INCH-POUND

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26 November 1996
SUPERSEDING
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27 February 1974

DETAIL SPECIFICATION

HMX

(CYCLOTETRAMETHYLENETETRANITRAMINE)

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers requirements, examinations and tests for two grades of HMX for use in explosive compositions as follows:

Grade A - 93 percent minimum purity with the remainder being RDX

Grade B - 98 percent minimum purity with the remainder being RDX

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are needed to meet the requirements specified in sections 3, 4, and 5 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, 4, and 5 of this specification, whether or not they are listed.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document, should be addressed to: Commander, U.S. Army ARDEC, ATTN: AMSTA-AR-EDE-S, Picatinny Arsenal, NJ 07806-5000 by using the Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSMC N/A FSC 1376

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 listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation (see 6.2).

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieves, Standard for Testing Purposes

STANDARDS

DEPARTMENT OF DEFENSE

MIL-STD-1168 - Lot Numbering of Ammunition
MIL-STD-1235 - Single and Multilevel Continuous Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-1751 - Safety and Performance Tests for Qualification of Explosives
DOD-STD-2101 - Classification of Characteristics

(Unless otherwise indicated, copies of above specifications, standards, and handbooks are available from DODSSP - Customer Service, the Standardization Document Order Desk, 700 Robbins Avenue, Bldg. 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 are those cited in the solicitation.

DRAWINGS

Holston Army Ammunition Plant/Holston Defense Corp. Engineering Drawing No. 7651-1028.48, 7651-1028.49 & 7651-1028.50 - Holston Impact Machine

Naval Sea Systems Command, Code Ident 10001,
Drawing No: LD70518 - Impact Machine

(Copies of other Government documents, drawings and publications required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting activity.)

2.3 Non-government publications. The following document(s) form a part of this document to the extent specified herein. unless otherwise specified, the issues of the following documents which are DOD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issues of the documents cited in the solicitation. (see 6.2)

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM Designation E300 - Recommended Practice for Sampling Industrial Chemicals

(Applications for copies should be addressed to the American Society for Testing Materials, 100 Bar Harbor Drive, West Conshohocken, PA 19428-2959.)

ANSI/ISO/ASQC A8402-1994 - Quality Management and Quality Assurance Vocabulary

(Application for copies should be addressed to American Society for Quality Control, 611 East Wisconsin Ave, Milwaukee, WI 53202)

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, supersedes applicable laws and regulations unless a specific exemption has been obtained. (See contract provisions for additional precedence criteria.)

3. REQUIREMENTS

3.1 First article. When specified in the contract or purchase order (see 6.2), a sample shall be subjected to first article inspection in accordance with the technical provisions herein (see 4.4).

3.2 Materials. HMX shall conform to the chemical and physical requirements specified in Table I, when tested as specified in the applicable paragraphs (See also 4.6, 6.10 and 6.12).

TABLE I. Chemical and physical requirements for HMX

Test	Grade A	Grade B	Test Paragraph
Alpha HMX, wt % (max),	0.10	0.10	4.7.1
HMX content(Beta), wt % (min)	93	98	4.7.1
RDX content, wt % (max)	7	2	4.7.1
Melting point, Celsius (deg. C), (min)	277	277	4.7.2
No. of Insoluble particles, on USS No. 40 sieve (max)	0	0	4.7.3
on USS No. 60 Sieve (max)	5	5	
Acetone insoluble material, wt % (max)	0.05	0.05	4.7.4
Inorganic insoluble, wt % (max)	0.03	0.03	4.7.5
Acidity, wt % (max)	0.02	0.02	4.7.6
Impact sensitivity, cm (min), (See Note 1)			
ERL, type 12, 2.5 kg	17	17	4.7.7.3
Holston impact method			
Short method (for acceptance only)	26	26	4.7.7.4.1
Long method	26	26	4.7.7.4.2
Granulation	(See Table II)	(See Table II)	4.7.8
Workmanship	(See 3.4)	(See 3.4)	4.7.9

Note 1 - Either the ERL, type 12, 2.5 kg impact sensitivity test method or the Holston impact sensitivity test method may be used. The ERL impact test method is described in MIL-STD-1751. The ERL, type 12 machine is shown in the Naval Sea System Command impact machine drawing (see 2.2.2). The Holston impact machine is shown in the Holston AAP impact machine drawing (see 2.2.2).

3.2.1 Granulation. HMX shall be designated as Class 1 to Class 6 in compliance with the granulation requirements given in Table II, when tested in accordance with 4.7.8.

Table II. Granulation

Through U.S. Standard Sieve No.	Class 1 Wt. Percent	Class 2 Wt. Percent	Class 3 Wt. Percent	Class 4 Wt. Percent	Class 5 Wt. Percent	Class 6 Wt. Percent
8				100		
12			99 min	85 min		99 min
35				25 ± 15		
50	90 ± 6	100	40 ± 15			90 min
100	50 ± 10		20 ± 10	15 max		65 ± 15
120		98 min				
200	20 ± 6		10 ± 10			30 ± 15
325	8 ± 5	75 min			98 min	15 ± 10

3.2.2 Use of virgin HMX only. HMX covered by this specification shall consist of virgin HMX only (see 6.7).

3.3 Process controls. The contractor shall submit a Process Control document to the Government specifying the process variables which are considered crucial for the production of HMX. The Process Control Document shall be submitted 30 days prior to commencement of production in accordance with 4.2.

3.4 Workmanship. The manufacturer shall use procedures and controls which assure that the HMX produced does not contain foreign material such as dirt, rust, paint or metal chips, etc., and that the safety and reliability of the explosive are not compromised. Compliance with this requirement shall be as specified in 4.7.9.

3.5 Product certification. No HMX material whether produced for military or commercial use may be represented as meeting the requirements of this detail specification unless it complies with all requirements contained in Section 3. The process control document (see 3.3), test data for first article inspection (see 4.4) and conformance inspection (see 4.5) shall be submitted to the Technical Agency (see 6.9) for evaluation, approval and certification on compliance with MIL-DTL-45444.

4. VERIFICATION

4.1 General provisions. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements (examinations and tests) as specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The government reserves the right to perform any of

the inspections set forth in this specification where such inspections are deemed necessary to ensure supplies and services conform to prescribed requirements. Reference shall be made to ANSI/ISO/ASQC A8402-1994 in order to define the terms used herein. All items must meet all requirements of Section 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling inspection, as part of manufacturing operations, is an acceptable practice to ascertain conformance to requirements, however, this does not authorize submission of known defective material, either indicated or actual, nor does it commit the government to accept defective material.

4.1.1 Submission of product. At the time each completed lot of items deliverable under the contract is submitted to the Government for acceptance, the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product being submitted (see 6.3):

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

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

c. Certificates of analysis on all materials used directly by the contractor when such material is controlled by Government specifications, shall be available upon request by the contracting officer.

d. Number of pounds of HMX in the lot.

e. Date submitted.

The certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the agent's authority to bind his principal. Substantiation of the agent's authority will not be required with the subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.

4.2 Process control document. A Process Control Document shall be submitted to the Technical Agency (see 6.9). The document shall contain a description of the process, all materials used, process conditions, procedures, production and inspection equipment used to produce HMX meeting the requirements of this specification. In addition, whenever there is a change that requires lot interfix change (per MIL-STD-1168) such as the source of materials, process conditions, procedures, etc., from those in the previous process control document, the Technical Agency shall be notified of the new process and process control change(s) and the reason for the change(s) within 10 business days from the change(s).

4.3 Classification of inspections. The inspection requirements specified herein are classified as follows:

a. First article inspections (See 4.4)

b. Conformance inspection (See 4.5)

4.3.1 Inspection conditions. Unless otherwise specified, all inspections shall be performed in accordance with the test conditions specified in 4.5.

4.4 First article inspection.

4.4.1 Submission. The contractor shall submit a first article sample as designated by the Contracting Officer for evaluation in accordance with provisions of 4.4.2, (see 6.2c). The first article shall consist of five kilograms (11 pounds) of the composition obtained by sampling as described in 4.6.1. The samples shall be obtained from a production batch which has been produced by the contractor using the same production process, procedures and equipment as will be used in fulfilling the contract. All materials shall be obtained from the same sources of supply as will be used in regular production. All samples submitted shall have been produced by the contractor using the same production process, procedures, equipment as will be used in regular production, and shall be accompanied by certificates of analysis. Prior to submission, the contractor shall inspect the sample to the degree necessary to assure that it conforms to the requirements of the contract and submit a record of this inspection with the sample. A sample containing known defects will not be submitted unless specifically authorized by the Contracting Officer, (see 6.2).

4.4.2 Inspections to be performed. The sample may be subjected by the Government to any or all of the examinations or tests specified in 4.5.2.1 of this specification and any or all of the applicable drawings. (See DOD-STD-2101 for definitions of critical, major and minor classification of characteristics.)

4.4.3 Rejection. If any sample fails to comply with any of the applicable requirements (see 3.), 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 requirements.

4.5 Conformance inspection.

4.5.1 Inspection lot formation. A lot shall consist of one or more batches of HMX produced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions. Each batch shall consist of that quantity of HMX that has been subjected to the same unit chemical or physical process intended to make the final product homogeneous (homogeneity criteria). Units of product selected for inspection shall represent only the inspection lot from which they are drawn and shall not be construed to represent any prior or subsequent quantities presented for inspection. All material submitted for inspection in accordance with this specification shall comply with the homogeneity criteria specified herein, regardless of the type of inspection procedure which is being applied to determine conformance with requirements. The lot shall be submitted for inspection in accordance with MIL-STD-1235 when applicable. The criteria and procedures for the assignment of lot numbers shall be in accordance with MIL-STD-1168.

4.5.2 Examinations and tests.

a. Classification characteristics. Conformance examinations and tests are specified in the following classification of characteristics paragraphs. The contractor's 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 the specification (4.7.9.1 to 4.7.9.5), attributes sample inspection shall be conducted in accordance with Table III below, using the inspection levels stated in the classification of characteristics paragraphs. (See DOD-STD-2101 for definitions of critical, major and minor classification of characteristics.)

Table III Attributes sample inspection

Lot Size	Inspection Levels I	II	III
2 to 8	*	*	*
9 to 15	*	*	*
16 to 25	*	*	*
26 to 50	*	*	32
51 to 90	*	*	32
91 to 150	*	125	32
151 to 280	*	125	32
281 to 500	*	125	32
501 to 1200	*	125	80
1201 to 3200	*	125	80
3201 to 10000	1250	125	125
10001 to 35000	1250	315	125
35001 to 150000	1250	315	125
150001 to 500000	1250	500	200
500001 and over	1250	500	200

Numbers under inspection levels indicate sample size; asterisks (*) indicate one hundred percent inspection. If sample size exceeds lot size, perform one hundred percent inspection. Accept on zero and reject on one or more for all inspection levels.

b. Alternative conformance inspection provisions. Unless otherwise specified herein or provided for in the contract, alternative conformance procedures, methods or equipment, such as statistical process control, tool control, variables sampling or other types of sampling plans, etc., may be used by the contractor when they provide, as a minimum, the level of verification required by the provisions herein. Prior to applying such alternative procedures, methods or equipment, the contractor shall describe them in a written proposal submitted to the Government for evaluation (see 6.11). When required, the contractor shall demonstrate that the effectiveness of each proposed alternative is equal to or better than the specified conformance inspection provision(s) herein. In case of dispute as to whether the contractor's proposed alternative(s) provides equivalent assurance, the provisions of this specification shall apply. All approved alternative conformance inspection provisions shall be specifically incorporated into the contractor's quality program or inspection system, as applicable.

4.5.2.1 Classification of characteristics - HMX

Classification	Examination/test	Conformance criteria	Requirement paragraph	Inspection method ref.
Critical	None defined			
Major				
101	Alpha HMX content	4.6	3.2	4.7.1
102	HMX content	4.6	3.2	4.7.1
103	RDX content	4.6	3.2	4.7.1
104	Melting point	4.6	3.2	4.7.2
105	Insoluble particle	4.6	3.2	4.7.3
106	Acetone insoluble material	4.6	3.2	4.7.4
107	Inorganic insoluble	4.6	3.2	4.7.5
108	Acidity	4.6	3.2	4.7.6
109	Impact sensitivity	4.6	3.2	4.7.7
110	Granulation	4.6	3.2.1	4.7.8
111	Workmanship	4.6	3.4	4.7.9
Minor	None defined			

4.6 Sampling and inspection equipment.

Hazard Notice

The materials described herein are flammable or explosive or both. Consequently, they present hazards in manufacture, handling, storage and shipment. The contractor should recognize these hazards and take appropriate measures to guard and protect against fire, explosion, adverse environment, corrosive atmosphere, rough handling and electrically-induced incidents.

4.6.1 Sampling. A representative sample of approximately 300 grams shall be taken from each batch of HMX in accordance with ASTM Procedure E300 for solids. Samples shall be chosen in accordance with MIL-STD-1235, CSP-1 Plan, Inspection Level II, AQL 4.0%. (AQL are provided as indices to simplify the use of the table in MIL-STD-1235, but have no other meaning relative to the sampling plan.) The samples shall be tested for compliance with requirements of 3.2, Table I of this specification. 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 the applicable methods given in paragraph 4.7. 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 the contractor will return to 100 % inspection until "i" successive batches are accepted as required by MIL-STD-1235.

4.6.2 Inspection equipment. Commercially available inspection equipment shall be used for all examinations and tests specified in 4.5 and 4.7. The contractor is responsible to have the necessary equipment available, use it correctly and to assure that proper calibration procedures are followed. The designation of an instrument of a particular make or model number is not intended to preclude the use of an equivalent make or model which is capable of performing the required test. Government approval of all inspection equipment is required prior to its use for acceptance purposes. Inspection equipment designs shall be submitted to the Technical Agency (see 6.9).

4.7 Methods of inspection. The following tests shall be performed using prescribed analytical procedures for replicate determination given in standard and analytical textbooks. Also, unless otherwise specified herein, all chemicals and reagents shall be ACS Grade or reagent grade chemicals (see 6.10 for use of equivalent test methods).

4.7.1 Purity of HMX. The alpha HMX, beta HMX and RDX content of HMX shall be determined by x-ray diffraction as follows:

A Philip Electronic Instrument x-ray diffractometer (or equivalent) equipped with voltage and current stabilizer, scintillation detector and copper target tube (1) shall be used for the determination of alpha HMX, beta HMX and the RDX content of HMX. The energy source tube of the instrument should be capable of an excitation of 40kV and a filament current of 20 ma. A pulse height analyzer capable of passing copper Ka (alpha) radiation should be used. A nickel filter may be used to remove copper Kb (beta) radiation. Tube voltage and filament current can be selected by analyzing five of the calibration standards at two different settings. The x-ray diffraction patterns for alpha HMX, RDX, and beta HMX indicate characteristic diffractions at 17.81 degrees and 25.10 degrees two theta respectively for RDX and alpha HMX in the presence of beta HMX. (These angles may vary slightly according to the alignment of the instrument). Background intensities for RDX and alpha HMX can be measured at 16.90 degrees and 24.10 degrees two theta, respectively. Beta HMX, the major component, is determined by difference.

Note 1: The x-ray equipment shall be operated in accordance with the manufacturer's instructions.

4.7.1.1 Beta HMX, alpha HMX and RDX, for use as calibration standards.

4.7.1.1.1 HMX (beta) and RDX calibration standards. High purity HMX (beta) and high purity RDX suitable to be used as calibration standards are obtainable from Holston AAP/HDC; ARDEC, Picatinny Arsenal and the Naval Surface Warfare Center. (See 6.8). The HMX and RDX used for high purity standards shall have a minimum purity of 99.90%.

4.7.1.1.2 Preparation of alpha HMX calibration standard. Add 4 grams of the purified beta HMX to 80 ml of (70% by weight) nitric acid. Heat until the HMX dissolves. Filter the solution with filter paper and cool slowly to 30 degrees C. After one hour, filter the slurry using a medium porosity crucible. Wash the precipitate thoroughly with distilled water. Dry in a vacuum oven at 60 degrees C for two hours. Alpha morphology shall be verified by microscopy or infrared spectroscopy before the alpha HMX is used as a calibration standard.

4.7.1.2 Purity analysis of RDX standard. Prepare RDX solvent by stirring 1,2 dichloroethane at room temperature for four hours in contact with HMX crystals. Solubility of HMX in 1,2 dichloroethane at room temperature (24 degrees C) is 0.02 gram per 100 ml. Accurately weigh approximately 0.2 gram of the RDX calibration standard to the nearest 0.1 milligram into a tared 125 ml glass stoppered conical flask. Add 100 ml of 1.2 dichloroethane that has been saturated with HMX. Secure the glass stopper and shake the flask on a wrist action shaker for one hour. Accurately weigh a fine porosity 30 ml fritted glass crucible and place it on a filtering flask. Apply vacuum to the filtering flask and transfer the sample residue from the conical flask to the crucible. Rinse the contents of conical flask into the crucible with saturated 1,2 dichlorethane. Wash residue in the crucible 2 times with 100 ml portions of diethylether. Suck dry under vacuum for 15 minutes. Allow the crucible to come to room temperature and weigh crucible and insoluble residue. The insoluble residue is HMX. Therefore, the percentage of RDX in the sample is:

$$\frac{(A-B) \times 100}{A}$$

Where:
A - is the original sample weight in grams.
B - is the insoluble residue weight in grams.

4.7.1.3 Preparation of calibration standard samples. Prepare 5 gram samples of the calibration standard mixtures listed in Table IV as follows: Accurately weigh to the nearest 0.1 milligram the ingredients listed in Table IV (use purified materials which pass through a US Standard Sieve No. 260) in the proper proportions to yield a 5.0 gram sample. Place the ingredients in 250 ml Erlenmeyer flasks and mix thoroughly for a minimum of three hours with the aid of a wrist action shaker.

Table IV Composition of calibration standard mixtures

Beta HMX, %	Alpha HMX, %	RDX, %
99.70	0.30	0.00
99.40	0.60	0.00
99.00	1.00	0.00
98.00	2.00	0.00
97.00	3.00	0.00
96.00	4.00	0.00
95.00	5.00	0.00
99.00	0.00	1.00
98.00	0.00	2.00
97.00	0.00	3.00
96.00	0.00	4.00
95.00	0.00	5.00
94.00	0.00	6.00
93.00	0.00	7.00
92.00	0.00	8.00
91.00	0.00	9.00
90.00	0.00	10.00

4.7.1.4 Intensity measurement and construction of calibration curve. Use X-ray diffraction apparatus to measure the angular intensities of the calibration standard mixtures at 16.90, 17.81, 24.10 and 25.10 degrees two theta as outlined in 4.7.1.5. Use the X-ray diffraction data obtained for the calibration standard mixtures to plot the calibration standard curves as indicated below:

RDX - Plot the corrected intensity (cps) at 17.81 degrees two theta vs. RDX concentration, weight percent. (See example in Fig. 1).

Alpha HMX - Plot the corrected intensity (cps) at 25.10 degrees two theta vs. alpha HMX concentration, weight percent. (See example in Fig. 2).

Correction curve for determining alpha HMX in the presence of RDX: Plot the corrected intensity (cps) at 25.10 degrees vs. RDX concentration, weight percent, for those samples free of alpha HMX. (See example in Fig. 3).

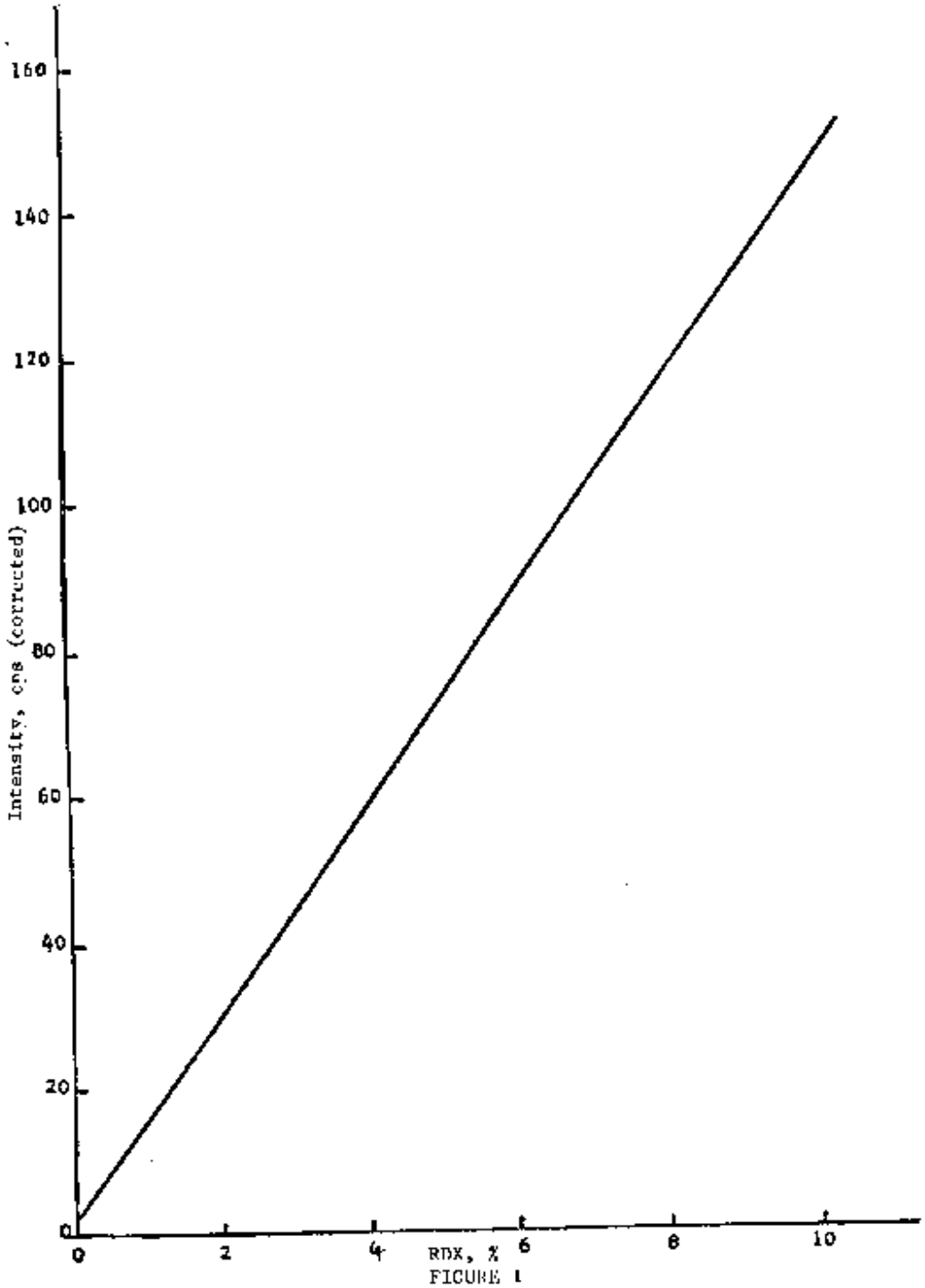

FIGURE 1

CALIBRATION CURVE FOR THE DETERMINATION OF RDX IN PRESENCE OF BETA HMX

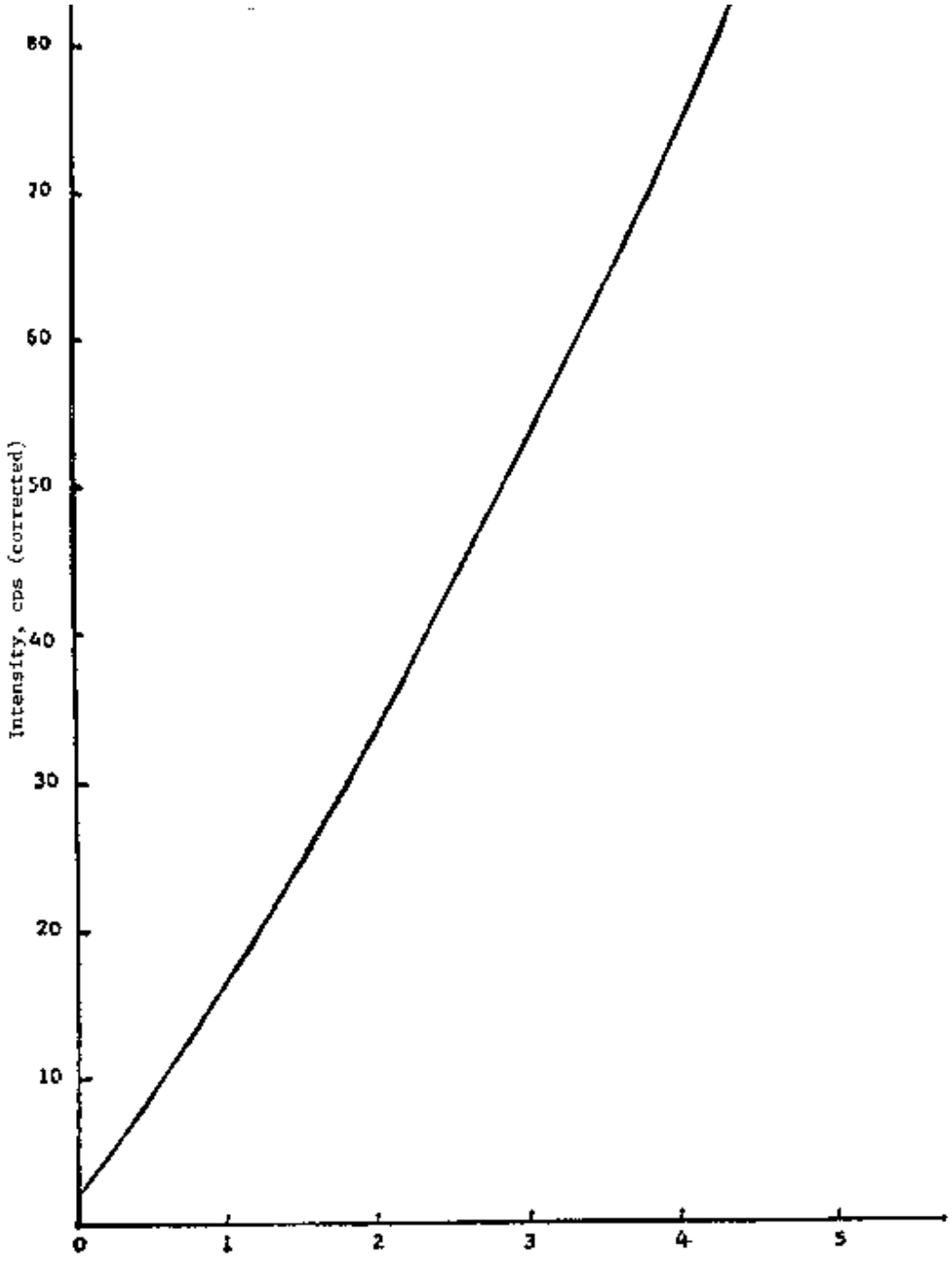

FIGURE 2

CALIBRATION CURVE FOR THE DETERMINATION OF ALPHA HMX IN PRESENCE OF BETA HMX

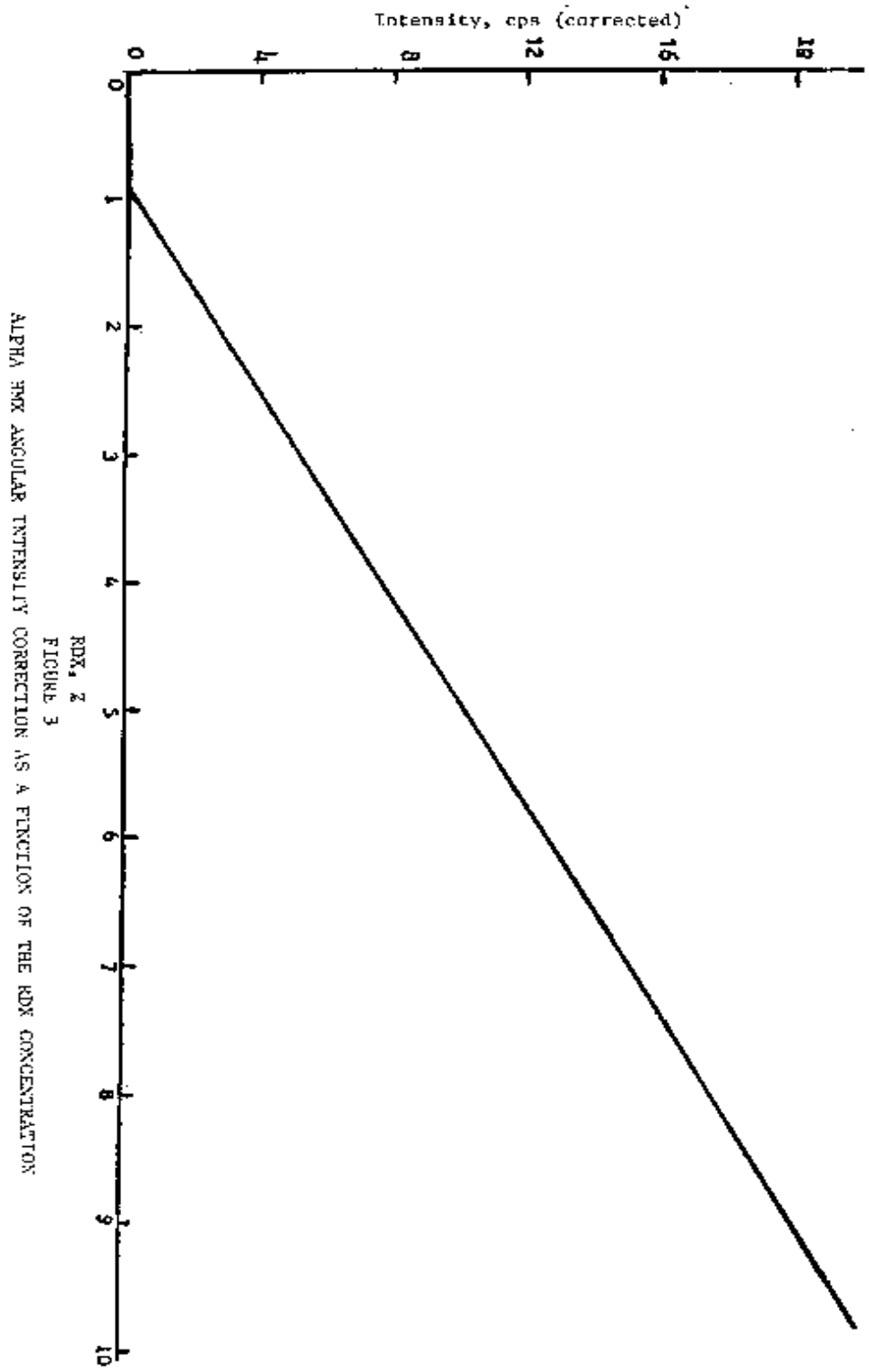

FIGURE 3

ALPHA HMX ANGULAR INTENSITY CORRECTION AS A FUNCTION OF THE RDX CONCENTRATION

4.7.1.5 Analysis of HMX sample. Reduce the HMX sample particle size to less than 62 micron. HMX particle size is usually small enough to permit analysis as received. It may however, occasionally be necessary to lightly crush the material in a mortar. Recrystallized HMX requires grinding in a mortar to reduce the particle size. This is accomplished safely by grinding 0.1 gram portions in a small mortar. A sample size of approximately 0.4 gram is required to properly fill the cavity type sample holder. Press sample into cavity of sample holder. Place aluminum sample holder (grooved side down) on a very smooth surface such as polished stainless steel. Place sample into cavity of holder and press with spatula. Add additional powdered sample and hand press by placing a stainless steel block over the cavity area and exerting pressure with hand (50 to 100 lbs is sufficient). Remove sample and holder and examine surface of sample on grooved side of sample holder. Sample surface must not have any voids, cracks, etc. Remove loose explosive from sample holder. The grooved side of the sample holder must be completely free of particles prior to insertion into the diffractometer. Remove shield from diffractometer (shutters must be closed at all times when intensity measurements are not being made). Insert sample. The groove on a sample holder must be coincident with the groove on the goniometer axis of rotation. The rear edge of the sample holder must be flush against the sample stage. Replace the shield being careful not to move the sample. If the electronic panel is maintained ready, move toggle switch to the up position. Set diffractometer to 16.90 degrees two theta. Open shutters on X-ray tube tower by pulling out as far as possible. After 30 seconds, push toggle switch toward scan to print out time registered on rate meter dekatron tubes. Reset goniometer to 17.81 degrees two theta. Repeat above. Reset goniometer to 24.10 degrees two theta. Repeat above. Reset goniometer to 25.10 degrees two theta. Repeat. Close shutters on X-ray tower. When no additional samples are ready for analysis, turn off electronic panel by moving toggle switch downward.

4.7.1.5.1 Interpretation of data. Record the counts that are accumulated in 100 seconds, that are printed out under the respective two theta angle. Place the decimal point two places to the left in the printout to obtain the representative counts per second (cps). Subtract cps at 16.90 degree two theta from cps at 17.81 degrees two theta. This is the intensity (cps) due to the RDX percent. Opposite the cps obtained, read the RDX concentration from Fig. 1 (RDX calibration curve). Subtract cps at 24.10 degrees two theta from the cps at 25.10 degrees two theta. This intensity is due to alpha HMX. If the RDX concentration is greater than 1%, determine from Fig. 3 the correction required by reading the cps opposite the RDX percentage. Subtract this correction from the cps. From Fig. 2, read percentage alpha HMX opposite the intensity, (corrected or uncorrected).

4.7.1.6 Alternate method for HMX purity and RDX content. A high performance liquid chromatographic (HPLC) method shall be used to determine HMX purity by quantifying the concentration of RDX impurity in the HMX product.

4.7.1.6.1 HPLC method conditions. The HPLC system used in the analysis of HMX product and appropriate method calibration standards shall be comprised of a column, mobile phase, pumping system, sample injection system, detector system and data capture system. The column used in the testing shall be a microBondapak CN (10 micron particle size, 3.9 mm x 150 mm Internal Dimensions; see 6.6.1) or a direct column equivalent. The mobile phase shall be a quaternary system consisting of the following components and volumetric proportions: 70% by volume 2,2,4-trimethylpentane (isooctane), 15% by volume of acetonitrile, 7.5% by volume chloroform, and 7.5% by volume methanol. All solvents used in the mobile phase shall be vendor certified as HPLC grade chemicals. Prior to use in testing, the mobile phase should be thoroughly degassed using either vacuum degassing techniques or by purging with an inert gas. The injector component within the HPLC system shall be equipped with a sample loop capable of delivering a 20 microliter injection volume of sample to the HPLC column. The detection system used in this HPLC testing shall utilize a ultraviolet (UV) detector having a light wavelength of 254 nanometers.

4.7.1.6.2 Analysis of HMX samples and standards. Prior to the analysis of either HMX samples or standards, the pumping of mobile phase through the system shall be initiated at a flow rate of 1.5 milliliters per minute. The HPLC system shall be allowed to equilibrate at ambient conditions for approximately 10 minutes or until the flow of mobile phase and the output reading on the UV detector are stable. After the HPLC system has stabilized, the sample loop on the HPLC injector system shall be filled with an aliquot of the HMX sample or standard. Using either manual or automated injection techniques, the aliquot of sample in the 20 microliter sample loop on the injector should be transferred to the HPLC column. The peak area data from the HPLC chromatogram for both the HMX and RDX components in the samples and standards should be calculated and retained using the data collection system (i.e. computer, computing integrator, etc.).

4.7.1.6.3 Preparation and analysis of HMX calibration standards. Calibration standards having the approximate concentrations provided in Table V shall be prepared. The RDX and HMX used in the calibration standards shall have a minimum purity of 99.9%. Prepare the calibration standards by weighing the HMX and RDX components to the nearest 0.1 milligram in tared 250-ml glass volumetric flasks with ground glass stoppers. Add approximately 100 milliliters of HPLC grade acetone to the flasks. Cap the flasks and place the standards in an ultrasonic bath for 10 minutes or until the RDX and HMX are completely dissolved and uniformly mixed. Allow the standard mixtures to cool to ambient temperature, and then fill to the mark of the volumetric flasks using HPLC grade acetone.

Table V. Composition of HMX/RDX Standards

HMX		RDX	
Percent	Weight, grams	Percent	Weight, grams
99.5	0.4975	0.5	0.0025
98.0	0.4900	2.0	0.0100
96.5	0.4825	3.5	0.0175

Each calibration standard which is prepared shall be tested using the conditions and methodology outlined in Sections 4.7.1.6.1 and 4.7.1.6.2.

4.7.1.6.4 Preparation and analysis of HMX samples. Dry approximately 1 gram of the HMX sample to be tested in an oven at 100 ± 5°C for a minimum of one hour. Using an analytical balance, weigh approximately 0.05 gram of the dried HMX to the nearest 0.1 milligram in a clean, glass 30-ml bottle with a Teflon lined lid. Add 25.0 milliliters of HPLC grade acetone to the bottle using an automatic repipette. Cap the bottle and place the samples in an ultrasonic bath for 10 minutes or until the HMX is completely dissolved and the solution is uniformly mixed.

The HMX samples which are prepared shall be tested using the conditions and methodology outlined in 4.7.1.6.1 and 4.7.1.6.2. Use the chromatographic peak areas for HMX and RDX in the sample to determine the HMX purity and the RDX content using the following calculations:

$$\text{Percent (HMX)} = \frac{100 \times A \times B}{C \times W}$$

Where:

A = The HMX chromatographic peak area (or height) obtained for the HMX sample.

B = The weight of HMX in the calibration standard in grams. (Use the calibration standard which provided a peak area closest to the area obtained for the sample.)

C = The HMX peak area obtained for the calibration standard having the weight, B. above.

W = The total weight of the HMX sample in grams.

$$\text{Percent (RDX)} = \frac{100 \times E \times F}{G \times W}$$

Where:

E = The RDX chromatographic peak area (or height) obtained for the RDX in the HMX sample.

F = The weight of RDX in the calibration standard in grams. (Use the calibration standard which provided a peak area closest to the area obtained for the sample.)

G = The RDX peak area obtained for the calibration standard having the weight, F. above.

W = The total weight of the HMX sample in grams.

In the development of a calibration curve using the three HMX/RDX standards, upon establishing the linearity of the HPLC method response across the concentration range being studied, a single-point calibration using the standard which provided a peak area closest to the area obtained for the sample can be used to determine the HMX and RDX concentrations in the HMX product.

4.7.2 Melting point. A Fisher-Johns or equivalent hot state melting point apparatus shall be used to determine the melting point of HMX. The thermometer of this apparatus shall be calibrated using appropriate melting point standards. A portion of the sample shall be ground in small agate mortar and a very small quantity (approximately 0.05 gram) of the finely pulverized sample placed between two clean 18 millimeters (mm) diameter cover glasses. These shall be gently but firmly pressed together and placed in the circular depression on the stage. The powerstat shall be turned up and the unit allowed to heat. The heating rate may be very rapid to within 15 degrees C of the melting point. Thereafter a heating rate of approximately 1 degree C per minute shall be used. When the sample begins to melt, the thermometer shall be read and the temperature reading adjusted to reflect the thermometer calibration correction shall be reported as the melting point.

4.7.3 Insoluble particles. Weigh three samples of 17 ± 0.1 grams each (dry weight) from the prepared HMX sample. Transfer each sample into a 1000 ml beaker and add 500 ml of acetone to each beaker. Heat the contents of the beakers until the explosives are in solution. Combine the solutions and filter through USSS 40 and USSS 60 sieves. The filtrate shall be retained and used for testing acetone and inorganic insolubles. The screens shall be dried. Any particles remaining on the screens will be counted and reported as insoluble particles.

4.7.4 Acetone insoluble. Use the residue and filtrate from 4.7.3 or weigh an approximately 10 gram portion of the dried sample accurately to the nearest 0.01 gram, place the sample in a 600 ml beaker and add 400 ml of filtered acetone. Place the beaker and contents on a steam bath (cover with a watch glass if necessary) and stir occasionally until all of the HMX has dissolved. Filter the HMX solution through a 25 ml medium porosity porcelain filtering crucible, which has been ignited at 700 ± 20 degrees C, and tared to the nearest 0.1 milligram. Transfer any remaining insoluble material from the beaker to the crucible with the aid of a stream of acetone from a wash bottle. Wash the material remaining in the crucible three times with 20 ml portions of acetone and aspirate until the odor of acetone is no longer noticeable. Dry the crucible in an oven at 105 ± 5 degrees C for thirty minutes, cool in a desiccator and weigh to the nearest 0.1 milligram. Reserve the crucible and contents for determination of inorganic insoluble materials given in 4.7.5.

Calculate the acetone insoluble material as follows:

$$\text{Percent acetone insoluble material} = \frac{(A-B) \times 100}{W}$$

Where:
A = The final weights of the acetone insoluble material and the crucible in grams.
B = The tare weight of the crucible in grams.
W = The HMX sample weight in grams.

4.7.5 Inorganic insoluble material. Ignite the sample reserved from the acetone insoluble material determination, 4.7.4, in a muffle furnace at 700 ± 10 degrees C for 30 ± 5 minutes, cool in a desiccator and weigh to the nearest 0.1 milligram.

Calculate the inorganic insoluble material as follows;

$$\text{Percent inorganic insoluble material} = \frac{(C-B) \times 100}{W}$$

Where:
C = The weight of the crucible and contents after ignition in g.
B and W are as given in 4.7.4.

4.7.6 Acidity. Weigh an approximate 10 gram portion of the dried sample accurately to the nearest 0.01 gram, place in a clean 800 ml beaker and add 500 ml of acetone. Place the beaker on a steam bath and heat with occasional stirring until the sample has completely dissolved. One hundred ml of distilled water shall be added and the mixture shall be cooled and titrated with 0.05 normal (N) sodium hydroxide using phenolphthalein or methyl red indicator. A blank shall be run and the results of the titration of the specimen shall be corrected for acidity of reagents. The acidity shall be calculated on the dry basis to percent acetic acid as follows:

$$\text{Percent acid (as acetic acid)} = \frac{6.0\ (S-B)\ N}{W}$$

Where:
S = ml of sodium hydroxide solution used in sample.
B = ml of sodium hydroxide solution used in blank.
N = normality of sodium hydroxide solution.
W = weight of specimen on a dry basis, in grams.

4.7.7 Impact sensitivity.

4.7.7.1 Safety. Proper safety should be exercised in determining the impact sensitivity of HMX. Compliance with local safety rules is required. The following safety precautions apply to both the ERL (4.7.7.3) and the Holston (4.7.7.4) impact sensitivity test methods.

Observe normal safety precautions -- pay particular attention to the following rules:

a. Operate the exhaust fan while tests are being performed.

b. Always operate the machine with a safety shield in place.

c. Secure the weight with a safety chain while cleaning the machine.

d. Remove waste explosives from the room after completion of each test.

4.7.7.2 Interferences. The following interferences apply to both the ERL (4.7.7.3) and the Holston (4.7.7.4) impact sensitivity test methods.

a. A machine loosely assembled or not in proper alignment may produce incorrect values.

b. A rough surface or cracks on the anvil or plunger may produce lower sensitivity values.

c. Insufficient or unevenly distributed sample may produce incorrect values.

d. A sample containing glass, metal or other gritty matter foreign to the product may produce low sensitivity values. (A low sensitivity value indicates more sensitive material.)

e. Wet samples or samples containing oil, greases and soft plastics may produce high sensitivity values.

4.7.7.3 ERL, Type 12, 2.5 kg impact sensitivity method. The impact sensitivity of HMX shall be determined using the ERL type 12 impact machine with a 2.5 kg drop weight on a 35 mg sample.

The following ERL test method is described in MIL-STD-1751:

(a) Sample preparation. Pass a dry sample of HMX through a sample splitter until a representative portion of approximately 10 gram is obtained. A small scoop will be used to measure the HMX powder sample. Weigh a few scoops on a balance to determine the volume necessary to obtain 35 mg. Subsequent 35 mg samples shall be measured on a volume basis using the small scoop. The sample shall be poured directly onto a 180A garnet sand paper (1" x 1" square) with the sand side up resting on a flat surface. Approximately 25 samples of 35 mg HMX each on a one inch square sand paper shall be prepared. Each of the samples shall be weighed on the garnet paper, (any abnormally high or low weight should be adjusted), and placed on the anvil for testing.

(b) Performance of the test. Test data shall be recorded on a worksheet which can be on paper (hard copy) or on a computer. Record sample name, ID number or lot number, sample preparation (powder, cube, etc.), requester, operator, date, temperature, and humidity. The drop heights used for the test are as follows:

64.0 cm
50.5 cm
40.5 cm
32.0 cm
25.5 cm
20.0 cm
16.0 cm
12.5 cm
10.0 cm
8.0 cm
6.5 cm
5.0 cm

NOTE: The actual starting height for HMX should be determined from test data on HMX previously tested.

Before each drop, the striker and anvil shall be cleaned. Ethanol or acetone on a wipe shall be used to clean the surfaces. A spatula or sandpaper may also be used to clear any further residue from previous tests. A 35 mg sample (on the sand paper) shall be placed on the anvil. Gently place the striker on top of the sample. Close the shield. Turn on the electromagnet, and using the hoist, raise the weight to the desired height. Face the opposite direction of the impact machine and turn the electromagnet switch to the "off" position, allowing the weight to fall and strike the top of striker. Lift and secure the weight, remove the striker to determine whether the drop was a positive (explosion) or negative (nonexplosion) reaction. Any reaction observed is considered a go: smoke, flame, sparks, odor, burn marks on the striker or anvil, a noise above the ambient drop weight noise. A positive reaction shall be recorded as "1", and a negative reaction as a "0". When a positive reaction is achieved, the next drop height shall be one level lower. When a negative reaction is achieved, the next drop height shall be one level higher.

NOTE: The actual test begins when a positive reaction is followed by a negative reaction, or a negative reaction is followed by a positive reaction. It may be necessary to make several test shots before the starting point of the test is recorded.

Repeat 4.7.7.3 (b) until all 25 samples prepared in 4.7.7.3 (a) are drop-tested and recorded.

(c) Test result calculations: The test result calculation may be preprogrammed in a computer and automatically calculated based on the results recorded in the computer. If the test was recorded on paper, enter all data into the impact spreadsheet. In either case, a hard copy of the results shall be immediately printed following the testing for record keeping. The following is an explanation of the calculation for the height in centimeters where 50 percent of the explosions occur:

The lowest drop height with recorded data is assigned an "I" value of 0. Each successively greater height is assigned values of 1, 2, etc. The following values are calculated:

ni = # of negatives at each log height.
N = sum of ni
A = sum of (i*ni)
B = Sum of i*i*ni
C = log interval of lowest drop height
M = C + 0.1(A/N + 0.5)
Height (at 50% explosion) = 10 raised to the power of M.
S (standard deviation, log units) = 0.162*[(NB - A*A)/N*N + 0.0219]

The ERL, type 12, 2.5 kg impact machine shall be tested for accuracy each day that it is used for testing HMX in accordance with the specification. For this purpose an HMX sample having a known sensitivity range shall be used. The results shall be recorded on a chart showing the minimum and maximum values for the sample (control limits) and the mid point of the control limits range. If the test result for the control sample falls outside or near the minimum or maximum value of the control limits, the impact machine shall be checked and adjusted if necessary to assure that the platform for the anvil is level and the guide rails for the drop weight is upright. Drop height is also checked periodically with vertical weighed line to ensure that the impact machine is upright. Drop tests with carbon paper for determination of points of impact shall be conducted, and impact machine shall be adjusted as required.

4.7.7.4 Holston impact sensitivity. The Holston impact sensitivity of HMX shall be determined using a Holston impact machine.

4.7.7.4.1 Holston short impact method. Pass a dry sample of HMX through a sample splitter or roll and quarter on glazed paper until a representative sample of about 5 grams is obtained. Position 13 brass percussion caps (0.303 inch diameter and 0.20 inch in height) with open end up on a flat surface. Fill a 0.025 g loading spoon with HMX, remove the excess material by passing the flat edge of a wooden spatula across flat surface of the spoon and transfer the portion remaining in the spoon to one of the percussion caps. Repeat the HMX loading procedure until all of the percussion caps are loaded. Be sure the HMX is evenly

distributed in each of the percussion caps. Turn on the exhaust fan in order to remove the dust and fumes from the impact machine enclosure. Using laboratory tongs place a loaded percussion cap on the anvil of the impact machine. Insert a plunger through the guide hole above the anvil and into the open end of the percussion cap. Turn the electromagnet power switch on. Turn the gear handle until the base of the lower electromagnet arm coincides with the 26 cm mark on the right guide bar. Lower the safety shield and lift the weight vertically until it is held in place by the electromagnet. Face the opposite direction of the impact machine and turn the electromagnet switch to the OFF position allowing the weight to fall and strike the plunger. Lift the weight and raise the safety shield and allow the weight to rest on the safety shield. Examine the percussion cap to determine if an explosion has occurred. If an explosion has occurred, the percussion cap is usually disintegrated. A partial explosion may be recognized by inspection of the cap for parts of the rim having been blown away. An explosion may also be recognized by a sharp report or smoke in the area of the plunger, Clean away all of the unexploded material and percussion cap parts from the anvil, plunger and base plate with a brush or cloth prior to placing the next test sample in place.

a. If the above test is a non-explosion, place a minus sign on line zero (0) above Test No. 1 on the short impact method test card. (See below for a sample test card.) Repeat the procedure with one of the loaded percussion caps, and continue testing on line zero until an explosion occurs or the acceptance (A) zone has been reached. If an explosion occurs, move up to the next line on the card and continue testing until an acceptance or rejection is determined.

b. If the above test was an explosion, place a plus sign on Line 1 above Test No. 1. Should a second explosion occur, record the plus sign on Line 2 above Test No. 2. The sample would be rejected and a long impact must be performed. If the second sample is not an explosion, continue testing and record explosions or non-explosions on their appropriate line until acceptance or rejection is determined.

c. If the sample being tested is rejected, a long impact value must be determined in accordance with 4.7.7.4.2. Otherwise (if the sample passes the short impact test) report sample as accepted on impact.

Sample test card for Holston short impact test method

SHORT METHOD
IMPACT SENSITIVITY

RUN HMX AT 26 CM
RUN RDX AT 33 CM

IF BATCH IS REJECT - RUN IMPACT BY LONG METHOD
USE OTHER SIDE OF CARD

ACCEPT _____
REJECT _____
LONG METHOD _____CM

	4								R	R	R	R	R	R
NUMBER	3			R	R	R	R	R	o	o	o	o	o	A
OF	2		R	o	o	o	o	o	o	o	o	o	A	A
EXPLOSIONS	1	o	o	o	o	o	o	o	o	o	o	A	A	A
	0	o	o	o	o	o	A	A	A	A	A	A	A	A
		1	2	3	4	5	6	7	8	9	10	11	12	13

TEST NUMBER

NOTE: A - Zone for acceptance

R - Rejected by the short method, and a test with the long impact method (see 4.7.7.4.2) must be performed.

4.7.7.4.2 Holston long impact method. Pass a dry sample of HMX through a sample splitter or roll and quarter on glazed paper until a representative sample of about 5 gram is obtained. Position 25 brass percussion caps (0.303 inch diameter and 0.20 inch in height) with open end up on a flat surface. Fill a 0.025 g (25 mg) loading spoon with HMX, remove the excess material by passing the flat edge of a wooden spatula across flat surface of the spoon and transfer the portion remaining in the spoon to one of the percussion caps. Repeat the HMX loading procedure until all of the percussion caps are loaded. Be sure the HMX is evenly distributed in each of the percussion caps. Turn on the exhaust fan in order to remove the dust and fumes from the impact machine enclosure. Using laboratory tongs place a loaded percussion cap on the anvil of the impact machine. Insert a plunger through the guide hole above the anvil and into the open end of the percussion cap. Turn the electromagnet power switch on. Turn the gear handle until the base of the lower electromagnet arm coincides with the 30 cm mark on the right guide bar. Lower the

safety shield and lift the weight vertically until it is held in placed by the electromagnet. Face the opposite direction of the impact machine and turn the electromagnet switch to the OFF position allowing the weight to fall and strike the plunger. Lift the weight and raise the safety shield and allow the weight to rest on the safety shield. Examine the percussion cap to determine if an explosion has occurred. If an explosion has occurred, the percussion cap is usually disintegrated. A partial explosion may be recognized by inspection of the cap for parts of the rim having been blown away. An explosion may also be recognized by a sharp report or smoke in the area of the plunger, Clean away all of the unexploded material and percussion cap parts from the anvil, plunger and base plate with a brush or cloth prior to placing the next test sample in place. Repeat the procedure with the remaining HMX loaded percussion caps until twenty trials have been recorded. Raise the electromagnet 5 cm after each non-explosion and lowering the electromagnet 5 cm after each explosion. The first non-explosion after an explosion shall be considered as the starting point of the twenty trials. Record the electromagnet height for this trial in cm as the first of the twenty trials. Place a minus sign (-) opposite the height in cm indicating the trial resulted in a non-explosion. Record the electromagnet height for each subsequent trials and either a minus sign to indicate a non-explosion or a plus sign (+) to indicate an explosion.

Calculate the percentage explosions at a given height using the following equation:

$$\text{Explosions, 50\% point} = \frac{A \times 100}{B}$$

Where:
A = The number of explosions at a given height.
B = The total number of explosions and non-explosions at the given height.

Calculate the impact sensitivity as follows:

$$\text{Impact sensitivity, cm} = C - \frac{5(D-50)}{D-E}$$

Where:
C = The lowest height in cm at which more than 50% explosions occurred.
D = The percentage of explosions greater than 50%
E = The percentage of explosions less than 50%.
5 = The difference in height in cm of each test.

The Holston impact machine shall be tested for accuracy each day that it is used for testing HMX in accordance with the specification. For this purpose an HMX sample having a known sensitivity range shall be used. The test shall be run in accordance with 4.7.7.4.2. The results shall be recorded on a

chart and shall fall within the minimum and maximum values for the control sample (control limits).

4.7.8 Granulation. The granulation shall be determined by the following procedure:

A 50 ± 0.05 gram sample of HMX (dry weight), weighed accurately to the nearest 0.01 gram, shall be transferred to a 600 ml beaker containing approximately 300 ml of a 2 percent solution of a suitable wetting agent such as dioctyl sodium sulfosuccinate (see 6.5). With the aid of a rubber policeman attached to a glass stirring rod, the mixture shall be stirred for a few minutes wetting the sample thoroughly and breaking up as many of the aggregate as possible.

A spray nozzle (see 6.6.2) under tap water pressure, shall be used to quantitatively transfer this mixture to the uppermost sieve of a set of the specified 8-inch U.S. Standard sieves complying with Specification RR-S-366, nested in order of decreasing size, the largest mesh being placed on top. This assembly should be set up near a water tap and drain. Provisions shall be made to prevent transfer of the explosive to the drain. The pressure of the spray shall be adjusted so that when the spray strikes the sample at an angle approximately perpendicular to the screen from a height of 2 to 3 inches, it is possible to wash the sample back and forth across the sieve without splashing any of the material over the side of the sieve. The spray should be moved about the screen at a rate such that the spray would traverse the diameter of the screen 1 to 2 times per second. The wet agglomerates shall be gently crushed on the top sieve with the aid of a rubber policeman and the washing of the material back and forth across the sieve with the water spray shall be continued until all the agglomerates have been broken and only individual crystals larger than the mesh of the sieve remain on the sieve. The rubber policeman shall be used in breaking up the agglomerates on only the uppermost sieve. The top sieve shall be removed and a few drops of a 10 percent solution of the wetting agent shall be added to the material on the next sieve and washed with the water spray as before for a maximum of 2 minutes or until no change is noticed in the amount of crystals remaining. This procedure shall be repeated for each of the sieves.

After the washing has been completed, the portions remaining on each of the screens shall be quantitatively transferred by means of a suitable transfer funnel into a separate previously tared fritted glass filtering crucibles of coarse porosity as follows:

The screen shall be held in an almost vertical position and with a moderate spray of water from the spray nozzle, the material shall be gently washed to the lower part of the screen by drawing the spray back and forth across the screen, beginning at the top and moving slowly down the screen as the crystals move

down. When the material has been collected at the lower part of the sieve, it can readily be washed into the crucible with a stream of water from a wash bottle.

The crucible shall be aspirated during the transfer process and the crucible and contents shall be aspirated for approximately 2 minutes after the transfer has been completed. The suction shall be turned off and 15 ml of anhydrous methanol added to the crucible and contents; the methanol shall be allowed to remain in contact with the explosive for approximately 5 seconds and then removed with the aid of suction. The contents of the crucible shall be washed once more with anhydrous methanol as described above and then the crucible and contents shall be aspirated until the odor of methanol is no longer discernible. The crucible and contents shall be dried in an oven maintained at 105 ± 2 degrees C for 15 minutes (or equivalent method), cooled in a desiccator and weighed.

The weight of material retained on each sieve shall be determined and the percentage through each sieve calculated on the basis of the dry weight of the HMX sample to determine compliance with 3.2.1 (Table II).

4.7.9 Workmanship. Visually observe the production of HMX and examine the samples submitted for testing for compliance with the requirements given in 3.4.

Attribute sample inspection shall be conducted in accordance to Table III (see 4.5.2) using the inspection levels in Table III as stated in the classification of characteristics paragraphs below (4.7.9.1 to 4.7.9.5).

NOTE: See DOD-STD-2101 for definitions of critical, major and minor classification of characteristics.

4.7.9.1 Classification of characteristics - loaded drum with tied bag, cloth (See 5.1 and 6.4.1)

Classification	Examination/test	Conformance criteria	requirement paragraph	inspection method ref.
Critical	None defined			
Major				
101	Foreign matter	Level II	3.4	Visual
102	Bag pierced or torn	Level II	3.4	Visual
103	Not tied or improperly tied	Level II	3.4	Visual
Minor	None defined			

4.7.9.2 Classification of characteristics - empty polyethylene bag. (See 5.1 and 6.4.1)

Classification	Examination/test	Conformance criteria	Requirement paragraph	Inspection method ref.
Critical	None defined			
Major				
101	Seam splits when pressure is applied along entire length of seam.	Level II	3.4	Visual/manual
102	Bag damaged	Level II	3.4	Visual
Minor	None defined			

4.7.9.3 Classification of characteristics - loaded drum with tied polyethylene bag or sealed rubber bag. (See 5.1 and 6.4.1)

Classification	Examination/test	Conformance requirement	Requirement paragraph	Inspection method ref.
Critical	None defined	Level II	3.4	Visual
Major				
101	Bag improperly closed.	Level II	3.4	Visual
102	Bag damaged.	Level II	3.4	* See note
103	Insufficient solution.	Level II	3.4	* See note
104	Insufficient alcohol in solution.	Level II	3.4	* See note
105	Net weight.	Level II	3.4	* See note
Minor	None defined.			

NOTE: *Inspection will be accomplished by witnessing procedures used by contractor to comply with requirements.

4.7.9.4 Classification of characteristics - loaded drum with cover and locking ring before closing. (See 5.1 and 6.4.2)

Classification	Examination/test	Conformance criteria	Requirement paragraph	Inspection method ref.
Critical	None defined			
Major				
101	Top chime bent, deformed or cut.	Level II	3.4	Visual
102	Bottom chimes collapsed (annular groove closed or partially closed), or deformed.	Level II	3.4	Visual
103	Body bulged, cut or dented	Level II	3.4	Visual
104	Gasket in cover missing or damaged	Level II	3.4	Visual
105	Cover bent, creased or deformed in gasket area or around edge	Level II	3.4	Visual
106	Locking ring damaged so as to prevent closing	Level II	3.4	Visual/ manual
Minor				
201	Outer body surface seriously scuffed or metal scratched through galvanized surface	Level II	3.4	Visual
202	Nicks or dents in chimes or cover not affecting function	Level II	3.4	Visual
203	Locking ring bent or deformed	Level II	3.4	Visual

4.7.9.5 Classification of characteristics - sealed drum. (See 5.1 and 6.4.2)

Classification	Examination/test	Conformance criteria	Requirement paragraph	Inspection method ref.
Critical	None defined			
Major				
101	Alcohol solution leaking from drum	Level II	3.4	Visual/manual
102	Cover or locking band improperly seated	Level II	3.4	Visual
103	Seal missing or improperly applied	Level II	3.4	Visual
Minor				
201	Marking missing, incorrect or illegible	Level II	3.4	Visual

5. PACKAGING

5.1 Packaging. Packaging requirements shall be as specified in contract or order (see 6.2 and 6.4).

6. NOTES

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

6.1 Intended use. HMX is to be used as an energetic ingredient in a number of explosive compositions for applications where high energy is needed in ammunition items.

6.2 Acquisition requirements. The acquisition document must specify the following:

a. Title, number and date of this specification.

b. Issue of DODISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.1 and 2.2).

c. Provisions for submission/resubmission of first article samples. A first article quantity, or portion thereof, as directed by the Contracting officer in accordance with contract provision: Instruction Regarding Submission of First Article, should also be submitted whenever there is a lapse in HMX production of a period in excess of 90 days, or whenever a change occurs in the manufacturing process, or in sources of constituents or process materials, drawings or specifications which could affect product uniformity as determined by the Government.

d. Acceptance and description sheet - Acceptance and description sheets should be prepared for each lot in accordance with MIL-STD-1171.

e. Quantity required and delivery schedules.

f. Grade and granulation class required (see 1.1 and 3.2).

g. Levels of preservation and packing (see 5. and 6.4).

h. Acceptance and description sheets (see 6.3).

i. Item hazard classification (see 6.4.5).

6.3 Consideration of data requirements. When this specification is used in a procurement contract which incorporates DD Form 1423, contract data requirements list, acceptance and description sheets (for explosives) are required for each lot. These sheets should be prepared and submitted by the contractor in accordance with MIL-STD-1171 or otherwise as directed by the contract. In addition, the following data requirements should be considered when this specification is applied on a contract, per the Contract Data Requirements List, DD Form 1423:

Reference	DID Number	DID Title	Suggested Tailoring
6.4.4	DI-PACK-81059	POP Test Report	None

6.4 Acceptable packaging requirements. The following packaging and marking requirements have been used for packaging HMX, and is found to be acceptable to the Government. These requirements should be included in the contract or order for the procurement of HMX because HMX is a hazardous energetic material. (Caution: If the following paragraphs are to be incorporated in a contract, they must be modified, using standard contract language, to make them compulsory requirements.)

6.4.1 Preservation and packaging.

6.4.1.1 Level B and Level C. Unless otherwise specified by the procuring activity, HMX will be preserved and packaged as follows: Not more than 50 pounds dry weight, of the wet HMX should be packed in a cloth bag described in 6.4.1.1.2. Filled

cloth bags should be placed in a rubber bag, rubberized cloth bag or two polyethylene bags, described in 6.4.1.1.1, placed one inside the other. The bags should be free of foreign matter. A water-isopropyl alcohol solution in a 40 percent isopropyl alcohol and 60% water ratio should be added to the outer bag. The amount of water added has to comply with DOT requirements (49 CFR 100-199). Inner and outer bags should be securely tied, with the polyethylene bags individually tied, using non-metallic tape or cord. The tops of the outer bags should be gathered and formed into a gooseneck when being tied.

6.4.1.1.1 Polyethylene bags. The polyethylene bags should comply with Type II Style 1 of Specification PPP-B-26 except that closure will not be heat sealed. The bag size should be approximately 31 inches flat width by 55 inches in length. The bag seams have to meet the seam strength test with an AQL of 0.65 percent at 50 percent of the breaking strength of the polyethylene film. The seam strength test should be performed in accordance with Method A of ASTM D882 using one-inch wide specimens. Seams should also be examined by separating the bag face and applying pressure manually along the entire length of the seam. Seams which can be opened at any point in this manner are not acceptable.

6.4.1.1.2 Cloth bags. The cloth bags should be made from white cotton twill, free of size and brighteners, complying with the general requirements of Specification CCC-C-461 and the following detailed requirements:

Yarn	Carded
Yarn Ply	Warp 12/1 Fill 18/1
Weight	7.2 + 0.5 oz/sq yd (Greige) 6.0 + 0.7 oz/sq yd (after bleaching)
Yards per inch (minimum)	Warp 78 Fill 47
Breaking Strength (minimum)	Warp 170 Fill 70
Weave	2 x 1 twill

A suggested bag size is 19 inches wide by 29 inches in depth. The tie tape may be attached to the bag by sewing. Alternatively, double filled gray cloth cotton duck weighing not less than 12 oz. per square yard may be used for the bag.

6.4.2 Packing.

6.4.2.1 Level B. A maximum of 300 pounds dry weight of crystalline HMX packaged in accordance with 6.4.1 should be packed in an outer bag which has been placed in a drum complying

with Department of Transportation Specification 1A2, Paragraph 178.504 of the Code of Federal Regulation 49 CFR 100-199.

6.4.2.2 Level C. A maximum of 225 pounds dry weight of crystalline HMX packaged in accordance with 6.4.1 should be packed in a fiber drum complying with DOT specification 1G, Paragraph 178.508 of the Code of Federal Regulation 49 CFR 100-199 (approximate gross weight of 300 pounds) and the following requirements. The drum should be approximately 18 1/2 inches in diameter by 26 inches in height, inside dimensions. The drum should have a 24-gauge steel cover with a rubber gasket, level locking band with provisions for sealing wire and wide bottom chime (2-inch minimum formed height). All metal parts should be hot dip galvanized and chimes should be welded. Bottom chimes should be 0.028 inch steel and top chimes should be 0.025 inch steel. The body should be wound with hot melt or thermoplastic adhesive and the seam covered with a hot sealed tape. The inner surface of the bottom and body should have a laminated liner of 0.002 inch thick polyethylene. The bottom crimp has to be caulked. The finished drum including closure should be leak tight construction suitable for liquid contents.

6.4.3 Marking. Containers should be marked as required by the Code of Federal Regulation 49 CFR 100-199. In addition, shipments should be marked in accordance with Standard MIL-STD-129-1. Each container should be clearly labeled with the lot number and net weight of its contents. The HMX contents of the container have to be from the same lot.

6.4.4 Performance oriented packaging (POP). The exterior pack cited above should meet all of the POP test requirements in accordance with the Code of Federal Regulations, Title 49 (49CFR), including testing, and retesting as specified in Paragraph 178.601 (e) of 49 CFR. A POP test report should be generated in accordance with DI-PACK-81059 following the tests. POP test report may be waived if an acceptable non-government analogy can be made IAW 49CFR to another pack which has successfully completed the testing. This analogy must also be documented IAW DI-PACK-81059. When completed, either POP test report must be kept on file by the contractor and must also be submitted to the U.S. Army Research Development and Engineering Center, ATTN: AMSTA-AR-AEP, Picatinny Arsenal, New Jersey, 07806-5000. (NOTE: If a POP test report is prepared against an acceptable analogy, the analogy POP test report must also be submitted to AMSTA-AR-AEP). The POP marking symbol applied to the exterior pack should be that belonging to the organization which conducted the POP testing.

6.4.5 Item hazard classification. All U.S. manufacturers should make certain that the item is tested in accordance with Part 173, Subpart C, Section 173.58 (a) of 49 CFR, Parts 106-180 to assign proper Class and Division for all explosives (division 1.1, 1.2, 1.3 and 1.4 explosives.) Registration with the Associate Administrator of Hazardous Materials safety is required

in accordance with Part 173, Subpart C, Section 173.56 (b) (l) or 173.56 (c) of 49 CFR so that proper markings in accordance with Part 172, Subpart D, Section 172.301 (a) and 172.320 (a) are met.

All foreign manufacturers should make certain that the dangerous goods are tested in accordance with United Nations Committee of Experts on the Transportation of Dangerous Goods (as published in UN Document ST/SG/AC. 10.11 latest revision, Recommendations for the Transport of Dangerous Goods - Tests and Criteria) to determine the proper class and division (Class 1-9 and Division 1.1 -1.6 for explosives). Registration for air and vessel transport is required with each manufacturing country's National Competent Authority. The Hazard Classification letter of competent authority is issued in accordance with part 2, paragraph 1.3 of the International Civil Aviation Organization (ICAO) technical instructions and approves the hazard classification and compatibility group assignment and assigns the appropriate shipping name to the dangerous goods. The proper packaging, marking and labeling is contained in the united Nations Committee of Experts on the Transport of Dangerous Goods (as published in UN Document ST/SG/AC. 10.1, latest revision, recommendations on the Transport of Dangerous Goods).

For air transport the dangerous goods must comply with the provisions of the International Air Transport Associate (IATA) Dangerous Goods Regulations and for vessel transport, the dangerous goods must comply with the provisions of the Intergovernmental Maritime Organization's International Maritime Dangerous Goods Code (IMDG Code).

6.4.6 Referenced documents for packaging. The following list of documents referenced in 6.4 should be included in the contract or purchase order as requirement documents. Document users are cautioned that they must meet all requirements of these documents if cited in the contract or purchase order.

(a) Federal and military specifications/standards. (See 2.2.1 for issues of documents and address to obtain these documents.)

MIL-STD-129-1 - Marking for Shipment and Storage - Ammunition and Explosives
CCC-C-461 - Cloth, Twill, Uniform Cotton
PPP-B-26 - Bag, Plastic, Polyethylene

(b) Other Government documents, drawings and publications. Unless otherwise specified, the issues of documents are those cited in the solicitation.

CODE OF FEDERAL REGULATIONS

49 CFR 100-199 - Department of Transportation Rules and Regulations for the Transportation of Explosives and other Dangerous Articles

(The Interstate Commerce Commission Regulations are now a part of the Code of Federal Regulations, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. Orders for the above publication should cite, "49 CFR 100-199 (latest revision)".)

(c) Non-government publications. The issues of the following documents which are DOD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issues of the documents cited in the solicitation. (see 6.2)

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM Designation D882 - Tensile Properties of Thin Plastic Sheeting

(Applications for copies should be addressed to the American Society for Testing Materials, 100 Bar Harbor Drive, West Conshohocken, PA 19428-2959.)

6.5 Dioctyl sodium sulfosuccinate. Dioctyl sodium sulfosuccinate is commercially available as Aerosol OT.

6.6 Approved equipment.

6.6.1 MicroBondapak (CN) HPLC column. The MicroBondapak CN column (Catalog No. WAT086688) is available from the Millipore Corporation, Waters Chromatography Division, headquartered in Milford, MA.

6.6.2 Spray nozzle. A spray nozzle can be made by fitting a Gooch crucible (Number 5, Coors porcelain crucible containing about 75 openings approximately 0.07 centimeters (cm) in diameter has been found satisfactory) over a Number 8 one hole rubber stopper fitted with a short piece of glass tubing to which is attached a length of rubber hose approximately 1 cm inside diameter. The free end of the hose is connected to the water tap and the Gooch crucible at the other end acts as a spray nozzle.

6.7 Virgin HMX. Virgin HMX is produced with a feedstock to the recrystallizer that is solely composed of slurried HMX coming directly from the HMX synthesis process.

6.8 High purity HMX (beta), RDX calibration standards. High purity HMX (beta) and RDX calibration standards can be obtained from the following addressees:

a. Commander, Holston Army Ammunition Plant, 4509 West Stone Dr., Kingsport, TN 37660-9982.

b. Commander, ARDEC, ATTN: AMSTA-AR-AEE-W, Picatinny Arsenal, NJ 07806-5000.

c. Naval Explosive Ordnance, Technology Division, Bldg. 2172, ATTN: Mr. Lou Wasserzug (Code 50A24), 2008 Stampneck Rd., Indian Head, MD 20640.

6.9 Technical agency. Picatinny Arsenal is the technical agency that is responsible for the preparation and revision of the specification. All correspondence to the Technical Agency should be submitted through the contracting officer to: Commander, ARDEC, ATTN: AMSTA-AR-QAT-P, Picatinny Arsenal, NJ 07806-5000.

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

6.11 Submission of alternative conformance inspection provisions. Unless otherwise specified in the contract, proposed alternative conformance inspection provisions should be submitted by the contractor for evaluation by the technical activity responsible for preparation of this specification. (See 6.9)

6.12 International standardization agreement. Certain provisions of this specification are the subject of international standardization agreement NATO STANAG 4284. When amendment, revision or cancellation of this specification is performed, the departmental custodians will inform their respective Departmental Standardization Offices so that appropriate action may be taken respecting the international agreement concerned.

6.13 Subject term (key word) listing.

Explosive compositions
Her majesty explosive
High melting explosive

6.14 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

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.

Custodians
Army - AR
Navy - OS
Air Force - 99

Preparing activity:
Army-AR

(Project 1376-0038)

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.

2. The submitter of this form must complete blocks 4, 5, 6, and 7.

3. The preparing activity must provide a reply within 30 days from receipt of the form.

NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced documents(s) or to amend contractual requirements.

I RECOMMEND A CHANGE:	1. DOCUMENT NUMBER **MIL-DTL-45444C**	2. DOCUMENT DATE (YYMMDD) **961126**

3. DOCUMENT TITLE

HMX (CYCLOTETRAMETHYLENETETRANITRAMINE)

4. NATURE OF CHANGE (*Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)*

5. REASON FOR RECOMMENDATION

6. SUBMITTER

a. NAME *(Last, First, Middle Initial)*	b. ORGANIZATION	
c. ADDRESS (*Include Zip Code)*	d. TELEPHONE (*Include Area Code)* (1) Commercial (2) AUTOVON (*if applicable)*	7. DATE SUBMITTED (*YYMMDD)*

8. PREPARING ACTIVITY

a. NAME **U. S. Army ARDEC**	b. TELEPHONE (*Include Area Code)* (1) Commercial **(201) 724-6671**	(2) AUTOVON **880-6671**
c. ADDRESS (*Include Zip Code)* **ATTN: AMSTA-AR-EDE-S, B-12** **Picatinny Arsenal, NJ 07806-5000**	IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT: Defense Quality and Standardization Office 5203 Leesburg Pike, Suite 1403, Falls Church, VA 22041-3466 Telephone (703) 756-2340 AUTOVON 289-2340	