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NATO STANDARD

AOP-4798

ENERGETIC MATERIALS, SPECIFICATION FOR DADNE (1,1-DIAMINO-2,2-DINITROETHYLENE/FOX-7)

Edition A, Version 1

DATE

RATIFICATION DRAFT 1



NORTH ATLANTIC TREATY ORGANIZATION

ALLIED ORDNANCE PUBLICATION

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NATO LETTER OF PROMULGATION

[Date]

1. The enclosed Allied Ordnance Publication AOP-4798, Edition A, Version 1, ENERGETIC MATERIALS, SPECIFICATION FOR DADNE (1,1-DIAMINO-2,2-DINITROETHYLENE/FOX-7), which has been approved by the nations in the CNAD AMMUNITION SAFETY GROUPE (A/C 326), is promulgated herewith. The agreement of nations to use this publication is recorded in STANAG 4798.
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Lieutenant General, GRC (A)
Director, NATO Standardization Office

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CHAPTER 1 INTRODUCTION

1.1. AIM

The aim of this agreement is to ensure that DADNE (FOX-7) shall possess properties which make it suitable for military use and to provide, within NATO, an acceptable basis for the procurement and certification of DADNE.

1.2. AGREEMENT

Participating nations agree that DADNE proposed for military use, except when ordered for particular uses, shall meet all the requirements of Table A-1 of this document. The test procedures used to verify the requirements of Table A-1 are described in Annex B. The methods used and the results obtained shall be quoted on the test certificate of Annex C.

1.3. GENERAL

1.3.1. Composition

DADNE shall consist of 1,1-diamino-2,2-dinitroethylene corresponding to the chemical formula $C_2H_4N_4O_4$ and to the structural formula given in Figure 1 below.

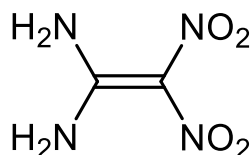


Figure 1: DADNE structural formula

1.3.2. Manufacturing process

Any data or information concerning the proposed manufacturing process must be provided in confidence at the request of the purchaser. Any deviation from this accepted process must be noted and the product thus manufactured put aside until the purchaser has determined its approval or rejection.

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1.3.3. Appearance and granulation

1. The material shall be in the form of yellow crystals and shall conform to the granulation requirements specified in the contract between manufacturer and purchaser. In his order, the purchaser must clearly specify the granulation he requires.
2. At the purchaser's request, the manufacturer shall provide microphotography of DADNE crystals. Magnification of the microphotography shall permit clear viewing of individual particle shape (needles, spheres, irregular crystals etc.).

1.3.4. Sampling

The sample size taken from each lot should be at least 200 g. The sampling technique must lead to a representative sample of each lot and has to be agreed to by the purchaser.

1.3.5. Drying procedure

See test procedures, Annex B.

1.3.6. Rejection criteria

Failure of a representative sample of DADNE to meet the requirements specified in Table A-1 shall cause rejection of the lot from which the sample was taken.

1.3.7. Warning

This AOP calls for the use of substances and test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way, absolves the user from statutory obligations relating to health and safety at any stage during use.

Refer to the information given in the safety data sheet and national regulations for each of the components used throughout this AOP.

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**ANNEX A TO
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ANNEX A PHYSICAL AND CHEMICAL PROPERTIES
--

A.1. MINIMUM REQUIREMENTS

Table A-1 shows the minimum requirements for the properties of DADNE (FOX-7).

Characteristics	Required value	Test method (see Annex B)
Purity	min 98% DADNE	B-1
Acidity (as sulphuric acid)	max 0.01%	B-2
Insoluble matter in DMSO	max 0.05%	B-3
Gritty matter		
On sieve US 40 (0.425 mm)	Nil	B-4
On sieve US 60 (0.250 mm)	max 1 piece/10g	B-4
Decomposition temperature	informative (typically first peak between 200- 240°C and second peak above 250°C)	B-5
Thermal stability	max 1.0 cm ³ /g after 40 hours at 120°C	B-6

Table A-1: Minimum requirements for properties of DADNE

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ANNEX B TEST PROCEDURES

B.1.a DETERMINATION OF DADNE PURITY BY HPLC ANALYSIS

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the purity of DADNE.

NOTE 1: This test procedure is recommended for all DADNE (crystalline and crude) if the sample is unknown. For quality check, test procedure B.1.b. is recommended.

2. PRINCIPLE

Water/ammonia solutions of accurately weighed samples of DADNE are analysed using liquid chromatography (LC) combined with spectroscopic detection (UV) at 250 nm. The DADNE purity is calculated comparing the area of the DADNE peak with an external purified standard. Figure 1 shows the preparation pathway and the usual by products/impurities. Figure 2 shows a chromatogram using the example method as described in this AOP.

NOTE 2: Other methods and columns can be used after thorough evaluation. C18 columns did not work well with the analytes due to their polarity.

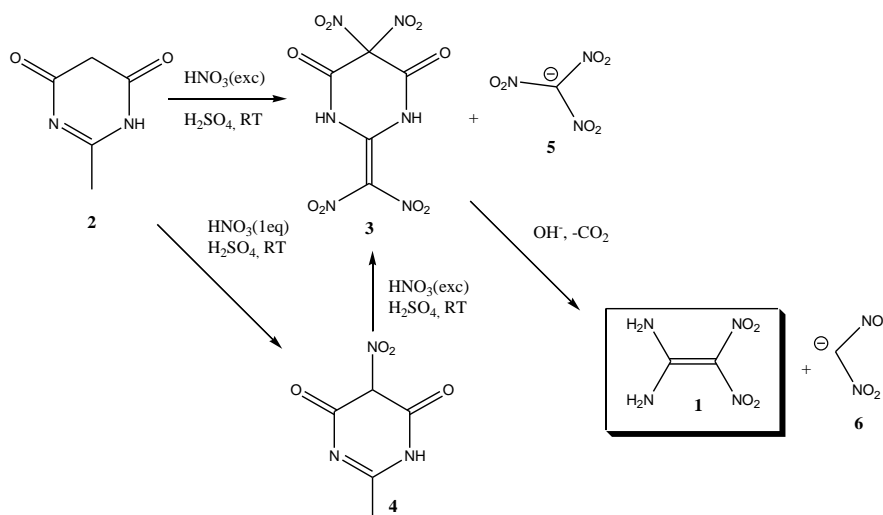


FIGURE 1: DADNE (1) and by-products from synthesis. 2-methyl-pyrimidine-4,6-dione (2), 2-dinitromethylene-5,5-dinitro-dihydro-pyrimidine-4,6-dione (3), 2-methyl-5-nitro-1H-pyrimidine-4,6-dione (4), nitroform (5) and dinitromethane (6).

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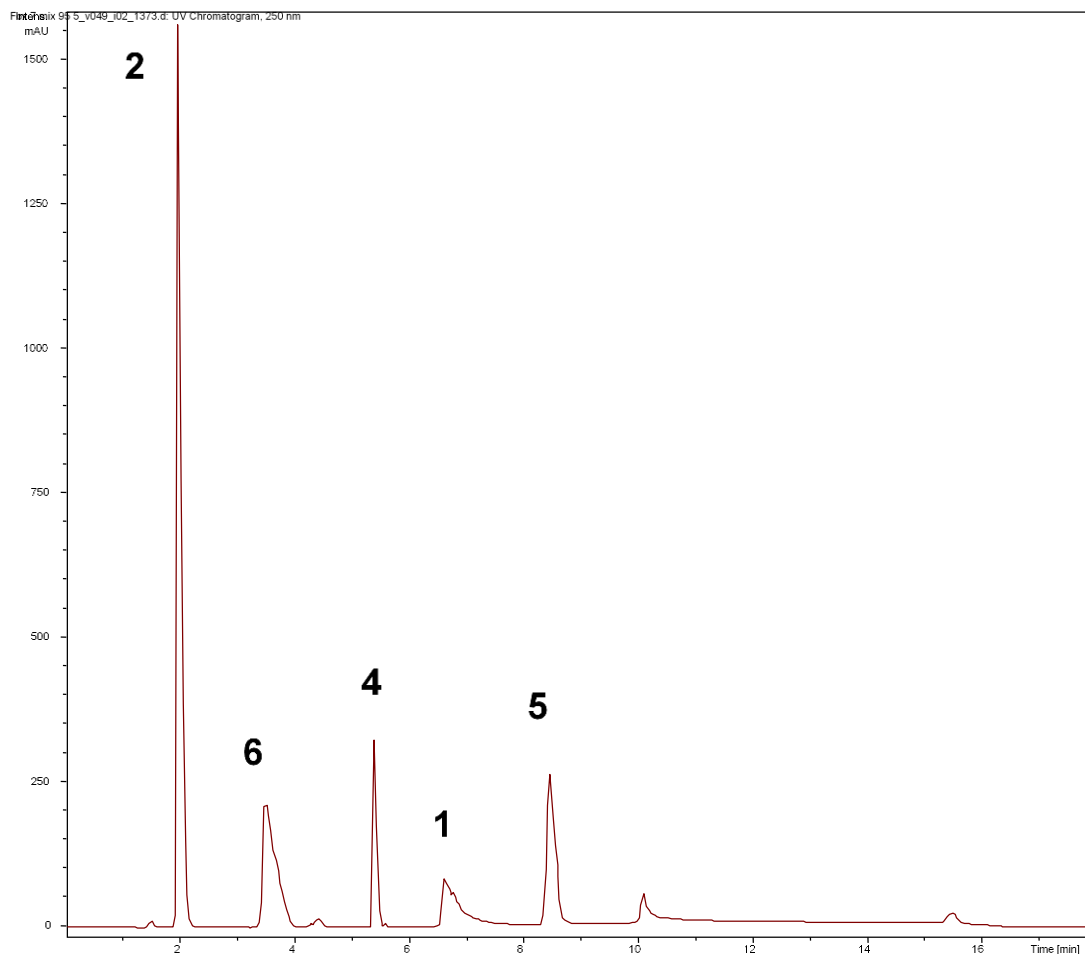


FIGURE 2: Chromatogram of DADNE (1) and by-products from synthesis. 2-methyl-pyrimidine-4,6-dione (2), 2-methyl-5-nitro-1H-pyrimidine-4,6-dione (4), nitroform (5) and dinitromethane (6). 2-dinitromethylene-5,5-dinitro-dihydro-pyrimidine-4,6-dione (3) is not shown in the chromatogram since it is not stable.

3. APPARATUS

- a. A porous graphitic carbon (PGC) column that yields suitable peak shape, resolution and retention (e.g. Hypercarb PGC, 100 mm × 4.6 mm, 3 μm particles from Thermo Quest, UK). The temperature is maintained at 55°C.

NOTE 3: The temperature (55°C) was chosen to optimize the peak shape. At room temperature, peaks tend to be broader.

- b. The mobile phase is a gradient mixture of two eluents. Eluent A consists of 100% water. Eluent B consists of 93.3% acetonitrile, 4.7% water, and 2%

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ammonia (v/v). The analysis starts with A/B 95/5 for 2 min with a flow rate of 1 cm³/min. During the next 10 min the mixture is changed to A/B 0/100 and then held there for 3 min. HPLC parameters (e.g. flow rates, mobile phase ratios, injection volumes, run times) may be adjusted to maintain adequate resolution and shape of the DADNE peak and any impurities.

- c. UV detection is done at 250 nm (e.g. by a Waters UV detector model 996 photodiode array detector).

4. CHEMICALS

- a. Acetonitrile, HPLC-grade or equivalent
- b. Deionized Water, HPLC-grade or equivalent
- c. Ammonia, water solution 25%, analytical grade or equivalent

5. STANDARD PREPARATION

DADNE shall be purified using e.g. preparative HPLC or by at least two recrystallizations to ensure absolute purity.

NOTE 4: Samples with lower purity values (e.g. min 98.0%) may also be used as long as the actual purity value is known. Standard mass values will then be adjusted accordingly for the purity of the material.

Recrystallization: Add 1 g of DADNE to 100 cm³ of boiling water. Boil under stirring for 5 min. Add 10 cm³ of water and boil for another 5 min, if required. Filter while hot. Dry the filtered powder to a constant weight.

Standard solution preparation: Prepare five calibration solutions containing 10, 20, 30, 40, and 50 µg DADNE in 1 cm³ of 0.1 % ammonia/water solution (v/v).

6. SAMPLE PREPARATION

- a. Obtain samples of DADNE that have been dried to constant weight in a 50°C standard oven.
- b. For each batch to be tested, prepare three 30 µg/cm³ samples in a 0.1% ammonia/water solution (v/v). Use high density polyethylene beakers in order

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to avoid reactions between beaker and analytes, which may occur by using glass beakers. 5 μ L of this solution is injected.

7. RUN SAMPLES

Verify all HPLC parameters are correct and allow the system to equilibrate. Enter necessary information into data acquisition program. Run analysis on standard vials. The five standards shall be used to generate a linear equation using a least-squares regression. The R², or the fraction of the total squared error of the linear equation, shall not be less than 0.995. If this is not the case, analysis of further samples shall not be continued until a series of standards can be analyzed to provide such a linear equation. Enter necessary information into data acquisition program. Run analysis on sample vials. Run also a sample without DADNE in the same way as above to correct the result from baseline noise.

8. CALCULATION

Computer software package is used to establish a calibration equation based on the concentration of DADNE in the three calibration standards and the HPLC chromatographic peak areas obtained from the analyses of these standards. Using the calibration equation, and the peak areas obtained for the test samples, the concentrations of the components can be determined. Results will be reported as % purity DADNE.

9. REFERENCES

Latypov, N. V. *et al.* On the Synthesis of 1,1-Diamino-2,2-dinitroethene (Fox 7) by nitration of 4,6-Dihydroxy-2-methylpyrimidin. *Organic Process Research & Development* 11(1), 2007, 56-59.

B.1.b DETERMINATION OF DADNE PURITY BY UV-VIS ANALYSIS

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the purity of recrystallized DADNE, where the by-products described in B.1.a have never been observed.

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2. PRINCIPLE

Water solutions of accurately weighed samples of DADNE are analysed with UV-VIS spectrophotometry. The DADNE purity is calculated from a calibration towards a purified standard, *vide infra*. All observations of by-products stated above apply, see B.1.a.

3. APPARATUS

- a. A well maintained UV-VIS spectrophotometer is used (e.g. Lambda 650 from PerkinElmer, SE). The analyses are performed at room temperature. The lamps of the instrument should be lit well in time before the analysis according to the specifications of the instrument (e.g. 1 hour).
- b. UV detection is performed at 350 nm in 1 cm quartz cuvettes.

4. CHEMICALS

- a. Deionized water, spectrophotometric grade or equivalent

5. STANDARD PREPARATION

DADNE shall be purified by recrystallization from water. If crude DADNE is used, it should be recrystallized twice.

Recrystallization: Add 1 g of DADNE to 100 cm³ of boiling water. Boil under stirring for 5 min. Add 10 cm³ of water and boil for another 5 min, if required. Filter while hot. Dry the filtered powder to a constant weight.

- a. Calibration curve and linearity check
Standard solution preparation: Prepare five calibration solutions containing 5, 6, 7, 8, and 9 mg DADNE in 1 dm³ of water. The calibration samples should be weighed to an accuracy within ± 0.4 to the milligram to ensure separate calibration points. The weights should be recorded with two decimals to the mg. Obtain a calibration curve. A correlation coefficient of 0.99 is recommended. Remove the end-points, if they are not within the linear range. If so, use five points within the linear range and weigh the DADNE to an accuracy of ± 0.2 to the milligram.

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This calibration curve can be used for the quantification of DADNE in the sample. A new calibration curve should be obtained, if the lamps have been switched off.

NOTE 5: The dissolution of DADNE is slow, especially for larger crystals. Stirring overnight in 750 cm³ of water and subsequent transfer and dilution in a volumetric flask is recommended. This applies to calibration and samples.

b. One-point calibration

For routine analyses, a one-point calibration can be used. The weight should be recorded with two decimals to the mg. Then prepare a solution of 7 ± 0.2 mg of DADNE in 1 dm³ of water. The accuracy required in the weighing depends on the linearity of the spectrophotometer, *vide supra*.

6. SAMPLE PREPARATION

- a. Obtain samples of DADNE that have been dried to constant weight in a 50°C standard oven.
- b. For each lot to be tested, prepare three 7 ± 0.2 mg samples in a 1 dm³ of water. The weights should be recorded with two decimals to the mg.

7. RUN SAMPLES

Measure the absorbance at 350 nm and record the results.

8. CALCULATION

Calibration curve: Use the quantification feature in the software of the instrument to obtain the concentration of DADNE in the lot, if available. If not, calculate it by other means.

One-point calibration: Use the absorbance at 350 nm from a reference standard at the same concentration (e.g. 7 mg of DADNE in 1 dm³ of water).

Calculation of DADNE purity:

$$\% \text{ DADNE} = \text{sample/reference} \times 100$$

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Where: sample = concentration or absorbance from sample of DADNE lot; reference = concentration or absorbance from reference standard.

B.2. ACIDITY

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the acid content in DADNE.

2. PRINCIPLE

The acidity of DADNE is determined by dissolving the sample completely in dimethyl sulfoxide (DMSO). After adding of deionised water the solution is titrated with sodium hydroxide solution. The acidity is calculated as equivalent to percentage sulfuric acid.

3. APPARATUS

- a. Titration equipment with pH-electrode. Choose the dynamic titration mode if available. An example of a titration equipment setup is shown in Figure 3.
- b. Magnetic stirrer
- c. Burette size: apparatus specific

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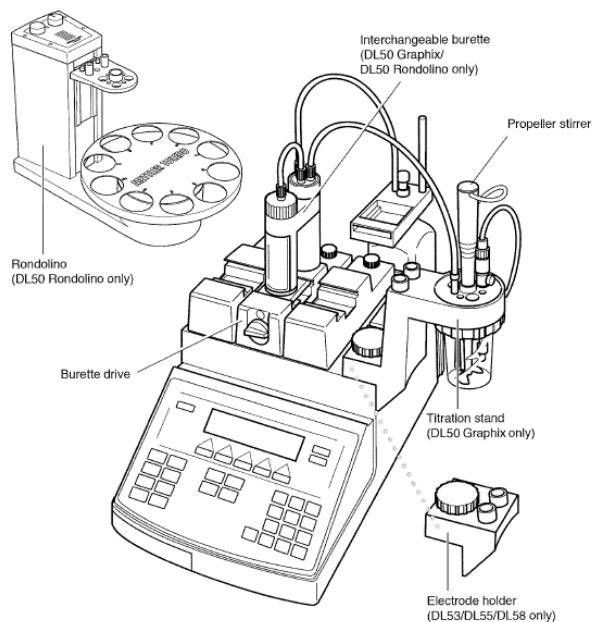


FIGURE 3: An example of a titration equipment setup.

4. CHEMICALS

- a. Dimethyl sulfoxide (DMSO), PA grade or equivalent
- b. Deionised water
- c. Sodium hydroxide solution 0.05N, standardised against e.g. potassium hydrogen phthalate

5. PROCEDURE

- a. Calibrate the titration equipment before use.
- b. Place 10.0 (± 0.01) g of DADNE, dried to constant weight in a standard oven at 50°C, in a 250 cm³ beaker.
- c. Add 40 cm³ of dimethyl sulfoxide (DMSO) to the beaker with DADNE and stir until completely dissolved.
- d. Add 120 cm³ deionised water.
- e. Titrate with sodium hydroxide solution until the equivalent point is detected.
- f. Prepare and titrate a sample without DADNE in the same way as above.

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6. CALCULATION

The acidity is calculated on the basis to percentage sulphuric acid as follows:

$$\text{Percentage acid (as sulfuric acid)} = \frac{(S-B) \times N \times 4.9}{W}$$

Where:

S = volume of sodium hydroxide solution used in sample (cm³)

B = volume of sodium hydroxide solution used in blank (cm³)

N = normality of sodium hydroxide solution

W = weight of sample (g)

4.9 = factor for sulfuric acid.

B.3. INSOLUBLE MATTER

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the insoluble matter in DADNE.

2. PRINCIPLE

Insoluble matter is determined by dissolving DADNE in dimethyl sulfoxide (DMSO) and filtering the solution through a glass filter crucible. The insoluble matter in the glass filter crucible is dried and weighed.

3. APPARATUS

- a. Glass filter crucible, porosity G3 (10-20 micron)
- b. Vacuum filtering equipment suitable for the glass filter crucible
- c. 200 cm³ beaker
- d. Heated water bath, 80-90°C

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- e. Heated standard oven, 50°C
- f. Desiccator

4. CHEMICALS

- a. Dimethyl sulfoxide (DMSO), PA grade or equivalent
- b. Acetone, technical grade or equivalent

5. PROCEDURE

- a. Obtain samples of DADNE that have been dried to constant weight in a 50°C standard oven.
- b. Place 10.0 (± 0.01) g of DADNE in a 200 cm³ beaker. Note weight as W.
- c. Add 40 cm³ of DMSO to the beaker with DADNE.
- d. Heat the solution using a water bath, 80-90°C, until all DADNE is dissolved in the DMSO.
- e. Weigh the empty glass filter crucible. Note weight as W1.
- f. Filter material through a pre-tarred porous crucible, porosity G3 (10-20 micron).
- g. Wash the residue on the filter with acetone. Dry the crucible in the oven at 50°C until constant weight is obtained.
- h. Cool the crucible in a desiccator and weigh it to an accuracy of 0.01g. Note weight as W2.

6. CALCULATION

Calculation of undissolved solids:

$$\text{Insoluble matter in DMSO}(\%) = \frac{(W2 - W1) \times 100}{W}$$

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Where W1 = weight of the crucible (g)
 W2 = weight of tare and undissolved solids (g)
 W = DADNE sample weight (g)

B.4. GRITTY MATTER

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the gritty matter in DADNE.

2. PRINCIPLE

The gritty matter is determined by dissolving DADNE in dimethyl sulfoxide (DMSO) and filtering the solution through sieves. The number of gritty particles are then determined.

3. APPARATUS

- a. US sieve no. 40 (0.425 mm) and US no. 60 (0.250 mm), diameter 3 inch (76 mm)
- b. Water bath
- c. 200 cm³ beaker
- d. Heated standard oven, 50°C

4. CHEMICALS

- a. Dimethyl sulfoxide (DMSO), PA grade or equivalent
- b. Acetone, technical grade or equivalent

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5. PROCEDURE

- a. Obtain samples of DADNE that have been dried to constant weight in a 50°C standard oven.
- b. Place 10.0 (\pm 0.1) g of DADNE in a 200 cm³ beaker.
- c. Add 40 cm³ of DMSO to the beaker with DADNE.
- d. Heat the solution using a water bath, 80-90°C, until all DADNE is dissolved in the DMSO.
- e. Pour the solution through the US sieve no. 40 and sieve no. 60 (connected to each other).
- f. Rinse the beaker with hot DMSO and pour the solution through the US sieve 40 and sieve 60.
- g. Wash the residue on the sieves with acetone.
- h. Dry the sieves in the oven at 50°C.
- i. The number of particles on the sieves are determined by ocular examination.

B.5. DECOMPOSITION TEMPERATURE

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the decomposition temperature of DADNE.

DADNE shows two solid phase transitions and two decomposition steps during thermal analysis. The second solid phase transition is also associated with loss of solvent residues from recrystallization (Figure 4). The first exotherm is believed to originate from the decomposition of amorphous parts in the DADNE crystal, whereas the second exotherm (see Figures 5-8) show the decomposition of the crystalline parts (Tichmanis *et. al.* (2004)).

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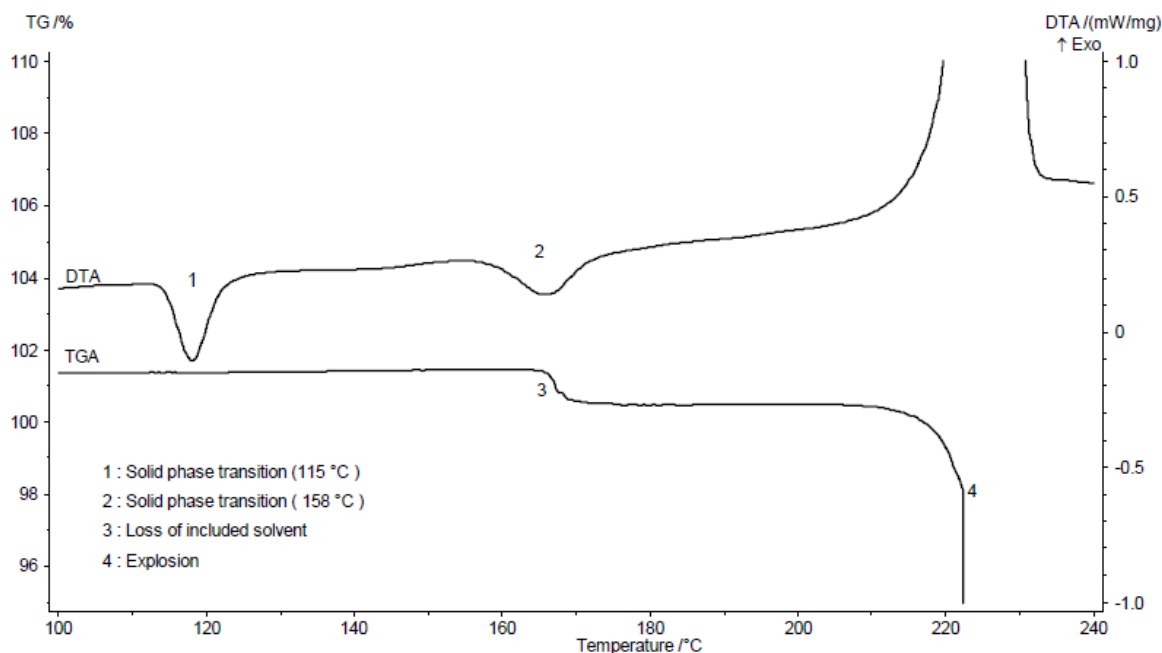


FIGURE 4: DADNE DTA/TG thermogram. Picture from reference Tichmanis *et. al.* (2004).

2. PRINCIPLE

The initial temperature of decomposition of DADNE is measured by Differential Scanning Calorimetry (DSC). The DSC method is described in more detail in STANAG 4515.

3. APPARATUS

- a. A Differential Scanning Calorimeter should be used which is capable of meeting the heating rate and sample size parameters as specified below.
- b. Crucibles (pans), e.g. 40 mm³ aluminum crucibles

4. CHEMICALS

- a. Indium for calibration of the DSC
- b. Nitrogen gas

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5. SAMPLE PREPARATION

- a. The DADNE sample is analysed as received.

6. PROCEDURE

- a. Make sure that the DSC is calibrated before use. The melting point of standard Indium should be within specifications.
- b. Place 0.3 - 1.0 mg of the DADNE sample in a suitable sized crucible.

NOTE 6: Depending on the type of crucible used, open or closed lid and the particle size of the DADNE sample, the shapes and onset of the exotherms may vary, see Figure 5-7.

- c. Heat at a rate of 10K/min to a temperature of at least 350°C in nitrogen atmosphere.

NOTE 7: If a heating rate of 5K/min is used, the onset temperature of the first exotherm is slightly lower, see Figure 8.

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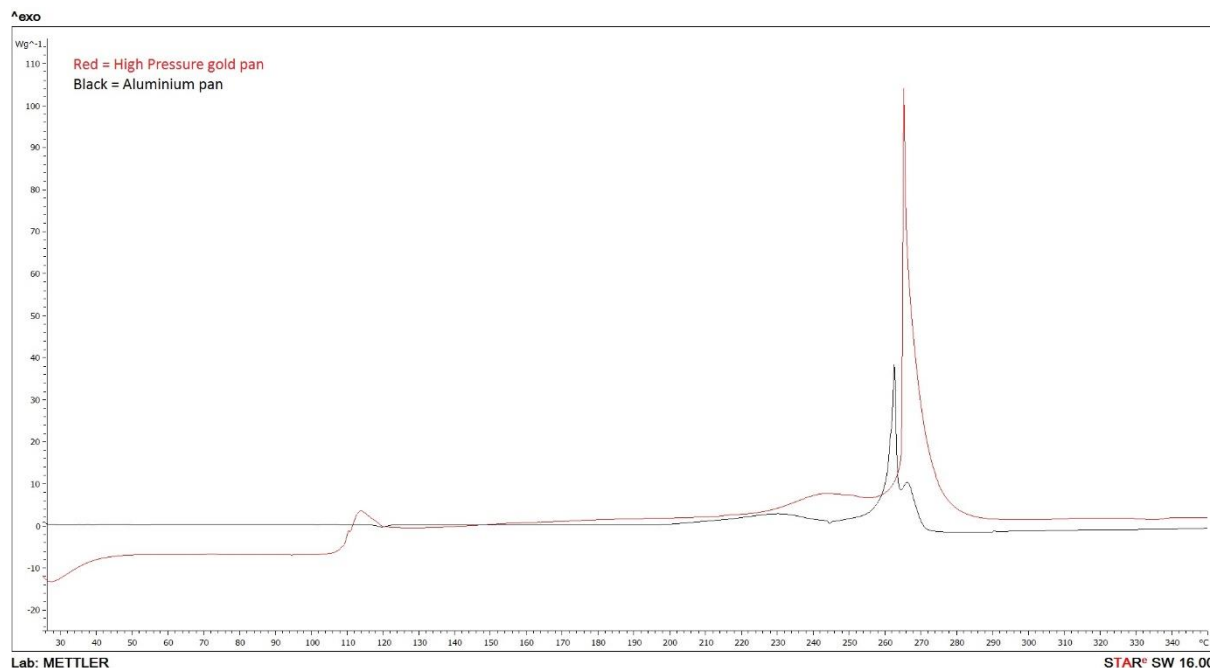


FIGURE 5: DADNE DSC thermogram. Red = High pressure gold pan. Black = 40mm³ closed aluminium crucible pan. There is a phase transition at 389 K (120 °C), which was studied by Evers et al. This transition leads to a drop in density of FOX-7 from 1.865 g/ml at 373 K to 1.825 at 393 K. This leads to an expansion that compressed the air in the high pressure cup, which raises the temperature for a moment. The Al low pressure cups are flexible enough to conceal this effect, when the lid is closed. With a pierced lid, it is not observed.

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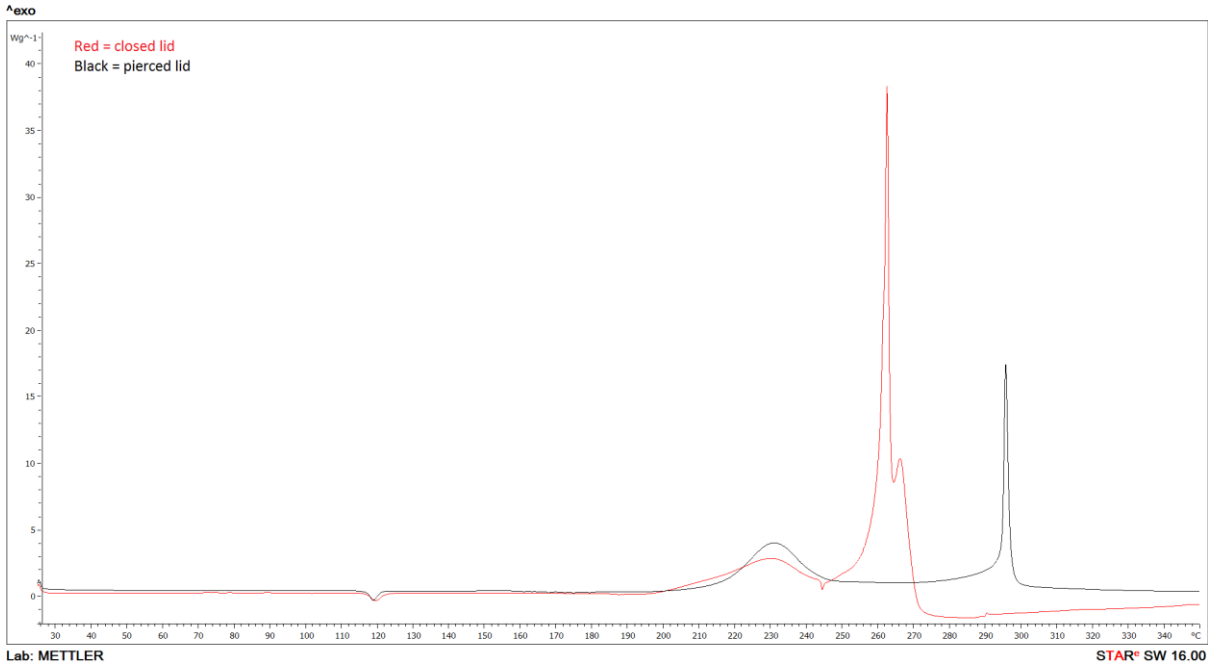


FIGURE 6: DADNE DSC thermogram. Red = closed lid. Black = open (pierced) lid. 40mm³ aluminium crucible pan.

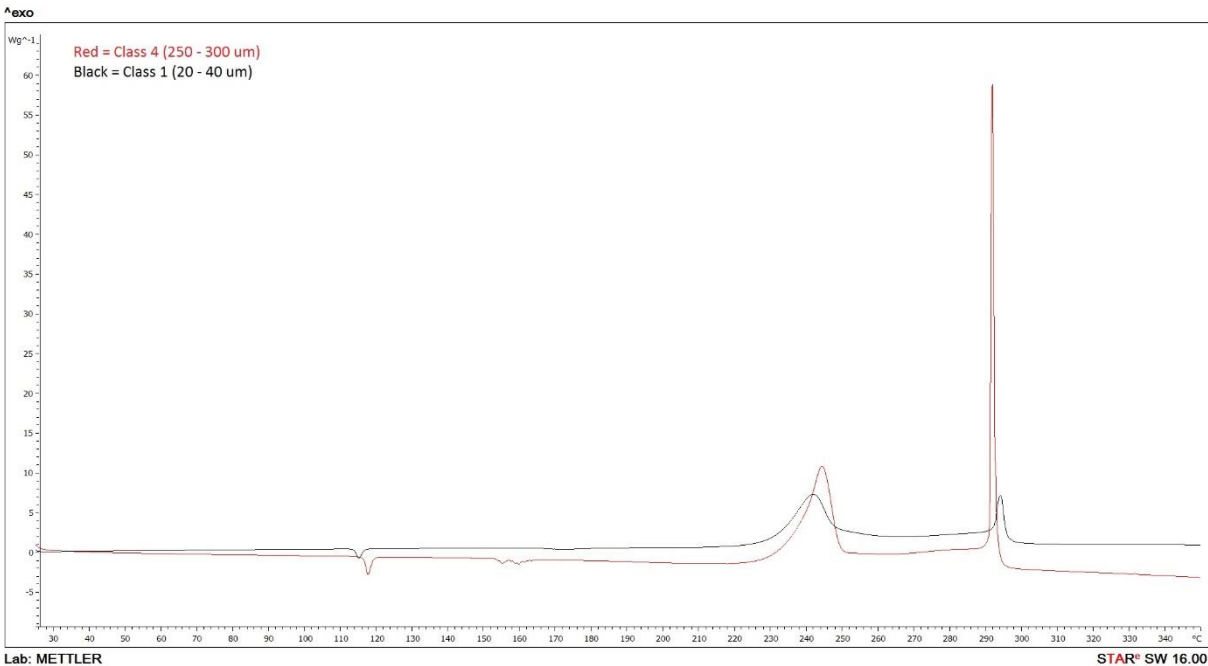


FIGURE 7: DADNE DSC thermogram. Red = large particles (250-300µm). Black = small particles (20-40µm). 40mm³ pierced aluminium crucible pan.

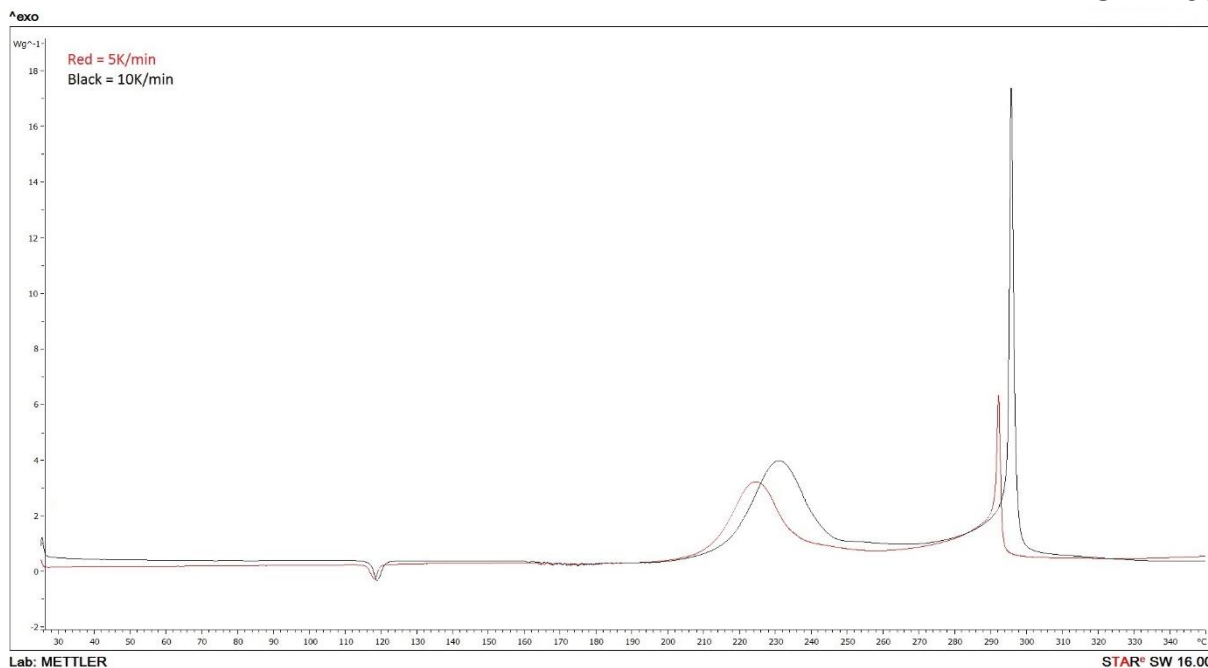
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Lab: METTLER STAR® SW 16.00
FIGURE 8: DADNE DSC thermogram. Red = 5K/min. Black = 10K/min. 40mm³ pierced aluminium crucible pan.

7. CALCULATION

a. Find the exothermal peaks and evaluate the initial temperature (T_i) for the exothermal peaks using the guidelines from STANAG 4515.

NOTE 8: T_i , the initial temperature, is defined in STANAG 4515 as the temperature at which the first deflection from the base line is observed for a chemical reaction or a phase transition.

b. Report the initial temperature(s), T_i , as the exothermal decomposition onset temperature(s) for the sample. Evaluation can normally be performed using the software running the DSC.

8. REFERENCES

Evers J. *et al.* α - and β -FOX-7, Polymorphs of a High Energy Density Material, Studied by X-ray Single Crystal and Powder Investigations in the Temperature Range from 200 to 423 K. *Inorg. Chem.* 45, 2006, 4996-5007.

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Ticmanis, U. *et al.* Kinetics and chemistry of thermal decomposition of FOX-7. *Proc. 35th Int. Ann. Conf. ICT*, 2004, p. 70/1-13.

B.6. VACUUM THERMAL STABILITY

1. SCOPE

The apparatus and test procedure herein described is suitable for the measurement of the thermal stability of DADNE.

2. PRINCIPLE

The vacuum stability test is used to assess the thermal stability of an energetic material by measuring the volume of gas evolved on heating the energetic material under specified conditions. This method is based on STANAG 4556 where a test temperature of 120°C and a duration of 40 hours are used.

3. APPARATUS

- a. A Vacuum Thermal Stability equipment that fulfils the STANAG 4556, Transducer method, procedure 1A.
- b. Heated metal block, 120 (\pm 0.2) °C
- c. Vacuum pump with capacity <10 mbar
- d. Heated oven, 50°C
- e. Glass tubes, which is capable of meeting the specifications in STANAG 4515 and sample size parameters as specified below.

4. SAMPLE PREPARATION

- a. Dry a minimum amount of 12 g of DADNE sample to constant weight in a 50°C standard oven.

NOTE 9: A minimum of two tests shall be conducted on each sample lot to be tested.

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- b. Weigh 5.0 (± 0.01) g of the dried DADNE sample and transfer it to the glass tube in duplicate.

5. PROCEDURE

- a. Connect the glass tube to the test equipment. Evacuate the equipment until the pressure is below 6.7 mbar. Let the equipment stabilize for minimum 30 minutes. Check the pressure regularly during this time to be sure that the equipment does not leak.
- b. Register the start pressure (P_1) and the temperature near the glass tube (t_1) and put the glass tube into the thermostatic bath. Check the temperature regularly during this time to ensure that the temperature is constant.
- c. After 40 hours remove the glass tube from the bath and let the equipment cool down for minimum 30 minutes. Register the final pressure (P_2) and temperature (t_2).

6. CALCULATION

Calculate the volume of gas, V , (at 273 K and 1.013 bar) per weight of the sample, m , liberated during the test, as follows:

$$\frac{V}{m} = \left[V_c + V_t - \frac{m}{d} \right] \cdot \left[\frac{P_2 \cdot 273}{273 + t_2} - \frac{P_1 \cdot 273}{273 + t_1} \right] \cdot \frac{1}{1.013} \cdot \frac{1}{m}$$

- Where :
- V = volume of gas liberated from the sample (cm^3 at 273 K and 1.013 bar)
 - V_c = volume of the transducer and adapter (cm^3)
 - V_t = volume of the glass tube (cm^3)
 - m = mass of the sample (g)
 - d = density of the sample (g/cm^3),
(Density of DADNE = $1.89 \text{ g}/\text{cm}^3$)
 - P_1 = calculated pressure at the beginning of the test (bar)
 - P_2 = calculated pressure at the end of the test (bar)
 - t_1 = room temperature at the beginning of the test ($^{\circ}\text{C}$)
 - t_2 = room temperature at the end of the test ($^{\circ}\text{C}$)

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**ANNEX C TO
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ANNEX C TEST CERTIFICATE FOR DADNE (FOX-7)

AOP-4798 – TEST CERTIFICATE FOR DADNE (FOX-7)

Report Reference Number

(Unique Reference Number)

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TEST SITE INFORMATION**Laboratory**

(Name of Laboratory)

Date:

(Date that form was completed)

Date Tested:

(Date of test period)

POC:

(Point of contact)

DADNE SAMPLE INFORMATION**Identification:**

(Trade name and/or Identity code)

Manufacturer:

(Name of Manufacturer)

Lot, Batch of Consignment Number:**Date of Manufacture or Receipt:****Quantity:****TEST RESULTS**

PROPERTY	METHOD	VALUE FOUND	UNIT
Purity	HPLC and/or UV-VIS		% DADNE
Decomposition temperature(s)	DSC		°C (initial temperature, T _i)
Acidity	Titration by pH electrode		% H ₂ SO ₄
Insoluble matters in DMSO			%
Gritty particles			No of particles retained on 0.25 mm sieve (US No. 60)
			No of particles retained on 0.425 mm sieve (US No. 40)
Thermal stability	Vacuum stability test		cm ³ /g
Comments:	Data Sent To: (Name and address of person receiving this information)		

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