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5 February 1990
SUPERSEDING
WS 20617
22 January 1985
(See 6.11)

MILITARY SPECIFICATION

CYCLOTETRAMETHYLENETETRANITRAMINE (HMX)

This specification is approved for use by the Naval Sea Systems Command, Department of the Navy and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification defines one type of cyclotetramethylenetetranitramine, referred to herein as HMX, which has been precipitated from acetone and screened through 80-mesh tensile bolting cloth (see 6.6.2) and two types of processed HMX.

1.2 Classification. The HMX shall be classified as follows:

a. Type I - HMX (see 6.8)

b. Type II - HMX, dry

c. Type III - HMX, ground (3.2 micrometers, μm)

NOTE: The processing of the type II and type III HMX shall be in accordance with the applicable manufacturing process control document.

2. APPLICABLE DOCUMENTS

2.1 Government documents.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Ordnance Station, Standardization Branch (3730), Indian Head, MD 20640-5000, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A FSC 1376

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2.1.1 **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).

STANDARDS

FEDERAL

FED-STD-313	Material Safety Data, Transportation Data and Disposal Data for Hazardous Materials Furnished to Government Activities

MILITARY

MIL-STD-129	Marking for Shipment and Storage

(Unless otherwise indicated, copies of federal and military specifications and standards are available from: Military Specifications and Standards, Bldg. 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094).

2.1.2 **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.

CODE OF FEDERAL REGULATIONS (CFR)

49 CFR 100-199	Transportation

(Application for copies of CFRs should be addressed to the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402-0001.)

2.2 **Non-Government publications.** The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the 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 E 11	Wire-Cloth Sieves for Testing Purposes (DoD adopted)
ASTM E 161	Precision Electroformed Sieves (Square Opening Series) (DoD adopted)

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103-1187.)

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AMERICAN TRUCKING ASSOCIATION, INC.

National Motor Freight Classification

(Application for copies should be addressed to the American Trucking Association, Inc., Attn: Traffic Dept., 2200 Mill Road, Alexandria, VA 22314-4677.)

NATIONAL RAILROAD FREIGHT COMMITTEE

Uniform Freight Classification (UFC) 6000

(Application for copies should be addressed to the National Railroad Freight Committee, 222 South Riverside Plaza, Suite 1120, Chicago, IL 60606-5945.)

(Non-Government standards and other publications are normally available from the organizations that prepare or distribute the documents. These documents also may be available in or through libraries or other informational services.)

2.3 **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.

3. REQUIREMENTS

3.1 **First article.** When specified (see 6.2), a sample shall be subjected to first article inspection (see 6.3) in accordance with 4.3.

3.2 **Material content.** Material content shall consist of not less than 98.0 percent (%) cyclotetramethylenetetranitramine basically in beta form, with no greater than 2.0% cyclotrimethylenetrinitramine (RDX). Alpha polymorph of HMX shall be no greater than 0.5%.

3.3 **Melting point.** Melting point of the HMX shall be not less than 275 degrees Celsius (°C).

3.4 **Insoluble particles.** There shall be no acetone insoluble particles retained on a U.S. Standard Series (USSS) No. 40 sieve and not more than 5 acetone insoluble particles retained on a USSS No. 60 sieve per 50 grams (g) of type I HMX.

3.5 **Total acetone insoluble material.** Total acetone insoluble material shall be not greater than 0.05%.

3.6 **Inorganic insoluble material.** Inorganic insoluble material shall be not greater than 0.03% (see 4.5.5 NOTE).

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3.7 **Acidity.** Acidity, as acetic acid, shall be not greater than 0.02%.

3.8 **Granulation.** Contractor certification verifying that all the type I HMX has been screened through 80-mesh tensile bolting cloth shall be required (see 6.2).

3.8.1 **Types II and III.** Manufacturer or processor certification verifying that all type II and type III HMX has been made from type I HMX shall be required (see 6.2).

3.9. **Particle size distribution.**

3.9.1 **Particle size distribution for batches.** Particle size distribution by weight of each type I HMX batch shall conform to the limits specified in table I.

TABLE I. Batch particle size distribution.

Cumulative Weight Distribution	Particle Diameter Range
10 % level	0.9 - 51.3 μm
50 % level	57.0 - 130.3 μm
90 % level	126.0 - 200.4 μm

3.9.2 **Particle size distribution of allocated groups.** Type I HMX shall be allocated into blend groups and the calculated particle distribution of the allocated blend groups shall meet the limits specified in table II. Contractor certification verifying that the calculated particle distribution of the allocated blend groups meets the limits specified in table II shall be required (see 6.2). The certification shall also include the following:

a. Allocation number. Allocations shall be numbered in series and the numbers shall not repeat.

b. Batch number of each batch in the group.

c. Weight of each batch.

d. Total weight of each group.

e. Particle size distribution of allocated group.

f. Title, number, date, and revision of this specification.

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TABLE II. Particle size distribution of allocated HMX.

Cumulative Weight Distribution	Particle Diameter Range
10 % level	28.0 - 32.0 μm
50 % level	96.6 - 103.4 μm
90 % level	166.1 - 171.9 μm

3.9.3 **Particle size distribution of type III HMX.** Particle size distribution by weight of type III ground HMX shall conform to the limits specified in table III.

TABLE III. Type III particle size distribution.

Cumulative weight percent less than stated diameter	Spherical diameter range
10 %	0 - 2.0 μm
50 %	2.1 - 4.2 μm
90 %	4.4 - 9.4 μm

3.10 **Types II and III moisture content.** The maximum moisture content for types II and III shall be 0.04%.

3.11 **Shelf life.**

3.11.1 **Types I and II HMX.** Shelf life of types I and II HMX shall be considered to be unlimited when stored at ambient temperature in sealed containers.

3.11.2 **Type III HMX.** Shelf life of type III ground HMX shall be one year when stored in sealed containers at ambient conditions.

3.11.2.1 **Shelf life extension.** The type III HMX shelf life may be extended for one year periods provided the particle size distribution and moisture requirements are met.

3.12 **Safety.**

3.12.1 **Toxic products and safety.** The material shall have no adverse effect on the health of personnel when used for its intended purpose in accordance with the precautions of the Material Safety Data Sheets (MSDSs).

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to the appropriate departmental medical service who will act as an advisor to the contracting agency (see 4.1.2).

3.12.2 **Material Safety Data Sheets (MSDS).** The contractor shall prepare and submit a MSDS in accordance with FED-STD-313 as specified in the contract (see 6.2 and 6.9).

3.13 **Workmanship.** The HMX shall be uniform in quality and free of visible impurities which would render the HMX unsuitable for the intended use.

4. QUALITY ASSURANCE PROVISIONS

4.1 **Responsibility for inspection.** Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements (examinations and tests) 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 the specification where such inspections are deemed necessary to ensure supplies and services conform to prescribed requirements.

4.1.1 **Responsibility for compliance.** All items shall meet all requirements of sections 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 ensuring 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.

4.1.2 **Toxicological product formulations.** The contractor shall have the toxicological product formulations and associated information available for review by the contracting activity to evaluate the safety of the material for the proposed use.

4.2 **Classification of inspections.** Inspection and testing of the HMX shall be classified as follows:

a. First article inspection (see 4.3).

b. Quality conformance inspection (see 4.4).

4.3 **First article inspection.** First article inspection shall consist of the examination and tests specified in table IV (see 4.4 and 6.2). Failure to meet all requirements specified herein shall be cause for rejection of the first article sample.

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4.4 **Quality conformance inspection.** Quality conformance inspection shall consist of the examination and tests specified in table IV and verification of contractor certification required in 3.8, 3.8.1 and 3.9.2. Failure to meet the requirements specified herein shall be cause for rejection of the lot.

TABLE IV. Quality conformance inspection.

Examination and tests	Requirement	Test Method
Visual examination	3.8, 3.9.2, 3.12, 3.13 & section 5	4.5.1 & 4.5.9
Material content test	3.2	4.5.2
Melting point test	3.3	4.5.3
Insoluble particles test	3.4	4.5.4
Acetone insoluble material test	3.5	4.5.5
Inorganic insoluble material test	3.6	4.5.6
Acidity test	3.7	4.5.7
Particle size distribution test	3.9	4.5.8

4.4.1 **Sampling for inspection.**

4.4.1.1 **Visual examination.** Sampling for visual examination to determine conformance to section 5 shall consist of a random selection of at least 10% of the lot. Sampling for visual examination to determine conformance to 3.13 shall consist of the sample selected in accordance with 4.4.1.2.

4.4.1.2 **Sampling for tests.** Each batch shall be sampled by probing to obtain thief samples from a minimum of 12 well separated places over the surface of the batch. The 12 thief samples shall be composited to give one sample per batch. The particle size distribution of each batch shall be determined to verify conformance to the requirements of table I. Individual batches shall be accepted based on batch particle size distribution (table I). For all tests other than particle size, reduced sampling shall be used (see 6.4).

4.4.2 **Lot.** For inspection purposes, lots shall be defined as follows:

a. A lot of allocated type I HMX shall consist of one or more batches.

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b. Type II lot - A lot of type II HMX shall consist of material dried from a lot of type I HMX.

c. Type III lot - A lot of type III HMX shall be a quantity ground in one or more grinds from a single lot of type II HMX.

4.4.2.1 **Group.** Each group shall consist of all the batches that are combined in one allocation to meet the requirements of table II.

4.4.2.2 **Batch.** Each batch shall consist of that quantity of material subjected to the same unit chemical or physical mixing or grinding process intended to make the final product homogeneous.

4.5 **Inspection methods.** The following inspection methods shall be used. Unless otherwise specified in the method, all weights, volumes, temperatures, and times shall be measured to the nearest specified unit or decimal.

NOTE: Reagent grade chemicals shall be used for chemical reactions in the conduct of all tests defined herein. Solvents and indicators may be commercial nonreagent grade materials.

4.5.1 **Visual examination.** Samples selected in accordance with 4.4.1.1 and 4.4.1.2 shall be visually examined to determine compliance with 3.13 and section 5. Contractor certification verifying conformance to 3.8, 3.8.1 and 3.9.2 shall be examined (see 6.2).

4.5.2 **Material content.** HMX, RDX, and the alpha polymorph of HMX content shall be determined in accordance with the following procedures.

4.5.2.1 **Equipment.**

4.5.2.1.1 **X-ray diffractometer.** Equipment with voltage and current stabilizers, copper target tube with a nickel filter, curved graphite crystal monochromator and scintillation counter.

4.5.2.1.2 **Vacuum oven.**

4.5.2.1.3 **Burrell wrist action shaker or equivalent.**

4.5.2.1.4 **USSS No. 230 sieve.**

4.5.2.2 **Materials.**

a. Glacial acetic acid (buffer solution) - reagent grade.

b. Sodium acetate (buffer solution) - reagent grade.

c. Filter paper - medium retention grade.

d. 1, 2-dichloroethane (EDC) - technical grade.

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e. Dimethyl sulfoxide (DMSO) - reagent grade.

f. Acetone - technical grade.

g. Nitric acid - reagent grade.

4.5.2.3 Test procedure.

4.5.2.3.1 Preparation of standard sample materials. Materials to be used for standards shall be prepared as follows:

4.5.2.3.1.1 Beta HMX.

a. Obtain a 0.5 ± 0.1 pound sample of a production batch of HMX.

b. Heat the sample with four parts of buffer solution at 90 ± 5°C for 2 hours. (Buffer solution: 6.0 milliliter (mL) glacial acetic acid and 13.6 gram (g) sodium acetate diluted to 1 liter). (pH 4.6).

c. Filter, then dry the HMX at 100 ± 5°C for 2 hours. Wash the dry HMX with technical grade EDC. Use twice the quantity of EDC required to remove the residual cyclotrimethylenetrinitramine (RDX) as indicated by the original purity analysis.

d. Wash with 50% aqueous acetone solution.

e. Dry at 100 ± 5°C for 2 hours minimum.

4.5.2.3.1.2 Alpha HMX.

a. Add 4.0 ± 0.8 g of the HMX as purified in 4.5.2.3.1.1 to 80 mL of 70% nitric acid.

b. Heat until the HMX dissolves. Dissolution may be difficult unless heated at high temperatures, i.e., 100 ± 5°C or above. Lesser quantities can be recovered by filtering to remove undissolved HMX while retaining the filtrate at high temperatures.

c. Cool the solution to 30 ± 6°C over a period of 1 hour.

d. Filter, wash the solids free of acid with distilled water, and dry in vacuum oven at not more than 15 millimeters (mm) Hg absolute pressure and 60 ± 12°C for 2 hours minimum.

4.5.2.3.1.3 RDX.

a. Obtain a 100 ± 20 g sample of a production batch of RDX.

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b. Heat the sample with four parts of buffer solution (4.6 pH) (see 4.5.2.3.1.1b) at 90 ± 5°C for 2 hours minimum.

c. Filter, then dry the RDX at 100 ± 5°C for 2 hours.

d. Heat 1 part RDX and 1-1/2 parts DMSO to 92 to 96°C.

e. Add sufficient DMSO, up to 1 part, to dissolve all the RDX.

f. Digest at 92 to 96°C for 30 minutes minimum.

g. Add distilled water until solution becomes cloudy.

h. Reheat until solution clears, then cool rapidly to room temperature and filter.

i. Wash with 50 percent aqueous acetone.

j. Dry at 100 ± 5°C for 2 hours minimum.

k. Analyze a small sample for purity by the EDC method as follows:

(1) Place the sample in a beaker and add four parts (by weight) of a buffer solution and heat at 90 ± 18°C for two hours minimum. (Buffer solution: 6.0 mL glacial acetic acid and 13.6 g of sodium acetate diluted to one liter (pH 4.6)).

(2) Filter the solution using a medium porosity crucible.

(3) Dry the HMX at 100 ± 5°C for two hours minimum.

(4) Wash the dry HMX with 200 mL of EDC to remove residual RDX.

(5) Wash with 50% aqueous acetone solution.

(6) Dry at 100 ± 5°C for two hours minimum.

l. Repeat 4.5.2.3.1.3b through 4.5.2.3.1.3j until a pure product is indicated by X-ray diffraction.

4.5.2.3.2 **Mixing standard sample materials.** Beta HMX, alpha HMX, and RDX, in pure form obtained from 4.5.2.3.1.1, 4.5.2.3.1.2 and 4.5.2.3.1.3 shall be mixed as follows:

a. Grind not more than 0.1 g of recrystallized beta HMX, alpha HMX and RDX at one time, to a particle size of 62 micrometers or less. Use only material which passes through a USSS No. 230 sieve.

NOTE: Dry explosives shall be ground only when they are behind a suitable safety shield.

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b. In 250-mL flasks, mix beta HMX and alpha HMX or RDX as needed to prepare 5 ± 1 g samples of each of the following compositions:

Beta HMX, Percent	Alpha HMX, Percent	RDX, Percent
99.70	0.30	0.0
99.40	0.60	0.0
99.00	1.00	0.0
98.00	2.00	0.0
97.00	3.00	0.0
96.00	4.00	0.0
95.00	5.00	0.0
99.00	0.00	1.0
98.00	0.00	2.0
97.00	0.00	3.0
96.00	0.00	4.0
95.00	0.00	5.0
94.00	0.00	6.0
93.00	0.00	7.0
92.00	0.00	8.0
91.00	0.00	9.0
90.00	0.00	10.0

c. Mix standard sample materials on a wrist-action shaker for a minimum of 3 hours.

4.5.2.3.3 **Intensity measurement of standard sample composition.** Angular intensities of the standard sample composition shall be measured at 16.90 degrees, 17.81 degrees, 24.10 degrees and 25.10 degrees two theta as follows:

a. Press standard sample composition (see 4.5.2.3.2b) into cavity of sample holder.

b. Insert sample holder in path of X-ray beam.

c. Set X-ray diffractometer.

(1) Tube excitation shall be 40 kilovolts (kV).

(2) Filament current shall be 20 milliamperes (mA).

d. Set goniometer to 16.90 degrees two theta and count the background for 100 seconds.

(1) When counting is completed, activate recorder to print out time registered on rate meter dekatron tubes.

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e. Reset goniometer to 17.81 degrees two theta and count the RDX for 100 seconds.

f. Repeat step 4.5.2.3.3d(1).

g. Reset goniometer to 24.10 degrees two theta and count the background for 100 seconds.

h. Repeat step 4.5.2.3.3d(1).

i. Reset goniometer to 25.10 degrees two theta and count the alpha HMX for 100 seconds.

j. Repeat step 4.5.2.3.3d(1).

k. Repeat the above operations until all standard samples have been tested.

l. Deactivate X-ray equipment, or place in a standby condition.

4.5.2.3.4 Construction of calibration curves.

4.5.2.3.4.1 For RDX. For each standard sample, correct the intensity counts per second (cps), obtained at 17.81 degrees two theta by subtracting the background count obtained at 16.90 degrees two theta. Plot the corrected cps vs RDX weight percent as shown on figure 1.

4.5.2.3.4.2 For alpha HMX. For each standard sample, correct the intensity cps obtained at 25.10 degrees two theta by subtracting the background count obtained at 24.10 degrees two theta. Plot the corrected intensity cps vs alpha HMX weight percent as shown on figure 2.

4.5.2.3.4.3 Corrective curve for determining alpha HMX in presence of RDX. For the samples free of alpha HMX, correct the intensity cps obtained at 25.10 degrees two theta by subtracting the background count obtained at 24.10 degrees two theta. Plot the corrected intensity cps vs RDX weight percent (free of alpha HMX) as shown on figure 3.

NOTE: Calibration curves shall be re-determined when major changes are made to X-ray spectrograph, i.e., replacement of X-ray tube.

4.5.2.3.5 Preparation of sample to be analyzed. The sample to be analyzed shall be prepared as follows:

a. Reduce sample particle size to less than 62 micrometers.

b. Press sample material into cavity of sample holder.

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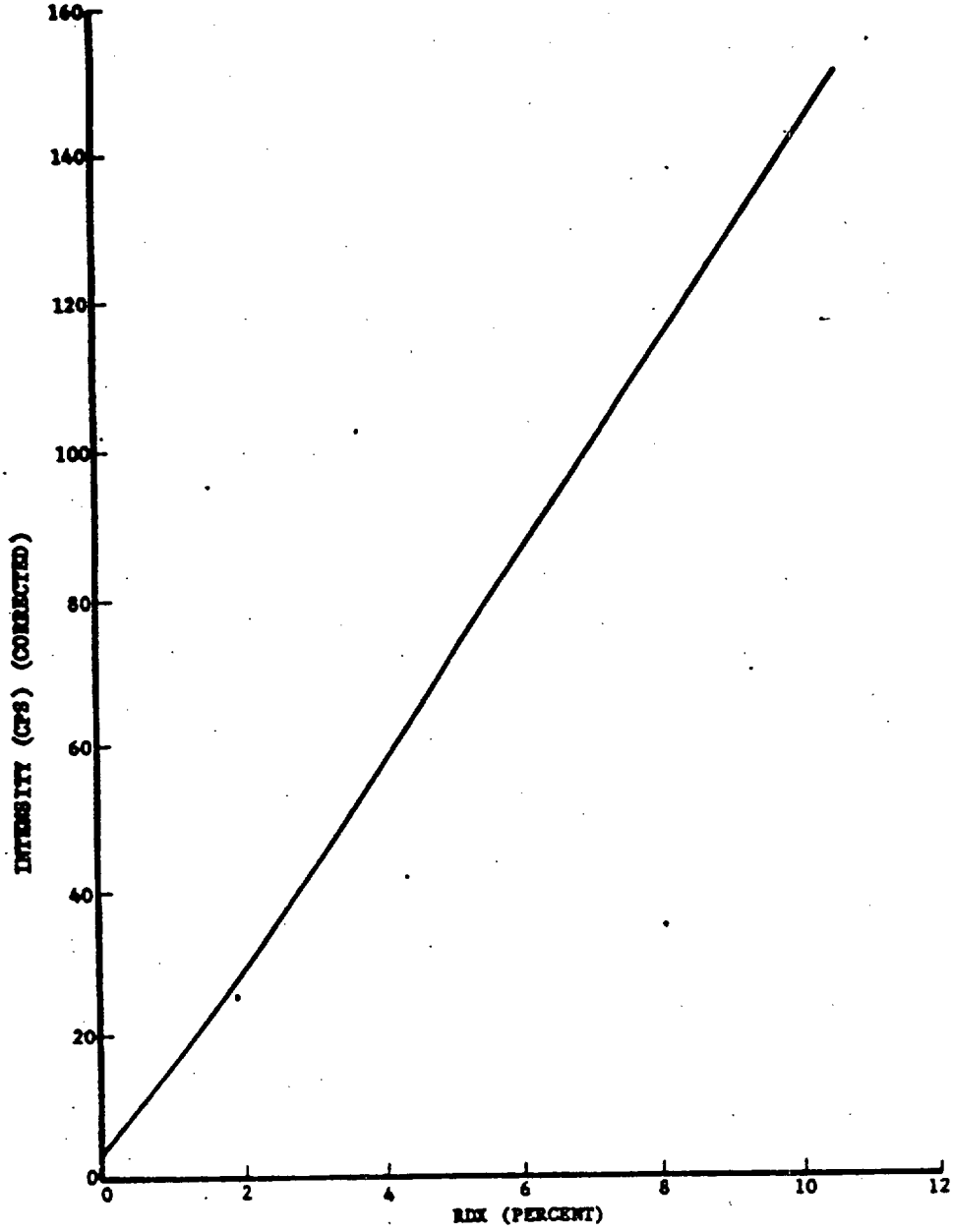

FIGURE 1. Calibration curve for the determination of RDX in the presence of beta HMX.

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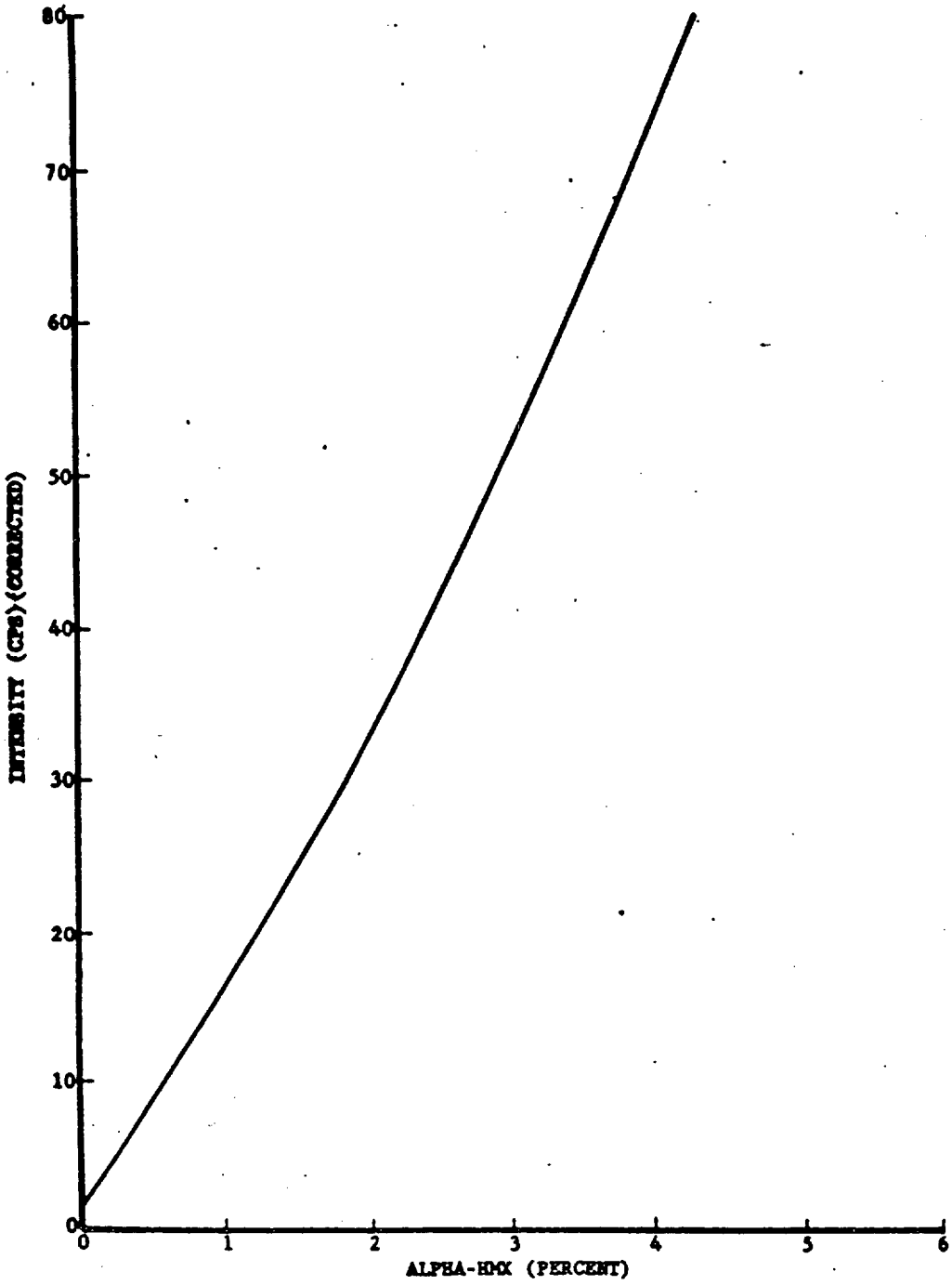

FIGURE 2. Calibration curve for the determination of alpha HMX in the presence of beta HMX.

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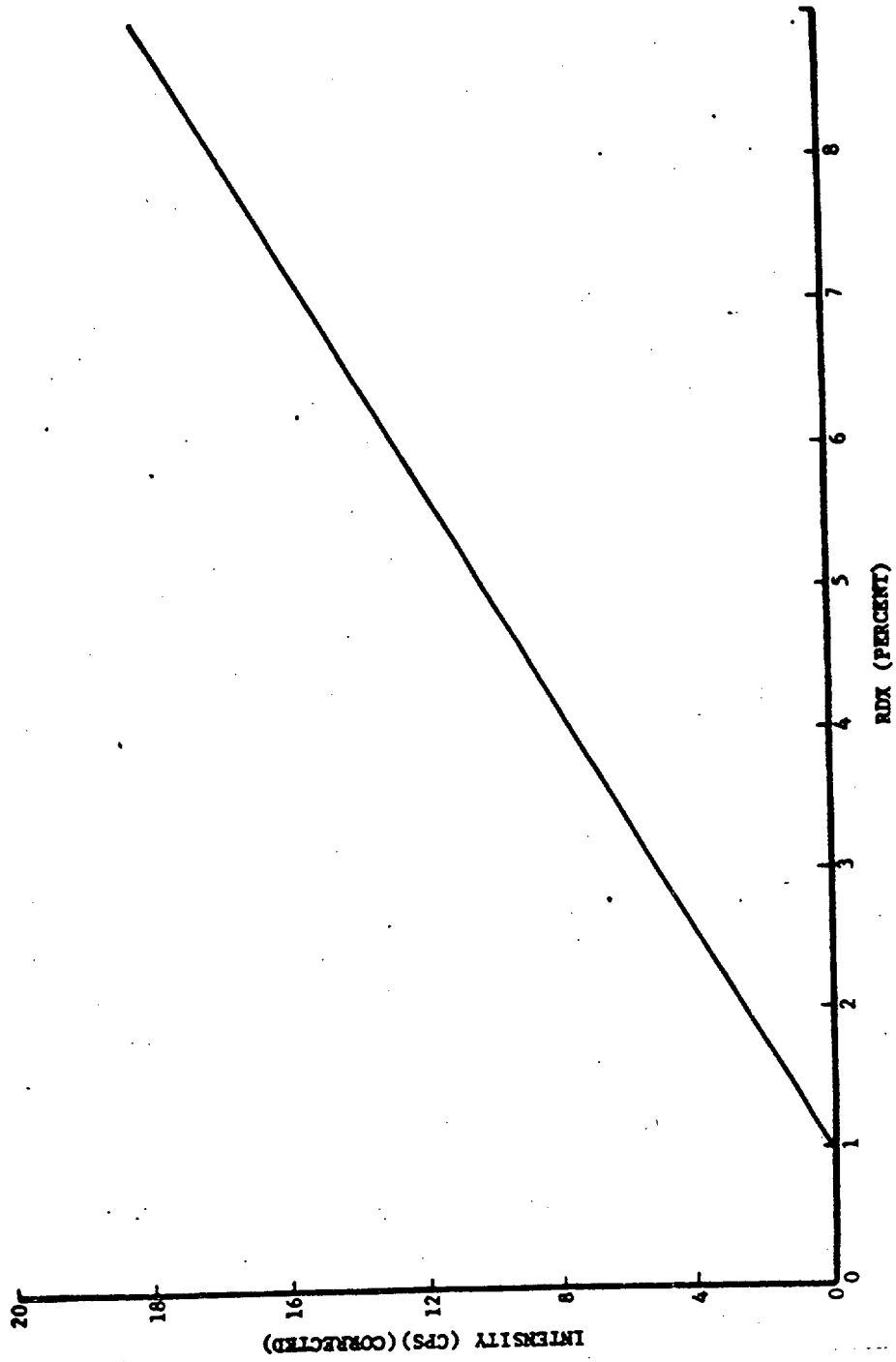

FIGURE 3. Alpha HMX angular intensity correction as a function of the RDX concentration.

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4.5.2.3.6 Intensity measurement of sample to be analyzed. Angular intensities of the sample to be analyzed shall be measured at 16.90 degrees, 17.81 degrees, and 25.10 degrees two theta as follows:

a. Place sample holder (see 4.5.2.3.5b) in path of X-ray beam.

b. Set X-ray diffractometer.

(1) Tube excitation shall be 40 kV.

(2) Filament current shall be 20 mA.

c. Set goniometer to 16.90 degrees two theta and count the background for 100 seconds.

(1) When counting is complete, activate recorder to print out time registered on rate meter dekatron tubes.

d. Reset goniometer to 17.81 degrees two theta and count the RDX for 100 seconds.

(1) Repeat step 4.5.2.3.6c(1).

e. Reset goniometer to 24.10 degrees two theta and count the background for 100 seconds.

(1) Repeat step 4.5.2.3.6c(1).

f. Reset goniometer to 25.10 degrees two theta and count the alpha HMX for 100 seconds.

(1) Repeat step 4.5.2.3.6c(1).

g. Repeat the above operations until all the samples in the lot to be analyzed have been tested.

h. Deactivate X-ray equipment, or place in stand-by condition.

4.5.2.3.7 Interpretation of data. Determine the intensity (cps) due to presence of RDX and intensity due to alpha HMX as follows:

a. Calculate intensities in counts per second by dividing time (seconds) into total counts for each setting (see 4.5.2.3.6c, 4.5.2.3.6d and 4.5.2.3.6e).

b. Subtract cps at 16.90 degrees two theta from cps at 17.81 degrees two theta. This is the intensity (cps) due to RDX present.

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c. Opposite the cps obtained in 4.5.2.3.7b, read the RDX concentration from RDX calibration curve.

d. Subtract cps at 24.10 degrees two theta from the cps at 25.10 degrees two theta. This is the intensity due to alpha HMX.

e. If the RDX concentration is greater than 1 percent, determine from figure 3 the correction required by reading the cps opposite the RDX percentage. Subtract this correction from the cps obtained in 4.5.2.3.7d.

f. From figure 2, read percentage alpha HMX opposite the intensity (corrected or uncorrected obtained in 4.5.2.3.7d or 4.5.2.3.7e).

4.5.3 **Melting point.** Melting point shall be determined in accordance with the following:

a. Pulverize 10 ± 2 milligrams (mg) of wet sample with an agate mortar and pestle.

b. Place 5 ± 1 mg of pulverized sample between two clean micro-cover glasses. Spread sample evenly by pressing and rotating top cover glass.

c. Place cover glasses containing sample on stage depression of Fisher-Johns, or equivalent, hot stage melting point apparatus.

d. Turn on power switch and heat the sample rapidly to 250 ± 50°C.

e. Reduce the setting to obtain a heating rate of 1°C per minute.

f. When sample begins to melt, determined by visual observation through a magnifying glass, record temperature of heating block to the nearest 1°C.

4.5.4 **Insoluble particles.** Insoluble particles shall be determined in accordance with the following:

a. Weigh 50.0 ± 0.1 g (dry weight basis) of HMX. Transfer 1/3 of the 50 g sample to a 1000-mL beaker.

b. Add 400 ± 80 mL of acetone to the beaker. Place a stirring rod in the beaker, cover with a watch glass, heat the contents of the beaker on a steam-heated hot plate, and stir periodically.

c. When the HMX is in solution, filter through 3-inch USSS No. 40 and 60 sieves.

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d. Dissolve each of the other 1/3 portions of the sample with 400 ± 80 mL of acetone and filter, repeating b and c above for each 1/3 portion.

e. Wash the sieves with acetone to remove any remaining traces of HMX.

f. Dry the sieves; then count, examine and identify, and record any particles remaining on the sieves.

4.5.5 **Acetone insoluble material.** The total acetone insoluble material shall be determined in accordance with the following:

a. Prepare a tared filtering crucible by washing the filter with acetone, igniting at 700 ± 140°C to constant weight.

b. Place the crucible on a vacuum flask and apply vacuum.

c. Quantitatively transfer the acetone solutions obtained in 4.5.4 to the crucible using acetone from a wash bottle to rinse the beaker.

d. Quantitatively transfer all insoluble particles retained on the USSS No. 40 and No. 60 sieves obtained in 4.5.4 into the filtering crucible.

e. Wash the crucible three times with acetone from a wash bottle to remove any trace of HMX.

f. Dry the crucible in a dryer. Cool in a desiccator and weigh.

g. Calculate percent acetone insoluble material as follows:

$$\text{Percent acetone insoluble material} = \frac{W2 - W1}{W} \times 100$$

Where: W1 = weight of empty crucible, g

W2 = weight of crucible and residue, g

W = weight of dry specimen, g.

NOTE: When the acetone insoluble material is greater than 0.03 percent, the residue shall be tested for inorganic insoluble material (see 4.5.6). When the acetone insoluble material is 0.03 percent or less, it will not be necessary to perform the inorganic insoluble material test. The inorganic insoluble material will be recorded as less than 0.03 percent.

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4.5.6 **Inorganic insoluble material.** Inorganic insoluble material shall be determined in accordance with the following:

a. Place the filtering crucible and residue obtained in 4.5.5 inside a solid bottom porcelain crucible. Ignite the crucibles for 10-minutes at 700 ± 140°C in a muffle furnace. Cool in a desiccator and reweigh.

b. Calculate percent inorganic insoluble material as follows:

$$\text{Percent inorganic insoluble} = \frac{W3 - W1}{W} \times 100$$

Where: W1 = weight of empty crucible, g (from 4.5.5)

W3 = weight of crucible and contents after ignition, g

W = weight of dry specimen, g (from 4.5.5).

4.5.7 **Acidity.** The acidity shall be determined in accordance with the following:

a. Accurately weigh a 10 ± 2 g specimen of HMX, transfer to a 1000-mL beaker and add 500 ± 100 mL of acetone, and cover with a watch glass.

b. Heat the beaker and contents on a steam bath maintained at 50 ± 10°C until the HMX is completely dissolved.

c. Allow the solution to cool to 35 ± 7°C, add 100 ± 20 mL of distilled water to precipitate the HMX, mix by swirling and titrate with 0.05 normal (N) sodium hydroxide using phenolphthalein indicator.

d. Run a blank titration to correct the results of the specimen titration for acidity of reagents.

e. Calculate the acidity on the dry basis as percent acetic acid as follows:

$$\text{Percent acidity (as acetic acid)} = \frac{6\ (VS - VB)\ N}{W}$$

Where: 6 = 100 X milliequivalent weight of acetic acid, g

VS = mL of sodium hydroxide solution used in sample titration

VB = mL of sodium hydroxide solution used in blank titration

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N = normality of sodium hydroxide solution

W = weight of specimen on a dry basis, g

4.5.8 **Particle size distribution.** Particle size distribution of type I HMX shall be determined in accordance with 4.5.8.1 through 4.5.8.3.

4.5.8.1 **Equipment.**

a. Precision electroformed sieves conforming to the construction and precision of ASTM E 161. Sieves for analysis shall be: 200, 180, 150, 130, 100, 80, 60, 40, 20, and 10 μm.

b. Ultrasonic bath - Blackstone Model SP-2 or Branson Model AP-10, or equivalent.

c. Sieving apparatus - Pantex.

4.5.8.2 **Reagents.** Reagents shall be in accordance with the following:

a. Triton X-100 (see 6.7).

b. Isobutyl acetate - industrial grade.

c. Eluant - Saturate the isobutyl acetate at room temperature with HMX for at least 24 hours minimum. During HMX saturation, add 0.25 g Triton X-100 for each gallon of isobutyl acetate. Filter the solution, using a fine porosity funnel, to remove crystalline HMX prior to use.

NOTE: In the event of large temperature changes or prolonged periods of standing, examine eluant to assure the absence of crystals.

4.5.8.3 **Test procedure.** The screen analysis procedure shall be as follows:

a. Dry sample in a 110 ± 22°C oven, then mix samples by rolling the container.

b. Select sieves. Weigh to the nearest mg and record weights of each of the sieves.

c. Weigh 5 ± 1 g of type I HMX on analytical balance and place in a 100-mL beaker with 25 ± 5 mL of isobutyl acetate eluant.

d. Place sample beaker in ultrasonic bath for a maximum of 3 minutes and manually stir.

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e. Place weighed sieves in a stack of descending micrometer order on the sieving apparatus.

NOTE: The sieves should have a small hole drilled in the side at the top. An aluminum rivet should be bradded in this hole. The rivet should have a hole drilled in it and a vertical filed indentation. The rivet will prevent the sieves from turning in the stack and the file mark will prevent an eluant lock.

f. Pressure settings (static) for filtered plant air shall be as follows:

Rotation - 40 to 55 pounds per square inch (psi)

Tappers - 30 to 35 psi

Vibrators - 50 psi

Eluant tanks - 3 to 4 psi

Eluant flow - 100 mL/min

g. Start rotation and vibration, pour samples through stacked sieves.

h. Wash sample and container with 50 to 75 mL of eluant and pour on sieve stack.

i. Place cover on sieve stack and start tappers.

NOTE: Eluate will build up on the 20μm sieve if tappers are started after eluant is added.

j. After more than half of the initial eluant has passed the 20μm sieve, turn on eluant flow.

k. Pass 1500 $\pm$ 300 mL of eluant through sieve stack. Catch in a beaker. Eluate should be clear at this point; if not, continue until clear.

l. Turn off vibrator.

m. When flow ceases turn off tappers and rotation.

n. Remove sieves and wash down sides with eluant, when necessary, catching washings on the next lower screen.

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p. Remove and record weights of individual sieves.

q. Calculate percent type I HMX retained on each sieve.

4.5.9 **Type I HMX allocation.**

4.5.9.1 **General description.** The calculation (see 4.5.9.7) is accomplished using an algorithm in which a step-wise optimization procedure selects type I HMX batches to meet the required type I HMX weight and particle size distribution for type I HMX blends.

4.5.9.2 **Restrictions for HMX blends.**

a. All of a batch of HMX shall be allocated for a blend.

b. Blends shall be selected essentially chronologically (oldest first) provided required particle distributions are met.

4.5.9.3 **Blender-dryer.** In the blender-dryer all of an allocated batch shall be used in one blend. Blender-dryer allocations shall not exceed 4850 pounds.

4.5.9.4 **V-blender-dryer.** Allocated HMX batches shall be evenly split between V-blender runs. V-blender dryer allocations shall not exceed 6,000 pounds.

4.5.9.5 **Input.** Ten points (see 4.5.8.3q) shall be used to describe the particle size distribution curve for HMX batches (unblended, unground).

4.5.9.6 **Information required from type I HMX blend allocation.**

a. Allocation number.

b. Batch number of each batch in allocation.

c. Weight of each batch in allocation.

d. Total weight of allocation.

e. Particle size distribution of allocation.

4.5.9.7 **Method.**

4.5.9.7.1 **Definition of terms.**

F(X) = Distribution function evaluated at a diameter X. (Evaluated in percent.)

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X_a = Equal to $F^{-1}(a)$. X_{10} represents the diameter (in micrometers) where 10 percent by weight of the particles have a diameter less than or equal to X_{10}.

W_i = Weight of the i'th type I HMX batch.

XL_a = Lower limit of X_a for meeting the required particle size distribution.

XU_a = Upper limit of X_a for meeting the required particle size distribution.

DW = Desired weight.

$$\text{Cost} = \sum_{J=1}^{6} \quad \text{Min } (0, \sum_i C_{ij})$$

4.5.9.7.2 Constraints. Constraints are defined in terms of the a = (10, 50, and 90 percent) particle size distribution probability levels. For blends made from batches i=1,...,m, the distribution function F at a diameter X is obtained from $F(X) = \Sigma F_i\ (X)\ W_i\ / \Sigma W_i$ where F_i is the distribution function of the i'th type I HMX batch, and W_i is the weight of the i'th batch.

a. The constraint functions can be expressed as follows:

$$\frac{\sum F_i\ (XL_{a_j})\ W_i}{\sum_i W_i} \leq a_j \quad \text{and} \quad \frac{\sum_i F_i\ (XU_{a_j})\ W_i}{\sum_i W_i} \geq a_j$$

or

$$\sum_i \left[F_i\ (XL_{a_j}) - a_j\right] W_1 \leq 0 \text{ and } \sum_i \left[F_i\ (XU_{a_j}) - a_j\right] W_i \geq 0$$

b. Assume

$$C_{i,2j-1} = -\left(F_i\ (XL_{a_j}) - a_j\right) W_i$$

$$C_{i,2j} = \left(F_i\ (XU_{a_j}) - a_j\right) W_i$$

then the constraint equation becomes

$$\sum_i C_{ij} \geq 0 \quad \text{for} \quad j = 1,6.$$

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c. Batches are selected one at a time by finding the one which maximizes the cost function,

$$\text{Cost} = \sum_{j=1}^{6} \text{Min}\left(0, \sum_{i} C_{ij}\right),$$

where i ranges over the batches already selected, and the new batch to be tested. When the cost function is zero, the constraint equations are met.

4.5.10 **Type III HMX particle size distribution.** Determine particle size distribution using a Coulter Counter (CC) with Isoton II electrolyte in accordance with the following:

a. Obtain a representative sample of sufficient size to cover the bottom of a 100-mL beaker. Add 40 ± 8 mL of isopropyl alcohol (IPA). Immerse the beaker so that the water level in an ultrasonic bath is just below the level of the IPA. Vibrate the sample and alcohol ultrasonically for one minute ± 5 seconds.

b. Place a 50 micrometer orifice in position. Calibrate with a mono-sized 5 micrometer microsphere standard. Recalibrate before each succeeding 8-hour period of use.

c. With a pipet, take a small sample from the beaker while stirring. Discharge entire sample in the pipet into fresh electrolyte in the CC beaker while stirring and flush pipet.

d. CONCENTRATION INDEX meter should indicate not more than 0.10. If it does indicate more than 0.10, adjust the sample size accordingly.

e. Analyze the sample according to the procedure recommended by Coulter Electronics.

f. Calculate the particle diameters at the 10, 50, and 90 percent cumulative weights by interpolating between the pair of adjacent channels that include the desired percent weight (10, 50, or 90).

g. Record the particle diameters calculated at the 10, 50, and 90 percent cumulative weights.

4.5.10.1 **Retest.** When the particle size distribution of a type III HMX batch does not fall within the ranges given in table III, two independent additional samples shall be taken from the batch. Particle size distribution of these independent samples shall each be determined one time in accordance with 4.5.10.

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4.5.11 **Types II and III HMX moisture content.** Moisture content of the HMX shall be determined in accordance with the following procedures:

a. Weigh a 10 ± 2 g sample to the nearest 0.1 mg.

b. Eject two to five g of the sample into 100 mL of previously neutralized Karl Fischer (KF) carrier solution.

c. Weigh back the sample to the nearest 0.1 mg to determine the weight of the sample added to the carrier solution.

d. After the sample has dissolved or become dispersed, begin titration. Titrate to a 30-second endpoint.

e. Continue titration and allow titrator to complete a second 30-second endpoint. Record the volume of titrant used to the nearest 0.01 mL.

f. Calculate the percent water as follows:

$$\text{Percent water} = \frac{VE}{10W}$$

Where: V = volume of neutralized KF reagent, mL

E = water equivalent of KF reagent, mg/mL

W = weight of sample, g

g. Record the moisture content to the nearest 0.001 percent.

5. PACKAGING

5.1 **Packaging.** Unless otherwise specified in the contract or order (see 6.2), packaging shall be level C as specified herein.

5.1.2 **Level C.** Type I HMX shall be packaged to afford adequate protection against loss, contamination, deterioration, and damage during shipment from the supply source to the first receiving activity and during storage under the shelf life period and conditions specified in 3.11.1. Containers in the same shipment shall be of the same size. The packaging shall conform to UFC 6000, National Motor Freight Classification, 49 CFR 171-178, or to other carrier rules and regulations as applicable to the mode of transportation.

5.2 **Marking.**

5.2.1 **Standard marking.** In addition to any special marking required by the contract or order (see 6.2), interior and exterior containers shall be marked in accordance with MIL-STD-129.

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5.2.2 **Special marking.** In addition to the marking requirements of 5.2.1, external packing shall be marked and labeled in accordance with 49 CFR 171-178 and shall include the following:

a. Title, number, and date of this specification.

b. Name of product.

c. Batch number.

d. Lot number.

e. Net weight (dry basis, alcohol packaged).

f. Date of manufacture.

g. Manufacturer's name and address.

5.3 **Shipping.** The contractor shall ship type I HMX batches as allocated herein, except that one allocated group may be split to fill the shipping conveyance. That part of a group not shipped shall be part of the next shipment.

6. NOTES

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

6.1 **Intended use.** The HMX is intended for use as an ingredient in the Standard Missile MK 104 Dual Thrust Rocket Motor solid propellant.

6.2 **Acquisition requirements.** Acquisition documents 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 2.1.1 and 2.2).

c. Whether a first article inspection is required (see 3.1, 4.3 and 6.3).

d. DOD FAR 52.246-15 (see 3.8, 3.8.1 and 3.9.2).

e. That a MSDS is required (see 3.12.2 and 6.9).

f. Packaging required, if other than as specified in 5.1.

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g. Special marking, if other than as specified in 5.2.2.

h. Safety precautions (see 6.5).

6.3 **First article.** When a first article inspection is required, the contracting officer should provide specific guidance to offerors that the item(s) should be a first article sample, (see 3.1), and the number of items to be tested as specified in 4.3. The contracting officer should also include specific instructions in acquisition documents regarding arrangements for examinations, approval of first article test results, and disposition of first articles. Invitations for bids should provide that the Government reserves the right to waive the requirement for samples for first article inspection to those bidders offering a product which has been previously acquired or tested by the Government, and that bidders offering such products, who wish to rely on such production or test, must furnish evidence with the bid that prior Government approval is presently appropriate for the pending contract. Bidders should not submit alternate bids unless specifically requested to do so in the solicitation.

6.4 **Sampling for acceptance tests.** In the past, for other than particle size distribution, one sample was taken from the first batch and one sample from each fifth batch produced, in series. The batches between consecutively sampled acceptable batches were acceptable. Those between an acceptable and an unacceptable batch were tested for the rejection attribute, individually, forward and backward from the tested unacceptable batch. Batches were acceptable between batches found to be acceptable by attribute test and the preceding or succeeding batch tested.

6.5 **Hazard notice.** The material described herein is flammable or explosive or both. Consequently, it presents 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. The safety precaution requirements of the "Contractor's Safety Manual for Ammunition, Explosives, and Related Dangerous Material" (DOD 4145.26M) are applicable.

NOTE: When this specification is used as part of the description of work to be accomplished by a Government activity, the safety precaution requirements of "Ammunition and Explosives Ashore" (OP 5) are applicable.

6.6 **Definitions.**

6.6.1 **Blend.** A blend shall consist of two or more batches which have been combined to give a homogeneous particle distribution.

6.6.2 **80-mesh tensile bolting cloth.** Stainless steel wire mesh with nominal 224 micrometer openings.

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6.7 Recommended reagent. Triton X-100, supplied by J. T. Baker Chemical Co., Philipsburg, NJ, 08865, is a reagent recommended for use in determining type I HMX particle size distribution.

6.8 Possible material. The following material has been found to meet the requirements of this specification. This is only given for information and is not restrictive.

HMX, Grade B, 80-screen - manufactured by Holston Defense Corporation, Kingsport, TN.

6.9 Material Safety Data Sheets. Contracting officers will identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

6.10 Subject term (key word) listing.

Propellant
Rocket Motor, Dual Thrust, Mk 104
Standard Missile

6.11 Supersedure information. MIL-C-82783 incorporates the following engineering change proposal (ECP), notice of revision (NOR) and specification change notice (SCN):

ECP	NOR	SCN
MTA078 (2/7/89)	MTA078.1 (12/23/88)	SCN 1 (5/8/89)

Preparing Activity:
NAVY-OS
(Project 1376-N362)

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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 document(s) or to amend contractual requirements.

I RECOMMEND A CHANGE:	1. DOCUMENT NUMBER MIL-C-82783	2. DOCUMENT DATE (YYMMDD) 5 February 1990

3. DOCUMENT TITLE
CYCLOTETRAMETHYLENETETRANITRAMINE (HMX)

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 NAVAL ORDNANCE STATION (CODE 3730) INDIAN HEAD, MD 20640-5000

a. NAME	b. TELEPHONE (Include Area Code) (1) Commercial (2) AUTOVON
c. ADDRESS (Include Zip Code)	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