17.07.2013

IND/ME/1011

MASTER COPY

2,3 DIMETHYL-2,3 DINITROBUTANE OR

SYM TETRAMETHYL DINITROETHANE(DMNB)

V. Thanigavelan 4/10/19

MASTER COPY

DEPARTMENT OF DEFENCE PRODUCTION
MINISTRY OF DEFENCE
CONTROLLERATE OF QUALITY ASSURANCE
(MILITARY EXPLOSIVES)
AUNDH ROAD, PUNE - 411 020.

RECORD OF AMENDMENTS

Amendment Sr. No	Date	Details of amendment	Amendment carried out by (Name & Date)
D.C.NO. 3607-ME/13.3.2000		Sealed Provisionally	

CONTENTS

0.	FOREWORD	---	01
1.	SCOPE	---	01
2	RELATED SPECIFICATIONS AND DOCUMENTS	---	02
3	MATERIAL	---	02
4.	MANUFACTURE	---	03
5.	PRE INSPECTION OF STORE/CONSIGNMENT	---	03
6	QUALITY ASSURANCE	---	03
7.	PACKAGING	---	05
8.	MARKING	---	06
9.	WARRANTY	---	07
10.	SAFETY OF OPERATION	---	07
11.	DEFENCE STORES CATALOGUE NUMBER	---	07
12.	SUGGESTIONS FOR IMPROVEMENT	---	07
13.	APPENDICES	---	08 to10

0.0 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 shall be used for tender enquiry, procurement, manufacture and quality assurance of the item covered by this specification.

0.3 The Quality Assurance Authority for this store is The Controller, Controllerate of Quality Assurance (Military Explosives), Aundh Road, Pune - 411 020. Enquiries regarding this specification relating to technical or any other contractual conditions shall be referred to the Quality Assurance Authority mentioned above/named in the tender or contract.

0.4 Copies of this specification can be obtained on payment from -

The Controller
Controllerate of Quality Assurance (Mily Explosives)
Aundh Road,
Pune-411 020.

1.0 SCOPE

1.1 This specification prescribes requirements, method of sampling and test of 2,3 Dimethyl 2,3 Dinitrobutane or Sym Tetramethyl Dinitroethane (DMNB) and provides guidance to suppliers/manufacturers and Quality Assurance agencies

1.2 This specification is meant to govern manufacture, supply and Quality Assurance of 2,3 Dimethyl 2,3 Dinitrobutane or Sym Tetramethyl Dinitroethane (DMNB)

1.3 The store is suitable for use in the manufacture of Plastic Explosives -1 (marked) as a marking agent.

2.0 RELATED SPECIFICATIONS AND DOCUMENTS

2.1 The related documents as mentioned in 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 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 this IND/ME Specification :

i IS:138 -1992 (Revision No. 3) Reaffirmed 2009 Amds 1	-	Ready Mixed Paint, Marking, for packages and Petrol Containers - Specification
ii JSG 0112-1997	-	General methods of tests and assessment of impurities in chemicals/materials used in the manufacture of explosives
		and ammunition.
iii IS-460:Part-I :1985(Revision No. 3) Reaffirmed 2008, Amds 1	-	Specification for Test Sieves Part-I wire cloth test sieves

2.3 Copies of this specification and of related specifications may be obtained on payment basis from the agencies mentioned below:

IND/ME Specification	The Controller Controllerate of Quality Assurance (Mity Explosives)
	Aundh Road, Pune - 411 020
IS Specification	The Bureau of Indian Standards, Manak Bhavan, 9 B. S. Zafar Marg,
	New Delhi-110 002
CQA(A) Specification	The Controller,
	Controllerate of Quality Assurance (Ammn)
	Khadki Pune - 411 003
JSG Specification	The Director,
	Directorate of Standardisation
	SDC, J-13, Ministry of Defence , 'H' Block
	Nirman Bhavan, New Delhi -110 011

3.0 MATERIAL

The material shall essentially consist of 2,3 Dimethyl 2,3 Dinitrobutane or Sym Tetramethyl Dinitroethane (DMNB) $CH_3\ C(CH_3).C(CH_3)\ CH_3$ It shall be in the

$NO_2 \quad NO_2$

form off white crystalline powder, free from visible impurities foreign matter and gritty particles.

4.0 MANUFACTURE

4 1 The material shall be manufactured by a process which has authoritative approval The QA Officer/QA authority shall be informed regarding the process used and shall be informed with prior notification any proposed deviation there from All the deviation from the approved process however slight, shall be recorded immediately and all the material affected shall be kept aside pending till the decision of the Quality Assurance Officer/Quality Assurance Authority.

5.0 PRE INSPECTION OF STORES/CONSIGNMENT

5.1 Manufacturers/contractors must satisfy themselves that 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 resident QA Officer nominated under the terms of contract. A declaration by the contractor that necessary pre-inspection has been carried out on the stores tendered will be submitted along with a challan. The declaration will also indicate the method followed in carrying out pre-inspection showing the features checked/tested and will have test certificate attached to the challan/declaration

5.2 If the QA Officer finds that pre-inspection of the batch as required above has not been carried out, the batch is liable for rejection.

6.0 QUALITY ASSURANCE

6.1 INSPECTION

6.1.1 2,3 Dimethyl 2,3 Dinitrobutane (DMNB) and the packages in which it is contained shall be subjected to inspection by and to the approval of QA Officer/QA Authority.

6 1.2 Samples of the material and of the packages may be taken from any portion of the batch/lot/consignment and at any stage of manufacture.

6.1.3 The foregoing provisions shall equally apply to the prime contractors and subcontractors if any.

6.2 SAMPLING

6.2.1 Normally two representative samples of 100 g each shall be drawn from a batch or lot of 5 Kg (50 bottles of 100 g each in a batch or lot of 5 Kg)

6.2.2 All precautionary measures for handling of the store as stated in the standing instructions shall be strictly complied with. In the absence of these, instructions from the QAO shall be sought.

6.3 TESTING

6.3.1 The sample shall be tested/examined as per Clause 3 above and shall also conform to the following test requirements :

6.3.2 TEST REQUIREMENTS

SR. NO.	CHARACTERISTICS	PASSING STANDARD	TEST METHOD
I	Visual examination	White crystalline powder free from visual impurities, foreign matter and gritty particles	
II	Insoluble matter coarse gritty particles retained on 63 micrometre IS sieve	Nil	Appendix 'A'
III	Matter insoluble in Benzene, Percent by mass	Max 0.10	Appendix 'B'
IV	Melting Point degree Celsius, Min.	210	Appendix 'C'
V	Infra red spectrum	IR spectra of test sample and standard should be superimposable	Appendix 'D'
VI	Water content, Percent by mass.	Max 0.20	By Karl Fischer

VII	Purity, Percent by mass	Min. 97	Appendix 'E'
VIII	Water soluble Sulphate as Na_2SO_4, Percent by mass	Nil	Method No. 8 (JSG 0112-1997)
IX	pH of aqueous extract Min Max	 6.5 7.5	Method no. 13 (JSG 0112-1997)
X	Ash, Percent by mass	Max 0 .08	Method no 2(a) (JSG 0112-1997)
XI	Chloride as NaCl. Percent by mass	Max 0.02	Method no 20 (JSG 0112-1997)
XII	Bromide as Br , Percent by mass	Max 0.05	— do —
XIII	Sieving Requirements retained on 850 Micrometer IS-Sieve, Percent, max.	2.0	Appendix 'F'(Particulars of IS sieves referred to will be found in IS: 460-1985)

6.4 QUALITY OF REAGENTS

6.4.1 Unless otherwise specified, pure chemicals and distilled water shall be employed in tests. "Pure chemicals" shall mean the chemicals that do not contain impurities which affect the results of analysis.

6.5 CRITERIA FOR CONFORMITY

6.5.1 The batch/lot shall be declared as conforming to the specification if the samples drawn as per the sampling plan satisfy Clause 3 and the test requirements given in Clause 6.3.2 above.

7.0 PACKAGING

7.1 The 2,3 Dimethyl 2,3 Dinitrobutane (DMNB) shall be packed in a suitable polythene bottle with tight fitting lid, so as to avoid ingress of moisture Quantity per package shall be 100 g each

7.2 Any other form of packing shall have prior approval of QA Officer/QA Authority.

7.3 The inclusion of any foreign matter or impurities in any of the packages shall render the whole batch/consignment liable for rejection.

8.0 MARKING

8.1 All the packages containing the material shall be indeliably and legibly marked with the following details as applicable :

a) Nomenclature and Specification No. of the store
b) Name and address of the consignee
*c) A/T No or S. O. No. and Date
d) Lot/Batch No and date of manufacture
*e) Consignment No
f) Gross and Net Mass
g) Consecutive No of package and total No of packages
h) Date of Supply
*i) Contractor's initials or recognised trade mark
* Not applicable when the store is manufactured in Ordnance Factories

8.2 In addition to the above, the QA Officer/QA Authority may suggest some more markings/identification suitable at the time of inspection.

8.3 The paint used for marking shall conform to IS 138 (latest issue) Any paint used on the container and the paint or other materials used for marking shall be of good quality to the satisfaction of QA officer and shall not contain Lead or Lead compounds calculated as metallic Lead together exceeding 0.5 percent.

9.0 WARRANTY

9 1 The store supplied against the contract shall deem to have been warranted against defective material and performance by the manufacturer/contractor/supplier for a period of 12 months from the date of receipt of the store at the consignee's end and if during this period any of the store supplied is found defective the same shall be replaced by the manufacturer/contractor/supplier free of charge at the consignee's premises.

10.0 SAFETY OF OPERATION

10.1 Nothing in this specification shall relieve the manufacturer/supplier/user of his responsibility for the safety of operation in manufacture, storage, transport or use of the store.

11.0 DEFENCE STORES CATALOGUE NUMBER

11.1 The Defence Stores Catalogue Number allotted to this store is

12.0 SUGGESTIONS FOR IMPROVEMENT

Any suggestions for improvement in this document shall be forwarded to The Controller CQA(ME), Aundh Road, Pune - 411 020.

- sd -

Date:- 17 July 2013

(Dr C. NESAMANI),
Controller,
CQA(ME), Aundh Road,
PUNE - 411 020

APPENDIX 'A'

Insoluble matter, Coarse, gritty particles

Place a known mass of the material (about 10 g) in a beaker, add 200 ml Acetone and heat on a hot water bath until all the material gets dissolved. Pour the solution through a 63 Micrometre IS sieve, transfer whole of the material from the beaker to sieve and finally wash the sieve with hot Acetone

Examine the retained material for gritty particles, if any. Repeat the experiment and report the results Retention on 63 Micrometre IS sieve should be Nil.

APPENDIX 'B'

Matter insoluble in Benzene

Weigh 5 g of DMNB and dissolve in 200 ml Benzene in a 500 ml beaker Transfer the contents of the beaker completely into the Gooch crucible Wash the contents with 50 ml hot Benzene Repeat washing with another 50 ml hot Benzene Dry the crucible in an oven at 100 -105° C for one hour, cool in a dessicator and weigh.

$$\text{Total insoluble matter in Benzene, Percent by mass} = \frac{M_2 - M_1}{M} \times 100$$

where

M_1 = mass of the empty crucible

M = mass of the sample

APPENDIX 'C'

Melting point

Take a glass capillary closed at one end and fill it with the fine , dry powder sample upto a height of 7 to 10 mm. Place the capillary in an electrically heated Melting point apparatus and maintain the rate of heating at 3 to 5 °Celsius per minute. When the sample melts note the reading on the thermometer Repeat the experiment for atleast three times and report the average results.

APPENDIX 'D'

IR spectra

IR spectra of the test sample and the standard sample should be superimposable The spectra are recorded using an IR spectrometer using Potassium bromide matrix. Samples from trade (Aldrich make with 98% purity or any other make with melting point 2100 Celsius or more) may be treated as a standard.

APPENDIX 'E'

Purity

Sample preparation

Weigh 0.0004 to 0 0005 g of DMNB and dissolve in 100 ml acetone

Preparation of standard solution

Weigh 0 0004 to 0.0005 g of standard DMNB and dissolve in 100 ml acetone

a) Instrumental Parameters

Chromatograph	Gas Chromatograph
Detector	Electron capture detector based on Ni 63 Radioactive source
Column	Capillary BP-10 at 1200 C

Injector temperature	: 240°C
Carrier gas	: IOLAR Nitrogen
Detector temperature	: 340^{0}C
Oven temperature	: 120° C

b) Quantitative Analysis

i) Inject 0.5µl standard and obtain the chromatograph .

ii) Then inject 0 5µl DMNB sample and obtain the chromatograph

iii) Formulaused for analysis

$$\% \text{ DMNB} = \frac{\text{Area of DMNB in sample x weight of standard DMNB}}{\text{Area of DMNB in standard x weight of test DMNB}} \times 100$$

iv) Repeat the standard and sample for atleast 5 times and report the average results

APPENDIX 'F'

Sieving Requirement

Transfer about 20 ± 5 g of the standard DMNB, accurately weighed, to 850 micrometer IS sieve and tap the sieve gently over sheet of white paper until a stage is reached at which it is shown by further tapping that no significant amount of fines are passing. Break the lumps with light pressure.

All should pass through 850 micrometer IS sieve. Any material remaining on the sieve will indicate the presence of oversize particles of DMNB Record as percentage of matter retained on the sieve and report accordingly.

$$\% \text{ Retention} = \frac{\text{Mass of retention}}{\text{Mass of sample}} \times 100$$