NATO STANDARD

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ENERGETIC MATERIALS, SPECIFICATION FOR RDX (CYCLOTRIMETHYLENETRINITRAMINE)

Edition A, Version 1

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NORTH ATLANTIC TREATY ORGANIZATION

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NORTH ATLANTIC TREATY ORGANIZATION (NATO)

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NATO LETTER OF PROMULGATION

21 February 2023

1. The enclosed Allied Ordnance Publication AOP-4022, Edition A, Version 1, ENERGETIC MATERIALS, SPECIFICATION FOR RDX (CYCLOTRIMETHYLENETRINITRAMINE), which has been approved by the nations in the CNAD AMMUNITION SAFETY GROUP (CASG - AC/326), is promulgated herewith. The agreement of nations to use this publication is recorded in STANAG 4022.

2. AOP-4022, Edition A, Version 1, is effective upon receipt.

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4. This publication shall be handled in accordance with C-M(2002)60.

Dimitrios SIGOULAKIS
Lieutenant General, GRC (A)
Director, NATO Standardization Office

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RECORD OF RESERVATIONS

CHAPTER	RECORD OF RESERVATION BY NATIONS

Note: The reservations listed on this page include only those that were recorded at time of promulgation and may not be complete. Refer to the NATO Standardization Document Database for the complete list of existing reservations.

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RECORD OF SPECIFIC RESERVATIONS

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TABLE OF CONTENTS

CHAPTER 1 INTRODUCTION

1.1. AIM

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

1.2. AGREEMENT

Participating nations agree that RDX, proposed for military use, shall meet all the physical and chemical requirements of ANNEX A, Table 1 of this document. The test procedures used to verify the requirements of ANNEX A, Table 1 are described in ANNEX B and the rejection criteria shall be in accordance with ANNEX B, Paragraph 1.2 of this document.

1.3. USE

RDX is intended for use as an explosive material in primary, booster, and main-fill high explosives, as well as gun propellant formulations.

1.3. IMPLEMENTATION

This AOP is considered implemented when a nation has issued the necessary orders and instructions, putting the contents of this agreement into effect.

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ANNEX A REQUIREMENTS FOR RDX

A.1. MANUFACTURING PROCESS

Two types of RDX shall be defined as Type I or Type II, according to the manufacturing process. Any new or modified manufacturing process must be noted. Data or information concerning the manufacturing process must be provided in confidence at the request of the purchaser. Product manufactured by a new or modified process must be put aside until the purchaser has determined its approval or rejection. Type I and Type II RDX are defined as follows:

Type I = RDX made by the nitric acid process
Type II = RDX made by the acetic anhydride process

A.2. DEFINITION OF LOT

For continuous processes, a lot is defined as the total quantity of material that is offered for acceptance at one time. For batch processes, a lot shall consist of one batch or multiple, blended batches of RDX, produced by one manufacturer in accordance with the same specification and process, or same specification revision, under the same set of operating conditions. Each batch shall consist of that quantity of RDX that has been subjected to the same chemical or physical process.

A.3. DRYING PROCEDURE

Samples taken from a water-wetted supply must be dried before testing. The sample is dried in a thin layer in an oven at 60 +/- 5°C until a constant weight is reached.

A.4. PHYSICAL AND CHEMICAL PROPERTIES

The requirements for the physical and chemical properties for RDX (cyclotrimethylenetrinitramine) shall be as specified in Table A-1. Testing Procedures are given as ANNEX B of this document.

Property	**Specification**		**Procedure**
	TYPE I	TYPE II	
Melting point, °C	≥ 200	≥ 190	B-2
HMX content, wt. %	≤ 5	4 - 17	B-3
RDX + HMX content, wt. %	≥ 99	≥ 99	B-4
Acetone insoluble matter, wt. %	≤ 0.05	≤ 0.05	B-5
Inorganic matter (ash), wt. %	≤ 0.03	≤ 0.03	B-6
Acidity (quantity)	(as nitric acid)	(as acetic acid)	B-7
Grade 1 (for detonators), wt. %	≤ 0.01	≤ 0.01	
Grade 1A (for other uses), wt. %	≤ 0.05	≤ 0.02	
Gritty particles			B-8
0.25 mm aperture sieve, particles/50g	≤ 5	≤ 5	
0.425 mm aperture sieve, particles/50g	0	0	
Impact sensitivity	See B-9	See B-9	B-9
Vacuum stability*, mL/g after 48 hrs at 100°C	< 2	< 2	B-10
Granulation	Table A-3	Table A-3	B-11
*Note: This requirement to perform Vacuum Stability testing may be waived at the discretion of the purchaser. Vacuum Stability is not required when RDX is to be purchased as part of a composition, provided the final composition requires such testing with its own specific testing and acceptance criteria			

Table A-1. Specification for RDX.

Compound	**Abbreviation**
cyclotrimethylenetrinitramine	RDX

Table A-2. Abbreviation of compounds.

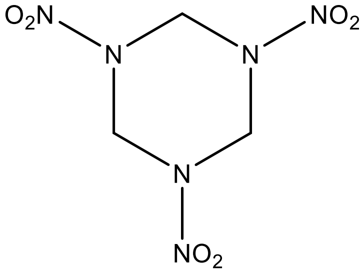

Figure A-1. Structural formula of RDX.

The requirements for granulation of RDX shall be as specified in Table A-3, unless otherwise specified by the purchaser.

Through		Class								
U.S. Standard Sieve No.	Mesh size (µm)	Class 1	Class 2	Class 3	Class 4	Class 5	Class 6	Class 7	Class 8	Class 9
8	2360				100					
12	1700			99-100						
20	850	96-100								96-100
35	500		98-100		0-40				100	
50	300	80-100	90-100	30-50				96-100	98-100	35-70
60	250						96-100			
80	180						91-100			
100	150	30-90	50-80	10-30				82-98	90-100	5-45
120	125						67-93			
170	90						43-80			
200	75	5-45	20-46	0-20				31-61	55-80	0-40
230	63						22-50			
325	45					97-100	8-36		40-60	

Table A-3. Granulation requirements for RDX classes.

A.5. APPEARANCE

Product shall be a white crystalline powder, free from any obvious color inhomogeneity or visible foreign matter. Microphotography showing crystal shape and surface shall be provided at the request of the purchaser.

A.6. SAFETY REQUIREMENTS

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

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ANNEX B TEST PROCEDURES

B.1. SAMPLING AND REJECTION

B.1.1. Sampling

A lot of RDX shall consist of the total quantity of material that is offered for acceptance at one time. For batch processes, a lot shall consist of one batch or multiple blended batches of RDX produced by one manufacturer in accordance with the same specification or same specification revision under the same set of operating conditions. Each batch shall consist of that quantity of RDX that has been subjected to the same chemical or physical process.

A representative sample is one that consists of at least 200 g of RDX taken at random from each lot by a sampling procedure that has been agreed to by the purchaser. Type II RDX is a very heterogeneous material, with the HMX impurity largely present as relatively fine, discrete HMX crystals. Type I RDX is also heterogeneous, but the problem is not so severe due to the lower concentration of HMX. Great care must be taken to ensure that the samples taken for analysis are representative of the entire composition. Suggested methods to obtain a representative sample include:

i. Mix the sample with an automated blender and subdivide with a rotary sample divider; or
ii. Mix the sample thoroughly with a blade and pile it up into a conical heap. Perform this operation two more times. Then divide the cone equally into as many parts as are needed for the analysis.

It will be necessary to dry samples before testing if the RDX samples are taken from a water-wetted supply. The sample is dried in a thin layer (less than 1 cm or 0.39 inches) in an standard oven at 60 ± 5°C until a constant weight is reached.

B.1.2. Rejection Criteria

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

B.2. MELTING POINT DETERMINATION

B.2.1. Principle and Test Description

The melting point is the temperature at which the solid material is converted to liquid state upon heating with a constant heat rate. It may be determined by either Differential Scanning Calorimetry (DSC) [STANAG 4515], the Capillary Tube Method, or a Fischer-Johns hot stage (or equivalent) melting point apparatus, as described in the following sections. The test certificate shall indicate the test method used. The melting point shall be reported along with the heating rate. If DSC is used, the thermogram shall also be included in the report.

B.2.2. Differential Scanning Calorimetry Method

This method is the preferred method for melting point determination per STANAG 4515

B.2.2.1. Apparatus and Materials

a. A DSC capable of generating a heat rate of 5°C/min is to be used. It must also be capable of automatically recording the differential heat flow between the sample and the reference material to the required sensitivity and precision. For comparison purposes, the same heating rate (5°C/min) and environmental conditions shall be used for both calibration and sample runs.
b. A balance capable of weighing to the nearest 0.01 mg.
c. Sample containers manufactured from a material that is not only inert to the explosive under test conditions, but should also have high thermal conductivity. Materials suitable for these kinds of uses are aluminum, gold, and platinum. Secure aluminum pinhole lid to sample pan.
d. A gas supply for purging. The gas flow should remain constant during the test. Due to its high efficiency as a purge gas, helium is susceptible to minor changes in flow rate. While the superior thermal properties of helium make it the purge gas of choice at low temperatures, another gas such as nitrogen is preferred at high temperatures (above about 100°C), unless the instrument is capable of very accurate control of the gas flow rate.
e. Inert reference material. The reference material used shall not be thermally active over the temperature range to be used. For most applications, alumina has been found suitable. However, newer instruments having multiple thermocouples extend the heat flow equation, resulting in corrections for the differences in sample pan mass. Addition of an inert reference material in this case would be counterproductive. Consult the reference manual for the instrument being used to determine whether an inert reference material should be used.

B.2.2.2. Procedure

a. Calibrate the equipment in accordance with STANAG 4515 and/or the operator’s manual of the manufacture, using the same conditions (type of specimen holder, heating rate, purge gas, and purge gas flow rates) as they will be used for the measurements of the RDX samples.
b. Weigh approximately 0.5 – 1.0 mg of a representative portion of the sample into the tared sample container. Ensure that good thermal contact is achieved between the sample and the container by lighting pressing the material into the container.
c. Conduct the analysis in accordance with STANAG 4515, using a heating rate of 5°C/min over the temperature range from 160°C to the end of the endothermic peak, normally a maximum of 300°C is sufficient. Record the thermogram produced.

B.2.2.3. Calculation of Melting Point

The melting point is determined from the thermogram by extrapolation of the baseline before the endotherm onset. A tangent is also extrapolated from the steepest part of the endotherm onset. The temperature at which these extrapolations intersect is the melting point.

B.2.3. Capillary Tube Method

Analysis should be done according to AOP-4682, article 2.1.1

B.2.4. Fischer-Johns Method

This method should only be used as an alternative to B.2.2., since samples with heating rates lower than 5°C/min may be affected by sample decomposition.

B.2.4.1. Apparatus and Materials

a. Fischer-Johns hot stage melting point apparatus or equivalent.
b. Two cover glasses with sizes appropriate to the instrument being used.
c. Agate mortar and pestle.
d. Short range, short stem thermometers full immersion type, having a range of 195 - 205°C and 185 - 195°C, graduated at 0.1°C intervals.
e. A portion of dry RDX sample, ground with caution to a fine powder.

B.2.4.2. Procedure

a. Place approximately 0.05 g of the finely ground sample between the two clean cover glasses. Gently but firmly press the two together and place on the stage.

b. Turn on the powerstat and allow the unit to heat. The heating rate may be very rapid to within 15°C of the melting point. Thereafter, a heating rate of approximately 1°C/min shall be used.
c. When the sample begins to melt, record the temperature as the melting point of RDX.

B.3. HMX CONTENT IN RDX VIA HPLC/UPLC ANALYSIS

B.3.1. Principle

The HMX content in RDX shall be measured by Liquid Chromatograph (HPLC or UPLC) analysis. The LC method shall be performed to determine the HMX concentration in a sample from its peak area. The total HMX concentration will be reported as an average value of weight percent to two (2) decimal places based on a minimum of 5 measurements, to be no more than 1% for Type I and 4 – 17% for Type II RDX. The chromatographic conditions shall be reported along with the sample results.

B.3.2. Test Description

LC shall be used to analyze injections of standardized solutions of HMX/RDX in order to create a calibration table. This calibration curve will be used to determine the amount of HMX in the RDX sample based on the observed peaks. The method varies depending on whether the sample is Type I or Type II RDX as outlined in the following sections. Due to the large sample size required to measure the low levels of HMX, this method is not suitable for the direct quantification of RDX.

B.3.3. Apparatus and Materials

a. A liquid chromatograph (HPLC or UPLC) with a suitably sized sample loop (e.g. 5 µL), a detection system equipped with a UV detector and an integrator or computerized data acquisition system.
b. 10-mL, 25-mL, and 100-mL Class A glass volumetric flasks. Larger volumetric flasks are permitted to allow for elimination of the dilution step in the sample preparation.
c. Analytical column with a C18 stationary phase, such as a RESTEK Ultra Cyano column with 5 µm particle size, 4.6 mm inside diameter, and 250 mm length, or equivalent.
d. HPLC grade solvents (e.g. acetonitrile, acetone, methanol) with a purity > 99.8% and miliQ water
e. Certified HMX-standard (e.g. Accustandard), suitable for use as primary analytical standard >99.5%. This may be prepared by repeated recrystallization of industrial-grade HMX (preferably Type III according to STANAG 4284) from acetonitrile. Total peak area of UV-absorbing impurities in HMX calibration

standard shall not exceed 0.5% of HMX peak area at 230 nm wavelength, solvent peak area excluded.

f. Certified RDstandard (e.g. Accustandard), suitable for use as primary analytical standard, >99.5%. This may be prepared by repeated recrystallization of industrial-grade RDX (preferably Type I according to this STANAG) from acetonitrile. Total peak area of UV-absorbing impurities in RDX calibration standard shall not exceed 0.5 % of RDX peak area at 230 nm wavelength, solvent peak area excluded.
g. Appropriate internal standard (e.g., diethyl phthalate or ethyl centralite) of Analytical Reagent quality. NOTE: Some analysts prefer to use ethyl centralite as a standard since it is a solid, whereas diethyl phthalate is a liquid. Either of these compounds is acceptable, and other compounds can be used provided they do not interfere with the analysis of the peaks for RDX and HMX.

B.3.4. Calibration Standards Preparation

A minimum of 5 concentrations should be used to create a calibration curve. The concentrations should be spanning the expected RDX and HMX concentration range in the sample and lying in the concentration range suitable for the LC-separation (depending on column and injection volume) The standards shall be prepared according to the following procedures and recommended concentrations (Tables B-1 and B-2).

<u>Stock Standard Solution of HMX:</u>

a. Weigh 50 mg HMX to the nearest 0.1 mg into a 100 mL volumetric flask
b. Weigh 50 mg of the internal standard to the nearest 0.1 mg and add to the flask
c. Add enough LC grade acetonitrile to ensure dissolution
d. Bring to volume with additional acetonitrile and mix well

<u>Type I RDX:</u>

a. Transfer the appropriate amount of RDX (according to Table B-1) into a glass, class A, 25 mL volumetric flask by either weighing directly or transferring via serial dilution of a stock solution
b. Transfer the appropriate amount of HMX (Table B-1) in the same flask via serial dilution due to the low concentrations
c. Add roughly 10-15 mL if HPLC grade acetonitrile to the flask
d. Stir until the RDX is completely dissolved and the solution is uniformly mixed
    i. An ultrasonic bath may be used to assist dissolution
    ii. Allow sample to cool to ambient temperature prior to bringing to final volume
e. Fill to the mark of the flask using LC grade acetonitrile

RDX		HMX	
Concentration (mg/mL)	Weight (g) in 25 mL	Concentration (mg/mL)	Volume of HMX Stock Solution (mL) in 25 mL
5.2	0.130	0.005	0.250
5.0	0.125	0.020	1
4.8	0.120	0.100	5

Table B-1. Composition of RDX/HMX Standards for Type I RDX.

<u>Type II RDX:</u>

a. Transfer the appropriate amount of RDX (according to Table B-2) into a glass, class A, 100 mL volumetric flask by either weighing directly or transferring via serial dilution of a stock solution
b. Transfer the appropriate amount of HMX (Table B-2) in the same flask via serial dilution due to the low concentrations
c. Add roughly 10-15 mL if LC grade acetonitrile to the flask
d. Stir until the RDX is completely dissolved and the solution is uniformly mixed
    iii. An ultrasonic bath may be used to assist dissolution
    iv. Allow sample to cool to ambient temperature prior to bringing to final volume
e. Fill to the mark of the flask using LC grade acetonitrile

RDX			HMX	
Concentration (mg/mL)	Weight (g) in 100 mL	Volume of RDX Stock Solution (mL) in 100 mL	Concentration (mg/mL)	Volume of HMX Stock Solution (mL) in 100 mL
0.55	0.055	11	0.005	1
0.47	0.047	9	0.025	5
0.40	0.040	8	0.100	20

Table B-2. Composition of RDX/HMX Standards for Type II RDX.

B.3.5. Sample Preparation

a. Accurately weigh approximately 500 mg of dry sample to the nearest 0.1 mg into a clean, class A 100 mL volumetric flask
b. Accurately weigh approximately 50 mg of internal standard to the nearest 0.1 mg into the same volumetric flask
c. Add roughly 60-70 mL of HPLC grade acetonitrile; stir until completely dissolved and uniformly mixed
    i. An ultrasonic bath may be used to assist dissolution
    ii. Allow sample to cool to ambient temperature
d. Fill to the mark of the flask using LC grade acetonitrile

e. Filter sample through a 0.45 µm filter prior to introduction into the chromatographic system
f. <u>For Type II RDX only</u>:
   i. Transfer 1 mL of filtered solution to a clean, class A, 10 mL volumetric flask
   ii. Dilute to mark with LC grade acetonitrile
   iii. Ensure sample is uniformly mixed before performing analysis
g. Repeat steps a through e so that each sample is prepared in triplicate

B.3.6. LC Analysis

a. The following two LC operating conditions (Table B-3) are given for information only to present two possible instrument set-up that has proven effective in resolving RDX, HMX, and observed impurity peaks. Other conditions may be used provided that baseline separation of the peaks for solvent, internal standard, HMX, and RDX are achieved.

	CN Column	C18 Column
Mobile phase	30% acetonitrile 60% water 10% methanol (by volume)	56% MeOH 44% MiliQ
Flow rate	1.0 mL/minute	Depending on the system and particle 0.2 to 1.0 mL/min
UV wavelength	230 nm or 254 nm	230 nm with multi wavelength detector also 210 nm
Column	e.g. RESTEK Ultra Cyano (CN)	
Temperature	30°C	30°C
Sample size	5.0 microliters	1 to 5 microliter, depending on column I.D.

Table B-3. HPLC Instrument Parameter Guide for RDX and HMX Analysis.

b. The concentration of HMX and RDX are calculated from its peak area. A qualitative measure of impurities other than HMX contained in the sample can be gauged by the presence of other peaks in the chromatographic trace. Lower wavelengths typically provide a better response for observing impurities.
c. The operation and analysis procedures of the exact LC equipment used shall be followed for maximum accuracy and precise area measurements of the LC trace.

B.3.6.1. HPLC Procedure

a. Initiate the LC system in accordance with the manufacturer's operating instructions
b. Enter the recommended conditions (Table B-3)
c. Allow the LC to equilibrate for a minimum of 10-20 minutes or until the flow of mobile phase and the output reading on the UV detector are stable
d. Once stabilized, fill the sample loop on the injector system with an aliquot of the sample or standard and initiate the run
    a. Standard and sample solutions are introduced into the system using a syringe or autosampler - a 5 µL injection volume is recommended
    b. All injections must be performed under the same operating conditions
    c. For statistical control, a sufficient number of injections of each solution must be made to ensure reproducibility of the system. It is recommended that at least two (2) injections be performed for the working calibration standards and samples.
    d. A calibration check standard should be run before and after samples with no more than six (6) hours between calibration and verification.

B.3.7. HMX Response Factor (RF_{HMX}) and Concentration Calculations

The peak areas of HMX and the internal standard for all the injections of the calibration standard and sample should be calculated using the data collection system (i.e. computer, computing integrator, etc.). The results of the injection are averaged for the sample and the standard. The areas of the injections should agree within 1% relative to each other. If this criterion is not met, the sample should be rejected and a new sample must be prepared and analyzed.

The response factor for HMX shall be calculated per the following equation:

$$\mathrm{RF_{HMX}} = \frac{\mathrm{w_{H-C}} \times A_{IS-C}}{\mathrm{w_{IS-C}} \times \mathrm{A_{H-C}}},$$

Where: w_{H-C} = Weight of pure HMX in calibration standard in grams (g),
w_{IS-C} = Weight of internal standard in calibration standard in grams (g),
A_{H-C} = Average area of HMX peak from the injections of the calibration standard solution (mAU),
A_{IS-C} = Average area of internal standard peak from the injections of the calibration standard solution (mAU),
RF_{HMX} = Response factor for HMX.

From this, the content of HMX for each sample is to be calculated for each of the three sample preparations as well as the average of the determinations as weight percent (wt. %) per the following:

$$\%\ \mathrm{HMX} = \frac{A_H \times w_{IS} \times RF_{HMX}}{\mathrm{A_{IS}} \times \mathrm{w}} \times 100\,,$$

Where: RF_{HMX} = Response factor for HMX as determined above by calibration standards,
A_H = HMX peak area as the average of five injections (mAU),
A_{IS} = Internal standard peak area as the average of five injections (mAU),
w_{IS} = Weight of the internal standard in milligrams (mg)
w = Weight of the sample in milligrams (mg).

B.4. RDX CONTENT VIA LC ANALYSIS

B.4.1. Principle

The RDX content shall be measured by Liquid Chromatograph (HPLC or UPLC) analysis. The LC method shall be performed to determine the RDX concentration in a sample from its peak area. The total RDX concentration will be reported as an average value of weight percent to two (2) decimal places. Additionally, the total concentration of RDX + HMX will be reported as an average value of weight percent, to be no less than 99% for both Type I and Type II RDX. The chromatographic conditions shall be reported along with the sample results.

B.4.2. Test Description

LC shall be used to analyze standardized solutions of RDX with varying amounts of HMX in order to create a calibration table (curve). This table will be used to determine the amount of RDX in the sample based on the observed peaks. The method varies depending on whether the sample is Type I or Type II RDX as outlined in the following sections.

B.4.3. Apparatus and Materials

a. A liquid chromatograph (HPLC or UPC) with a suitably sized sample loop (e.g. 5 µL), a detection system equipped with a UV detector and an integrator or computerized data acquisition system.
b. 10-mL, 25-mL, and 100-mL Class A glass volumetric flasks. Larger volumetric flasks are permitted to allow for elimination of the dilution step in the sample preparation.
c. Analytical column, such as a RESTEK Ultra Cyano column with 5 µm particle size, 4.6 mm inside diameter, and 250 mm length, or equivalent.
d. HPLC grade solvents (e.g. acetonitrile, acetone, methanol, water).
e. RDX, suitable for use as primary analytical standard, >99.5%. This may be prepared by repeated recrystallization of industrial-grade RDX (preferably Type I according to this STANAG) from acetonitrile. Total peak area of UV-absorbing impurities in RDX calibration standard shall not exceed 0.5 % of RDX peak area at 230 nm wavelength, solvent peak area excluded.

f. Appropriate internal standard (e.g., diethyl phthalate or ethyl centralite) of Analytical Reagent quality. NOTE: Some analysts prefer to use ethyl centralite as a standard since it is a solid, whereas diethyl phthalate is a liquid. Either of these compounds is acceptable, and other compounds can be used provided they do not interfere with the analysis of the peaks for RDX.

B.4.4. Calibration Standards Preparation

Three (3) or more calibration standards spanning the anticipated RDX concentration range contained in the sample shall be prepared. For Type II RDX, both the %RDX and %HMX may be obtained within a single analysis provided the calibration standards contain both RDX and HMX spanning the anticipated concentration ranges and baseline resolution of the peaks is achieved under the conditions employed. The calculations of both RF_{HMX} and %HMX would be performed analogously to that of RDX.

a. Transfer the appropriate amount of RDX into a glass, class A, 100 mL volumetric flask
    i. Smaller or larger flasks can be used provided there is no loss in either the accuracy or precision of the weighing operation
    ii. Dilution may be necessary from more concentration standard solutions to allow for larger initial weights
b. Add an equal mass of the internal standard to be used
c. Prepare a separate calibration check standard (CCS) at approximately the middle point concentration of the calibration standards. This should contain both RDX and HMX to ensure the two are sufficiently resolved for integration of the RDX peak
d. Add roughly 10-15 mL of HPLC grade acetonitrile to each flask
e. Stir until the standard solution is completely dissolved and uniformly mixed
    v. An ultrasonic bath may be used to assist dissolution
    vi. Allow sample to cool to ambient temperature prior to bringing to final volume
f. Fill to the mark of the flask using HPLC grade acetonitrile

B.4.5. Sample Preparation

a. Accurately weigh approximately 0.5 g of dry RDX sample to the nearest 0.1 mg into a clean, class A 100 mL volumetric flask
b. Accurately weigh approximately 0.2 g of internal standard to the nearest 0.1 mg into the same volumetric flask
c. Add roughly 60-70 mL of HPLC grade acetonitrile; stir until completely dissolved and uniformly mixed
    i. An ultrasonic bath may be used to assist dissolution
    ii. Allow sample to cool to ambient temperature
d. Fill to the mark of the flask using HPLC grade acetonitrile
e. Transfer 1 mL of filtered solution to a clean, class A, 10 mL volumetric flask

f. Dilute to mark with HPLC grade acetonitrile
g. Ensure sample is uniformly mixed before performing analysis
h. Filter sample through a 0.45 µm filter prior to introduction into the chromatographic system
i. Repeat steps a through h so that each sample is prepared in triplicate

B.4.6. HPLC Analysis and Procedure

The HPLC operating conditions outlined in Procedure B.3.6. (Table B-3) are also applicable for this method.

a. Initiate the HPLC system in accordance with the manufacturer's operating instructions
b. Enter the recommended conditions (Table B-3)
c. Allow the HPLC to equilibrate for a minimum of 10-20 minutes or until the flow of mobile phase and the output reading on the UV detector are stable
d. Once stabilized, fill the sample loop on the injector system with an aliquot of the sample or standard and initiate the run
    a. Standard and sample solutions are introduced into the system using a syringe or autosampler - a 5 µL injection volume is recommended
    b. All injections must be performed under the same operating conditions
    c. For statistical control, a sufficient number of injections of each solution must be made to ensure reproducibility of the system. It is recommended that at least two (2) injections be performed for the working calibration standards and samples.
    d. A calibration check standard should be run before and after samples with no more than six (6) hours between calibration and verification.

B.4.7. RDX Response Factor (RF_{RDX}) and Concentration Calculations

The peak areas of RDX and the internal standard for all the injections of the calibration standard and sample should be calculated using the data collection system (i.e. computer, computing integrator, etc.). The results of all the injections are averaged for the sample and the standard. The areas of the injections should agree within 1% relative to each other. If this criterion is not met, the sample should be rejected and a new sample must be prepared and analyzed.

The response factor for RDX shall be calculated per the following equation:

$$\mathrm{RF}_{RDX} = \frac{\mathrm{w}_{\mathrm{R-C}} \times A_{IS-C}}{\mathrm{w}_{\mathrm{IS-C}} \times \mathrm{A}_{\mathrm{R-C}}},$$

Where: w_{R-C} = Weight of pure RDX in calibration standard in grams (g),
w_{IS-C} = Weight of internal standard in calibration standard in grams (g),
A_{R-C} = Average area of RDX peak from the injections of the calibration standard solution (mAU),

A_{IS-C} = Average area of internal standard peak from the injections of the calibration standard solution (mAU),
RF_{RDX} = Response factor for RDX.

From this, the content of RDX for each sample is to be calculated and reported for each of the three sample preparations as well as the average of the determinations as weight percent (wt. %) per the following:

$$\%\ \mathrm{RDX} = \frac{A_R \times w_{IS} \times RF_{RDX}}{\mathrm{A_{IS}} \times \mathrm{w}} \times 100\,,$$

Where: RF_{RDX} = Response factor for RDX as determined above by calibration standards,
A_R = RDX peak area as the average of five injections (mAU),
A_{IS} = Internal standard peak area as the average of five injections (mAU),
w_{IS} = Weight of the internal standard in milligrams (mg)
w = Weight of the sample in milligrams (mg).

The RDX + HMX content shall also be reported, which is the sum of the averages of the determinations of %RDX (from above) and %HMX (from Procedure B.3.7.).

B.5. ACETONE INSOLUBLE MATTER CONTENT

Analysis should be done according to AOP-4682, article 3.6.1 Inorganic Matter (Ash)

B.6. INORGANIC MATTER CONTENT

Analysis should be done according to AOP-4682, article 3.4.1 Insoluble Impurities

B.7. ACIDITY

B.7.1. Principle

This method shall be performed to determine the acidity of RDX. The acidity shall be reported as a value of weight percent of nitric acid for Type I RDX, to be no greater than 0.01% for detonators (Grade 1) or 0.05% for other uses (Grade 1A). The acidity shall be reported as a value of weight percent of acetic acid for Type II RDX, to be no greater than 0.01% for detonators (Grade 1) or 0.02% for other uses (Grade 1A).

B.7.2. Test Description

A sample of RDX will be titrated against a standardized sodium hydroxide solution. The acidity is calculated based on the volume of sodium hydroxide used to reach the end-point.

Analysis should be done according to AOP-4682, article 3.1.1 Acidity and Alkalinity

B.8. GRITTY PARTICLES ANALYSIS

Analysis should be done according to AOP-4682, article 3.7.1 Gritty Particles

B.9. IMPACT SENSITIVENESS

B.9.1. Principle

This test shall be performed to assess the extent of sensitivity of RDX to impact. The sensitivity shall be reported as the lowest height in centimeters (cm) at which more than 50% of explosions occur.

B.9.2. Test Description

The purchaser may require that the manufacturer provide impact sensitiveness data. Impact sensitiveness shall be assessed in accordance with STANAG 4489. The purchaser may request additional information on the performance of the manufacturer’s impact apparatus, such as the impact sensitivities of other known explosives (e.g. PETN, HMX, or TNT) or archival values of RDX impact sensitiveness measurements made on the same instrument.

B.9.3. Test Methods

i. ERL, Type 12, 2.5 kilogram (kg), Rotter, BAM in accordance with STANAG 4489

B.10. VACUUM STABILITY TEST

B.10.1. Principle

The vacuum thermal stability of RDX shall be measured to determine the thermal stability. It shall be reported as the amount of gas evolved after heating for 48 hours at 100°C. The criteria for this test is that the evolved volume is not greater than 2 mL/g.

B.10.2. Test Description

The vacuum stability shall be assessed in accordance with NATO STANAG 4556 for Energetic Materials: Vacuum Stability Test. For RDX, the weight of explosive used is between 1.0 and 5.0 g, depending on the precision of the apparatus being used and the volume of gas that can be tolerated for safety reasons. The test temperature is 100 ± 0.5°C and the test duration is 48 hours.

B.11. GRANULATION ANALYSIS

B.11.1. Sieving Method

Analysis should be done according to AOP-4682, article 2.4.2 Wet Sieving

B.11.2. Low Angle Laser Light Scattering (LALLS) Method

Analysis should be done according to AOP-4682, article 2.4.3

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