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27 February 1974
SUPERSEDING
MIL-H-45444A (MU)
24 April 1961

# MILITARY SPECIFICATION

## HMX

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

1. SCOPE

1.1 This specification covers HMX for use in ammunition

1.2 Classification. HMX shall be of the following grades (see 6.7) as specified.

Grade A - 93 percent minimum purity
Grade B - 98 percent minimum purity

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

PPP-B-26 - Bag, Plastic, Polyethylene
RR-S-366 - Sieves; Standard, for Testing Purposes
CCC-C-461 - Cloth, Twill, Uniform Cotton

STANDARDS

MILITARY

MIL-STD-105 -Sampling Procedures and Tables for Inspection by Attributes (ABC-STD-105)
MIL-STD-109 -Quality Assurance Terms and Definitions
MIL-STD-129 -Marking for Shipment and Storage
MIL-STD-1168 -Lot Numbering of Ammunition
MIL-STD-1235 -Single and Multilevel Continuous Sampling Procedures and Tables for Inspection by Attributes

FSC: 1376

3.3.2 When HE ... heat treated as specified ...

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer).

2.2 Other publications. The following document forms a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitations for bids shall apply.

CODE OF FEDERAL REGULATIONS

Title 49 Transportation, CFR 49 Part 100-199

(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 Officer, Washington,D.C. 20402. Orders for the above publications should cite, "49 CFR 100-199 (latest revision)")

3. REQUIREMENTS

3.1 HMX shall consist of cyclotetramethylenetetranitramine (HMX), in the beta form only, when tested as specified in 4.3.1 Grade A shall consist of 93% HMX minimum and Grade B shall consist of 98% HMX min.

3.2 RDX content. Grade A shall consist of 7% RDX maximum and Grade B shall consist of 2% max. RDX when tested as specified in 4.3.1.

3.3 Melting point. The melting point of the HMX shall be a minimum (min) of 277 degrees Centigrade (°C), when tested as specified in 4.3.2.

3.4 Total acetone insoluble material. The total acetone insoluble material shall be 0.05 percent maximum (max.) when tested as specified in 4.3.3.

3.5 Inorganic insoluble material. The inorganic insoluble material shall be 0.03 percent, max., when tested as specified in 4.3.4.

3.6 Insoluble particles. There shall be no insoluble particles retained on a U.S. Standard Number (No.) 40 sieve and not more than 5 insoluble particles retained on a U.S. Standard No.60 sieve, when tested as specified in 4.3.5.

3.7 Acidity. The acidity, as acetic acid, shall be 0.02 percent, max., when tested as specified in 4.3.6.

3.8 Granulation.The HMX shall be in accordance with the granulation requirements as specified in Table I when tested as specified in 4.3.7.

TABLE I (see 6.7)

| Through U.S. Standard Sieve No. | Class 1 Percent | Class 2 Percent | Class 3 Percent | Class 4 Percent | Class 5 Percent | Class 6 Percent |
|---|---|---|---|---|---|---|
| 8 | | | | 100 | | |
| 12 | | | 90 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 |

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection.Unless otherwise specified in the contractor or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order the supplier 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 assure supplies and services conform to prescribed requirements. Reference shall be made to MIL-STD-109 to define terms used herein.

4.1.1 Submission of product. At the time each completed lot of items deliverable under the contractor 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 6.1c):

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 subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.

4.2 Inspection provisions

4.2.1 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. The lot shall be submitted for inspection in accordance with MIL-STD-105 (or MIL-STD-1235 when applicable). The criteria and procedures for the assignment of lot numbers shall be in accordance with MIL-STD-1168.

4.2.2 Examination. Sampling plans and procedures for the following classifications of defects shall be in accordance with MIL-STD-105, except that inspection for critical defects shall be 100 percent. Contractor's

sampling plans, if used, shall be approved by the Government and shall provide, as a minimum, the protection afforded the Government by the sampling plans in MIL-STD-105. Continuous sampling plans in accordance with MIL-STD-1235 may be used if approved by the procuring activity. Also, at the option of the procuring activity, AQL's and sampling plans may be applied to the individual characteristics listed, using an AQL of 0.40 percent for each major defect and an AQL of 0.65 percent for each minor defect, except where 100 percent inspection is specified.

4.2.2.1 Bag, cloth (see 5.1)

| Categories | Defects | Method of Inspection | Code No. |
|---|---|---|---|
| Critical: | None defined | | |
| Major: | AQL 0.40 percent | | |
| 101. | Foreign matter | Visual | 01001 |
| 102. | Bag pierced or torn | Visual | 01002 |
| Minor: | None defined. | | |

4.2.2.2 Polyethylene bag (see 5.1.1.1)

| Categories | Defects | Method of Inspection | Code No. |
|---|---|---|---|
| Critical: | None defined | | |
| Major: | AQL 0.40 percent | | |
| 101. | Seam splits when manual pressure is applied along entire length of seam | Visual/ Manual | 04001 |
| 102. | Bag damaged | Visual | 04002 |
| Minor: | None defined | | |

4.2.2.3 Sealed rubber bag or suitable water tight material (see 5.1)

| Categories | Defects | Method of Inspection | Code No. |
|---|---|---|---|
| Critical: | None defined. | | |

| | | | |
|---|---|---|---|
| Major: | AQL 0.65 percent | | |
| 101. | Bag improperly closed | Visual/Manual | 02001 |
| 102. | Gross weight, max. | Balance | 02002 |
| 103. | Bag damaged | Visual | 02003 |
| 104. | Solution missing, insufficient or leaking out | Visual | 02004 |

Minor: None defined

4.2.2.4 Sealed drum (see 5.2)

| Categories | Defects | Method of Inspection | Code No. |
|---|---|---|---|
| Critical: | None defined | | |
| Major: | AQL 0.40 percent | | |
| 101. | Sealing improper | Visual/Manual | 03001 |
| 102. | Drum damaged | Visual | 03002 |
| Minor: | AQL 0.65 percent | | |
| 201. | Marking missing, incorrect or illegible | Visual | 03003 |

4.2.3 Sampling. A representative sample of approximately 300 grams of HMX shall be selected from each batch for testing. The inspection of the samples shall be in accordance with MIL-STD-1235 CSP-1 Plan, Inspection Level II, AQL 4.0%. 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 4.3. 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. The classification and code number shall be as given in Table I.

| | | |
|---|---|---|
| HMX analysis (see 3.1) | Major Defect | Code No. 05001 |
| RDX content (see 3.2) | Major Defect | Code No. 06001 |
| Melting point (see 3.3) | Major Defect | Code No. 07001 |
| Total acetone insoluble material (see 3.4) | Major Defect | Code No. 08001 |
| Inorganic insoluble material (see 3.5) | Major Defect | Code No. 09001 |
| Insoluble particles (see 3.6) | Major Defect | Code No. 10001 |
| Acidity (see 3.7) | Major Defect | Code No. 11001 |
| Granulation (see 3.8) | Major Defect | Code No. 12001 |

4.3 Test methods and procedures

4.3.1 Purity Analysis of HMX

4.3.1.1 Determination of Alpha HMX, Beta HMX and RDX Contents by X-ray diffraction

4.3.1.1.1 Introduction. The X-ray diffraction patterns for alpha HMX, RDX and beta HMX indicate characteristic diffractions at 17.710 degrees and 25.10 degrees two theta, respectively, for RDX and alpha HMX in the presence of beta HMX. 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.

4.3.1.1.2 Apparatus. A Philips Electronic Instrument X-ray diffractometer (or equivalent) equipped with voltage and current stabilizer, scintillation detector and copper target tube.(1)

4.3.1.1.2.1 General optimum instrumental conditions. The tube should be capable of an excitation of 35 kv and a filament current of 20 ma. A pulse height analyzer capable of passing copper $K_\alpha$ radiation should be used. A nickel filter may be used to remove copper $K_\beta$ radiation. Tube voltage and filament current can be selected by analyzing five of the calibration standards at two different settings.

4.3.1.1.3 Preparation of calibration curves

4.3.1.1.3.1 Purification of Beta HMX, Alpha HMX and RDX

4.3.1.1.3.1.1 Beta HMX. Obtain a sample (approximately 1/2 pound) of a production batch of high purity HMX. Place the sample in a beaker and add four parts (by weight) of a buffer solution and heat at 90°C for two hours. (Buffer solution: 6.0 ml glacial acetic acid and 13.6 gm

(1) The X-ray equipment shall be operated in accordance with the manufacturer's instructions.

sodium acetate diluted to one liter (pH=4.6)). Filter the slurry using a medium porosity crucible. Dry the HMX at 100°C for two hours. Wash the dry HMX with 200 ml of 1,2 - dichlorethane (EDC) to remove residual RDX. Then, wash with 50% aqueous acetone solution. Dry at 100°C for two hours.

4.3.1.1.3.1.2 Alpha HMX. 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°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°C for two hours.

4.3.1.1.3.1.3 RDX. Obtain a 100gm sample from a production batch of high purity RDX. Heat the sample with four parts, (by weight) of buffer solution at 90°C for two hours. The buffer solution is prepared as described in 4.3.1.1.3.1.1. Filter the slurry using a medium porosity crucible. Dry the RDX at 100°C for two hours. Heat 1 part by weight, RDX and 1 1/2 parts by volume, dimethyl sulfoxide (DMSO) to 92-96°C. In addition, add up to 1 part DMSO to sufficiently dissolve all the RDX. Digest at 92-96°C for 30 minutes. Add distilled water until solution becomes cloudy. Reheat until the solution clears, then cool rapidly to room temperature and filter. Wash and dry a small sample of the precipitate for purity analysis by the EDC procedure as described in paragraph 4.3.1.1.3.1.3.1. Repeat the above procedure until a very pure product is indicated. Wash with 50% aqueous acetone. Dry at 100°C for 2 hours.

4.3.1.1.3.1.3.1 Purity analysis of RDX

4.3.1.1.3.1.3.1.1. Special reagent. The special reagent shall be as follows: 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°C) is 0.02 gm per 100 ml.

4.3.1.1.3.1.3.1.2 Procedure. The determination of the RDX content shall be conducted as follows: Use a portion of the sample prepared for analysis as specified in paragraph 4.3.1.1.3.1.3. Weigh 0.2 gm of the sample accurately into a 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 dichloroethane. 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:

$$\frac{Wg - Wr \times 100}{Wg}$$

Where:

Wg - is original sample weight, gm
Wr - is insoluble residue weight, gm

4.3.1.1.3.2 Preparation of standard mixtures. The standard mixtures must be thoroughly mixed. Mix the samples in a wrist action shaker for a minimum of three hours. In 250 ml Erlenmeyer flasks, mix beta HMX, alpha HMX, and RDX (use purified material which passes USSS No. 260) as needed to prepare 5 gm samples of the following composition:

| 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 |

4.3.1.1.3.3 Intensity measurements. Measure the angular intensities at 16.90, 17.81, 24.10 and 25.10 degrees two theta as outlined in 4.3.1.1.4 and 4.3.1.1.5.

4.3.1.1.3.4 Construction of calibration curves. On separate sheets of graph paper, construct calibration curves for the following:

RDX - Plot the corrected intensity (cps) at 17.810 degrees two theta vs RDX concentration, weight percent. (ex. Curve I).

Alpha HMX - Plot the corrected intensity (cps) at 25.10 degrees two theta vs alpha HMX concentration, weight percent. (ex. Curve II).

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. (ex. Curve III).

4.3.1.1.4 Sample preparation

4.3.1.1.4.1 Reduce samples particle size to less than 62 micron. HMX particle size is sufficiently small enough to permit analysis as received. It may 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 gm portions in a small mortar. A sample size of approximately 0.4 gm is required to properly fill the cavity type sample holder.

4.3.1.1.4.2 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.

4.3.1.1.5 Analysis of sample

4.3.1.1.5.1 Intensity measurements. 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 16.900 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 17.810 degrees two theta. Repeat above. Reset goniometer to 24.100 degree two theta. Repeat above. Reset goniometer to 25.100 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.

4.3.1.1.5.2 Interpretation of data. Record the counts (times $10^{-1}$) that are accumulated in 100 seconds, that are printed out under the respective two theta angle. Place the decimal point one place to the left in the printout to obtain the representative counts per second (cps). Subtract cps at 16.900 degrees two theta from cps at 17.810 degrees two theta. This is the intensity (cps) due to the RDX percent. Opposite the cps obtained, read the RDX concentration from curve 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 curve 3 the correction required by reading the cps opposite the RDX percentage. Subtract this correction from the cps. From curve 2, read percentage alpha HMX opposite the intensity, (corrected or uncorrected).

4.3.1.2 Alternate method, RDX analysis by the EDC method

4.3.1.2.1 Special reagent. The special reagent shall be prepared as stated in 4.3.1.1.3.1.3.1.1.

4.3.1.2.2 Procedure. The determination of the RDX content shall be conducted as follows: Weigh a 0.2 gm dried sample accurately into a 125 ml glass stoppered conical flask. Then follow the procedure as stated in 4.3.1.1.3.1.3.1.2.

4.3.2 Melting point

4.3.2.1 Apparatus. The Fischer-Johns hot state melting point apparatus shall be used for this determination.

4.3.2.2 Procedure. The instrument shall be plugged into the proper electrical outlet and the toggle switch shall be pushed to the "on" position allowing power to flow to both the illuminating lamp and the powerstat. The magnifier shall be adjusted to give a clear view of the stage. A portion of the sample shall be ground in a small agate mortar and a very small quantity (approximately 0.05 gm) of the finely pulverized sample placed between two clean 18MM 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°C of the melting point. Thereafter a heating rate of approximately 1°C per minute shall be used. When the sample begins to melt, the thermometer shall be read and the temperature reading be recorded as the melting point.

4.3.3 Determination of acetone insoluble material. An accurately weighed dry specimen of approximately 10 grams (gm) of HMX shall be placed in a 600 milliliter (ml) beaker and 400 ml of acetone shall be added. The beaker shall be covered with a watch glass and heated on a steam bath until the HMX is dissolved. The acetone solution shall be filtered through a tared filtering crucible, which was prepared by washing the filter with acetone, igniting and weighing. Care shall be taken to transfer all the insoluble matter to the crucible. The residue shall be washed three times with acetone and



be 60 ; the thickness of the chisel 1/8 inch shall be cause for rejection

aspirated until the odor of acetone is not longer discernible. The crucible shall be dried for 30 minutes in an oven at 105 ± 5°C, cooled in a desiccator and weighed. The increase in weight shall be calculated as percent acetone insoluble material in the dry specimen.

4.3.4 Determination of inorganic insoluble material. The material in the crucible, obtained as directed in 4.3.3, shall be ignited, cooled in a desiccator and reweighed. The increase in weight over the original tare weight shall be calculated as percent inorganic insoluble material in the dry specimen.

4.3.5 Determination of insoluble particles. An accurately weighed specimen of approximately 50 gm of HMX shall be transferred to a thimble of soxhlet extractor and placed in the soxhlet apparatus or other suitable extractor. Sufficient acetone shall be added to the flask and the specimen shall be extracted on a steam bath until all HMX is dissolved. All the remaining insoluble material in the thimble shall be transferred to a U.S. Standard No. 60 sieve complying with Specification RR-S-366. The particles shall be counted and examined. Particles shall be brushed on a U.S. Standard No. 40 sieve, and any that are retained shall be counted and examined.

4.3.6 Determination of acidity. An accurately weighed specimen of approximately 10 gm of HMX shall be transferred to a 800 ml beaker, 500 ml of acetone shall be added, and the beaker and contents shall be heated on a steam bath until the HMX is 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:

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

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

4.3.7 Determination of granulation. The granulation shall be determined by the following procedure.

4.3.7.1 Preparation of sample for analysis. Place a dried sample (about 55 gm) on a piece of glazed weighing paper, 8 1/2 by 11 inches, and thoroughly mix by rolling (roll a minimum of 20 times). Quarter the sample by standard techniques and take a 2 gm sample, weighed to the nearest milligram. Repeat this procedure for additional 2 gm samples. A grounded metal sample splitter (riffler) may be used to obtain the representative samples.

4.3.7.2 Preparation of wash solution. A wash solution of ethanol saturated with HMX (at room temperature) shall be made.

4.3.7.3 Apparatus. The mechanical sieve washer shall be set up as shown in figures 1, 2, 3 and 4.

4.3.7.3.1 Operation of mechanical sieve washer and pressure tank. The pressure tank shall be filled with wash solution through the filling funnel.The valve of the tank shall be closed after the solution has been added. A controlled air supply, adjusted to 1.5 pounds per square inch (psi) is used to pressurize the tank. The pressure in the tank shall not exceed 1.5 psi. The tank shall be at least two thirds full before starting an analysis. The petcock at the base of the pressure tank is opened, which permits wash liquid to flow through the spray head. The flow of wash liquid through the spray head shall be adjusted to a rate of 150 ml per minute. The sieves shall be rotated at approximately 20 revolutions per minute by regulating the air supply to the motor via the air motor pressure regulating valve.

4.3.7.4 Procedure. Tare each sieve, which has been previously washed in water and acetone and dried, to the nearest milligram on a analytical balance. Arrange nest with sieve in diminishing sieve sizes, and attach nest to rotating sieve holder. Adjust the lower spray head to within approximately one inch of the top sieve surface. Wet the top sieve in the wash liquid from a wash bottle and transfer a 2 gm sample to the sieve. Wash each sieve successively from the coarsest to the finest for two minutes each, taking care to wash down the sides of each sieve with a wash bottle. Check sieves while sample is being washed for indication of liquid back up. Release back-up by lifting screens and breaking liquid seal between sieves. Air dry the sieves until odor of alcohol is no longer discernible. Then dry the sieves containing HMX in an oven for 30 minutes at 50°C. Cool and weigh to nearest milligram; record weight retained on each sieve. Weights of sub-sieve material is calculated by difference. If desired, subsieve particles may be collected on a fritted crucible for analysis on some type of classifier adapted to this size material. The percent retained in each size range starting with subsieve size material, is divided by weight of sample and multiplied by 100 to give percentage in each size range.

4.3.7.5 Alternate method

4.3.7.5.1 Procedure. An accurately weighed specimen of approximately 50 gm (dry weight) 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 6.2). With the aid of a rubber policeman attached to a glass stirring rod, the mixture shall be stirred for a few minutes, wetting the specimen thoroughly and breaking up as many of the aggregates as possible. A spray nozzle (see 6.3) 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. The sieves shall be nested in

order of decreasing size, with the largest mesh being placed on top. This assembly should be set up near a water tap and drain. Provision should 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 specimen at an angle approximately perpendicular to the screen from a height of 2 to 3 inches, it is possible to wash the specimen 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 transverse the diameter of the screen one to two times per second. The wet agglomerates shall be gently crushed on the top sieve with the side of a rubber policeman. 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 sieves 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 which shall then be washed with the water spray as before for five 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 portion remaining on each of the screen shall be transferred to separate 400 ml beakers as follows: The screen shall be held in an almost vertical postion 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 beaker with a stream of water from a wash bottle. The explosive in each of the beakers shall be quantitatively transferred to separate previously tared fritted glass filtering crucibles of medium porosity. The crucibles shall be aspirated during the transfer process and the crucibles and contents shall be aspirated for approximately two 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 explosives for approximately five 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°C for 15 minutes, cooled in a desiccator and weighed. The weight of material retained on each sieve shall be determined and the percentage passing through each sieve calculated on the basis of the dry weight of the HMX specimen.

## 5. PREPARATION FOR DELIVERY

### 5.1 Preservation and packaging

5.1.1 Level A. Unless otherwise specified by the procuring activity, the HMX shall be thoroughly mixed to form a slurry or cake containing not less than 10 percent by weight of a solution made as follows: 40 percent by weight of isopropyl alcohol and 60 percent water. Not more than 50 pounds, dry weight, of the wet HMX shall be packed in a cloth bag described in 5.1.1.2. Not more than six bags shall be placed in a rubber bag, rubberized cloth bag or two polyethylene bags, described in 5.1.1.1, placed one inside the other. Inner and outer bags shall be securely tied with the polyethylene bags individually tied, using non-metallic tape or cord. The tops of the outer bags shall be gathered and formed into a gooseneck when being tied.

5.1.1.1 The polyethylene bags shall comply with Type II, Style 1 of Specification PPP-B-26 except that closure will not be heat sealed. The bag size shall be large enough to prevent strain on the bag when it is placed in the drum and filled; the length shall be sufficient to allow gathering of the top and folding into a gooseneck when tying. The bag seams shall meet the seam strength test with an AQL of 0.65 percent. Seams shall also be examined by separating the bag faces

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.

5.1.1.2 Cloth bag. The bags for 50 pounds of HMX shall be made from cotton twill conforming to Specification CCC-C-461, Type VI. The cloth shall be white and free from size or brighteners. 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 cotton duck weighing not less than 12 oz per square yard may be used for the bag.

5.2 Packing

5.2.1 Level A. The large bag containing a maximum of 300 pounds dry weight of crystalline HMX shall be placed in a drum complying with Department of Transportation specification 5 or 5B of the Code of Federal Regulations 49 CFR 100-199.

5.2.2 Level C.Packing requirements shall be the same as Level A except that a Department of Transportation Specification 21C fiber drum may be used.

5.3 Marking. Containers shall be marked as required by Code of Federal Regulations 49 CFR 100-199. In addition, shipments shall be marked in accordance with Standard MIL-STD-129. Each container shall be clearly labelled with the lot number and net weight of its contents. When a container holds HMX from more than one lot, all lot numbers shall be shown on the label and each of the small bags inside the container shall be clearly and permanently labelled with the lot number of its contents.

6. NOTES

6.1 Ordering data. Procurement documents should specify the following:

a. Title, number and date of the specification.
b. Grade and class required.
c. Acceptance and description sheets - These sheets shall be prepared for each lot in accordance with MIL-STD-1171.

6.2 Dioctyl sodium sulfosuccinate is commercially available as Aerosol OT.

6.3 A spray nozzle can be made by fitting a Gooch crucible (Number 4, 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.

6.4 Hexachlorobutadiene and tetrachloroethylene are obtainable from Eastman Kodak Company.

6.5 Water tight material. The following materials have been found satisfactory. Scotchpak 45A x 48, .0045 inch thick or Scotchpak Brand Barrier Material 140A22, .0140 inch thick.

6.6 Determination of Alpha HMX and RDX in the Presence of Beta HMX by X-ray diffraction, Holston Defense Corporation, Kingsport, Tennessee, Control No. 20-P-28 Series A, November 1965, Russell A. Jackson.

6.7 In accordance with the designation of classifications, paragraph 4-222 of Defense Standardization Manual 4120.3-M, the following designation change is implemented in this specification: "Grades I and II" and "Class a,b,c,d.e and f" as specified in MIL-H-45444A (Ord) are changed to "Grades A and B" and "Classes 1,2,3,4,5 and 6" respectively.

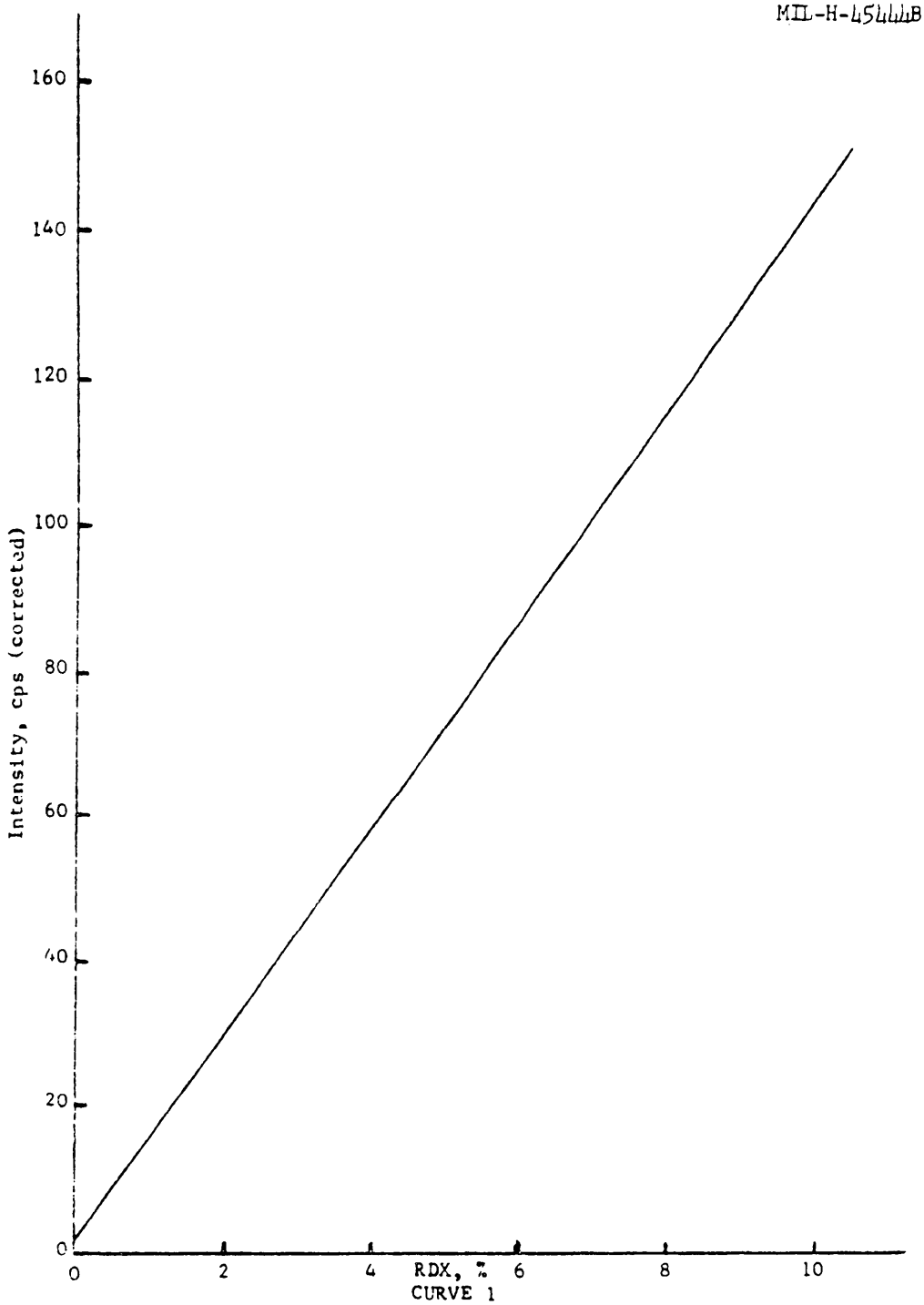
6.8 Gamma HMX. Gamma HMX is prepared by dissolving 1.0 gm of HMX in 280 ml of 50 percent acetic acid, in a 500 ml Erlenmeyer flask, by heating over a grounded hot plate. Any polymorph of HMX may be used. If beta HMX is used, it should be ground so that it will dissolve rapidly.

When solution is complete, cool the flask in an ice water bath with continuous swirling. The temperature should reach 20°C in three to four minutes. Filter with as little delay as possible and dry the crystals. The habit of gamma HMX prepared by the above procedure is plate like and the sensitivity will be about 20 centimeters.

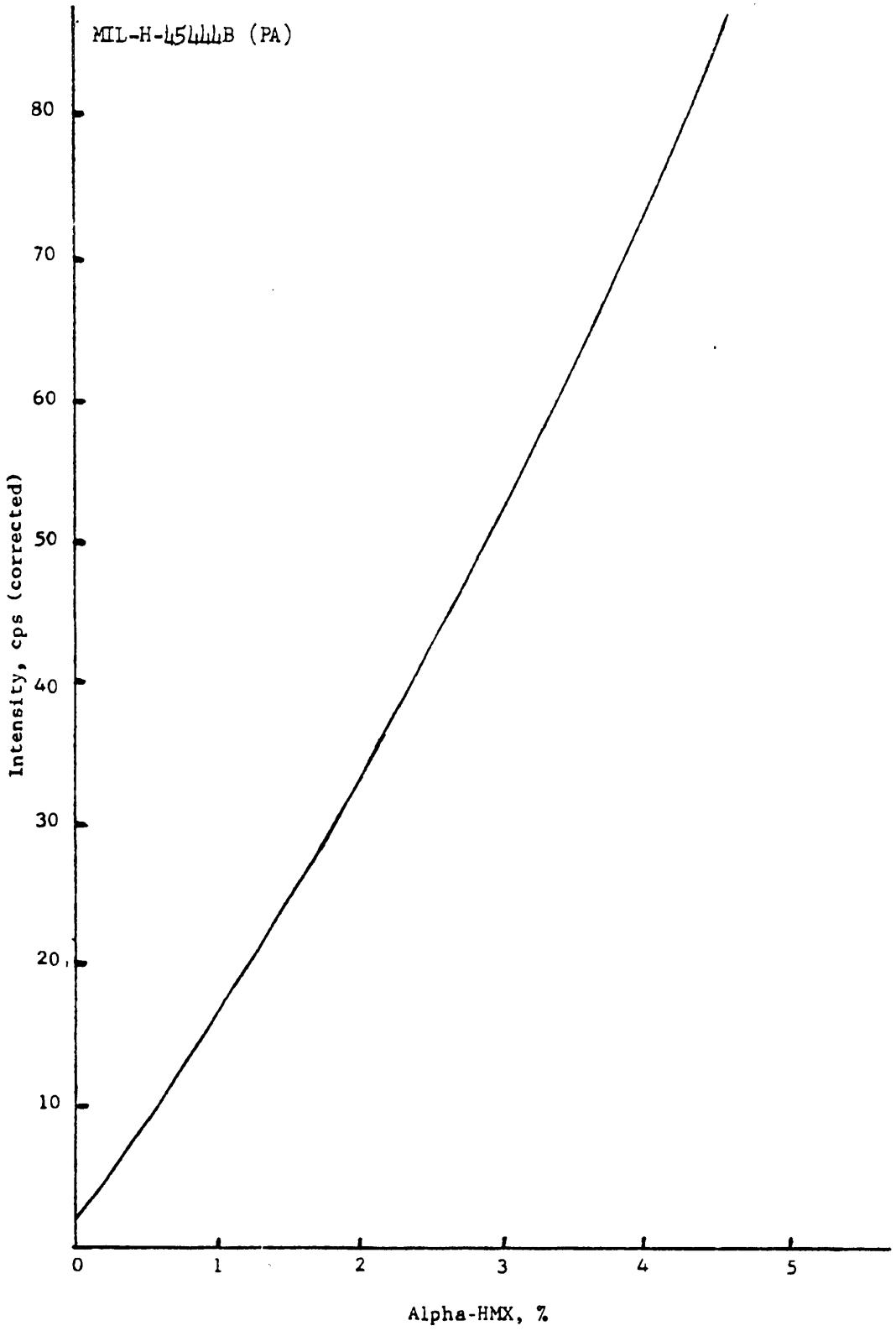
Custodian:
Army-PA

Preparing Activity
Army-PA

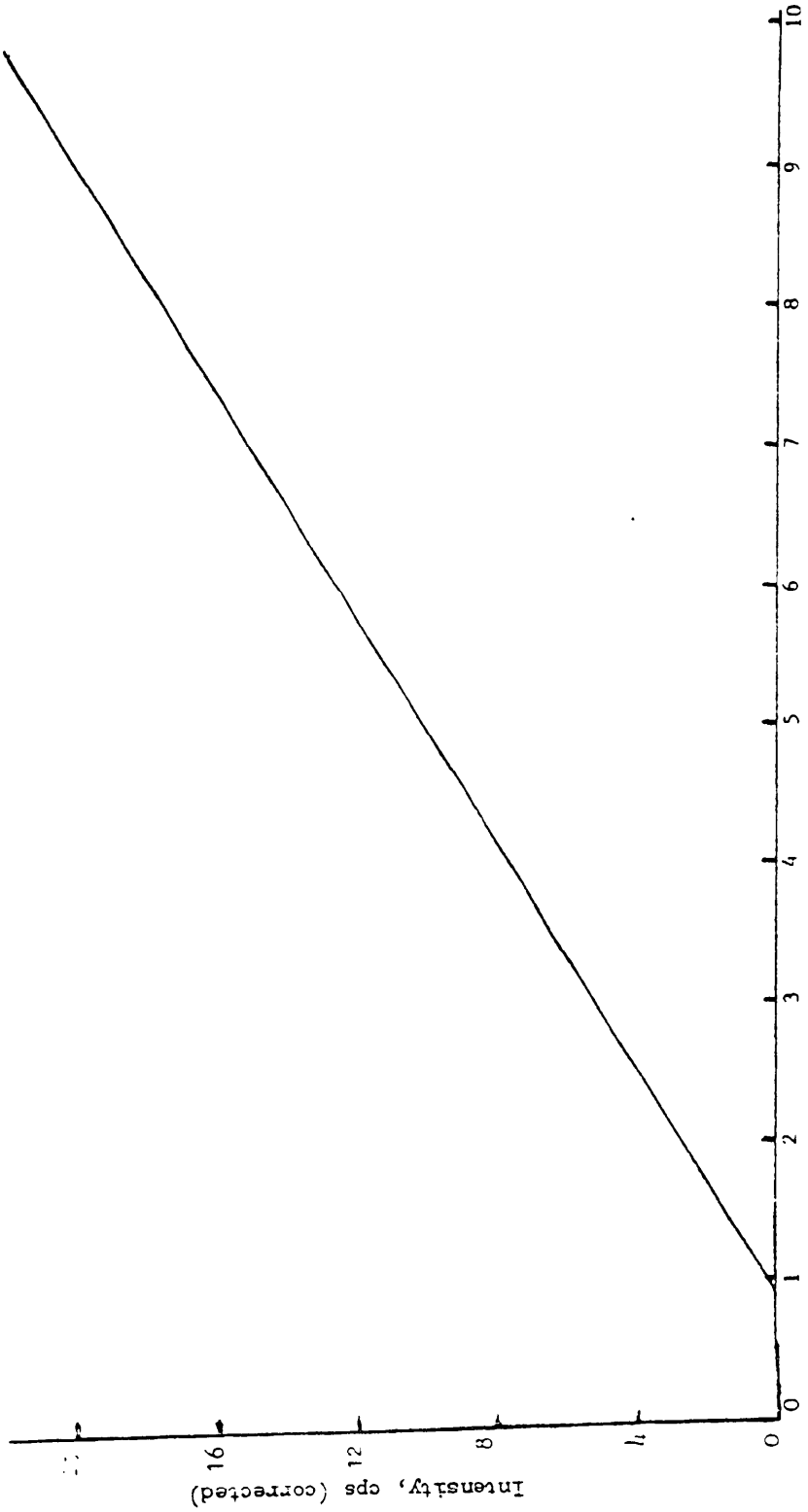
Project Number: 1376-A-020




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

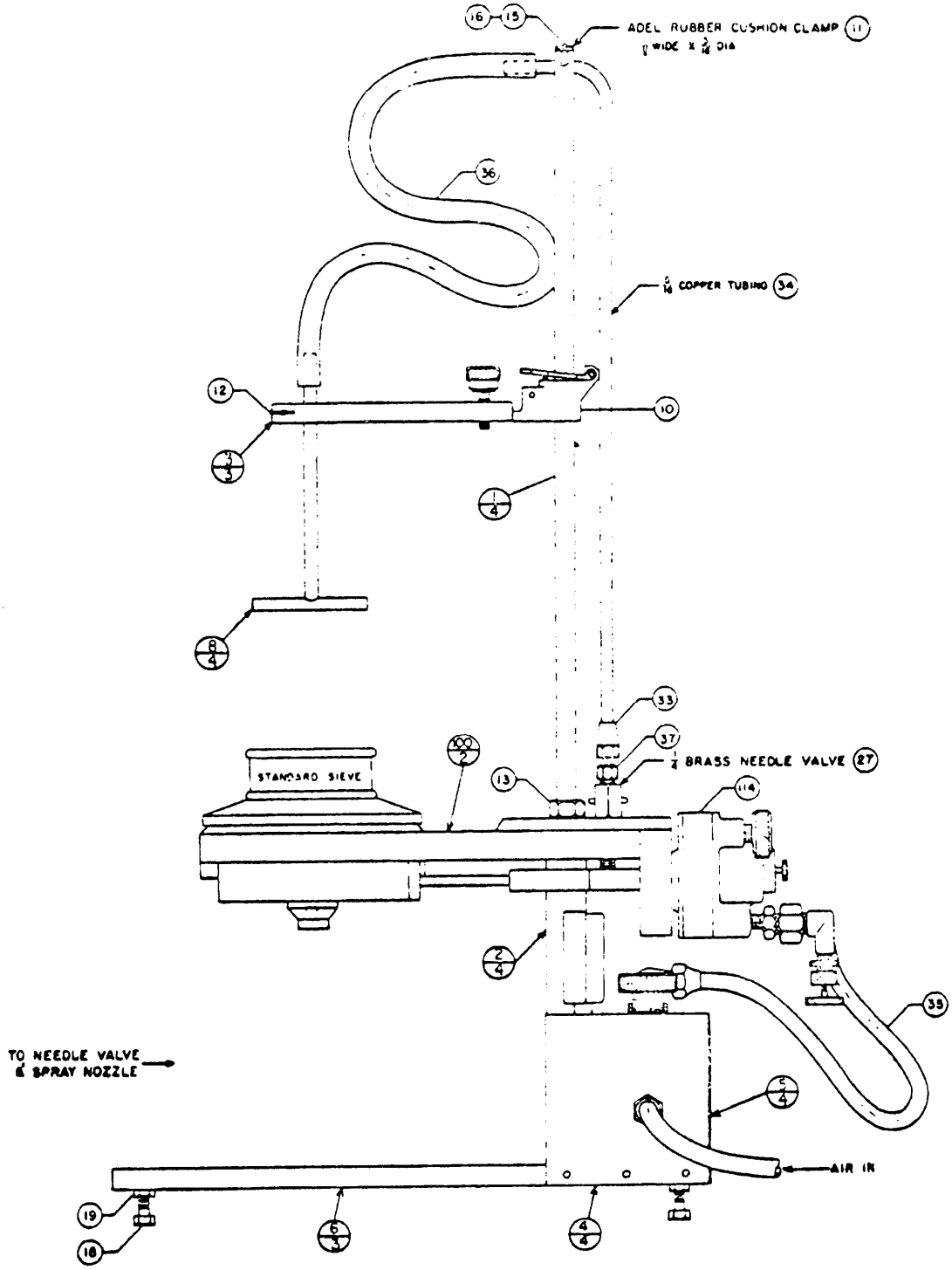



CURVE II
CALIBRATION CURVE FOR THE DETERMINATION OF ALPHA HMX IN PRESENCE OF BETA HMX



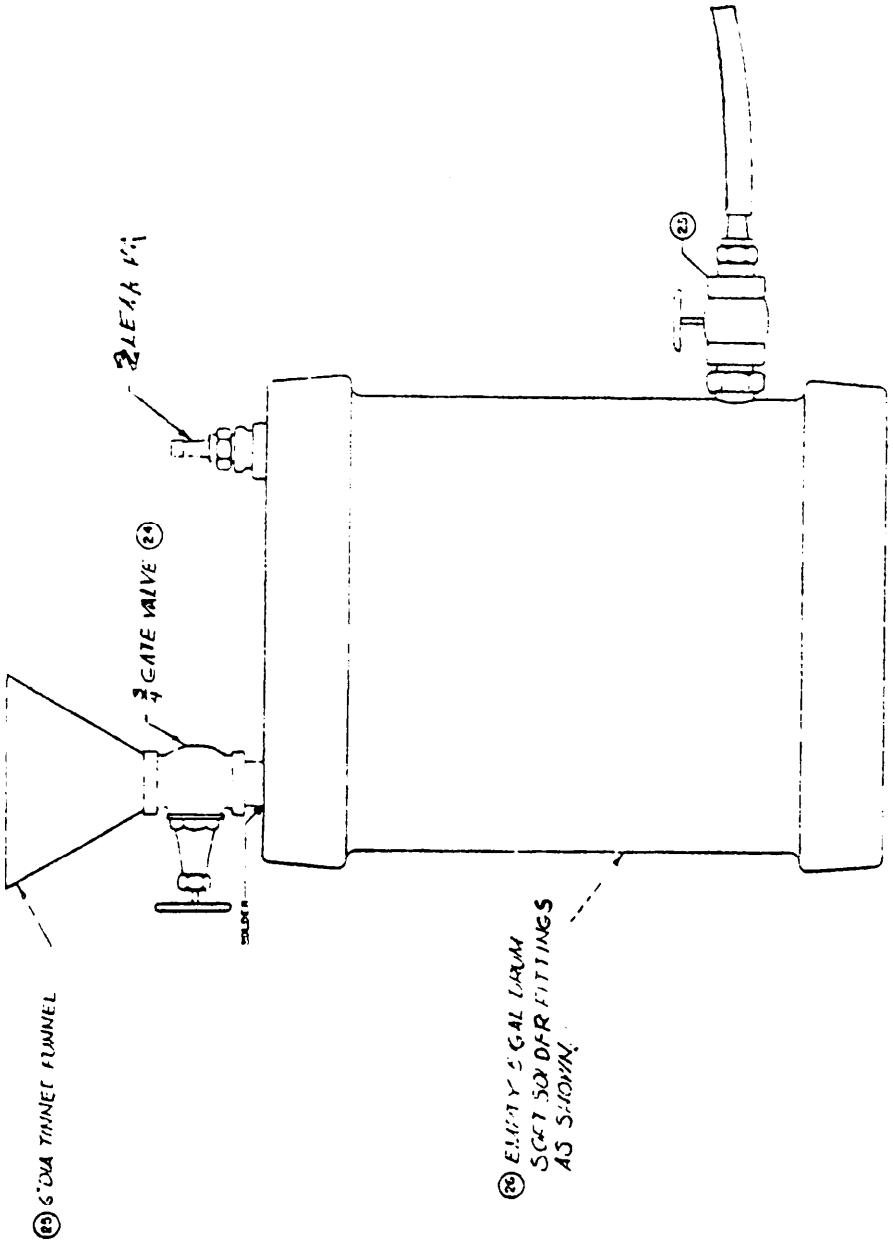

CURVE 111

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



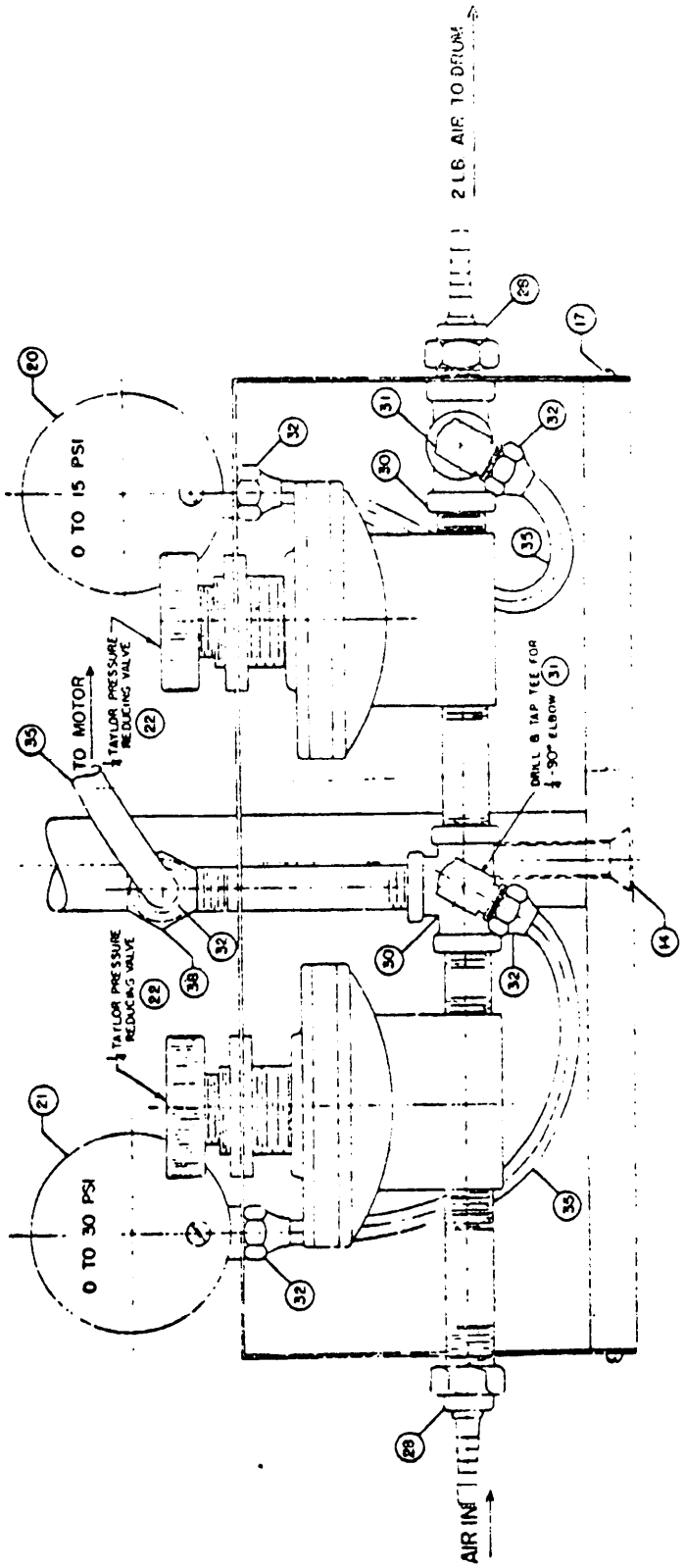

MECHANICAL SIEVE WASHER

FIGURE 1




STORAGE TANK

FIGURE 2




GAGE & VALVE ASSY REAR VIEW
FIGURE 3

BILL OF MATERIAL

| PART NUMBER | NUMBER REQD. | MTR | DESCRIPTION |
|---|---|---|---|
| 114 | 1 | | DEVILBISS AIR MOTOR |
| 10 | 1 | | FISCHER LEVELING BULB SUPPORT |
| 11 | 1 | | $\frac{5}{16}$ ADEL RUBBER CUSHION CLAMP |
| 12 | 1 | BRASS | THUMB SCREW - #10-32 - 1" LONG |
| 13 | 1 | SST | HEX. NUT - $\frac{1}{2}$ - 13NC |
| 14 | 1 | SST | MACH SCREW, FLAT HD. - $\frac{3}{8}$ - 16 - $1\frac{1}{2}$ LONG |
| 15 | 1 | SST | MACH. SCREW, FIL. HD - #5-40 - $\frac{3}{4}$ LONG |
| 16 | 1 | SST | WASHER, FLAT - #6 |
| 17 | 12 | BRASS | MACH SCREW, FIL. HD - #4-40 - $\frac{1}{16}$ LONG |
| 18 | 4 | BRASS | CAP SCREW, HEX HD - $\frac{1}{4}$ - 20 - 1 LONG |
| 19 | 4 | BRASS | HEX NUT - $\frac{1}{4}$ - 20 |
| 20 | 1 | 2" DIAL | PRESSURE GUAGE - $\frac{1}{4}$ - 0 TO 15 PSI |
| 21 | 1 | 2" DIAL | PRESSURE GUAGE - $\frac{1}{4}$ - 0 TO 30 PSI |
| 22 | 2 | | TAYLOR PRESS RED. VALVE - $\frac{1}{4}$ |
| 23 | 1 | BRASS | COCK - HOSE - $\frac{3}{8}$ |
| 24 | 1 | BRZ | GATE VALVE - $\frac{3}{4}$ |
| 25 | 1 | TINNED | FUNNEL - 6" DIA |
| 26 | 1 | | DRUM - 5 GAL |
| 27 | 1 | BRASS | NEEDLE VALVE - $\frac{1}{4}$ |
| 103 | 1 | BRZ | BOSTON WORM GEAR - # G-1041 |
| 115 | 1 | STEEL | BOSTON WORM - # HLUH |
| 116 | 1 | STEEL | NEW DEPARTURE BB # 4773L |
| 117 | 2 | STEEL | NICE BALL BEARING - # PR-2 |
| 118 | 1 | RUBBER | HOSE - $\frac{1}{4}$ x $2\frac{1}{4}$ - FOR COUPLING |
| 119 | 4 | BRASS | MACH SCREW, FIL HD - #6-32 - $1\frac{1}{2}$ LONG |
| 120 | 4 | BRASS | MACH SCREW, FIL. HD - #6-32 - $\frac{3}{8}$ LONG |
| 121 | 3 | BRASS | MACH. SCREW, FIL. HD - #6-32 - $\frac{1}{2}$ LONG |
| 122 | 1 | BRASS | MACH SCREW, FIL HD - $\frac{1}{4}$ - 20 - 2 LONG |
| 123 | 2 | BRASS | MACH SCREW, FIL HD - $\frac{1}{4}$ - 20 - $\frac{3}{4}$ LONG |
| 124 | 4 | BRASS | MACH SCREW, FIL HD - #4 - 40 - $\frac{3}{8}$ LONG |
| 125 | 1 | STEEL | GROOVED PIN - #0000 |
| 28 | 2 | BRASS | HOSE CONNECTION, |
| 29 | 6 | BRASS | NIPPLE, $\frac{1}{4}$, SUITABLE |
| 30 | 2 | BRASS | TEE, PIPE, $\frac{1}{4}$, |
| 31 | 2 | BRASS | ELBOW, FLARED, 90°, $\frac{1}{4}$ MALE FLARE TO MALE PIPE |
| 32 | 6 | BRASS | NUT, FLARED $\frac{1}{4}$ |
| 33 | 1 | BRASS | NUT, FLARED, $\frac{3}{16}$ |
| 34 | 2' | COPPER | TUBING, $\frac{3}{16}$ DIA |
| 35 | 3' | COPPER | TUBING, $\frac{1}{4}$ DIA |
| 36 | | RUBBER | TUBING, AS NEEDED |
| 37 | 1 | BRASS | COUPLING, $\frac{3}{16}$ MALE FLARE - $\frac{1}{4}$ FEMALE PIPE |
| 38 | 1 | BRASS | ELBOW, FLARED, 90°, $\frac{1}{4}$ MALE FLARE TO FEMALE PIPE |

FIGURE 4

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