the beaker and contents on a boiling waterbath to aid solution. Remove the beaker from the waterbath, cool and add 2.0 ml of bromine, a few drops at a. time, shaking after each addition. Replace the beaker on the waterbath and evaporate the contents to a volume of 5 ml - 10 ml, then add 400 ml of hot water and stir well. Continue to heat with occasional stirring on the waterbath until the precipitated tribromo compound is coagulated and the supernatant liquid is clear. Filter the precipitate through a tared G-4 sintered glass crucible (M_1) and wash well with hot water. Dry the crucible at 103°C - 105°C for 1 hour, cool in a desiccator for 20 minutes and reweigh (M_2), to constant mass. Record the gain in mass as the tribromo derivative of 2 -nitrodiphenylamine.

Calculation

$$\text{2 –nitrodiphenylamine Content Percent by mass} = \frac{(M_2 - M_1) \times 47.48 \times 100}{M \times (100 - \% \text{ volatile matter })}$$

APPENDIX 'H'

SULPHURIC ACID TEST

Take about 0.2 g of the sample and dissolve it in a 2 ml of dilute sulphuric acid (Relative density 1.5) and add ,the solution so obtained to 20 ml of concentrated sulphuric acid in a clear test tube. Observe the colour of the solution.

It must be a clear red coloured solution.

CMK/18-9-79/