INCH-POUND

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COMMERCIAL ITEM DESCRIPTION

DMDNB (2,3-dimethyl-2,-3-dinitrobutane)

The General Services Administration has authorized the use of this commercial item description, for all federal agencies.

1. SCOPE

1.1 Scope. This commercial item description covers reagent grade 2,3-dimethyl-2,-3-dinitrobutane.

2. SALIENT CHARACTERISTICS

2.1 Material. The material shall be 2,3-dimethyl-2,-3-dinitrobutane (DMDNB) meeting the following requirements:

(a) Assay, % DMDNB: $\geq$ 98.5%
(b) Melting point, degree C: 210 min. to 214 max.
(c) Appearance: The DMDNB shall be milled, and in a granular white crystalline solid form
(d) Water: Water content in the DMDNB shall be equal to or less than 0.50%
(e) Methanol: Methanol content in the DMDNB shall be less than 0.5%
(f) Chloride: The chloride content shall be less than 0.02% or 200 ppm
(g) FTIR: The DMDNB when subjected to FTIR analysis shall exhibit the spectrum conforming to the attached standard, (Fig. 1).

2.2 Workmanship. The best commercial practice shall be used in the manufacture of this product. Procedures and controls shall be employed to prevent product contamination. There shall be no contamination (such as dirt, paint chips, wood, etc.) that is obvious and readily observable. In addition, the DMDNB shall be free of contaminants which are hazardous, corrosive, reactive, or metallic.

Comments, suggestions, or questions on this document should be addressed to Commander, US Army ARDEC, Attn: RDAR-EIQ-SA, Picatinny Arsenal, New Jersey 07806-5000 or emailed to usarmy.picatinny.ardec.list-stdzn-branch@mail.mil. Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at https://assist.dla.mil.

AMSC N/A FSC 6810

DISTRIBUTION STATEMENT A. Approved for public release; distribution is unlimited.

3. REGULATORY REQUIREMENTS

3.1 Safety and health practices. This standard does not purport to address all of the safety concerns, if any, associated with the use of the DMDNB material. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

3.2 Manufacture safety data statement. Manufacture Safety Data Sheet shall be submitted in accordance with the requirements as specified in the Code of Federal Regulation, Part 29-1910 (OSHA). (For shipment to foreign users, the Manufacture Safety Data Statement may have to conform to the formatting and legislative requirements of the host country.)

4. QUALITY ASSURANCE PROVISIONS

4.1 Product conformance. The product provided shall meet the salient characteristics of this Commercial Item Description, conform to the producer's own drawings, specifications, standards, and quality assurance practices, and is the same product offered for sale in the commercial market. The Government reserves the right to require proof of such conformance prior to the first delivery and thereafter as may be otherwise provided for under provisions of the contract. For DMDNB product which has been sold on the market for less than two years, the manufacturer shall have to demonstrate process stability and to verify product quality by submitting analysis data from the most recent 15 consecutive production batches. Procurement shall not proceed until these data are evaluated and approval is obtained by the procuring agency. (See 6.2 and 6.5).

4.2 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of inspections for the verification of all salient characteristics as specified in Section 2 of this CID. The supplier may use his own or any other facility suitable for the performance of the inspection requirements specified herein, unless otherwise specified in the contract or disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the CID where such inspections are deemed necessary to assure supplies and services conform to the prescribed requirements.

4.2.1 Bid sample submission. Unless otherwise specified, a sample of 100 grams of DMDNB shall be submitted for inspection and approval in accordance with the terms of the procurement contract. The sample will be subjected to any or all of the quality inspections listed in this CID for verification of compliance to any or all requirements listed in this CID.

4.2.2 Test and inspection results. The contractor shall provide a summary of product test results (or an appropriate form of document of analysis in accordance with acceptable industry practices) to verify conformance to the salient characteristics requirements. (See 2). The contractor shall maintain a traceable record of inspection test data adhering to general laboratory practices for future reference if required.

4.3 Sampling techniques and inspection procedures. When not specified in this CID, the DMDNB shall be inspected using acceptable industry-wide sampling techniques and test procedures such as ASTM and ACS sampling test procedures. Also, unless otherwise specified herein, all chemicals and reagents shall be ACS Grade or Reagent Grade chemicals. Tests for

the liquid chromatography and the Fourier Transform Infrared Spectroscopy shall be performed as specified below (see 6.3).

NOTE: The test descriptions described below are not intended to be used as step-by-step laboratory procedures, rather they are intended to define the test method used to obtain the required characteristics. The user should prepare their own laboratory procedures taking into consideration and complying with all aspects of the following test method descriptions. In preparation of step-by-step laboratory procedure, the user should take into account safety precautions from the manufacturer, industry safety and health practices, and also determine the applicability of safety and environmental regulatory limitations. The test descriptions below do not purport to address all the safety concerns, if any, associated with their use. It is the responsibility of the user of the procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

4.3.1 Fourier transform infrared spectroscopy (FTIR). This method is strictly qualitative. Visual comparison of infrared spectrum of the sample with the reference spectra (lower graph, Figure 1) serves to identify the raw material. Each raw material will have a unique spectrum and any bands not specific to that spectrum will serve as a flag to indicate contaminants, or a spectrum that does not match the reference will serve to identify the sample as incorrectly labeled. Material with a spectrum that does not match that of the standard should not be accepted. It is recommended that FTIR be conducted by the receiving agency during acceptance of raw material.

4.3.1.1 FTIR analysis procedure.

(a) Apparatus. Fourier Transform Infrared Spectrometer, Nicolet 5DX, or equivalent, programmed in accordance with manufacturer's instruction for this specific application.

(b) Sample preparation. Add approximately 0.5 g of potassium bromide (KBr) and just a small amount (approximately 2.5 mg) of sample to a stainless steel chamber. Add a stainless steel ball bearing to the chamber and place a lid to the chamber. Place the chamber in the sample holder of the Wig-L-Bug vibrator (or equivalent), and vibrate for a minimum of 1 minute. Take out the ball bearing and pour the sample into a stainless steel die. Press the sample into the die with the hydraulic jack to a minimum of 20,000 psi. Remove the ends from the die DMDNB and place the die in a sample holder of the FTIR spectrometer for analysis.

(c) Analysis. Following the manufacturer's instruction, go to the available libraries in the FTIR computer, choose the correct library (where reference spectra for other solids, and for DMDNB are stored), select Analyze, then select Search. The top five spectrum matches will be shown on the screen. Select Stack Display of the sample graph stacking the sample spectrum onto the reference spectrum for visual comparison. Annotate all spectra before print out. Print the stack spectra (sample plus reference spectra) for visual comparison. Visually compare the spectrum of the sample with the reference spectrum for identification of the sample material.

NOTE: Sample spectrum that has been determined through comparison and analysis to be an exact match of the spectra in Figure 1 can be stored in the library of the FTIR computer for use as a standard reference.

4.3.2 Purity assay of DMDNB by HPLC (primary method). The DMDNB shall be analyzed by a reversed phase HPLC method using UV detection at 220 nm. The HPLC system used should be comprised of a column, mobile phase, pumping system, sample injection system, detection system and data integrator system.

4.3.2.1 Apparatus and devices. The following apparatus and devices or their equivalents shall be used to perform the HPLC assay analysis:

(a) Analytical column: Supelco Supelcosil LC-8-DB 4.6 mm x 25 cm p/n 5-8354
(b) Isocratic pump: Waters 2690 Alliance
(c) Detector: Waters Photo Diode Array 996 or Waters UV 484
(d) Integrator: Hewlett Packard 3395

4.3.2.2 Chemical reagents. Chemical reagents used in the test shall be HPLC grade acetonitrile, and deionized or HPLC grade water.

4.3.2.2.1 DMDNB. DMDMB used to prepare the standard solutions shall have a minimum purity of 99.99%. The impurity of the standards used shall be independently confirmed by a reputable chemical laboratory. If a standard with a purity of 99.9% or above is not available, a less pure standard can be used and the analysis result shall be corrected by multiplying the obtained purity by the fractional purity of the less pure standard. Whenever a sample which is purer than the certified standard is found, this purer sample can be and should be used as the new standard provided its assay purity is documented, and is traceable to an independently certified standard.

4.3.2.2.2 Mobile phased used. The mobile phase shall be 400 mL of HPLC grade acetonitrile in solution with 600 mL of deionized or HPLC grade water.

4.3.2.2.3 Diluent used. The diluent used shall be 400 mL of HPLC acetonitrile in solution with 600 mL of deionized water.

4.3.2.3 Preparation of standard and sample.

4.3.2.3.1 Standards.

4.3.2.3.1.1 Primary standard solution. Accurately weigh and record 0.1000 gram of DMDNB standard to the nearest 0.0001 gram and transfer the weighed sample to a 100 mL volumetric flask. Dilute to volume with acetonitrile and mix the contents thoroughly.

4.3.2.3.1.2 Secondary standard solution. Pipette 10 mL primary standard solution into a 100 mL volumetric flask. Dilute to volume with acetonitrile and mix thoroughly.

4.3.2.3.1.3 Injection standards. Pipette 5, 10, 15, and 20 mLs of the above secondary standard solution respectively into four 50 mL volumetric flask. Dilute to volume with Diluent (see 4.3.2.2.3) and mix thoroughly.

4.3.2.3.2 Samples.

4.3.2.3.2.1 Primary standard solution. Accurately weigh and record 3 grams of sample to the nearest 0.001 gram, and transfer the weighed sample to a 100 mL volumetric flask. Dilute to volume with acetonitrile and a small amount deionized water if necessary, and mix thoroughly.

4.3.2.3.2.2 Secondary standard solution. Pipette 5 mLs of the primary sample solution to a 100 mLs volumetric flask. Dilute to volume with Diluent. Mix thoroughly.

4.3.2.3.2.3 Injection sample. Pipette 2 mL of the secondary sample solution to a 100 mL volumetric flask. Dilute to volume with Diluent. Mix thoroughly.

4.3.2.4 HPLC system setting. The HPLC and its accessories shall be set to the following operating parameters:

UV detector: Wavelength – 220 nm; AUFS – 0.1; Filter – 1.0
Autosampler injector – Injection volume – 20 µL; Run time – 15 min.
Integrator: Attention – 6; Thresh – 5
Cht spd – 0.5; Pk wd – 0.04; AR REJ – 0
Isocratic pump: Flow rate – 1.5 mL/min

4.3.2.5 Analysis: Put an aliquot of each standard and sample in vials and put them into the Autosampler in the following sequence:

Vial 1 – 5 mL/50 mL Standard
Vial 2 – 10 mL/50 mL Standard
Vial 3 – 15 mL/50 mL Standard
Vial 4 – 20 mL/50 mL Standard
Vial 5 – Injection sample XXX

4.3.2.5.1 Standard and sample injection. Inject each standard and sample in duplicate.

4.3.2.5.2 Purity calculation. Percent purity shall be calculated as follows: From the printout of the integrator, transfer the area counts for the DMDNB peak of the standards at the different dilutions into the Excel spreadsheet in their corresponding cells. Similarly, transfer the area counts of the sample into their respective cells. Put the standard and sample weights into their respective cells. Input date, sample number, analyst, and time, etc. in their respective cells.

4.3.2.5.3 Assay value. The percent assay value is calculated automatically based in the following equation:

$$\%\text{DMDNB} = \frac{(\text{Sample area count}) \times (\text{Slope of DMDNB calibration curve}) \times (100)}{(\text{Sample weight in mg/100mL})}$$

NOTES:
(1) If a DMDNB standard with a purity of less than 99.9% is used for formulating the standard solutions, the resulting % purity shall be corrected by multiplying the analysis result with the fractional purity of the less-pure standard used. (See 4.3.2.2 above).

(2) Samples containing large amounts of chloride may be difficult to dissolve in acetonitrile with a small amount of water. For these samples it is necessary to add acetonitrile and water alternately in volumetric flask until the sample dissolves.

4.3.3 Purity assay of DMDNB by DSC (alternate method). The DMDNB shall be analyzed by differential scanning calorimetry (DSC).

4.3.3.1 DSC assay. The following apparatus and devices or their equivalents shall be used to perform the DSC assay:

(a) Aluminum hermetic DSC pans and lids.
(b) Analytical balance.
(c) TA Instruments DSC with operating system and Universal Analysis.
(d) Tweezers.
(e) Spatula.
(f) Encapsulating press.

4.3.3.2 Reagents.

(a) None.

4.3.3.3. Test control strategy.

4.3.3.3.1 DMDNB sample analysis. A DMDNB standard of known purity will be tested using the same preparation method and test procedure as for the sample on each day that the sample is analyzed.

4.3.3.4. Sample preparation.

(a) Tare sample pan on the analytical balance.
(b) Weigh 2.5 ± 0.5 mg of DMDNB into pan.
(c) Place a lid on the pan and transfer the pan to the well of the lower die on the encapsulating press.
(d) Place the spacer onto the top die with the big opening facing down. Lower the handle until it stops. Raise the lever.
(e) Remove the spacer and lower the handle of the press until it stops to seal the pan. Raise the lever.
(f) Use tweezers to transfer the pan from the press to the DSC auto sampler unit.

4.3.3.5. Procedure.

(a) Program the DSC to run the sample using a ramp of 5C° per minute from 35°C to 350°C.
(b) When the DSC run is complete, open Universal Analysis to obtain the scan.
(c) Click on “Analyze” in the top toolbar. Click on “Purity” in the drop down menu then click “Analyze” in the drop down menu.

(d) Drag the red markers to the beginning and end of the second endothermic peak. Right click the mouse. Click the "Accept Limits"

(e) Type in "176.2" for the molecular weight and click OK.

(f) Print the DSC scan.

4.3.3.6. Calculation. All calculations are performed by the software within the DSC instrument.

5. PACKAGING

5.1 Packaging. Preservation, packing, and marking shall be as specified in the contract order (see 6.1.d).

6. NOTES

6.1 Ordering data. Procurement documents should specify the following:

(a) Title, number, and date of this CID.

(b) Issue of Department of Defense Index of Standards and Specifications (DODISS) to be cited in the solicitation, and if required, the specific issue of the individual documents referenced.

(c) Quantity required.

(d) Packaging and marking requirements.

6.2 Sources of supply. DMDNB (2,3-dimethyl-2,-3-dinitrobutane) has been previously procured from Hampshire Chemical Corporation, 5529 US 60 East, Owensboro, Kentucky 42303, and found to be acceptable with regard to its chemical and physical properties, purity of the material, etc. for general laboratory applications. DMDNB from a new supplier intended for a particular application should be evaluated (qualified) for the specific application.

6.3 Equivalent test method. Prior approval of the Contracting Officer is required for use of equivalent test methods. A description of the proposed method should be submitted through the Contracting Officer to: Commander, ATTN: RDAR-EIQ-MG, ARDEC, Picatinny Arsenal, NJ 07806-5000. This description should include, but not be limited to, the procedures used, the accuracy and precision of the method, test data to demonstrate the accuracy and precision and drawings of any special equipment required.

6.4 Source documents. The Code of Federal Regulations (CFR) is available by mail order from the Superintendent of Documents, ATTN: New Order, PO BOX 371954, Pittsburgh, PA 15250-7954.

6.5 Technical agency. ARDEC, Picatinny Arsenal, (RDAR-EIQ-MG), is the technical agency that is responsible for the preparation and revision of this Commercial Item Description. This CID should not be revised without the expressed concurrence and approval from the Technical Agency. All correspondence to the Technical Agency should be submitted through the contracting officer to: Commander, ARDEC, ATTN: RDAR-EIQ-MG, Picatinny Arsenal, NJ 07806-5000.

Custodian:
Army – AR

Preparing Activity:
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