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3 September 1982
SUPERSEDING
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25 June 1968

MILITARY SPECIFICATION

HEXAMETHYLENETETRAMINE, TECHNICAL

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

1. SCOPE

1.1 Scope. This specification covers technical grade hexamethylenetetramine, $(CH_2)_6N_4$, commonly called hexamine or methenamine.

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications, standards, and handbooks. Unless otherwise specified, the following specifications, standards, and handbooks of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieve, Test

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-129 - Marking for Shipment and Storage

: Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed: to: Commander, US Army Armament Research and Development Command, ATTN: DRDAR-TSC-S, Aberdeen Proving Ground, MD 21010 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

2.1.2 Other Government documents, drawings, and publications. The following other Government documents, drawings, and publications form a part of this specification to the extent specified herein.

CODE OF FEDERAL REGULATIONS (CFR)

49 CFR 171 to 179 - Department of Transportation Hazardous Materials Regulations

(The Code of Federal Regulations is available from the Superintendent of Documents, US Government Printing Office, Washington, DC 20402. Orders for the above publication should cite "49 CFR 171 to 179.")

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

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

UNIFORM FREIGHT CLASSIFICATION RULES

(Application for copies should be addressed to the Uniform Classification Committee, Room 1106, 222 South Riverside Plaza, Chicago, IL 60606.)

ASTM STANDARDS

D1193 - Reagent Water
E203 - Water Using Karl Fischer Reagent

(Application for copies should be addressed to ASTM, 1916 Race Street, Philadelphia, PA 19103.)

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence.

3. REQUIREMENTS

3.1 Chemical characteristics. Hexamethylenetetramine shall conform to the chemical characteristics of table I when tested as specified therein.

TABLE I. Chemical characteristics

Characteristic	Percent by weight		Test paragraph
	Minimum	Maximum	
Assay	99.0	---	4.2.4.1
Formaldehyde	---	0.0	4.2.4.2
Ammonia	---	0.02	4.2.4.3
Chloride	---	0.02	4.2.4.4
Ash	---	0.1	4.2.4.5
Water content	---	0.5	4.2.4.6
Insoluble matter	---	0.05	4.2.4.7

3.2 Solubility in glacial acetic acid. Hexamethylenetetramine shall be completely soluble without turbidity in glacial acetic acid when tested as specified in 4.2.4.8.

3.3 Particle size. Hexamethylenetetramine shall conform to the particle size requirements of table II when tested as specified in 4.2.4.9.

TABLE II. Particle size

Sieve size	Percent passing	
	Minimum	Maximum
No. 16	98	—
No. 60	—	50
No. 100	—	30

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 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 the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the hexamethylenetetramine produced by one manufacturer, at one plant, from the same materials, and under essentially the same manufacturing conditions provided the operation is continuous. In the event the process is a batch operation, each batch shall constitute a lot (see 6.3).

4.2.2 Sampling.

4.2.2.1 For examination of packaging. Sampling shall be conducted in accordance with MIL-STD-105.

4.2.2.2 For test (see 6.5). Sampling shall be conducted in accordance with table III. A representative specimen of approximately one pound (lb) shall be removed from each sample container and placed in a suitable clean, dry container labeled to identify the lot and container from which it was taken.

TABLE III. Sampling for test

Number of containers in batch or lot	Number of sample containers
2 to 25	2
26 to 150	3
151 to 1,200	5
1,201 to 7,000	8
7,001 to 20,000	10
Over 20,000	20

4.2.3 Inspection procedure.

4.2.3.1 For examination of packaging. The sample unit shall be one filled container ready for shipment. Sample containers shall be examined for the following defects using an AQL of 2.5 percent defective:

(a) Contents per container not as specified
(b) Container not as specified
(c) Container closure not as specified
(d) Container damaged or leaking
(e) Container liner missing or not as specified
(f) Unitization not as specified
(g) Marking incorrect, missing, or illegible

4.2.3.2 For test. Each sample specimen taken in 4.2.2.2 shall be tested as specified in 4.2.4. Failure of any test by any specimen shall be cause for rejection of the lot represented.

4.2.4 Tests (see 6.5). Water in accordance with ASTM D1193, types as applicable, and reagent grade chemicals shall be used throughout the tests. Where applicable, blank determinations shall be run and corrections applied where significant. Tests shall be conducted as follows:

4.2.4.1 Assay. Weigh to the nearest milligram (mg) approximately 1 gram (g) of the specimen into a 250-milliliter (ml) flask. Dilute to 50 ml with water. Add 40 ml of 1N sulfuric acid and a few coarse crystals of carborundum or glass beads. Boil gently for approximately 5 hours or until the formaldehyde odor

has disappeared, adding water as needed to keep the volume over 50 ml. Cool and then titrate the excess sulfuric acid with freshly standardized 1N sodium hydroxide solution using methyl red as indicator and titrating to a yellow endpoint. Calculate the percent hexamethylenetetramine as follows:

$$\text{Percent hexamethylenetetramine} = \frac{3.505A(B-C)}{W}$$

where: A = Normality of sodium hydroxide solution,
B = Milliliters of sodium hydroxide solution required to titrate 40 ml of the 1N sulfuric acid,
C = Milliliters of sodium hydroxide solution required to titrate the excess sulfuric acid, and
W = Weight in grams of specimen.

4.2.4.2 Formaldehyde.

(a) Nessler's solution. Dissolve 10 g of potassium iodide in 15 to 20 ml of water. Slowly add a saturated aqueous solution of mercuric chloride, with stirring, until a slight permanent precipitate is formed. (If excess precipitate forms, add a few crystals of potassium iodide and dissolve.) Add a solution of 25 g of potassium hydroxide in 100 ml of water. Dilute to 250 ml with water.

(b) Procedure. Dissolve 0.500 g of the specimen in 50 ml of water in a Nessler tube. Add 2 ml of Nessler's solution prepared as specified in (a). Mix and allow to stand for 10 minutes. There shall be no formation of a grey turbidity or precipitate. The gradual formation of a pale yellow crystalline precipitate should be ignored. Save the Nessler tube and solution within for 4.2.4.3. (Appreciable quantities of ammonia in the specimen may mask the formation of a grey turbidity or precipitate.)

4.2.4.3 Ammonia.

(a) Color comparison standard. At the same time that the Nessler tube in the formaldehyde test is prepared (see 4.2.4.2), prepare another Nessler tube containing 50 ml of 0.0063 g per liter ammonium chloride solution and 2 ml of Nessler's solution. Mix and allow to stand for 10 minutes.

(b) Comparison. The intensity of any brown color produced in the formaldehyde test specimen (see 4.2.4.2) shall be compared with that of the standard. If lighter than the standard, the ammonia content is considered to be less than 0.02 percent. If darker than the standard, the solution shall be poured out from the specimen tube until the colors appear matched on looking down through the liquids. The depth of the liquid in both the standard and specimen tubes shall be measured and the percent ammonia calculated as follows:

$$\text{Percent ammonia} = \frac{(0.02)\ A}{B}$$

where: A = Depth of liquid in standard tube and
B = Depth of liquid in specimen tube.

Note. Do not dilute solutions as this causes precipitation.

4.2.4.4 Chloride. Dissove 5.0 g of the specimen in water to make 50 ml. Add 3 ml of 10-percent nitric acid and 1 ml of 0.1N silver nitrate solution and mix. Any turbidity produced shall be no greater than that in a simultaneously prepared standard containing 10.0 ml of standard sodium chloride solution (1 ml = 0.0001 g of chloride) and the same amounts of reagents, diluted to the same final volume as the specimen solution.

4.2.4.5 Ash. Weigh into a tared porcelain crucible, to the nearest milligram, approximately 5 g of the specimen. Carefully burn off the specimen and finally ignite to constant weight. Calculate the percent ash as follows:

$$\text{Percent ash} = \frac{100\ (A - B)}{W}$$

where: A = Weight in grams of the crucible and residue,
B = Weight in grams of the crucible, and
W = Weight in grams of the specimen.

4.2.4.6 Water content. Determine percent water content in accordance with ASTM E203.

4.2.4.7 Insoluble matter. Weigh into a 400-ml beaker, to the nearest milligram, approximately 50 g of the specimen. Dissolve in 200 ml of water by warming gently. Filter the solution through a tared filter crucible and wash well with hot water. Dry the residue at 105° ± 5° C for 2 hours, cool in a desiccator, and weigh. Calculate the percent insoluble matter as follows:

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

where: A = Weight in grams of filter crucible and residue,
B = Weight in grams of filter crucible, and
W = Weight in grams of specimen.

4.2.4.8 Solubility in glacial acetic acid. Dissolve 20.0 g of the specimen in 35 g of glacial acetic acid (99.90 percent), with stirring. The specimen shall dissolve completely, without turbidity, within 20 minutes at room temperature.

4.2.4.9 Particle size. Place 100 ± 1 g of the specimen on a sieve stack arranged from top to bottom as follows: Numbers 16, 60, 100, and bottom pan. Sieves shall conform to RR-S-366. Cover the sieve stack and place in a mechanical shaker geared to produce 300 ± 15 gyrations per minute and 150 ± 10 taps of the striker per minute. Vibrate for 5 minutes. Weigh the specimen remaining on the sieves and in the pan and calculate the percent passing.

5. PACKAGING

5.1 Unit packing, level C. Hexamethylenetetramine shall be unit packed level C in a quantity of 50 (+0.50 or -0) lb in a multiple wall paper shipping bag having a polyethylene film innermost liner and conforming to Uniform Freight Classification Rule 40 for Shipping Bag, Kraft Paper Plain or Extensible, wet-strength. The bag shall be closed in a manner to minimize the entry of moisture.

5.2 Packing, level C. Hexamethylenetetramine, unit packed as specified in 5.1, shall require no further protection for shipment other than unitization.

5.2.1 Unitization. Uniform quantities of bags of hexamethylenetetramine shall be unitized in a manner to assure integrity of the packs, acceptance by common carrier, and safe delivery and protection from supplier to first destination and for a period of 6 months.

5.3 Marking. Unit packs and unitized loads of hexamethylenetetramine shall be marked in accordance with MIL-STD-129 and all applicable Department of Transportation (DOT) regulations, and shall show date of manufacture and lot or batch number of the hexamethylenetetramine. Any overseas shipments shall be marked "HEXAMINE UN 1328" and shall bear the DOT FLAMMABLE SOLID label. In addition, all unit packs and unitized loads of hexamethylenetetramine shall bear the following precautionary marking:

"WARNING! FLAMMABLE - CAUSES IRRITATION.

Keep away from heat, sparks, and open flames. Avoid contact with eyes, skin, and clothing. Wash thoroughly after handling.

"FIRST AID: In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Call a physician. Flush skin with water. (Wash clothing before re-use.)"

6. NOTES

6.1 Intended use. Hexamethylenetetramine is intended for use in the manufacture of cyclonite (RDX) and homocyclonite (HMX) and as a stabilizer for mustard H.

6.2 Ordering data. Acquisition documents should specify the title, number, and date of this specification.

6.3 Batch. A batch is defined as that quantity of material which has been manufactured by some unit chemical process or subjected to some physical mixing operation intended to make the final product substantially uniform.

6.4 Significant places. For the purpose of determining conformance with this specification, an observed or calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding off method of ASTM E29.

6.5 Sampling and testing precautions. This specification covers inspection of chemical material which is potentially hazardous to personnel. Hexamethylenetetramine is a flammable solid and irritating material. Keep away from ignition sources. Avoid contact with skin and eyes. All applicable safety rules must be followed in the handling and processing of this material.

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