MIL-N-494A
22 MARCH 1968
SUPERSEDING
JAN-N-494
10 SEPTEMBER 1947

MILITARY SPECIFICATION

NITROGUANIDINE (PICRITE)

This specification has been approved by the Department of Defense and is mandatory for use by the Departments of the Army, the Navy, and the Air Force.

1. SCOPE

1.1 **Scope.** This specification covers nitroguanidine for use in the manufacture of propellants.

1.2 **Classification.** Nitroguanidine shall be of the following classes and types as specified (see 6.1 and 6.2):

Type I — Minimum purity 98 percent.
Type II — Minimum purity 99 percent.
Class 1 — Average particle diameter.— 6.0 microns maximum and 4.3 microns minimum (see table II).
Class 2 — Average particle diameter.— — Less than 3.3 microns (see table II).

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.

STANDARDS

MILITARY

MIL-STD-105 — Sampling Procedures and Tables for Inspection by Attributes.
MIL-STD-109 — Quality Assurance Terms and Definitions.
MIL-STD-129 — Marking for Shipment and Storage.
MIL-STD-286 — Propellants, Solid; Sampling, Examination and Testing.
MIL-STD-1233 — Procedure for Determining Particle Size Distribution and Packing Density of Powdered Material.
MIL-STD-1235 — Single and Multilevel Continuous Sampling Procedures and Tables for Inspection by Attributes.

(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 invitation for bids or request for proposals shall apply.

FSC 1375

TABLE I

Properties	Type I Percent Minimum	Type I Percent Maximum	Type II Percent Minimum	Type II Percent Maximum	Subparagraph
Purity (assay)	98.0	----	99.0	----	4.3.1
Ash content	.....	0.30	.....	0.30	4.3.2
pH value	4.5	7.0	4.5	7.0	4.3.3
Acidity (as sulfuric acid)	.....	0.60	.....	0.06	4.3.4
Total volatiles	.....	0.25	.....	0.25	4.3.5
Sulfates (as sodium sulfate)	.....	0.20	.....	0.20	4.3.6
Water insoluble impurities	.....	0.20	.....	0.20	4.3.7

CODE OF FEDERAL REGULATIONS

49 CFR 71-90 — Interstate Commerce Commission Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles.

(The Interstate Commerce Commissions regulations are now a part of the Code of Federal Regulations (1949 Edition — Current Revision) available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Orders for the above publication should cite "49 CFR 71-90 (Current Revision)".)

3. REQUIREMENTS

3.1 **Material.** Nitroguanidine, as received, shall be a white, free flowing, crystalline powder, when tested as specified in 4.3.

3.2 **Properties.** Nitroguanidine shall be in accordance with the chemical properties specified in table I, when tested in accordance with the applicable subparagraphs specified therein (see 6.4).

3.3 **Average particle size.** Nitroguanidine shall comply with the average particle size specified in table II, when tested in accordance with 4.3.8.

TABLE II

Class	Average particle size — Microns Minimum	Maximum
1	4.2	6.0
2	.....	Less than 3.3

3.4 **Workmanship.** The nitroguanidine and all containers shall be free from dirt, oil, grease, wood chips and other foreign material.

4. QUALITY ASSURANCE PROVISIONS

4.1 **General quality assurance provisions.** The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to 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 Standard MIL-STD-109 in order to define the terms used herein. Inspection shall be performed in accordance with this specification and other specifications referenced in any of the contractual documents.

4.1.1 *Contractor quality assurance system.* If the contractor desires to utilize a quality assurance system, which is at variance with the quality assurance provisions of 4.2 and 4.3 and other documents referenced herein, he shall submit a written description of the system to the contracting officer for approval prior to initiation of production. It shall include a description covering controls for lot formation and identification, inspections to be performed, inspection stations, sampling procedures, methods of inspection, (measuring and testing equipment), and provisions for control and disposition of nonconforming material. The written description will be considered acceptable when, as a minimum, it provides the quality assurance provisions required by the provisions of 4.2 and 4.3 and the other documents referenced herein. The contractor shall not be restricted to the inspection station or the method or inspection listed in this specification provided that an equivalent control is included in the approved

quality assurance procedure. In cases of dispute as to whether certain procedures of the contractors system provide equal assurance, the comparable procedure of this specification shall apply. The contractor shall notify the Government of, and obtain approval for, any changes to the written procedure that affects the degree of assurance required by this specification or other documents referenced herein.

4.1.2 *Submission of product.* At the time the completed lot of product 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 submitted:

(a) A statement that the lot complies with all quality assurance provisions of the approved current written description of the system.

(b) Quantity of product inspected.

(c) Results obtained for all inspection performed.

(d) Specification number and date, together with an identification and date of changes.

(e) Certificates of analysis on all material procured directly by the contractor, when such material is covered by referenced Government specification.

(f) Quantity of product in the lot.

(g) 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 agents authority to bind his principal. Substantiation of the agents 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.

4.1.3 *Government verification.* Using the contractors written quality assurance procedure (see 4.1.1), this detail specification, and other contractual documents as a guide, the Government inspector shall verify all quality assurance operations performed by the contractor. Verification shall be in accordance with (a) or (b) as applicable, the decision being the responsibility of the procuring activity. In either case, the inspector shall also ascertain, prior to acceptance, that all quality assurance provisions of other specifications referenced in any of the contractual documents have been complied with. Deviations from prescribed or agreed upon procedures discovered by the Government inspector shall be brought to the attention of the supplier. Disposition of the product and remedial action shall be as directed by the Government inspector and, depending on the nature of the deviation, may consist of lot rejection, screening, re-sampling, re-instruction of the suppliers employees, or other appropriate action:

(a) Verification at the point of manufacture shall be accomplished at unscheduled intervals in accordance with 4.1.3.1 and 4.1.3.2.

(b) Verification at the point of delivery shall be in accordance with 4.1.3.2.

4.1.3.1 *Surveillance.* Surveillance shall include, but is not limited to:

(a) Observation of procedures concerning lot formation and identification.

(b) Observation of sampling procedures and application of acceptance criteria.

(c) Determination that all required examinations and tests are performed in accordance with the prescribed procedures of this specification, or of approved equivalents thereto.

(d) Review of procedures for control and disposition of non-conforming material.

4.1.3.2 *Product inspection.* Product inspection shall consist of Government inspection of product which has been previously inspected by the contractor and found to meet the quality assurance provisions of this specification. The inspection by the Government shall be performed in order to determine that the product is of the quality required by this specification and that the contractors records are reliable.

4.2 Inspection provisions.

4.2.1 *Lot formation.* A lot shall consist of one or more batches of nitroguanidine, 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 nitroguanidine that has been subjected to the same mixing process intended to make the final product homogeneous.

4.2.2 *Examination.* Sampling plans and procedures for the following classification of defects shall be in accordance with Standard MIL-STD-105. Continuous sampling plans, in accordance with Standard 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 0.25 percent for each major defect and an AQL of 0.40 percent for each minor defect.

4.2.2.1 *Box, wooden, prior to sealing* (see 5.1.1).

Categories	Defects	Method of inspection	Code No. (see 6.3)
Critical:	None defined.		
Major:	AQL 0.40 percent		
101.	Foreign matter in nitroguanidine (see 3.4)	Visual	01001
102.	Liner in box cut, torn or punctured	Visual	01002
103.	Check material (see 3.1)	Visual	01003
Minor:	AQL 1.00 percent		
201.	Liner in box missing or inadequate	Visual	01004
202.	Insufficient packing material	Visual	01005

4.2.2.2 *Box, wooden, sealed* (see 5.1.1 and 5.2).

Categories	Defects	Method of inspection	Code No.
Critical:	None defined.		
Major:	AQL 0.25 percent		
101.	Box damaged	Visual	02001
Minor:	AQL 1.50 percent		
201.	Contents loose	Manual	02002
202.	Marking misleading or unidentifiable	Visual	02003
203.	Evidence of poor workmanship (see 3.4)	Visual	02004

4.2.2.3 *Fiberboard box, prior to sealing* (see 5.1.2).

Categories	Defects	Method of inspection	Code No.
Critical:	None defined.		
Major:	AQL 0.40 percent		
101.	Foreign matter in nitroguanidine (see 3.4)	Visual	03001
102.	Liner in box cut, torn or punctured	Visual	03002
103.	Check material (see 3.1)	Visual	03003
Minor:	AQL 1.00 percent		
201.	Liner in box missing or inadequate	Visual	03004
202.	Insufficient packing material	Visual	03005

4.2.2.4 *Fiberboard box sealed* (see 5.1.2 and 5.2).

Categories	Defects	Method of inspection	Code No.
Critical:	None defined.		
Major:	AQL 0.25 percent		
101.	Box damaged	Visual	04001
Minor:	AQL 1.50 percent		
201.	Contents loose	Manual	04002
202.	Marking misleading or unidentifiable	Visual	04003
203.	Evidence of poor workmanship (see 3.4)	Visual	04004

4.2.2.5 *Container prior to sealing* (see 5.1.3).

Categories	Defects	Method of inspection	Code No.
Critical:	None defined.		
Major:	AQL 0.25 percent		
101.	Foreign matter in nitroguanidine (see 3.4)	Visual	05001
Minor:	None defined.		

4.2.2.6 *Container, sealed* (see 5.1.3 and 5.2).

Categories	Defects	Method of inspection	Code No.
Critical:	None defined.		
Major:	AQL 0.25 percent		
101.	Container torn, cut or punctured	Visual	06001
Minor:			
201.	Marking misleading or unidentifiable	Visual	06002
202.	Evidence of poor workmanship (see 3.4)	Visual	06003

4.2.3 *Sampling by lot.* A random sample of containers shall be selected from each lot in accordance with the following table:

TABLE III

Lot Size	Sample Size
1 batch	8 containers
2 batches	12 containers
3 or more batches	16 containers

When lots are composed of 8 containers or less each container shall be sampled.

4.2.3.1 *Preparation of composite.* Equal primary samples in sufficient quantity to total 16 ounces shall be removed from each container. The individual primary samples shall then be combined in order to form a homogeneous composite sample of sixteen ounces and subjected to the tests specified in 4.3. If the composite sample fails to comply with any of the requirements specified, the lot shall be rejected.

4.3 The following test methods and procedures shall be used.

4.3.1 *Determination of purity.* Code No. 07001. The purity shall be determined by either the nitrometer method or the alternative chromous chloride method.

4.3.1.1 *Nitrometer method.* The nitrometer method specified in Standard MIL–STD–286, method 209.3 shall be used as the standard method for purity except that a 3 gram sample shall be crushed so that it will pass through a U. S. Standard Number 100 sieve. A 1 gram sample shall be dried in air at 46° Centigrade (° C.), overnight and then placed in an oven for 1 hour at 100° C. The sample

shall then be transferred to the nitrometer. The percent nitrogen shall then be converted to percent nitroguanidine by the following formula:

$$\text{Percent Nitroguanidine} = \frac{7.430R}{W}$$

Where: R = reading of measuring tube
W = sample weight
7.480 = correction factor

4.3.1.2 *Chromous Chloride Method.*

4.3.1.2.1 *Chromous Chloride (0.2 Normal (N)) Standardization.* A 110 grams of hydrated chromous chloride shall be dissolved in about 500 milliliters of distilled water contained in a 2-liter volumetric flask. Fifty millilitars of concentrated hydrochloric acid shall be added and diluted to the mark with distilled water. After mixing, the solution shall be poured into a 4-liter bottle containing an excess of amalgamated zinc. The mixture shall be allowed to stand overnight and maintained under this atmosphere at all times. A bright blue color indicates complete reduction of chromic ions (green) to chromous ions (blue). The chromous chloride solution shall be transferred to an automatic buret with a 2-liter reservoir.

4.3.1.2.2 *Ferric Alum (0.15N) Standardization.* The 0.15 Ferric Alum solution shall be prepared as specified in Standard MIL-STD-286, method 608.1.

4.3.1.2.3 *Procedure.* An accurately weighed portion of approximately 0.1 gram of sample shall be transferred to a 500-milliliter titration flask. Twenty-five milliliters of distilled water shall be added and shaken and heated until the sample is dissolved completely. The titration flask shall be placed in a magnetic stirrer and the contents shall be stirred for at least 10 minutes. The air shall be swept with a stream of carbon dioxide and the carbon dioxide atmosphere shall be maintained throughout the determination. Exactly 50 milliliters of 0.2N chromous chloride solution shall be added to the 500 millliliter titration flask. The contents shall be stirred for 5 minutes and 25 milliliters of 15 percent hydrochloric acid shall be added. Ten drops of 2 percent phenosafranin indicator shall be added and the excess chromous chloride shall be titrated with 0.15N ferric alum solution. The end point is a sharp color change from green to blood red. A blank determination using the same procedure and reagents but omitting the sample shall be conducted. The percentage of nitroguanidine shall be calculated according to the following equation:

$$\text{Percent Nitroguanidine} = \frac{1.735N\ (V_1 - V_2)}{W}$$

Where V_1 = ml of ferric alum used to titrate the total chromous chloride (blank).

V_2 = ml of ferric alum used to titrate the excess chromous chloride.

W = weight of sample in grams used in the analysis.

N = normality of ferric alum solution.

4.3.2 *Determination of ash content.* Code No. 08001. The ash content shall be determined as specified in Standard MIL-STD-286, method 106.1.

4.3.3 *Determination of pH value.* Code No. 09001. A portion of 5.0 grams (gms.) of the sample, weighed to the nearest milligram (mg) shall be transferred to an 8 ounce bottle which has been previously heated to 80° C. A 200 milliliter portion of freshly boiled distilled water, which has been allowed to cool to 80° C. shall be added. The bottle and contents shall be immersed in a constant temperature water bath, maintained at 80° ± 2° C. The bottle shall be removed for shaking at 3 minute intervals. As soon as the solution is complete, the bottle shall be removed and rapidly cooled to room temperature in cold running tap water. The pH of the solution shall be determined using a glass electrode pH meter. This solution shall be retained for the "Determination of Acidity" (see 4.3.4). Prior to the pH determination, the meter

shall be calibrated using standard buffer solutions, at pH 4.0 and pH 8.0.

4.3.4 *Determination of acidity.* Code No. 10001. The solution retained from the determination of the pH value shall be titrated with a 0.05 N sodium hydroxide solution to a reading of 7.6 on the pH meter. A blank determination shall be conducted on an equal volume of distilled water and the volume of sodium hydroxide used for the specimen shall be corrected. The percent acid present in the solution shall be calculated to sulfuric acid as follows:

$$\text{Percent sulfuric acid} = \frac{4.9\ (A-B)\ N}{W}$$

Where: A — ml of sodium hydroxide solution used for titrating specimen.

B — ml of sodium hydroxide solution used for titrating blank.

N — normality of sodium hydroxide solution.

W — weight of specimen in gms.

4.3.5 *Determination of total volatiles.* Code No. 11001. A portion of the sample, approximately 5.0 gms, weighed to the nearest 0.1 mg, shall be transferred to a tared aluminum dish (90 millimeters (mm) in diameter and 60mm deep) having a tightly fitting cover. The uncovered dish containing the specimen shall be heated for 2 hours in an oven maintained at 100° ± 2° C. At the end of this period the dish shall be covered, transferred to a desiccator until cool, and then weighed. The procedure of heating, cooling in a desiccator, and weighing shall be repeated at hourly intervals until 2 successive weighings do not differ by more than 0.2 mg, or the specimen increases in weight. The percent total volatiles shall be calculated as follows:

$$\text{Percent total volatiles} = \frac{(A-B)\ 100}{A}$$

Where: A — weight of specimen in gms prior to heating.

B — weight of specimen in gms after heating.

4.3.6 *Determination of sulfates.* Code No. 12001. A portion of the sample, approximately 5.00 gms, weighed to the nearest 0.1 mg, shall be dissolved in 200 milliliters of hot distilled water contained in a 400-milliliter beaker. If necessary the solution shall be filtered through a No. 42 Whatman filter paper, or equal. The solution shall be made acid by the addition of 1 milliliter of concentrated hydrochloric acid, which shall be followed by the addition of 1 millliter of 10 percent barium chloride solution. The solution shall be boiled for approximately 5 minutes and then allowed to stand, with occasional gentle stirrings, until cool. The solution shall then be allowed to stand for at least 10 hours. At the end of this period, the solution shall be heated to boiling and filtered through a previously ignited and tared Gooch crucible equipped with a fine asbestos mat. The precipitate shall be washed with small portions of warm distilled water, sucking each portion through before adding the next. The last few washings shall be tested with 1 percent silver nitrate solution to see that all chlorides have been removed. When free of chlorides the Gooch crucible shall be transferred to an oven maintained at 100° ± 2° C., followed by 1 hour in a muffle furnace, maintained at 600° C. The crucible shall then be transferred to a desiccator until cool, and weighed. The percent sulfate present in the specimen shall be calculated to sodium sulfate as follows:

$$\text{Percent sodium sulfate} = \frac{60.86A}{W}$$

Where: A — weight of residue in gms.

W — weight of specimen in gms.

4.3.7 *Determination of water insoluble impurities.* Code No. 13001. A portion of the sample of approximately 10.0 gms, weighed to the nearest 0.1 gm, shall be dissolved in 400 milliliters of boiling distilled water. The solution shall be filtered through a tared Gooch crucible equipped with a fine asbestos mat. The residue shall be washed with a total of 100 milliliters of boiling water, in 20 milliliter portions, letting each portion go through the crucible before adding the next.

The crucible shall be transferred to an oven, maintained at 105° ± 5° C. for 2 hours, cooled in a desiccator and weighed. The gain in weight shall be calculated as percent water insoluble impurities as follows:

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

Where: A = weight of crucible and residue in gms.

B = weight of crucible in gms.

W = weight of specimen in gms.

4.3.8 *Average particle size.* Code No.14001. The average particle size shall be determined in accordance with Standard MIL-STD-1233, method 100.

5. PREPARATION FOR DELIVERY

5.1 **Packing.**

5.1.1 *Level A.* Nitroguanidine shall be packed in wooden boxes in accordance with Code of Federal Regulations, ICC Specification 14, 15A or 16A. Wooden boxes shall be lined with strong paper or cloth bags of capacity not exceeding 50 pounds, packed with filling holes up.

5.1.2 *Level B.* Nitroguanidine shall be packed in fiberboard boxes in accordance with Code of Federal Regulations, ICC Specification 23F or 23H. Fiberboard boxes shall be lined with strong paper or cloth bags of capacity not exceeding 50 pounds, packed with filling holes up (see 6.1).

5.1.3 *Level C.* Nitroguanidine shall be packed in standard commercial containers complying with Code of Federal Regulations, Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles.

5.2 **Marking.** Containers shall be marked to insure safe handling as required by Code of Federal Regulations, Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, and, in addition, shipment shall be marked in accordance with Standard MIL-STD-129. Containers prepared for level B or level C shall be marked LTD MIL PK or MIN MIL PK, as applicable. When specified, exterior containers for overseas shipment shall be marked with Ordnance Corps symbol and the SM group of the item.

6. NOTES

6.1 **Ordering data.** Procurement documents should specify the following:

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

(b) Class and type required.

(c) Level of packing required, level B packing is intended to provide economical but limited protection, and should be specified only when it is determined that the nitroguanidine will be held in domestic covered storage 6 months or more from date of initial packing.

6.2 **Classification cross-reference.** Classes 1 and 2 were formerly class a and b respectively, of Specification JAN-N-494.

6.3 **Inspection code numbers.** The five digit code number assigned to the inspection herein are to facilitate future data collection and analysis by the Government.

6.4 **International Standardization Agreement.** Certain provisions of this specification are the subject of International Standardization Agreement, STANAG, No. 4026. When amendment, revision, or cancellation of this specification is proposed, the departmental custodians will inform their respective Departmental Standardization Offices so that the appropriate action may be taken respecting the international agreement concerned.

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

Custodians:

Army—Munitions Command
Navy—Bureau of Naval Weapons
International Interest (see 6.4)

Preparing activity:

Army—Munitions Command
Project Number: 1735-0435

INSTRUCTIONS: In a continuing effort to make our standardization documents better, the DoD rovides this form for use in submitting comments and suggestions for improvements. All users of military standardization documents are invited to provide suggestions. This form may be detached, folded along the lines indicated, taped along the loose edge *(DO NOT STAPLE)*, and mailed. In block 5, be as specific as possible about particular problem areas such as wording which required interpretation, was too rigid, restrictive, loose, ambiguous, or was incompatible, and give proposed wording changes which would alleviate the problems. Enter in block 6 any remarks not related to a specific paragraph of the document. If block 7 is filled out, an acknowledgement will be mailed to you within 30 days to let you know that your comments were received and are being considered.

NOTE: This form may not be used to request copies of documents, nor to request waivers, deviations, or clarification of specification 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.

(Fold along this line)

(Fold along this line)

DEPARTMENT OF THE NAVY

NO POSTAGE NECESSARY IF MAILED IN THE UNITED STATES

OFFICIAL BUSINESS
PENALTY FOR PRIVATE USE $300

BUSINESS REPLY MAIL
FIRST CLASS PERMIT NO. 12503 WASHINGTON D. C.

POSTAGE WILL BE PAID BY THE DEPARTMENT OF THE NAVY

Commanding Officer
Naval Weapons Station Earle
Colts Neck, NJ 07722

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

(See Instructions – Reverse Side)

1. DOCUMENT NUMBER	2. DOCUMENT TITLE

3a. NAME OF SUBMITTING ORGANIZATION	4. TYPE OF ORGANIZATION *(Mark one)*
	☐ VENDOR ☐ USER ☐ MANUFACTURER ☐ OTHER *(Specify)*: _____
b. ADDRESS *(Street, City, State, ZIP Code)*	

5. PROBLEM AREAS

a. Paragraph Number and Wording:

b. Recommended Wording:

c. Reason/Rationale for Recommendation:

6. REMARKS

7a. NAME OF SUBMITTER *(Last, First, MI)* – Optional	b. WORK TELEPHONE NUMBER *(Include Area Code)* – Optional
c. MAILING ADDRESS *(Street, City, State, ZIP Code)* – Optional	8. DATE OF SUBMISSION *(YYMMDD)*

(TO DETACH THIS FORM, CUT ALONG THIS LINE.)

DD FORM 82 MAR 1426

PREVIOUS EDITION IS OBSOLETE.