INCH-POUND

MIL-DTL-32202(AR)
4 April 2006
SUPERSEDING
AR-PD-132
16 August 2001

DETAIL SPECIFICATION

EXPLOSIVE, MELT CASTABLE: PAX-21

This specification is approved for use by the U.S. Army Armament Research, Development and Engineering Center (ARDEC), and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers the requirements and applicable tests for the manufacture and acceptance of EXPLOSIVE, MELT CASTABLE: PAX-21.

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all requirements documents cited in sections 3 and 4 of this specification whether or not they are listed.

2.2 Government Documents.

2.2.1 Specifications, standards, and handbooks. The following specifications, standards and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in solicitation or contract.

DEPARTMENT OF DEFENSE SPECIFICATION

MIL-DTL-398 - RDX (Cyclotrimethylenetrinitramine)

Comments, suggestions, or questions on this document should be addressed to (Commander ARDEC, ATTN: AMSRD-AAR-AIS-SS, Picatinny, NJ 07806-5000) or emailed to (ardec-stdzn@pica.army.mil). Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at http://assist.daps.dla.mil

AMSC N/A FSC 1376
DISTRIBUION STATEMENT D. Statement D, DoD & Contractors (Controlling DoD office is Energetics and Warheads Division, AETC, US Army, RDECOM-ARDEC)

DEPARTMENT OF DEFENSE STANDARDS

MIL-STD-1916 - DOD Preferred Methods for Acceptance of Product

MIL-STD-650 - Explosive: Sampling, Inspection, and Testing

(Copies of these documents are available online at http://assist.daps.dla.mil/quicksearch/ or http://assist.daps.dla.mil or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

2.2.2 Other Government documents, drawings and publications. The following other Government documents, drawings and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation (see 6.2).

DEPARTMENT OF DEFENSE TECHNICAL BULLETIN

TB 700-2 - Department of Defense Ammunition and Explosives Hazard Classification Procedures

(Copies of the above document are available from Chairman, Department of Defense Explosives Safety Board, Room 856C, Hoffman Building 1, 2461 Eisenhower Avenue, Alexandria, VA 22331-0600 or online at https://www.logsa.army.mil/pubs.htm)

2.3 Non-Government publications.

ASTM International

ASTM E 300 - Recommended Practice for Sampling of Industrial Chemicals

(Copies of ASTM standards are available online from http://www.astm.org/ and from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

UNITED STATES PHARMACOPEIA

USP-NF - Calcium Phosphate, Tribasic

(Application for copies should be addressed to: United States Pharmacopeia, 12601 Twin Brook Parkway, Rockvale, MD 20852.)

2.4 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence.

Nothing in this document, however, supercedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1. First Article. When specified (see 6.2 and 6.3), a sample shall be subjected to first article inspection in accordance with 4.2.

3.2 Materials.

3.2.1 Ingredients. All ingredients used in the manufacturing of PAX-21 shall meet the following requirements.

3.2.1.1 2,4-Dinitroanisole (DNAN). 2,4-Dinitroanisole (DNAN) shall conform to the chemical and physical requirements specified in TABLE I, when tested as specified in applicable paragraphs (See 4.6.1 through 4.6.8)

TABLE I. Chemical and physical requirements for DNAN.

Property		Test Paragraph
DNAN Purity (%min) (dry basis)	98	4.6.1 & 4.6.2
2,5-Dinitroanisole (%max) (dry basis): Below limit of HPLC detection	0	4.6.1 & 4.6.2
1-chloro-2,4-dinitrobenzene (% max) (dry basis)	0.25	4.6.1&4.6.2
2,4-dinitrophenol (% max) (dry basis)	0.01	4.6.1&4.6.2
Melting Point (°C min)	80.2	4.6.3
Moisture, (% max)	10	4.6.4 or 4.6.5
Undissolved solids, (% max) (dry basis)	0.15	4.6.6
Trace metal content (max ppm) (dry basis)	300	4.6.7
Physical form:	In flake or granular form	4.6.8

3.2.1.1.1 Workmanship for DNAN. The DNAN shall be uniform in quality and free from lumps, grit, visible impurities, foreign matter, or other defects that would render the DNAN unsuitable for the intended use (See 4.6.8).

3.2.1.1.2 Storage life for DNAN. Material stored in closed containers at ambient temperatures shall have a storage life of 36 months from date of acceptance testing. Material that exceeds its storage life may be extended in 24 month increments by demonstrating compliance with the chemical and physical properties in TABLE I.

3.2.1.2 n-Methyl-p-nitroaniline (MNA). n-Methyl-p-nitroaniline (MNA) shall conform to the chemical and physical requirements specified in TABLE II, when tested in accordance with 4.7.2 through 4.7.9 (see 6.11).

TABLE II. Chemical and physical requirements for MNA.

Property	Value	Test Paragraph
Color value (absorbance max)	0.148	4.7.2
MNA Purity (% min)	99	4.7.3
N,N-dimethyl-para-nitroaniline (DMNA) content (wt% max)	0.5	4.7.3
Para-chloronitrobenzene (PCNB) content (wt% max)	0.5	4.7.3
Moisture and volatile content (wt% max)	0.5	4.7.4
Acidity (mg KOH / g max)	0.3	4.7.5
Acetone insoluble matter (wt% max)	0.15	4.7.6
Iron content (wt% max)	0.005	4.7.7
Chloride content (wt% max)	0.005	4.7.8 or 4.7.9

3.2.1.2.1 Workmanship for MNA. The MNA shall be uniform in quality and free from lumps, grit, visible impurities, foreign matter, or other defects that would render the MNA unsuitable for the intended use. Inspection for workmanship shall be in accordance with 4.7.1.

3.2.1.2.2 Storage Life for MNA. Material stored in closed containers at ambient temperatures shall have a storage life of 36 months from date of acceptance testing. Material that exceeds its storage life may be extended in 24 month increments by demonstrating compliance with the chemical and physical properties in TABLE II.

3.2.1.3 Ammonium perchlorate. Ammonium perchlorate (AP) shall conform to the chemical and physical requirements described in TABLE III.

TABLE III. Physical and chemical properties of AP.

Property			Test Paragraph
	Min	Max	
Moisture Total (%)	-	0.13	4.8.2 or 4.8.3
Acid Insoluble Matter (%)	-	0.04	4.8.4 or 4.8.5
PH of aqueous solution	5	6.5	4.8.6
Chloride, as NH4Cl (%)	-	0.155	4.8.7 or 4.8.8
Sulfated Ash, as NaClO4 (%)	-	0.9	4.8.9 or 4.8.10
Chlorate, as NH4ClO3 (%)	-	0.02	4.8.11 or 4.8.12
Iron, as Fe2O3 (%)	-	0.0036	4.8.13
Purity (%)	98.2	-	4.8.14
Phosphate, as TCP (%)	0	.25	4.8.15
Bulk density, g/in3	17.5	-	4.8.16
Particle Size Distribution			
10 % less than (<)	300 microns	393 microns	4.8.17
50% less than (<)	350 microns	455 microns	4.8.17
90% less than (<)	425 microns	525 microns	4.8.17

3.2.1.3.1 Workmanship for AP. The ammonium perchlorate shall be uniform in quality and free from lumps, visible impurities, or other defects that would render the AP unsuitable for the intended use. Lumps that can be dispersed by hand pressure are acceptable. Lumps not dispersible by hand pressure shall not exceed 10 percent of the material by weight (See 4.8.18). AP shall be conditioned with 0.2 percent (nominal) tribasic calcium phosphate (TCP) as an anti-caking agent meeting requirements of the USP-NF.

3.2.1.3.2 Storage life for AP. Material stored in closed containers at ambient temperatures shall have a storage life of 18 months from date of acceptance testing. Material that exceeds its storage life may be extended in 18 month increments by demonstrating compliance with the chemical and physical properties in TABLE III.

3.2.1.4 RDX (cyclotrimethylenetrinitramine). A coarse and a fine RDX particle size will be used.

3.2.1.4.1 Coarse RDX. Coarse RDX shall conform to the chemical and physical requirements of Class 1 of specification MIL-DTL-398.

3.2.1.4.2 Fine RDX. Fine RDX shall be fluid energy milled from Class 1 RDX that meets the specification MIL-DTL-398. The particle size and moisture of this fine RDX shall conform to the distribution specified in TABLE IV.

TABLE IV. Physical and chemical analysis of fine RDX.

Property	Requirement	Test Paragraph
Moisture, (wt%)	0.066% max.	4.9.1
Particle Size Distribution, micron :		
10 % less than (<)	1.95±1 microns	4.9.2
50 % less than (<)	2.86±0.5 microns	4.9.2
90 % less than (<)	5.35±3 microns	4.9.2

The requirements in the table are based on the use of a Microtrac or a Malvern particle size analyzer. Alternate particle size distribution requirements may be established based on a minimum of 5 acceptable (based on application) PAX-21 batches.

3.2.1.4.3 Storage for fine RDX. Fine RDX shall be stored in closed containers, at ambient temperature, either sealed in plastic bags with tape or in purged containers, and shall have a storage life of 6 months from date of acceptance testing. Material that exceeds its storage life may be extended in 1-month increments by demonstrating compliance with the particle size distribution in TABLE IV.

3.2.2 PAX-21.

3.2.2.1 PAX-21 Nominal Formulation. The formulation shall conform to the weigh-up and addition as specified in TABLE V. Raw materials shall conform to the specifications as stipulated in paragraphs 3.2.1.1 through 3.2.1.4.

TABLE V. Nominal composition of PAX-21.

Material	Ingredient (wt %)	Paragraph
DNAN	34	3.2.1.1
MNA	0.25	3.2.1.2
AP	30	3.2.1.3
RDX (Coarse)	5	3.2.1.4.1
RDX (Fine)	30.75	3.2.1.4.2

3.2.2.2 PAX-21 Requirements. PAX-21 shall conform to the chemical and physical requirements specified in TABLE VI, when tested as specified in paragraph 4.10.1 through 4.10.11. For use of riser scrap and clean scrap PAX-21 material for melt pour operation, see 6.7 and 6.8.

3.2.2.3 Workmanship for PAX-21. The PAX-21 shall be uniform in color, texture, and free from undissolved lumps of ingredient materials, or oversized lumps of flakes, visible impurities, foreign matters, or other defects that would render the PAX-21 unsuitable for the intended use. (See 6.12). Workmanship inspection shall be conducted in accordance with 4.10.12.

TABLE VI. Chemical and physical requirements for PAX-21.

Property		Test Paragraph
DNAN, %	34 ± 2	4.10.1
MNA (%min)	0.15	4.10.1
MNA (%max)	0.35	4.10.1
RDX, %	35.75 ± 2	4.10.1
AP, %	30 ± 1	4.10.2
Melting Point DSC method, @5°C/min	88 ± 5	4.10.3
Exotherm Onset DSC method, @°C @ 5°C/min	202 ± 12	4.10.3
Viscosity, Kp (max) Brookfield, kP @205 ± 5°F Efflux Viscosity, sec (max) @205°F	 0.11 to 0.27 15	 4.10.4 4.10.5
Moisture (% max)	0.04	4.10.6
Flake Thickness, inches (max)	0.250	4.10.7
Flake Density (gm/cm3)	See 6.10	4.10.8
*Impact sensitivity ERL, Type 2, cm (min)	21	4.10.9
*Friction Sensitivity ABL Method, lb @ 8 fps	100 to 800	4.10.10
*Gap test, cards 50% Max	165*	4.10.11
* - See section 6.3.		

4. VERIFICATION

TABLE VII. Requirements/verification cross reference matrix

Method of Verification	Classes of Verification
1- Analysis 2- Demonstration (end item test) 3- Examination 4- Test	A - First Article B - Conformance

Section 3 Requirements	Description	Verification Method				Verification Class		Section 4
		1	2	3	4	A	B	
3.1	First Article	X		X	X	X		4.2
3.2	Materials				X	X	X	4.3.2.1-4.3.2.5
3.2.1	Ingredients				X	X	X	4.3.2.1-4.3.2.4
3.2.1.1	2,4-Dinitroanisole				X	X	X	4.3.2.1, 4.6
3.2.1.1.1	Workmanship for DNAN			X		X	X	4.6.8
3.2.1.1.2	Storage life for DNAN	X		X		X	X	4.3.2.1
3.2.1.2	n-Methyl-p-nitroaniline				X	X	X	4.3.2.2, 4.7.2- 4.7.9
3.2.1.2.1	Workmanship for MNA			X		X	X	4.7.1
3.2.1.2.2	Storage life for MNA	X		X		X	X	4.3.2.2
3.2.1.3	Ammonium perchlorate				X	X	X	4.3.2.3
3.2.1.3.1	Workmanship for AP			X		X	X	4.8.18
3.2.1.3.2	Storage life for AP	X		X		X	X	4.3.2.3
3.2.1.4	RDX				X	X	X	4.9
3.2.1.4.1	Coarse RDX				X	X	X	4.9
3.2.1.4.2	Fine RDX				X	X	X	4.3.2.4
3.2.1.4.3	Storage for fine RDX	X		X		X	X	4.3.2.4
3.2.2	PAX-21				X	X	X	4.3.2.5
3.2.2.1	PAX-21 nominal formulation	X				X	X	4.3.2.5
3.2.2.2	PAX-21 requirements	X			X	X	X	4.10
3.2.2.3	Workmanship for PAX-21			X		X	X	4.10.12

4.1 Classification of Verification. The verification requirements specified herein are classified as follows:

a. First Article Inspection (see 4.2).

b. Conformance Inspection (see 4.3).

4.2 First article inspection.

4.2.1 Submission. When specified a first article sample shall be provided for evaluation in accordance with section 4.2.2. The first article sample shall be 10 kilograms from the first production lot produced using the same production equipment, processes, procedures, and material supply sources (see 6.2.c and 6.3).

4.2.2 Inspections to be performed. The first article sample will be subjected by the government to any or all of the examination and tests specified in section 4.3.2.1 through 4.3.2.5 and Table VIII.

4.2.2.1 Rejection. If any sample fails to comply with any of the applicable requirements the first article quantity shall be rejected. The government reserves the right to terminate its inspection upon any failure of a sample to comply with any of the stated requirements.

Table VIII. First article inspection [1]

	TITLE PAX-21 EXPLOSIVE COMPOSITION		SHEET 1 OF 1		DRAWING NUMBER NEXT HIGHER ASSEMBLY
CATEGORY	EXAMINATION OR TEST	SAMPLE SIZE	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	PARAGRAPH REFERENCE/ INSPECTION METHOD
Critical	None defined				
Major [2]					
111	Impact Sensitivity	See 4.2.1	100% [3]	3.2.2.2	4.10.9
112	Friction Sensitivity	See 4.2.1	100%	3.2.2.2	4.10.10
113	Gap test	See 4.2.1	100%	3.2.2.2	4.10.11
Minor	None defined				

Note: (1) First article tests in addition to those listed in 4.3.2.1 thru 4.3.2.5
(2) Majors 101 thru 110 for PAX-21 are listed in 4.3.2.5
(3) 100% - Tests shall be conducted on sample(s) from each batch. (Also see 4.4.6).

4.3 Conformance inspection.

4.3.1 Lot formation. A lot shall consist of one or more batches of PAX-21 produced by one manufacturer, meeting all requirements of this specification, and produced using the same set of operating conditions. A lot of PAX-21 shall be made from a single lot of raw materials and a single grind lot of any applicable raw material. Lot formation shall be in accordance with the lot formation requirement of MIL-STD-1916, "Formation and identification of lots and batches".

4.3.1.1 Batch formation. A batch is defined as one mix made in a reactor or an equivalent mixing vessel. Each batch shall be tested for conformance to the requirements of this specification.

4.3.2 Examination and tests.

a. Classification characteristics. Conformance examinations and tests are specified in the following classification of characteristics paragraphs. A quality program or detailed inspection system shall provide assurance of compliance of all characteristics with the applicable drawing and specification requirements utilizing as a minimum the conformance criteria specified. When cited in this specification (4.3.2.1 to 4.3.2.5), attributes sampling inspection shall be conducted in accordance with paragraph 4.4 and Table IX as applicable. (See MIL-STD-1916 for the definitions of critical, major and minor classification of characteristics.)

Classification of characteristics

PARAGRAPH 4.3.2.1	TITLE: DNAN		SHEET 1 OF 1	DRAWING NUMBER NEXT HIGHER ASSEMBLY
CATEGORY	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	PARAGRAPH REFERENCE/ INSPECTION METHOD
Critical	None defined			
Major				
101	DNAN Purity	4.4.2	3.2.1.1	4.6.1 & 4.6.2
102	Exceed % max. of 1-chloro-2,4-dinitrobenzene	4.4.2	3.2.1.1	4.6.1
103	Exceed % max. of 2,4-dinitrophenol	4.4.2	3.2.1.1	4.6.1
104	Melting Point	4.4.2	3.2.1.1	4.6.3
105	% max. moisture	4.4.2	3.2.1.1	4.6.4 or 4.6.5
106	% max. undissolved Solids	4.4.2	3.2.1.1	4.6.6
107	Max. ppm trace Metal Content	4.4.2	3.2.1.1	4.6.7
Minor				
201	Workmanship	4.4.2	3.2.1.1.1	4.6.8

Classification of characteristics

PARAGRAPH 4.3.2.2	TITLE MNA SHEET 1 OF 1		DRAWING NUMBER NEXT HIGHER ASSEMBLY	
CATEGORY	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	PARAGRAPH REFERENCE/ INSPECTION METHOD
Critical	None defined			
Major				
101	Color Value	4.4.3	3.2.1.2	4.7.2
102	MNA Purity (% minimum)	4.4.3	3.2.1.2	4.7.3
103	DMNA content (wt. % max.)	4.4.3	3.2.1.2	4.7.3
104	PCNB content (wt. % max.)	4.4.3	3.2.1.2	4.7.3
105	Moisture and volatile content (wt. % max.)	4.4.3	3.2.1.2	4.7.4
106	Acidity (mg KOH / g max.)	4.4.3	3.2.1.2	4.7.5
107	Acetone insoluble matter (wt. % max.)	4.4.3	3.2.1.2	4.7.6
108	Iron content (wt. % max.)	4.4.3	3.2.1.2	4.7.7
109	Chloride content (wt. % max.)	4.4.3	3.2.1.2	4.7.8 or 4.7.9
Minor				
201	Workmanship	4.4.3	3.2.1.2.1	4.7.1

Classification of characteristics

PARAGRAPH 4.3.2.3	TITLE: Ammonium perchlorate SHEET 1 OF 1		DRAWING NUMBER NEXT HIGHER ASSEMBLY	
CATEGORY	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	PARAGRAPH REFERENCE/ INSPECTION METHOD
Critical	None defined			
Major				
101	Moisture total (%)	4.4.4	3.2.1.3	4.8.2 or 4.8.3
102	Acid insoluble matter (%)	4.4.4	3.2.1.3	4.8.4 or 4.8.5
103	pH of aqueous solution	4.4.4	3.2.1.3	4.8.6
104	Chloride (%)	4.4.4	3.2.1.3	4.8.7 or 4.8.8
105	Sulfated ash (%)	4.4.4	3.2.1.3	4.8.9 or 4.8.10
106	Chloride (%)	4.4.4	3.2.1.3	4.8.11 or 4.8.12
107	Iron (%)	4.4.4	3.2.1.3	4.8.13
108	Phosphate, as TCP (%)	4.4.4	3.2.1.3	4.8.15
109	Purity (%)	4.4.4	3.2.1.3	4.8.14
110	Bulk density (g/in3)	4.4.4	3.2.1.3	4.8.16
111	Nonconformance to Particle size distribution	4.4.4	3.2.1.3	4.8.17
Minor				
201	Workmanship	4.4.4	3.2.1.3.1	4.8.18

Classification of characteristics

PARAGRAPH 4.3.2.4	TITLE: Fine RDX SHEET 1 OF 1		DRAWING NUMBER NEXT HIGHER ASSEMBLY	
CATEGORY	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	PARAGRAPH REFERENCE/ INSPECTION METHOD
Critical	None defined			
Major				
101	Moisture (wt. %)	4.4.5	3.2.1.4.2	4.9.1
102	Nonconformance to Particle size distribution	4.4.5	3.2.1.4.2	4.9.2
Minor	None defined			

Classification of characteristics

PARAGRAPH 4.3.2.5	TITLE: PAX-21 SHEET 1 OF 1		DRAWING NUMBER NEXT HIGHER ASSEMBLY	
CATEGORY	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	PARAGRAPH REFERENCE/ INSPECTION METHOD
Critical	None defined			
Major				
101	DNAN content (wt. %)	4.4.6	3.2.2.2	4.10.1
102	MNA content (wt. %)	4.4.6	3.2.2.2	4.10.1
103	RDX and HMX content (wt. %)	4.4.6	3.2.2.2	4.10.1
104	AP content (wt. %)	4.4.6	3.2.2.2	4.10.2
105	Melting point	4.4.6	3.2.2.2	4.10.3
106	Exotherm onset	4.4.6	3.2.2.2	4.10.3
107	Viscosity	4.4.6	3.2.2.2	4.10.4 or 4.10.5
108	Moisture	4.4.6	3.2.2.2	4.10.6
109	Flake thickness	4.4.6	3.2.2.2	4.10.7
110	Flake Density	4.4.6	3.2.2.2	4.10.8
Minor				
201	Workmanship	4.4.6	3.2.2.3	4.10.12

4.4 Sampling. The tests performed in section 4.6 through 4.10 will be performed on a sample representative of the batch and taken in accordance with ASTM E 300 “Procedure for solids”.

Table IX. Drum sampling plan.

No. of Containers per lot	No. of Containers sampled per lot	Composite Sample 1	Composite Sample 2	Composite Sample 3
1	1	1	1	1
2	2	2	2	2
3 to 15	3	1	1	1
16 to 25	4	1	1	2
26 to 40	5	1	2	2
41 to 65	7	2	2	3
66 to 110	10	3	3	4
111 to 180	15	5	5	5
181 to 300	20	6	7	7
301 to 500	25	8	8	9
501 to 800	30	10	10	10
801 to 1300	35	11	12	12
1301 to 3200	40	13	13	14
3201 to 8000	50	16	17	17

4.4.1 Drum sampling. The individual container samples from a raw material lot shall be combined and blended to form three composite samples. An individual container sample shall not be split between composites, except where a lot consists of fewer than three containers. A single determination of each quality characteristic shall be performed on each of the three composite samples independently. When a lot consists of fewer than three containers, three samples shall be taken from each container. The sample portions obtained shall be blended and grouped to form three composite samples.

4.4.2 DNAN sampling. Sampling will be performed either on a drum loading stream or on drums already loaded. Samples for each DNAN lot shall be tested for conformance in accordance with Section 4.6. If any of lot fails to meet any of the test requirements, it shall be rejected.

4.4.2.1 DNAN sampling during drum loading. Samples selected during drum loading shall be taken as a composite sample from the loading stream.

4.4.2.2 DNAN drum sampling. Sampling of drums shall be according to Table IX. Composite samples shall be approximately 500 grams.

.

4.4.3 MNA sampling. Sampling will be performed on either a drum loading stream or on drums already loaded. Samples for each MNA lot will be tested for conformance in accordance with section 4.7. If any lot fails to meet any of the test requirements, it shall be rejected

4.4.3.1 MNA sampling during drum loading Samples selected during drum loading shall be taken as a composite sample of approximately 1 quart for acceptance testing.

4.4.3.2 MNA Drum sampling. Sampling of loaded drums shall consist of 10 percent of the fiberboard drums in a lot, but not more than 10 percent nor less than 3 drums selected to be representative of the lot. Remove representative portions from each drum to form a composite sample.

4.4.4 AP Sampling. AP sampling will be performed either on a drum loading stream or on already loaded drums. Samples for each AP lot shall be tested in accordance with section 4.8. If any lot fails to meet any requirements as specified in 3.2.1.3, the lot shall be rejected.

4.4.4.1 AP sampling during drum loading. Samples selected during drum loading by the supplier shall be taken as a composite sample from the loading stream.

4.4.4.2 AP drum sampling. Sampling of loaded drums shall be according to Table IX. Composite sample sizes shall be approximately 500 grams.

4.4.5 RDX Sampling. Sampling will be performed on either a drum loading stream, a continuous processing stream or on already loaded drums. Samples for each RDX lot shall be tested for conformance in accordance with Section 4.9. If any lot fails to meet any of the specified requirements, it shall be rejected.

4.4.5.1 Sampling from a loading stream. Samples selected during drum loading shall be taken as a composite sample from the loading stream.

4.4.5.2 Sampling during the FEM process line. In-line sampling of fine RDX shall be conducted during (or after) Fluid Energy Mill (FEM) grinding.

4.4.5.3 RDX drum sampling. Sampling of loaded drums (or other containers) shall be according to Table IX. Composite sample sizes shall be approximately 500 grams.

4.4.6 PAX-21 Sampling. The tests in section 4.10 shall be conducted on two 300-gram samples (two 5000 gram samples when testing for first article inspection applies) representative of the batch taken independently in accordance with ASTM E300 for solids. All batches shall be sampled regardless of test requirements for record keeping and traceability purposes. The selection of batches for inspection sampling and testing shall be in accordance with MIL-STD-1916 except that in lieu of Table IV

Continuous Sampling Plans (MIL-STD-1916), the following continuous sampling plan shall be used: I=7, f=1/3 and there shall be no reduced or tightened inspection level. If any sample fails to meet any test requirement, the batch represented by the sample shall be rejected. All batches produced between the time that the last batch was tested and accepted and the batch which failed shall be tested in accordance with applicable methods given in section 4.10. If any of these batches fail to meet any of the test requirements, that batch shall also be rejected. In addition, after any failure of a batch return to 100% inspection until "I=7" successive batches are accepted.

4.5 Methods of inspection. The following tests shall be performed using prescribed analytical procedures for replicate determination given in standard and analytical textbooks. Unless otherwise specified, all chemicals and reagents shall be ACS Grade or reagent grade chemicals. Alternate test method(s) may be used provided that the method has been evaluated and approved by the Technical Agency (see 6.4).

4.6 DNAN methods.

4.6.1 Purity.

4.6.1.1 DNAN purity is determined via HPLC.

4.6.1.2 Column conditions are as follows:

a. The column consists of a 125 x 4.0 mm Merck LiChrospher RP-8 or equivalent, 5 micron at 40°C.
b. The mobile phase is a 0.05% Trifluoroacetic Acid in water/ isopropanol/ acetonitrile mixed at a volume ratio of 70/ 5/ 25.
c. The flow rate is 1.5 mL / minute.
d. The injection volume is 5 mL.
e. The wavelength to quantify purity of 2,4-dinitroanisole is 380 nm.
f. The wavelength to quantify 1-chloro-2,4-dinitrobenzene is 215 nm.
g. The wavelength to quantify 2,4-dinitrophenol is 300 nm.

4.6.1.3 Sample preparation. Approximately 75 ± 0.1 mg of DNAN is weighed into a scintillation vial. An aliquot of acetonitrile is added to give a final concentration of 10.0 mg DNAN per mL. This solution is sonicated for 1 minute to facilitate dissolution of the sample. This solution is filtered through a 0.45 micron PTFE filter if insoluble material is present. The reference standard for DNAN should be prepared by the same method and to the same concentration.

4.6.1.4 Impurity Standard Preparation. At least five composite standards will be made with the following compounds. They should approximately cover the range listed below. Standards will be made in acetonitrile.

a. 2,4-dinitrophenol: 1 to 200 μg/mL
b. 1-chloro-2,4-dinitrobenzene: 1 to 200 μg/mL

Once these standards are made, a plot of concentration of these impurities versus their associated peak area is made. Using linear regression, these points are fitted with a line.

The slope of this line will be used to determine the percent of impurity in the sample (see 4.6.1.6).

4.6.1.5 Calculation of DNAN Purity:

$$\%DNAN = \frac{sample}{reference} \times 100$$

Where:

sample=Peak area from sample of DNAN lot
reference=Peak area from reference standard

4.6.1.6 Calculation of Impurities:

$$\%(W/w) = \frac{sample}{reference \times Slope} \times 100$$

Where:

sample = Peak area of impurity in sample
reference = 10,000 micrograms/mL
W/w = Constituent
Slope = Impurity calibration curve, peak area/microgram/mL

4.6.2 Purity alternate. An alternate method to determine the purity of 2,4 dinitroanisole and the percentage of 2,6 dinitroanisole present in a sample of 2,4 dinitroanisole.

4.6.2.1 Test Description. This method utilizes High Performance Liquid Chromatography (HPLC) to determine the purity of 2,4-Dinitroanisole (2,4-DNAN), and the amount of the structural isomer, 2,6-Dinitroanisole (2,6-DNAN), present in a sample of 2,4-DNAN. Acetonitrile solutions of accurately weighed samples of 2,4-DNAN are diluted to a concentration 50 ppm and analyzed via Reverse Phase HPLC. The analyses are compared to a linear regression generated from a series of standards of purified 2,4-DNAN and 2,6-DNAN. Synthetic samples are analyzed to ensure the accuracy and precision, and robustness of the method. Pure, LC-grade solvents and acetic acid should be used, as well as clean, undamaged, and calibrated equipment. The standard solutions to be analyzed for establishment of a linear regression should have an isomer ratio of 98% 2,4-DNAN / 2% 2,6-DNAN.

4.6.2.2 Apparatus and reagents.

a. HPLC Quaternary Pump (Thermo Separation Products Model No. P4000 or equivalent), or HPLC Binary Pump (Thermo Separation Products Model No. P2000 or equivalent).
b. HPLC Autosampler (Thermo Separation Products Model No. AS3000 or equivalent).
c. UV/Vis Variable Wavelength Detector (Thermo Separation Products Model No. UV1000 or equivalent).

d. Vacuum Membrane Solvent Degasser (Thermo Separation Products Model No. SCM1000 or equivalent).
e. Vacuum Dessicator and pump system.
f. Acetonitrile, Spec-grade.
g. Methanol, Spec-grade.
h. Deionized Water.
i. Glacial Acetic Acid, High Purity.
j. 2,4-DNAN, purified (see 4.6.2.3).
k. 2,6-DNAN, purified (see 4.6.2.3).

4.6.2.3 Standard Preparation.

a. 2,4-DNAN and 2,6-DNAN will be purified by recrystallization using a constant temperature bath at 75 degrees C. The initial concentration for 2,4-DNAN shall be 40%, when 50.0 g DNAN are completely dissolved in approximately 125 g Spec-Grade Methanol. . For absolute purity, three total recrystallizations are suggested.

The initial concentration for 2,6-DNAN shall be 30%, when 10.0 g of 2,6-DNAN are completely dissolved in approximately 35 g Spec-Grade Methanol. For absolute purity, three total recrystallizations using a constant temperature bath at 75 degrees C are suggested.

b. Standard solution preparation:

1. Obtain six clean, dry 100 mL volumetric flasks. Into one of the flasks, weigh (using an analytical balance accurate to 0.0001 g) 0.4900 g 2,4-DNAN and 0.0100 g 2,6-DNAN, and record the weight to the nearest 0.0001 g. Fill to the mark with spec-grade Acetonitrile. This stock solution is 5000 ppm and has an isomer ratio of 98.0% 2,4-DNAN / 2.0% 2,6-DNAN. Ensure that all DNAN is completely dissolved, and that the solution has been thoroughly mixed before proceeding to dilution.
2. Dilute the solution prepared in Step 1 as follows:
   (a) For a 5 ppm standard, pipette 0.10 mL of the stock solution into a 100 mL volumetric flask and fill to the line with spec-grade Acetonitrile. Label the flask accordingly.
   (b) For a 25 ppm standard, pipette 0.50 mL of the stock solution into a 100 mL volumetric flask and fill to the line with spec-grade Acetonitrile. Label the flask accordingly.
   (c) For a 50 ppm standard, pipette 1.00 mL of the stock solution into a 100 mL volumetric flask and fill to the line with spec-grade Acetonitrile. Label the flask accordingly.
   (d) For a 100 ppm standard, pipette 2.00 mL of the stock solution into a 100 mL volumetric flask and fill to the line with spec-grade Acetonitrile. Label the flask accordingly.

(e) For a 200 ppm standard, pipette 4.00 mL of the stock solution into a 100 mL volumetric flask and fill to the line with spec-grade Acetonitrile. Label the flask accordingly.

3. After ensuring that all solutions are thoroughly mixed, draw aliquots to fill HPLC vials and load into the autosampler tray.

4.6.2.4 Sample Preparation.

a. Obtain samples of 2,4-DNAN that have been dried for at least 12 hours in a 60°C steam-heated oven.

b. Weigh approximately 5.0000 g of the production material into clean, dry 100 mL volumetric flask. Fill the flask to the line with acetonitrile and label the sample accordingly. This is the stock solution from which the sample solution will be made. Ensure that all DNAN is completely dissolved and that the solution is thoroughly mixed before proceeding to dilution.

c. Using a Variable Volume Micropipettor, transfer 0.10 mL of the stock solution into a clean, dry 100 mL volumetric flask. Fill to the line with acetonitrile and label the sample flask accordingly. This should result in a solution with a sample concentration of 50 ppm. Ensure that the solution has been mixed thoroughly prior to drawing an aliquot for analysis on the HPLC system.

d. Repeat steps 2 and 3 for each individual sample to be tested.

e. Using a disposable plastic pipette, fill a 2 mL HPLC vial to ¾ capacity with the first sample solution and affix the vial cap firmly. Load the vial into the autosampler tray, noting its location.

f. Repeat Step 5 for each sample solution, using a fresh vial and pipette for each remaining sample.

4.6.2.5 Mobile Phase Preparation.

a. Into a 1 liter HPLC solvent bottle, add 500 mL distilled, deionized water.

b. Pipette 0.5 mL glacial (pure) Acetic Acid into the solvent bottle.

c. Add 500 mL distilled, deionized water to the 1 L solvent bottle. Swirl the solution briefly to ensure mixing of the acetic acid in the water.

d. Ensure that the tubing line reaches the bottom of the solvent bottle after the cap has been securely affixed to the top of the bottle. Place the solvent bottle in the bay on the top of the Online Degasser.

e. Flip the mixing switch on the Online Degasser from “Auto” to “Run,” and

then back to "Auto." It is good practice to purge the system after any modification of the solvent containers.

4.6.2.6 Run samples. Set appropriate HPLC parameters for data collection. A series of five standards, in concentrations of 5, 25, 50, 100, and 200 ppm will be used to generate a linear equation using a least-squares regression. The R^2, or the fraction of the total squared error of the linear equation, should be at least 0.990. If this is not the case, analysis of further samples should not be continued until a series of standards can be analyzed to provide such a linear equation.

Using purified standard 2,4-DNAN and 2,6-DNAN, a synthetic sample of known isomer ratio will be analyzed, in triplicate, alongside any production samples provided. Because the standards used to generate aforementioned linear equation are of 98% 2,4-DNAN / 2% 2,6-DNAN, the 50 ppm standard can be used as the synthetic sample.

Purge HPLC system. Enter necessary information into data acquisition program. Run analysis on sample vials. Collect the automatically printed chromatograms and reports. Use MS Excel (or equivalent) to analyze data. This includes using the chromatographic peak areas to determine the component percentages in the DNAN samples

4.6.2.7 Calculation to determine purity and concentration.

Computer software package is used to establish a calibration equation based on the concentration of 2,4-DNAN and 2,6-DNAN in the five 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 2,4-DNAN and 2,6-DNAN can be determined.

(Note: Consult with HPLC system operating instructions from the manufacturer for detailed operating procedure and parameter settings to be used. Instrument operating parameters should be verified and validated before used for actual analyses of production samples.)

4.6.3 Melting point.

a. Melting point is determined via Differential Scanning Calorimetry (DSC).

b. Weigh 6.000 grams to the nearest 0.5 milligrams. Place the DNAN into a 15 microliter aluminum cup and run at 20 C°/min.

c. Onset (Melting point) is determined by taking the intersection of a tangent line drawn from the steepest slope of the endotherm peak to the extrapolated baseline.

4.6.4 Moisture determination (Method I).

a. An automated volumetric Karl Fischer titrator will be required for this testing.

b. Standardize a pyridine based Karl Fischer titrant having approximate E-value of 1 to 25 mg/mL. A weighed or accurately syringed quantity of water shall be used as a reference standard.

c. The carrier solution shall be approximately 100 mL of a 1:1 mixture of pyridine and chloroform that has been allowed to equilibrate for a minimum of 10 minutes.

d. Weigh 2 ± 0.0001 grams of DNAN.

e. Place the DNAN in the carrier solution and allow a minimum of 10 minutes for the sample to dissolve completely. This is usually accomplished by using a pause or hold function of the titrator.

f. Titrate the sample to a 30 second amperometric endpoint. Because of solubility limitations the carrier should be replaced after two sample determinations.

g. Titrate an extraction blank on the carrier on each day of analysis to account for atmospheric moisture titrated during the 10-minute extraction time. Open and close the vessel as though a sample is being placed in the carrier and perform the same 10-minute extraction as with the sample. Record the volume of the blank titration.

h. The moisture content is calculated as follows:

$$\%Water = \frac{(Vol - Bl) \times E}{10 \times Wt}$$

Where:

Vol = Volume of KF titrant required to titrated the sample, mL
E = Titrant equivalence value, milligram/mL
Wt = Sample weight, gram
Bl = Volume of KF titrant used to titrate extraction blank, mL

Duplicate determinations are sufficient.

4.6.5 <u>Moisture determination (Alternative method)</u>.

a. Place 10 grams of DNAN into a pre-tared container.

b. Place in a vacuum oven for a minimum of 48 hrs at 145°F at 1.0 mm Hg.

c. Calculation of moisture:

$$\%Moisture = \left(1 - \frac{Wt - Tare}{St - Tare}\right) \times 100$$

Where:

Wt = End weight of container and DNAN, gram
St = Start weight of container and DNAN, gram
Tare = Weight of tared container, gram

4.6.6 Undissolved solids.

a. Place 1.0 ± 0.001 grams of DNAN in a 200 mL beaker.

b. Add 100 mL of methanol to this DNAN and stir for a minimum of 10 minutes.

c. Screen material through a pre-tared medium porous crucible.

d. Calculation of undissolved solids:

$$\%UndissolvedSolids = \frac{Wt - Tare}{St} \times 100$$

Where:

Wt = Weight of tare and undissolved solids, gram
Tare = Weight of the crucible, gram
St = Weight of DNAN, gram

4.6.7 Trace Metal Content.

4.6.7.1 Test Description. This procedure is used for the determination and quantitation of trace elements in dinitroanisole (DNAN). As a minimum, the DNAN sample shall be analyzed for trace metal contents for the following metals: aluminum, calcium, chromium, iron, magnesium, sodium, and zinc. Following an appropriate sample preparation the metals are determined simultaneously in solution. Sample solutions are nebulized into a radio-frequency inductively coupled plasma where desolvation and excitation occur. Atomic-line emission spectra characteristic of the metals found in the sample are produced and measured by an optical spectrometric technique involving dispersion by a grating and detection by a photosensitive device. A computer system collects and processes photocurrents from this device. Background correction must also be applied, except in cases of line broadening, and is usually handled by the computer system. Possible additional interferences also need to be monitored and appropriate compensation made. This method is not capable of distinguishing specific species of an element, but instead is an indication of the total amount of the given element present in the material.

4.6.7.2 Apparatus and reagents.

a. An inductively coupled plasma-atomic emission spectrometer (ICP-AES) that is capable of operating in a simultaneous or sequential mode and uses ionized argon gas as the plasma. System must be capable of fulfilling and complying with the requirements and description of the technique outlined above. System and processing of background corrected signals must be computer controlled.

b. Liquid argon gas supply.

c. Variable peristaltic pump capable of delivering sample and standard solutions to the nebulizer.

d. Assortment of digital air displacement pipetters that cover the range from 0.1 to 2500 µL with suitable tips and Repipet II dispensers or equivalent that cover the 0.0 to 10.0 mL range of volume.

e. Lab ware and the laboratory work area must be clean to avoid significant errors in determining trace levels of the analytes.

f. Assorted volumetric flasks.

g. Nitric and hydrochloric acid, concentrated. Required dilutions should be made using distilled water.

h. All water used in sample preparation and dilutions should be distilled water.

i. Stock metal solutions should be purchased from a reputable commercial vendor. All stock solutions are 1000 µg/mL with the exception of the potassium and sodium stock solutions, which should be 10,000 µg/mL.

j. Mixed calibration standards can be prepared by mixing appropriate volumes of the stock solutions in 500 mL volumetric flasks. Prepare by adding 20 mL of (1+1) nitric acid and 20 mL of (1+1) hydrochloric acid to the flask, followed by the aliquots of the required stock standard solutions and dilute with distilled water. Transfer each mixed standard to a clean polyethylene bottle that has not been previously used. The following recommended concentrations and combinations of metals for each standard are given below and may be used as is or modified to standardize the instrument for the quantitative determination of trace materials in the material.

k. Mixed Calibration Standard I is prepared by adding 0.25 mL of the stock solution of Ag, 0.5 mL of the stock solution of Ba, 1.0 mL of the stock solutions of B, Cd, Cu, and Mn, 2.5 mL of the stock solutions of Sb and Se, and 5.0 mL of the stock solutions of As and Ca to a 500 mL volumetric flask as prepared above and diluting as above.

NOTE: If the silver precipitates initially, add 15 additional mL of water and warm until the solution clears.

l. Mixed Calibration Standard II is prepared by adding 0.5 mL of the stock solution of Sr, 1.0 mL of the stock solution of K, 2.5 mL of the stock solution of Na,

and 5.0 mL of the stock solution of Mo to a 500 mL volumetric flask as prepared above and diluting as above.

m. Mixed Calibration Standard III is prepared by adding 1.0 mL of the stock solutions of Co and V and 5.0 mL of the stock solution of P to a 500 mL volumetric flask as prepared above and diluting as above.

n. Mixed Calibration Standard IV is prepared by adding 2.0 mL of the stock solution of Sn, 2.5 mL of the stock solutions of Cr and Zn, and 5.0 mL of the stock solutions of Al and Si to a 500 mL volumetric flask as prepared above and diluting as above.

o. Mixed Calibration Standard V is prepared by adding 0.5 mL of the stock solution of Be, 1.0 mL of the stock solution of Ni, 2.5 mL of the stock solution of Tl, and 5.0 mL of the stock solutions of Fe, Mg, and Pb to a 500 mL volumetric flask as prepared above and diluting as above.

p. A calibration blank that is used to establish the analytical curve is prepared by adding 20 mL of (1+1) nitric acid and 20 mL of (1+1) hydrochloric acid to a 500 mL flask and diluting to volume with deionized distilled water. This solution should be stored in a polyethylene bottle. Finally, the rinse blank should be prepared as the calibration blank and is used to flush the instrument sample uptake system and reduce memory interferences.

q. A laboratory performance check solution should be prepared from the stock standard solutions in a 2% nitric and hydrochloric acid matrix. Appropriate dilutions of the stock solutions should be made that will result in a concentration of 2.0 ppm for all elements except for K and P, which should be 10 ppm, and Na which should be 20 ppm.

r. A quality control sample should be prepared in the same acid matrix as the calibration standards at appropriate concentrations such as 1.0 ppm for most elements. This sample should likewise be stored in a polyethylene bottle and be obtained from a source independent of that from which the calibration standards were prepared.

s. A microwave unit that is capable of providing programmable power of at least 574 W and which can be programmed to be within ± 10W of the required power. The microwave unit cavity should be corrosion resistant and well ventilated. All electronics should also be protected against corrosion for safe operation. The unit should be use temperature feedback control to provide the primary control performance mechanism for the method, but it may also be pressure controlled and monitored. Temperature feedback control is necessary for reproducible microwave heating allowing flexibility in the reagents used to achieve total decomposition for the analysis. The microwave unit should meet temperature performance requirements that allow the temperature to be sensed to within ± 2.5 and to automatically adjust the microwave field output power within

2 seconds of sensing if this is not the case. Temperature sensors should be accurate to ± 2C°. A temperature calibration control mechanism should be developed for each specific set of reagent(s) combination(s), quantity, and specific vessel type and number. The system requires fluorocarbon digestion vessels with a volume of at least 45 mL and capable of pressures of at least 30 atm (30 bar or 435 psi) and having a controlled pressure relief. A rotating turntable should also be employed to ensure homogeneous distribution of microwave radiation in the unit. The speed of the turntable should be a minimum of 3 rpm. Follow the manufacturer's recommendations regarding the proper and safe operation of the microwave equipment and vessels.

4.6.7.3 Procedure.

a. Sample preparation requires digestion of approximately 100 milligrams of dinitroanisole. Weigh and record the amount of material weighed into each digestion vessel. Add 5.0 mL of concentrated nitric acid to all the digestion vessels. The determination may be performed in triplicate for each sample lot. Samples may also be spiked with known amounts of trace metals, blank spikes may be included, or blanks containing only nitric acid may be added to fill the digestion set. Digest using a microwave program that uses 1200 W of power if all twelve vessels are included in the microwave carousel. The program should ramp to a temperature of 200°C with a pressure limit of 400 psi and hold at the temperature of 200°C. After completion of the digestion, allow the vessels to cool, and add 20 mL of distilled water to complete sample preparation. The sample is now ready for analysis by ICP-AES.

b. Specific instrument operating conditions will be those recommended by the instrument manufacturer. Typical conditions are as follows: Incident rf power – 1100 watts, Reflected rf power - < 5 watts, Viewing height above the work coil – 15 mm, Injector tube orifice i.d. – 1 mm, Argon supply – liquid, Argon pressure – 60 psi, Coolant Ar flow rate – 19 L/min, Aerosol carrier Ar flow rate – 620 mL/min, Auxillary (plasma) Ar flow rate – 300 mL/min, and Sample uptake rate – 1.2 mL/min. The instrument should become thermally stabilized for a period of thirty minutes before use and optimization.

c. Optically profile the plasma following the manufacturer's instructions.

d. The instrument should be calibrated for operation following the manufacturers instructions using the calibration blank and standards previously prepared.

e. Standards and samples should be introduced to the instrument's nebulizer using the peristaltic pump.

f. Equilibrium should be reached in the plasma by aspirating the samples and standards into the plasma 30 seconds before integration of the background corrected signal.

g. Use at least three 5 second background corrected integration periods to obtain the atomic emission signal for use in the calculation of the standard curve and concentration data. The nebulizer and sample uptake system should be flushed with calibration blank between each sample and the standards for a minimum of sixty seconds to ensure that analyte memory effects are not occurring.

h. Immediately following the calibration of the instrument, the calibration blank and laboratory performance check solution should be analyzed. This solution should be also analyzed at the end of the sample run and after every tenth sample. The quality control sample should be analyzed with each daily analytical run.

4.6.7.4 Quality control. The laboratory performance check and quality control sample values for each analyte should be within 90% to 110% of the expected values. If this condition is not met the solution should be reanalyzed and if results for an analyte of interest are again outside ±10%, the instrument should be recalibrated. If a laboratory performance check fails, all samples analyzed following the last acceptable check should be reanalyzed.

4.6.7.5 Calculation. Instrument results for the targeted analytes should be multiplied by the volume (0.025 L), divided by the weight in grams, and by the dilution factor when appropriate to calculate the concentration of a given element in units of parts per million in the DNAN. If final results for trace elements in DNAN are desired in weight percent, divide the previous result by 10,000.

4.6.8 Visual examination. Unless otherwise specified, all visual examinations shall be conducted with an unaided eye, except for normal corrected vision (See also 3.2.1.1.1).

4.7 MNA methods.

4.7.1 Visual examination. Unless otherwise specified, all visual examinations shall be conducted with an unaided eye, except for normal corrected vision (See also 3.2.1.2.1).

4.7.2 Color. Color of the MNA shall be determined in accordance with the following:

4.7.2.1 Apparatus.

a. Spectrophotometer – visible range, with 10-millimeter pathlength cells.

b. Filter – medium-porosity glass fiber.

4.7.2.2 Reagents.

a. Acetone.

4.7.2.3 Procedure.

a. Weigh 5.00 ± 0.01 grams of sample and transfer it to a beaker. Add approximately 150 mL of acetone. Stir to completely dissolve the sample.

b. Filter the solution through a medium filter paper into a 250-mL volumetric flask. Wash the beaker and filter with acetone to obtain a qualitative transfer. Bring the flask to volume with acetone, and mix.

c. Zero the instrument, using acetone as a reference, at 500 nanometers (nm). Fill the cell with the sample and obtain the absorbance at 500 nm.

d. Obtain absorbances of the sample at 580 nm and 650 nm in the same manner as above, always rezeroing the instrument at each new wavelength.

e. Calculate the total absorbance (or color value) as follows:

$$\text{Total absorbance} = \text{abs}_{500nm} + \text{abs}_{580nm} + \text{abs}_{650nm}$$

4.7.3 Total unidentified impurities. The DMNA and PCNB content of the MNA shall be determined as follows:

4.7.3.1 Apparatus. The apparatus is a liquid chromatograph equipped with the following:

a. Detector – ultraviolet (UV).

b. Column – normal phase, capable of separating components of interest.

4.7.3.2 Reagents and standards.

a. 1,2-dimethoxyethane (glyme) – high performance liquid chromatograph (HPLC) grade.

b. n-heptane – HPLC grade.

c. N,N-dimethyl-p-nitroaniline.

d. Para-chloronitrobenzene (PCNB).

e. MNA – recrystallized from ethanol.

f. PCNB solution – Weigh approximately 0.125 gram of PCNB to the nearest 0.0001 gram in a 100-mL volumetric flask. Dissolve and dilute to volume with glyme. Mix well.

g. DMNA solution – Weigh approximately 0.125 gram of DMNA to the nearest 0.0001 gram in a 100-mL volumetric flask. Dissolve and dilute to volume with glyme. Mix well.

h. MNA solution – Weigh approximately 0.125 gram of MNA to the nearest 0.0001 gram in a 100-mL volumetric flask. Dissolve and dilute to volume with glyme. Mix well.

i. Working standard – Into a 250-mL flask, volumetrically pipet 5 mL of the DMNA standard solution, 5 mL of the MNA standard solution, and 5 mL of the PCNB standard solution. Add n-heptane to match the mobile phase concentration and dilute to volume with glyme. Mix well. The standard is now about 0.025 milligram/mL in PCNB and DMNA. This approximates the maximum allowable contents in the sample.

4.7.3.3 Procedure.

a. Weigh approximately 1.25 gram of sample to the nearest 0.0001 gram in a 250-mL volumetric flask. Dissolve in glyme and add n-heptane to match the mobile phase concentration. Dilute to volume with glyme. Mix well.

b. Analyze the sample solution and the working standard using liquid chromatographic conditions as follows:

1. Detector – set at 254 nm, and sensitivity sufficient to integrate clearly the peaks of interest.

2. Mobile phase – glyme/n-heptane ratio necessary to resolve the peaks of interest.

3. Flow rate – 1.0 to 1.5 mL per minute.

c. Integrate the PCNM, MNA, and DMNA peaks of the working standard. Integrate all peaks of the sample (excluding solvent and MNA peaks) up to a time of twice the DMNA retention time. The elution order of identifiable peaks is as follows: PCNB, DMNA, and MNA.

4.7.3.4 Calculations.

NOTE

Area or height may be used in calculations, but the same units (area or height) shall be used for the working standard and sample.

a. DMNA content.

1. Calculate the calibration factor, F, for DMNA as follows:

$$\text{Factor, F} = \frac{A}{C}$$

Where:
A = Peak parameter of DMNA in working standard
C = Concentration of DMNA in working standard, milligram/mL

2. Calculate the percent by weight of DMNA in the sample as follows:

$$\text{DMNA content, percent} = \frac{B \times D}{F \times W} \times 100$$

Where:
B = Peak parameter of DMNA in sample
D = Dilution factor, 250 mL
F = Calibration factor for DMNA as calculated above.
W = Weight of sample, milligram

b. PCNB content.

1. Calculate the calibration factor, F, for PCNB as follows:

$$\text{Factor, F} = \frac{A}{C}$$

Where:
A = Peak parameter of PCNB in working standard
C = Concentration PCNB in working standard, milligram/mL

2. Calculate the percent by weight of PCNB in the sample as follows:

$$\text{PCNB content, percent} = \frac{B \times D}{F \times W} \times 100$$

Where:
B = Peak parameter of PCNB in sample
D = Dilution factor, 250 mL
F = Calibration factor for PCNB as calculated above.
W = Weight of sample, milligram

c. Calculate the total unidentified impurities in the sample as follows:

$$F = \frac{A}{C}$$

Where:
A = Peak parameter of MNA in working standard
C = Concentration of MNA in working standard, milligram/mL

$$\text{Total unidentified impurities, percent} = \sum\left[\frac{B \times D}{F \times W}\right] \times 100$$

Where:
B = Peak parameter of each unidentified component in sample.
D = Dilution factor, 250 mL
F = Calibration factor for MNA as calculated above
W = Weight of sample, milligram

d. Calculate the total unidentified impurities in the sample as follows:

$$F = \frac{A}{C}$$

Where:
A = Peak parameter of MNA in working standard
C = Concentration of MNA in working standard, milligram/mL

$$\text{MNA Purity, percent} = \left(\frac{B \times D}{F \times W}\right) \times 100$$

Where:
B = Peak parameter MNA in sample.
D = Dilution factor, 250 mL
F = Calibration factor for MNA as calculated above
W = Weight of sample, milligram

4.7.4 Total moisture and volatiles. The total moisture content of the MNA shall be determined in accordance with MIL-STD-650, Method 101.6.

4.7.5 Acidity. The acidity of the MNA shall be determined as follows:

4.7.5.1 Reagents.

a. Weigh, to the nearest 0.0001 g, approximately 5 g of the MNA sample in a 150-ml beaker. Add approximately 100 ml of acetone and a magnetic stir bar. Stir the sample into solution and purge about 5 minutes with nitrogen to remove carbon dioxide (CO_2).

NOTE

The CO_2 is slowly absorbed by the solution and can affect the endpoint of the titration. To prevent this, keep the time between sparging and titration to a minimum. Other acceptable methods of excluding CO_2, such as covering of the beaker or continued sparging, are also acceptable.

b. Titrate the sample potentiometrically with 0.01-N KOH purging during the titration. A full-scale expansion of 750 millivolts (mV) has been found to be appropriate.

c. Perform a blank titration on acetone alone. Locate the equivalence points by conventional technique.

d. Calculate the acidity as follows:

$$\text{Acidity (milligram KOH/gram)} = \frac{(A-B)(N)(56.1)}{W}$$

Where:

A = Volume of KOH required to titrate the sample, mL
B = Volume of KOH required to titrate the blank (if blank requires no titrant, enter 0.00 ml), mL
N = Normality of titrant, equivalents/liter
56.1 = Gram of KOH/equivalent
W = Weight of the sample, gram

4.7.6 Acetone insoluble matter. Acetone insoluble matter shall be determined as follows:

a. Weigh, to the nearest mg, approximately 10 g of sample into a beaker with a capacity of not less than 500 ml. Add approximately 400 ml of acetone to the beaker and dissolve the sample.

b. Filter the solution through a tared medium porosity sintered glass filter crucible, rinsing out the beaker with acetone and passing the rinsings through the crucible.

c. Wash the residue in the crucible with acetone until the washings are colorless. Continue suction for not less than 5 additional minutes.

d. Dry the crucible at 100 ± 2 °C for not less than 1 hour, cool to ambient temperature in a desiccator, and weigh.

CAUTION
ACETONE IS AN EXTREMELY FLAMMABLE SOLVENT. INCOMPLETE REMOVAL COULD CAUSE AN OVEN EXPLOSION.

e. Calculate the acetone insoluble matter as follows:

$$\text{Acetone insoluble matter, percent} = \frac{B - A}{W} \times 100$$

Where:
A = Tare weight of crucible, gram
B = Weight of crucible and residue, gram
W = Weight of sample, gram

f. Report the percent acetone insoluble matter to the nearest 0.01 percent.

4.7.7 Iron content. The iron (Fe) content shall be determined as follows:

4.7.7.1 Apparatus. The apparatus shall be an atomic absorption spectrophotometer equipped and set for the following conditions:

a. Hollow cathode lamp for Fe – current appropriate for lamp.

b. Slit width – appropriate for equipment and element Fe.

c. Wavelength – 248.3 nm.

d. Aspiration rate – approximately 6 mL/minute.

e. Burner – air/acetylene oxidizing flame stoichiometric to oxidizing.

4.7.7.2 Reagents and standards.

a. Hydrochloric acid (HC1)

b. Commercial 1,000 parts per million (ppm) standard or Fe stock solution (1,000 micrograms/mL) – To make Fe stock solution, place 1.0

g of Fe wire, weighed to the nearest 0.1 mg, into a 100-ml beaker. Dissolve the wire in a minimum volume of 1:1 $HN0_3$. Transfer to a 1-liter flask and dilute to volume with deionized water.

c. Acetonitrile.

4.7.7.3 Preparation of standards.

a. Fe standard, 1 ppm: Into a 500-mLl volumetric flask, add 5.0 mL of a 100-ppm stock solution (made by diluting 50.0 mL of a 1,000-ppm stock solution to 500 mL), 45 mL of deionized water, and 50 mL of concentrated HC1. Dilute to volume with acetonitrile.

b. Fe standard, 2 ppm: Into a 500-mL volumetric flask, add 1.0 mL of a 1,000-ppm stock solution, 49 mL of deionized water, and 50 mL of concentrated HC1. Dilute to volume with acetonitrile.

c. Fe standard 4 ppm: Into a 500-mL volumetric flask, add 2.0 mL of a 1,000-ppm stock solution, 48 mL of deionized water, and 50 mL of concentrated HC1. Dilute to volume with acetonitrile.

d. Blank: Into a 500-mL volumetric flask, add 50 mL of deionized water and 50 mL of concentrated HC1. Dilute to volume with acetonitrile.

4.7.7.4 Preparation of samples.

a. Weigh, to the nearest 0.0001 g, 1.0 g of MNA into a 50-mL volumetric flask.

b. Add 5 mL of concentrated HC1 and 25 mL of acetonitrile. Heat the flask gently on a hot plate until all solids have dissolved.

c. Add 5 mL of deionized water and let the flask cool to room temperature.

d. Dilute to volume with acetonitrile and shake the flask vigorously until the contents are mixed.

4.7.7.5 Procedure.

a. Use the atomic absorption spectrophotometer to obtain absorbance values for the standards, sample, and blank. Subtract the absorbance value of the reagent blank from the sample and standard absorbance values. Use deionized water to zero the instrument.

b. Plot the absorbance of the standards versus the concentrations. Obtain the concentration of the sample solution from this graph. Since the plot of absorbance versus concentration is linear up to 5 micrograms/mL, a one-point calibration can be used in this range.

c. Calculate the percent Fe as follows:

$$\text{Percent Fe} = \frac{C \times D}{W \times \left(1 \times 10^{4}\right)}$$

Where:

C = Sample concentration as determined by comparison to standard solutions, micrograms/mL
D = Sample dilution volume, mL
W = Sample weight, gram

4.7.8 Chloride content. The chloride content shall be determined as follows:

4.7.8.1 Equipment and reagents.

a. Potentiometric titrator.

b. Silver billet electrode.

c. Modified calomel electrode containing a saturated solution of potassium nitrate.

d. Acetic acid – glacial.

e. Silver nitrate solution – 0.005 N.

4.7.8.2 Procedure.

a. Weigh, to the nearest mg, approximately 10 g of sample in a 250-mL beaker, and wash the sample with two successive 100-mL portions of boiling distilled water.

b. Filter each portion into a 400-mL beaker through a Whatman No. 40, or equivalent, filter paper, then cool the filtrate to approximately 25°C.

c. Acidify the extract with a few drops of glacial acetic acid and titrate potentiometrically with 0.005-N silver nitrate.

d. As a blank determination, omit the sample and filter 200 mL of boiling distilled water through a Whatman No. 40, or equivalent, filter paper and proceed as with the sample.

e. Calculate the water-soluble chlorides as follows:

$$\text{Chloride content, percent by weight} = \frac{(A-B)(N)\times 35.46}{1000\times W}\times 100$$

Where:

A = volume of silver nitrate required for sample titration, mL
B = volume of silver nitrate required for blank titration, mL
N = normality of silver nitrate solution, equivalents/liter
W = weight of sample, gram
35.46 = equivalent weight of chloride, gram/equivalent

4.7.9 Chloride content (alternate method). See 4.8.7 and 4.8.8 for procedure, (test procedure for chloride in the ammonium perchlorate test method section).

4.8 Ammonium Perchlorate testing methods.

4.8.1 Sampling. Samples for the following methods will be taken from the composite sample. They will be selected randomly from the sample using a riffler or equivalent.

4.8.2 Total Moisture (Method I).

a. Weigh a 2-3 gram sample to the nearest 0.001 gram.

b. Dissolve the sample in 100 milliliters of neutralized carrier solution (chloroform/methanol in a ratio of 70:30).

c. Titrate the moisture using a Karl Fischer titration system containing pyridine free titrant with an approximate equivalence value of 1.0 to 2.0 mg H_2O/mL titrant.

d. Consider the determination complete when an endpoint is reached which is stable for 30 seconds.

e. Calculate total moisture as follows:

$$\%Moisture = \frac{V\times E}{10\times W}$$

Where:

V = Volume of standardized Karl Fischer reagent, mL
E = Water equivalent of standardized Karl Fischer reagent, milligram/mL

W = Weight of sample, gram

Report moisture to nearest 0.01 percent.

4.8.3 Moisture determination (Alternative method).

a. Weight a 15 gram sample to the nearest 0.1 gram.

b. Dissolve the sample in approximately 100 mL of 3:1 pyridine-methanol carrier solution, which has been previously neutralized with a full concentration Karl Fischer reagent.

c. Consider the determination complete when an endpoint is reached.

d. Calculate moisture as follows:

$$\%Moisture = \frac{V \times E}{10 \times W}$$

Where:
V = Volume of standardized Karl Fischer reagent, mL
E = Water equivalent of standardized Karl Fischer reagent, milligram/mL
W = Weight of sample, gram

4.8.4 Acid insoluble matter (Method I).

a. Weigh a 25 gram sample to the nearest 0.1 gram.

b. Dissolve the sample in approximately 250 mL of distilled water and add 5 mL of 10 percent hydrochloric acid.

c. Filter the solution through a tared, medium-porosity, sintered glass crucible and wash with distilled water.

d. Dry the crucible for 90 ± 10 minutes at 105 ± 5°C, cool to room temperature and weigh to the nearest 0.1 mg.

e. Calculate acid insoluble matter by the following formula:

$$\%\ Acid\ Insoluble\ Matter = \frac{W3 - W2}{W1} \times 100$$

Where:
W1 = Sample Weight, gram
W2 = Crucible weight, gram

W3 = Weight of crucible plus residue, gram
Report results to the nearest 0.01 percent.

4.8.5 Acid Insoluble Matter (Alternative method).

a. Weigh a 50 gram sample to the nearest 0.1 gram.

b. Dissolve the sample in 350 ± 70 mL of water and add 5 mL of 1:1 by volume of hydrochloric acid.

c. Filter the solution through a micron membrane filter and wash with water.

d. Dry the crucible for 55 minutes at 105 ± 5°C, cool to room temperature, and weigh to the nearest 0.1 mg.

e. Calculate insoluble matter:

$$\%\ Acid\ Insoluble\ Matter = \frac{W3 - W2}{W1} \times 100$$

Where:
W1 = Sample weight, gram
W2 = Filter Weight, gram
W3 = Filter Weight plus residue, gram

4.8.6 pH of aqueous solution.

a. Weigh a 20 g sample to the nearest 0.1 g.

b. Dissolve the sample in 70 mL of neutral water.

c. Measure the pH of the solution with a pH meter previously calibrated with buffer solutions.

d. Report pH to nearest 0.1 unit

4.8.7 Chloride (Method I).

a. Weigh a 10 gram sample to the nearest 0.1 g.

b. Dissolve the sample in approximately 100 mL of distilled water and 5 mL of 6 N nitric acid.

c. Add 10.0 mL of standard 0.05 normal silver nitrate, plus 2 mL of nitrobenzene and 2 mL of 40 percent ferric alum indicator.

d. Back titrate the solution with standard 0.05 normal potassium thiocyanate.

e. Calculate percent chloride by the following formula:

$$\%Chloride = \frac{5.35 \times [(A \times B) - (C \times D)]}{W}$$

Where:

A = Volume of standard silver nitrate, mL
B = Normality of standard silver nitrate
C = Volume of standard potassium thiocyanate, mL
D = Normality of standard potassium thiocyanate
W = Sample Weight, gram

Report Chloride , as ammonium chloride to the nearest .001 percent

4.8.8 Chloride (Alternative method).

a. Weigh a 10 gram sample to the nearest 0.1 g.

b. Dissolve the sample in 70 ± 1.4 mL of water and 1 mL of concentrated nitric acid.

c. Titrate the solution, while stirring, with 0.0030 to 0.0040 normal silver nitrate using a silver billet electrode coated with silver chloride to detect endpoint.

d. Calculate percent chloride as follows

$$\%Chloride = \frac{53.5 \times A \times N}{10 \times W}$$

Where:

A = Volume of standard silver nitrate, mL
N = Normality of standard silver nitrate
W = Sample weight, gram

4.8.9 Sulfated Ash (Method I).

a. Weigh a 10 gram sample to the nearest 0.1 g into a tared silica crucible. Wet the sample with 50 percent sulfuric acid, place cover on the crucible and heat over a burner to initiate decomposition

b. After all reaction has ceased, heat the residue to evolve all fumes, and place the crucible into a muffle furnace for a minimum of 10 minutes at 800 ± 50°C.

c. Remove from the muffle furnace, cool to room temperature in desiccator, weigh to the nearest 0.1 mg, and save residue for the iron test.

d. Calculate sulfated ash:

$$\%SulfatedAsh = \frac{172 \times (B - C)}{A - C}$$

Where:

A = Sample Weight plus crucible weight, gram
B = Residue weight plus crucible weight, gram
C = Crucible weight, gram

Report sulfated ash, as sodium perchlorate, to the nearest 0.1 percent.

4.8.10 Sulfated Ash (Alternative method).

a. Weigh a 5 gram sample to the nearest 0.1 g into a tared silica crucible. Wet the sample with concentrated sulfuric acid, place a cover on the crucible and heat to initiate decomposition.

b. After all reaction has ceased heat the residue to evolve all fumes and place into a muffle furnace for a minimum of 10 minutes at 800 ± 50°C.

c. Remove from muffle furnace, cool to room temperature in a desiccator, weigh to the nearest 0.1 mg and save the residue for the iron test.

d. Calculate percent sulfated as sodium perchlorate as follows:

$$\%SodiumPerchlorate = \frac{1.72 \times (B - C)}{A - C} \times 100$$

Where:

A= Sample weight plus crucible weight, gram
B = Residue weight plus crucible weight, gram
C = Crucible weight, gram

4.8.11 Chlorate (Method I).

a. Weigh a 10 g sample to the nearest 0.01 g into an Erlenmeyer flask and add approximately 75 mL of distilled water.

b. Add 10.0 mL of 1 percent ferrous sulfate solution using pipette or buret and add 10 mL of mixed 15 percent sulfuric acid and 10 percent orthophosphoric acid solution.

c. While stirring, boil for 5 minutes then stopper and cool to room temperature.

d. Titrate with 0.01 N potassium permanganate to the first faint pink endpoint.

e. Carry at least two reagent blanks throughout the entire procedure.

f. Calculate percent chlorate as ammonium chlorate as follows:

$$\%AmmoniumChlorate = \frac{[(B \times N) - (S \times N)] \times 1.6917}{W}$$

Where:

B = Volume of potassium permanganate required to titrate blank, mL
S = Volume of potassium permanganate required to titrate sample, mL
N = Normality of potassium permanganate
W = Sample weight, gram

4.8.12 Chlorate (Alternative method).

a. Weigh a 10 g sample to the nearest 0.05 g into an Erlenmeyer flask and add approximately 75 mL of distilled water.

b. Add 1.50 ± 0.02 mL of 0.25 M ferrous sulfate solution using pipette or microburet and add 10 mL of mixed 15 percent sulfuric acid and 10 percent phosphoric acid solution.

c. While stirring, boil for 5 minutes then stopper and cool to room temperature.

d. Titrate with 0.01 N potassium permanganate to the first faint pink endpoint.

e. Carry at least two reagent blanks throughout the entire procedure.

f. Calculate percent chlorate as ammonium chlorate as follows:

$$\%AmmoniumChlorate = \frac{[(B \times N) - (S \times N)] \times 1.6917}{W}$$

Where:

B = Volume of potassium permanganate required to titrate blank, mL

S = Volume of potassium permanganate required to titrate sample, mL
N = Normality of potassium permanganate
W = Sample weight, gram

Report chlorate as ammonium chlorate to the nearest 0.01 percent.

4.8.13 Iron. Using the sulfated ash residue from paragraph 4.8.9 or 4.8.10.

(1) Preparation of standard iron curve.

a. Weigh 0.05 g, to the nearest 0.1 mg, of bright reagent iron wire. Place the wire into a 1000 mL volumetric flask. Add 2 mL of concentrated HCl and a few mL of water. Heat until solution is complete. Cool, dilute to volume, and mix well. Each mL of solution now contains 50 micrograms of iron

b. Measure various amount of solution into 100 mL volumetric flask with pipet.

c. Add 10.0 mL of 0.25 percent o-phenanthroline solution and 1.0 mL of 25 percent sodium citrate solution. Mix well and dilute to volume. Allow to stand for a least 30 minutes at not less than 20°C.

d. Develop color and read. Plot absorbance versus concentration of iron (Best line is fitted to points).

(2) Add approximately 1 mL of concentrated nitric acid and 5 to 10 mL of HCl to the residue from the sulfated ash test. Gently boil down, avoiding spattering, until the sample is just dry.

(3) Cool the residue, moisten with 2 drops of HCl, and quantitatively transfer to a 100 mL volumetric flask.

(4) Prepare a blank solution by adding 10 drops of HCl to 70 mL of water in a 100 mL volumetric flask and mixing well.

(5) Add to the sample and blank 1.0 mL of 10 percent hydroxylamine hydrochloride solution, mix and allow to stand for 15 minutes minimum.

(6) Add to the sample and blank 10.0 mL of 25 percent o-phenanthroline solution and 1.0 mL of 25 percent sodium citrate solution. Mix well and dilute to volume. Allow to stand for at least 30 minutes at not less than 20°C.

(7) Using a blank for comparison, determine the absorbance of the sample with a spectrophotometer set at a wavelength of 510 nm.

(8) Calculate iron as follows:

$$\%Iron = \frac{143 \times A}{W \times \left(1 \times 10^4\right)}$$

Where:
A = Concentration of iron from standard curve, micrograms/mL
W = Sample Weight, gram
Report percent iron , as ferric oxide to the nearest 0.0001 percent

4.8.13.1 Iron (alternate method). Atomic absorption, see 4.7.7 for procedure, (Iron content test in the MNA test method section.)

4.8.14 Purity.

a. Weigh 10 to 11 g of sample to the nearest 0.01 g.

b. Dissolve the sample in 160 mL of formaldehyde solution.

c. Mix well and allow to stand for 30 minutes.

d. Add four drops of phenolphthalein indicator and titrate with 1 N sodium hydroxide to a faint pink end point.

e. Calculate purity as ammonium perchlorate, as follows:

$$\%AP = \frac{11.75 \times A \times N}{W}$$

Where:
A = Volume of sodium hydroxide used for titration, mL
N = Normality of the sodium hydroxide
W = Sample weight, gram
Report purity as ammonium perchlorate to nearest 0.1 percent.

4.8.15 Phosphate.

a. Weigh a 25 gram sample to the nearest 0.05 grams into a 250 mL volumetric flask.

b. Add 150 mL of water and 25 mL of 70 to 72 percent perchloric acid. Dissolve the sample and dilute to volume with water.

c. Pipet a 10 mL aliquot into a 100 mL volumetric flask. Add 20 ± 4 mL of water and 15 mL of 70 to 72 percent perchloric acid. Cool to room temperature. Add 10 mL of ammonium vanadate to this solution. The

ammonium vanadate is prepared by dissolving 2.5 grams of ammonium vanadate in 400 ± 40 mL of hot water, adding 14 mL of perchloric acid, cooling, and diluting to 1 liter.

d. Add 10.0 mL of 10 percent ammonium molybdate solution (consisting of 100 to 110 g of ammonium molybdate, 81 percent molybdenum trioxide dissolved in water to make 1 liter). Dilute to volume with water, mix well, and allow to stand for at least 30 minutes at room temperature.

e. With a blank for comparison, determine the absorbance of each solution at a wavelength of 470 nm using a spectrophotometer with 1 cm silica cells.

f. Determine phosphate present from a standard curve.

g. Report phosphate as TCP to the nearest 0.01 percent.

4.8.16 Bulk Density.

a. Pour 50 to 100 mL of sample through a Scott volumeter, or equivalent, to purge the system. Discard purge material.

b. Pour 25 ± 5 mL of sample through the volumeter into the sample measure.

c. Sweep the excess sample from the top of the sample measure, with a spatula, starting the sweep from the corner of the measure with the spatula at approximately 45 degrees from horizontal. Exercise care to avoid vibrating the measure.

d. Weigh the measure and sample to the nearest 0.01 gram.

e. Calculate the bulk density as follows:

$$BulkDensity = \frac{S - T}{V}$$

Where:

S= Weight of measure plus sample, gram
T = Weight of empty measure, gram
V = Volume of volumeter or equivalent, in^3

f. Report bulk density to the nearest 0.1 g per cubic inch.

4.8.17 Particle Size Distribution.

a. Particle size is determined using a Microtrac or a Malvern particle size analyzer, (or equivalent).

b. Place 50 grams of AP in a Gilson Rotating Riffler or equivalent and divide the sample into 16 portions. Transfer one or more of these replicate portions into a beaker and blend with a spatula. Create 3 to 7 replicates samples from this beaker.

c. Take out approximately 0.5 grams of this portion and place into a 50 mL beaker.

d. Add one drop of a suitable surfactant to the beaker and manually mix with 0.5 mL of heptane.

e. Add 20 mL of heptane to the beaker. Sonicate for 5 minutes.

f. Add 150 ± 50 mL of filtered liquid to Microtrac Small Volume Circulator.

g. Quantitatively transfer approximately one mL aliquot of sample from the beaker to the recirculator.

h. Run triplicate samples as a minimum.

4.8.18 Lumps. Each sample selected shall be examined for non-dispersed material. If the sample contains lumps that cannot be dispersed with moderate pressure when held between thumb and fingers, the sample shall be weighed and lumps shall be weighed. Percent of weight as lumps shall be reported to nearest percent.

4.9 RDX methods.

4.9.1 Moisture.

a. Weigh a 1-2 gram sample to the nearest 0.001 gram.
b. Dissolve the sample in 100 milliliters of neutralized carrier solution (chloroform/methanol in a ratio of 70:30)
c. Titrate the moisture using a Karl Fischer titration system containing pyridine free titrant with an approximate equivalence value of 1.0 to 2.0 mg H_2O/mL titrant.
d. Consider the determination complete when an endpoint is reached which is stable for 30 seconds.

e. Calculate total moisture as follows:

$$\%Moisture = \frac{V \times E}{10 \times W}$$

Where:

V = Volume of standardized Karl Fischer reagent, mL
E = Water equivalent of standardized Karl Fischer reagent, milligram/mL
W = Weight of sample, gram

Report moisture to nearest 0.01 percent.

4.9.2 Particle size determination with Microtrac particle size analyzer. (For procedure using a Malvern particle size analyzer, see 4.9.3.)

a. Particle size distribution is determined using a Microtrac particle size analyzer, (or equivalent).

b. Place 50 grams of RDX in a Gilson Rotating Riffler or equivalent and divide the sample into 16 portions. Transfer one or more of these replicate portions into a beaker and blend with a spatula. Create 3 to 7 replicates samples from this beaker.

c. Take out approximately 0.5 grams of this portion and place into a 50 mL beaker.

d. Add one drop of a suitable surfactant to the beaker and manually mix with 0.5 mL of heptane.

e. Add 20 mL of heptane to the beaker. Sonicate for 5 minutes.

f. Add 150 ± 50 mL of filtered liquid to Microtrac Small Volume Circulator or equivalent.

g. Quantitatively transfer approximately one mL aliquot of sample from the beaker to the recirculator.

h. Run triplicate samples.

4.9.3 Particle size determination using a Malvern laser particle size analyzer. or equivalent.

a. Riffle approximately 50 grams of FEM RDX.

b. Transfer approximately 0.5 grams of the riffled material to a 50 mL beaker.

c. Add 10 mL of methanol to the beaker to serve as a wetting agent.

d. Add 15 mL of a surfactant solution (0.05% sodium metaphosphate or equivalent) to the beaker

e. Ultrasonicate the sample for 5 minutes.

f. Transfer sample to the Large Volume Sample Cell, containing 500 mL of distilled water, of the laser analyzer. Ensure obscuration range for laser is between 0.3 and 3.0.

g. Evaluate the sample for particle size in duplicate determinations.

h. Analyze duplicate samples.

4.10 PAX-21 Methods.

4.10.1 DNAN, HMX, RDX, MNA Content via HPLC.

4.10.1.1 Sample Preparation. Approximately 1 ± 0.1 gram of sample is weighed into a vial. An aliquot of acetonitrile is added to give a concentration of 50.0 mg PAX-21/mL. Solution is sonicated for 5 minutes. Verify that entire sample has dissolved except AP. Solution is diluted 1:4 with acetonitrile to a concentration of 10.0 mg PAX-21/mL.

4.10.1.2 Standard Preparation. At least five composite standards will be made with the following compounds. They should approximately cover the range listed below. Standards will be made in acetonitrile.

a. HMX: 2 to 400 μg/mL
b. RDX: 20 to 4000 μg/mL
c. MNA: 0.5 to 100 μg/mL
d. DNAN: 20 to 4000 μg/mL

Once these standards are made, a plot of concentration of these impurities versus their associated peak area is made. Using linear regression, these points are fitted with a line. The slope of this line will be used to determine the percent of each material in the sample (See 4.10.1.4).

4.10.1.3 Instrument conditions.

a. The column is a 125 x 4.0 mm Merck (or equivalent) LiChrospher RP-8, 5 m, w/guard @40°C.
b. The mobile phase is 0.05 % trifluoroacetic acid in water/isopropanol/acetonitrile at a ratio of 70/5/25.
c. The flow rate is 1.5 mL/minute.
d. The injection volume is 5 μL.
e. The wavelength for quantifying HMX and 1-chloro-2,4-dinitrobenzene is 215 nm.
f. The wavelength for quantifying RDX and 2,4-dinitrophenol is 300 nm.
e. The wavelength for quantifying MNA and DNAN is 380 nm.

4.10.1.4 Calculation of constituents.

$$\%(W/w) = \frac{sample}{reference \times Slope} \times 100$$

Where:

sample = Peak area of consistuent in sample
reference = 10,000 micrograms /mL
W/w = Constituent.
Slope = Calibration factor for constituent, Peak area/microgram/mL

NOTE: Report %RDX as the sum of RDX and HMX.

4.10.2 AP Content.

a. Standardize a 0.1 M solution of methanolic KOH to the nearest 0.0001 meq/mL using KHP (potassium-hydrogen phthalate) as a reference standard.

b. Weigh approximately 0.4 grams of sample into a beaker. Add 80 mL of acetone and a magnetic stir bar.

c. Titrate the sample solution with the standard methanolic KOH to a potentiometric inflection point. Record the volume at the inflection point.

d. Titrate an 80 mL acetone reagent blank.

e. Calculate the % AP as follows:

$$\%AP = \frac{(Vol - Bl) \times N \times 117.49}{Wt \times 1000} \times 100$$

Where:

Vol = Volume of KOH to titrate the sample solution, mL
Bl = Volume of KOH to titrate reagent blank, mL
N = Normality of KOH solution, eq/L
117.49 = Molecular Weight of AP
1000 = Conversion from milliliter to liter
Wt = Sample weight, gram

4.10.2.1 Alternate method for AP content - Ion Chromatography. Using an analytical balance, weigh a 1.0 gram sample of PAX-21 into a 100 mL volumetric flask.

a. Add 100 mL of acetone and ultrasonicate the sample until dissolved.

b. Pipette 1.0 mL of the above solution into a 10 mL volumetric flask and fill to volume with distilled water.

c. Analyze an aliquot of the solution by ion chromatography. The analytical

parameters are as follows:

Eluent solution—50 mM sodium hydroxide (note: discard solution after 5 days)
Eluent flow rate—1.5 mL/min
Sample loop—10 µL
Column—Dionex AG16 4 mm / AS 16 4 mm (or equivalent)
Suppressor—ASRS-1 4 mm / SRS Controller
Detector—Conductivity (100 µS)
Stock Standard Solution—1000 mg/L ClO_4^-. Dissolve 0.1231 grams of sodium perchlorate in distilled water and dilute to 100 mL in a volumetric flask.
Working standard—250 mg/L ClO_4^-. Pipette 2.5 mL of stock solution into a 10 mL volumetric flask and fill to volume with distilled water.

d. Analyze duplicate samples.

4.10.3 Melting Point and Exotherm Onset. A Differential Scanning Calorimeter will be used which is capable of meeting the heating rate and sample size parameters as specified below.

a. Place 4.0 ± 1.0 mg of the PAX-21 sample in a suitable sized aluminum crucible. Heat at a rate of 5 C°/min to a temperature of 350°C.

b. Determine the melting point based on the onset of the endotherm. The onset point is determined by the intersection of a tangent line drawn from the steepest slope of the endotherm peak to an extrapolated baseline.

c. Determine the exotherm by finding the onset of the exotherm peak. The onset point is determined by the intersection of a tangent line drawn from the steepest slope of the exotherm peak to an extrapolated baseline.

4.10.4 Viscosity (Method I).

a. Sample material coming out of the vessel. Material pour temperature should be at 205°F.

b. Take a sample of 50 grams in a container 50 mL beaker.

c. Take a viscosity using a Brookefield viscometer using a T-D spindle at 100 rpm.

d. Three viscosities should be taken during this run and average to the reported value.

4.10.5 Viscosity (Alternative Method).

4.10.5.1 Determination of viscosity (Efflux Method). The viscosity shall be determined in accordance with Method 212.1 of MIL-STD-650 except that the determination shall be made at a temperature of 205°F.

4.10.6 Moisture.

a. Standardize a pyridine based Karl Fischer titrant having approximate E-value of 1 to 25 mg/mL. A weighed or accurately syringed quantity of water shall be used as a reference standard.

b. The carrier solution shall be approximately 100 mL of a 1:1 mixture of pyridine and chloroform that has been allowed to equilibrate for a minimum of 10 minutes.

c. Weigh approximately 2 grams of sample to the nearest 0.0001 gram. The sample should be in pieces as small as possible, but no larger than 2 mm on any axis to facilitate dissolution.

d. Place the sample in the carrier solution and allow a minimum of 10 minutes for the sample to dissolve completely. This is usually accomplished by using a pause or hold function of the titrator.

e. Titrate the sample to a 30 second amperometric endpoint. Because of solubility limitations the carrier should be replaced after two sample determinations.

f. Titrate and extraction blank on the carrier on each day of analysis to account for atmospheric moisture titrated during the 10-minute extraction time. Open and close the vessel as though a sample is being placed in the carrier and perform the same 10-minute extraction as with the sample. Record the volume of the blank titration.

g. The moisture content is calculated as follows:

$$\%Water = \frac{(Vol - Bl) \times E}{(10 \times Wt)}$$

Where:

Vol = Volume of KF titrant required to titrated the sample, mL
E = Titrant equivalence value, mg/mL
Wt = Sample weight, gram
Bl = Volume of KF titrant used to titrate extraction blank, mL

4.10.7 Flake Thickness.

a. Thirty individual flakes from each composite sample will be used.

b. The thickness of the individual flakes will be taken using calipers or equivalent. The average thickness of each sample will be reported.

4.10.8 Density.

a. Density is measured using kerosene. The kerosene to be used will be Kerosene No. 1-k (low sulfur grade) as specified in ASTM D-3699. Determine and record the density of the kerosene before start of the test.

b. Approximately 5.0 grams of PAX-21 flake should be weighed to the nearest 0.1 mg.

c. Record the weight of the PAX-21 flake in air.

d. Record the weight of the PAX-21 flake in the kerosense.

e. Calculation of density:

$$DensityPAX-21=\frac{WtAir\times DenKer}{WtAir-WtIM}$$

Where:

WtAir = Weight of PAX-21 in the air, gram
WtIM = Weight of PAX-21 in kerosene, gram
DenKer = Density of kerosene, gram/cm3

Samples should be taken in triplicate and averaged.

4.10.9 Impact (Method I). The ERL, Type 12 impact test shall be conducted in accordance with MIL-STD-1751, Method 2, use of the noise meter for detection of reactions is optional. The PAX-21 test sample shall be prepared as outlined in MIL-STD-1751, Method 2. The samples shall be dried to a constant weight prior to testing. The height, which gives the 50% probability of reaction for the PAX-21 explosive, shall be obtained in the test.

4.10.10 Friction (ABL Method). This method should be performed in accordance with TB 700-2.

a. PAX-21 should be screened through a No. 8 and No. 20 U.S. standard mesh screen. At least 5 grams of material that is collected on the No. 20 mesh screen should be used for testing.

b. Adjust the valves to use the gauge for the pressure range of interest.

c. Close the valve on the hand pump and pump to lower the wheel onto the sample. Continue pumping until the desired pressure is reached. Because the area of the ram is 1 in^2, the pressure in psi equals the force on the wheel in pounds.

d. Press the button to release the clutch, and raise the pendulum to the

height for the desired speed.

e. While observing the sample, press the button, releasing the clutch. The pendulum will fall, striking the plate holder, causing it to move under the fixed wheel. Record sample as either a ‘go’ or ‘no-go’. A go is defined as:

(a) Noise greater than that produced by collision of the machine components
(b) Total consumption of sample

f. Continue the process. Each time, move the plate to a new plate surface, and index the wheel to a fresh surface. Place a new sample down for testing.

g. Start testing at 800 lbs and 8 fps. If 10 tests are conducted without a shot, testing is complete. If a shot is obtained, drop to 660 lbs and test at 8 fps, until 20 no-gos are obtained or a shot is obtained. Continue through successively lower force levels at 8 feet per second until all have been tested or 20 no-gos are obtained at a level. When 20 no-gos are obtained, stop testing and record this as the "threshold ignition level" or TIL.

h. After exhausting test levels at 8 fps, drop to 6 fps and start again at 800 pounds. Work through all levels at 6 fps, then drop to 4 fps, continuing to find the level where there are 20 no-gos (but where there WAS a go at the next higher level.) Velocity and speeds are shown in TABLE XI.

TABLE XI. Velocity and Force Values for ABL Friction.

Velocity (fps)	Applied Force (lbs)
8	800, 660, 560, 420, 370, 320, 240, 180, 130, 100, 50, 25
6	800, 660, 560, 420, 370, 320, 240, 180, 130, 100, 50, 25
4	800, 660, 560, 420, 370, 320, 240, 180, 130, 100, 50, 25
3	800, 660, 560, 420, 370, 320, 240, 180, 130, 100, 50, 25
2	800, 660, 560, 420, 370, 320, 240, 180, 130, 100, 50, 25

4.10.11 Gap test (50% Determination).

a. Gap test and configuration shall be performed as described in TB 700-2 (Section 7-3).

b. Card Gap pipes will be filled with PAX-21 in the melted state. Material should be cast in a manner that prevents voids or flaws.

c. A 'go' or 'no-go' of the witness plates will be recorded. A 'go' is when a clean hole the same diameter as the pipe is punched through the witness plate. Report as a fraction of 'no-gos' to total pipes tested.

4.10.12. Visual examination. The PAX-21 shall be examined with unaided eyes by personnel with normal vision or normal corrected vision. The PAX-21 shall be examined per requirements of Paragraph 3.2.2.3.

4.11 Inspection Equipment. Inspection equipment required to perform the inspections prescribed in this specification is identified in the Examination (4.3.2) and Test Methods and Proceudures (4.4 through 4.10) paragraphs herein. See 6.6 for details concerning responsibilities for inspection design and approval.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of material is to be performed by DOD personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's System Command. Packaging data retrieval is available from the managing Military Department or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. PAX-21 is intended to be used as a main charge explosive for insensitive munitions items. PAX-21 is only qualified as a main charge explosive fill in 60mm HE mortar cartridges.

6.2 Acquisition requirements.

a. Title, number, and date of this detail specification.
b. Quantity required and delivery schedules.
c. First article requirements (see 6.3)
d. Packaging requirements.
e. Certification for each ingredient.
f. Certification for the finished product.
g. Process control document (see 6.6)

6.3 Impact, friction, and Card Gap tests. These tests are for first article inspection only or to qualify a new process, critical process change, or new manufacturer. In order to qualify a new process, critical process change, and/or a new manufacturer, five consecutive batches of PAX-21 must conform to the chemical and physical requirements as specified by the first article inspection. The first article test should be required whenever a change occurs in the manufacture process, material used, drawing, specification, or source of supply that might significantly affect product quality as determined by the Government and whenever there is a lapse in PAX-21 production for a period in excess of six months. First article tests for a lapse in production should be conducted on sample(s) from one production sized batch.

6.4 Equivalent test methods. The test methods given in this specification are the official methods to be used. The contractor may request using other methods providing that the proposed method is equivalent (accuracy and precision) to the method given in this specification. Prior approval of the contracting officer is required for use of equivalent test methods. Submit copies of the test method and/or equipment designs to the Technical Agency: Commander, U.S. Army ARDEC, ATTN: AMSRD-AAR-QEM-C, Picatinny, NJ 07806-5000.

6.5 Process control document. A Process Control Document should be submitted to the Technical Agency. This document should contain a description of the process, all ingredients and percentages used, processing conditions and tolerances, and procedures used to produce PAX-21 that meets the requirements of this specification. An addendum to the Process Document will include the production equipment used to produce PAX-21. Whenever, there is a change from this Process Document, i.e. process conditions, the Technical Agency should be notified of the new process and process control change(s) and the reason(s) for the change(s) within 10 business days of the change. Submit documents to the Technical Agency: Commander, U.S. Army ARDEC, ATTN: AMSRD-AAR-QEM-C, Picatinny, NJ 07806-5000.

6.6 Submission of inspection equipment designs for approval. Submit equipment designs as required per the Contract Data Requirements Lists (DD Form 1423).

6.7 Riser scrap study. Previous analytical study of the riser scrap material obtained from production melt pour operations indicates that this material is sensitive to melt pour parameters regarding to homogeneity. The study indicated that during this specific melt pour process the riser resulted in an enriched ammonium perchlorate (AP) level and decreased level of 2,4 dinitroanisole (DNAN). This melt pour process resulted in out of spec material when 20% riser scrap was incorporated. Therefore, validation that the use of 20% riser scrap in actual production operations, will result in specification grade material upon incorporation, will be required and be approved by AMSRD-AAR-AEE-P personnel prior to approval of using the 20% riser scrap re-use plan by the contractor. Definitions per 6.8 should apply throughout the process.

6.8 Addition of 20% riser/clean scrap material. Riser scrap is defined as residual PAX-21 material in the funnel after post cooling removal operations. Clean scrap is defined as scrap formed in the kettle(s), clean pail(s)/pan(s), and typical loading/pour machine(s) used in loading and cooling operations. This material can be re-melted with virgin PAX-21 material for melt pour operation providing that:

a. The PAX-21 Riser scrap (and/or clean scrap) material should compose no more than 20% by weight of the total batch.

b. A PAX-21 riser study has been conducted by the contractor and its results validated and approved by AMSRD-AAR-AEE-P personnel, the riser material when remelted and mixed with virgin PAX-21 material will not adversely affect the overall mix, creating out-of-specification material.

c. Material from floor sweepings, drilling / facing operations, recovered in the ventilation system, or other such control apparatus IS NOT defined as Clean Scrap / Riser Scrap and will not be reused.

6.9 Submission of product. At the time each completed lot of items deliverable under the contract is submitted to the government for acceptance, the contractor should supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product being submitted:

a. A statement that the lot complies with all of the quality assurance provisions specified in the specification.

b. Specification number and date, together with identification and date of changes thereto.

c. Certificates of analysis on the ingredients and PAX-21 should be made available upon request by the contracting officer.

d. Quantity of PAX-21 in pounds in the lot.

e. Date submitted.

6.10. Flake density. Flake density should be in the approximate range of 1.68 g/c.c. to 1.76 g/c.c. Porous flakes in the samples may lead to lower flake density, but it does not necessarily result in a lower cast density because the entrapped air in the flakes will be evaporated during melt cast operation. Flake density is measured for information since flake density measurements deviating significantly from the normal range of acceptable products may be indicative of variation(s) in process operation and/or ingredients.

6.11 N-methyl-para-nitroaniline. Requirements and test methods for MNA are generally based on WS 18421 “Material Specification for n-methyl-para-nitroaniline”. Application for copies should be addressed to Director, Strategic Systems Programs, 2521 S. Clark St., Arlington, VA 22202.

6.12 Corrosive nature of PAX-21. Mixing and drying of PAX-21 ingredients with high moisture content in a heated kettle has found to be highly corrosive to the carbon steel mixing kettle. The rusts from the corroded kettle caused the PAX-21 to darken in color, brownish as opposed to being white. The corroded kettle eventually has to be replaced. Rust contamination or other forms of contamination by the production equipment, such as grease, oil, etc. are considered as foreign matters, their presence in PAX-21 is not allowed. (See 3.2.2.3)

6.13 Subject term (keyword) listing.

RDX-based explosive composition
Melt-cast explosive composition
Melt pour explosive composition
High explosive composition.

6.14 Notice. When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in anyway supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian:
Army - AR

Preparing activity:
Army - AR
Project 1376-2006-001

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at http://assist.daps.dla.mil.