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19 FEBRUARY 1962
SUPERSEDING
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MILITARY SPECIFICATION

NITROGLYCERIN

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 two types of nitroglycerin for use in propellants.

1.2 Classification. The nitroglycerin shall be of the following types, as specified in 6.1 and 6.2.

Type I — Using grade B glycerin.

Type II — Using partially polymerized glycerin.

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

FEDERAL

O-G-491 — Glycerin (Glycerol).

STANDARDS

MILITARY

MIL-STD-105 — Sampling Procedures and Tables for Inspection by Attributes.

MIL-STD-109 — Inspection Terms and Definitions.

MIL-STD-129 — Marking for Shipment and Storage.

MIL-STD-286 — Propellants; Standard for Methods of Sampling, Inspection and Testing.

PUBLICATIONS

QUARTERMASTER CORPS

ORD-M-608-11—Procedures and Tables for Continuous Sampling by Attributes.

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

2.2 The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

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 Commission regulations are now a part of the Code of Federal Regulations (1949 Edition — latest revision) available from the

FSC 1375

AF Heaven-Dayton Co., 28 June 1962 1000

Superintendent of Documents, Government Printing Office, Washington 25, D.C. Orders for the above publication should cite "49 CFR 71-90" (latest revision.)

3. REQUIREMENTS

3.1 **Material.** Materials shall be in accordance with applicable specifications.

3.1.1 *Glycerin.* The glycerin used in the manufacture of type I nitroglycerin and in the manufacture of partially polymerized glycerin for the manufacture of type II nitroglycerin shall comply with grade B of Specification O-G-491.

3.1.2 *Partially Polymerized glycerin.* The partially polymerized glycerin used in the manufacture of type II nitroglycerin shall contain 27 to 31 percent by weight of polymer glycerin, expressed as diglycerin.

3.2 **Moisture.** The moisture content shall be 0.5 percent maximum (max.), when determined as specified in 4.3.1. When nitroglycerin is used at the point of manufacture in the production of propellant by the water slurry method or by the water emulsion method, the moisture requirement shall not apply.

3.3 **Acidity or alkalinity.** The acidity as sulfuric acid or alkalinity as sodium carbonate shall be 0.002 percent, max., when determined as specified in 4.3.2.

3.4 **Nitrogen.**

3.4.1 *For type I.* The nitrogen content shall be 18.40 percent, minimum (min.), when determined as specified in 4.3.3.

3.4.2 *For type II.* The nitrogen content shall be 17.80 percent, min., to 17.90 percent, maximum, when determined as specified in 4.3.3.

3.5 **Stability.** When subjected to the 82.2° Centigrade (°C.) heat test, the nitroglycerin shall not change the color of the standard potassium iodide starch paper in less than 10 minutes when determined as specified in 4.3.4.

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. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. 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 non-conforming 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 of inspection listed in this specification provided that an equivalent control is included in the approved quality assurance procedure. In cases of dis-

pute as to whether certain procedures of the contractor's 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 covered by reference government specifications procured directly by the contractor.

(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 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.

4.1.3 *Government verification.* Using the contractor's 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 here 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 supplier's 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 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 in-

spected 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 contractor's records are reliable.

4.2 Inspection provisions.

4.2.1 *Lot formation.* Where the batch process is used a lot shall consist of one nitration charge. When the continuous process is used, a lot shall consist of not more than 3,000 pounds.

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 Handbook ORD-M608-11 may be used if approved by the procuring activity. Also, at the option of the Government, AQL's and sampling plans may be applied to the individual characteristics listed using an AQL of 0.25 percent for each major defect.

4.2.2.1 *Inside container, (prior to filling).*

Categories	Defects	Method of inspection
Critical—None defined.		
Major—AQL 0.25 percent.		
	101. Container material improper	Visual
Minor—None defined.		

4.2.2.2 *Inside container (filled).*

Categories	Defects	Method of inspection
Critical:		
	101. Leak in container	Visual
Major—AQL 0.25 percent		
	101. Marking incorrect, incomplete or illegible.	Visual
Minor—None defined.		

4.2.2.3 *Boot, rubber container for individual container (prior to insertion of container).*

Categories	Defects	Method of inspection
Critical—None defined.		
Major—AQL 0.25 percent		
	101. Boot cut, torn or punctured	Visual
	102. Boot leaks	Visual
Minor—None defined.		

4.2.2.4 *Boot, rubber container for individual containers (with container inserted).*

Categories	Defects	Method of inspection
Critical—None defined.		
Major—AQL 0.40 percent.		
	101. Boot height, improper	Visual
	102. V-shaped grooves missing or incomplete.	Visual
	103. Marking incorrect, incomplete or illegible.	Visual
	104. Container does not fit snugly	Manual
Minor—None defined.		

4.2.3 *Testing.*

4.2.3.1 *Sampling.* Using a rubber dipper, a sample of approximately 2 ounces shall be removed from each lot. The nitroglycerin shall be transferred to a rubber bottle with a rubber stopper. The bottle shall be labeled to show the following:

(a) Source of manufacture.

(b) Plant.

(c) Contract or purchase order number.

(d) Number of pounds in the lot.

(e) Lot number.

If the sample fails to comply with any of the requirements specified the lot shall be rejected.

After disposal of the nitroglycerin from the rubber sample bottle, the bottle shall be cleaned as follows:

(a) Rinse thoroughly with acetone.

(b) Flush generously with hot tap water (3 minutes under a direct flow is considered adequate).

(c) Rinse with distilled water and dry.

4.3 Test methods and procedures.

4.3.1 *Determination of moisture in nitroglycerin by Karl Fisher.* Weigh accurately a 5 to 10 gm portion of the sample (sample size shall be in accordance with the strength of the Karl Fisher reagent) of the nitroglycerin into a tared narrow-necked stoppered

flask. Titrate directly with the standardized Karl Fisher reagent until a brown tinge persists in the solution for 30 seconds. (Potentiometric endpoint indicators may also be used in titration with Karl Fisher reagent). Calculate the moisture content as follows:

$$\text{Percent moisture} = \frac{100\ (KF)}{W}$$

where:

K = ml. of Karl Fisher reagent used in titration.

F = reagent factor (gm. of water per ml. of reagent).

W = weight of nitroglycerin in gm.

4.3.2 *Determination of acidity or alkalinity.* The acidity or alkalinity shall be determined as follows: By means of a pipette, transfer a portion of approximately 10 gm of the sample to a tared beaker, reweigh and dissolve in 100 ml of benzene. Transfer the solution to a 250-ml separatory funnel and wash twice with 50-ml portions of neutral distilled water. Separate and combine the water washings in a 250-ml beaker. Add several drops of bromothymol blue indicator and titrate immediately. If yellow, titrate with 0.01 Normal (N) sodium hydroxide if blue titrate with 0.01N sulfuric acid. Run a blank determination on the volume of benzene and correct the volume acid or alkali required for titration. Calculate the percentage of acidity as sulfuric acid or alkalinity as sodium carbonate as follows:

$$\text{Percentage sulfuric acid} = \frac{4.90\ (Vv\text{-})N}{W}$$

where:

V = ml. sodium hydroxide solution required for sample.

v = ml. sodium hydroxide solution required for blank.

N = normality of sodium hydroxide solution.

W = weight of sample.

$$\text{Percentage sodium carbonate} = \frac{5.3\ (V\text{-}v)N}{W}$$

where:

V = ml. sulfuric acid solution required for sample.

v = ml. sulfuric acid solution required for blank.

N = normality of sulfuric acid solution.

W = weight of sample.

4.3.3 *Determination of nitrogen.*

4.3.3.1 *Standardization of the nitrometer.* Standardization of the DuPont 5-part form of Lunge's nitrometer, or equal, shall be made by method 209.3 of Standard MIL–STD–286.

4.3.3.2 *Dry filter paper.* Whatman number 42 or equivalent filter paper shall be transferred to a glass weighing dish equipped with a round-glass cover. The uncovered dish and contents shall be heated at 100° ± 2° C. for 2 hours, the dish stoppered, and cooled in a desiccator.

4.3.3.3 *Procedure.* Four dried filter papers prepared as specified in 4.3.3.2 shall be quickly transferred to a dry conical glass funnel and a portion of approximately 10 gm of the sample shall be added immediately. This shall be accomplished as rapidly as possible in order to avoid absorption of moisture by the filter papers. Suction shall be applied and the filtered nitroglycerin received in a moisture-free receptacle. A portion of from 0.70 to 0.75 gm of the filtered material shall be transferred to a tared 25-ml beaker and weighed accurately. Five ml of glacial acetic acid shall be added and the mixture stirred with a small glass rod until the nitroglycerin has dissolved. With the aid of the glass rod the solution shall be quickly transferred to the cup of the generating bulb of nitrometer and drawn into the generating bulb, making certain that no air is drawn into the generating bulb. Twenty-five ml of sulfuric acid (94.5 ± 0.5 percent) shall be measured into a graduate. The beaker and glass rod shall be rinsed by means of successive 5-ml portions of the acid, transferring each portion of the rinsing acid to the generating bulb cup and

drawing it into the bulb after agitating by means of the glass rod. Any air drawn into the bulb shall be forced out and the upper stopcock closed when the sample has been completely drawn into the generating bulb.

Caution: (The lower stopcock must remain open and the mercury reservoir adjusted so that the level of the mercury in the reservoir is 13 to 15 inches below the level of the mercury in the generating bulb.) The generating bulb shall be shaken gently keeping the lower end in a fixed position until most of the gas has been generated. The mercury reservoir shall be adjusted so that the mercury in the generating bulb drops nearly to the lower shoulder, the lower stopcock shall be closed, and the bulb shaken vigorously for 3 minutes. The bulb shall be replaced on the rack and allowed to remain there for 3 minutes. The bulb shall be removed from the rack and shaken vigorously for an additional 3 minutes and then replaced on the rack for 3 minutes. The above procedure shall be repeated. The gas shall be transferred to the measuring tube and the levels of mercury in the measuring and compensating tubes adjusted until they are approximately the same and the level of the mercury in the compensating tube is approximately at the standardization mark. The gas shall be allowed to stand for 20 minutes in order to permit equalization of the temperature of the gas in the two tubes. The levels of the mercury in the two tubes shall be adjusted so that they are the same, and the level of the mercury in the compensating tube is at the standardization mark. A leveling device shall be used which will make possible accurate adjustment of the levels. The reading on the measuring tube shall be noted and the percentage of nitrogen in the sample on a moisture-free basis shall be calculated as follows:

$$\text{Percentage of nitrogen} = \frac{A}{0.9983W}$$

where:

A = reading of the mercury level on the measuring tube.

W = weight of sample taken.

0.9983 = a factor based upon the experimental determined average moisture content of nitroglycerin subjected to the filtration treatment specified above.

4.3.4 *Determination of stability.* The stability shall be determined as follows: Filter a portion of the nitroglycerin sample through two thicknesses "S. and S." Number 604 filter paper or equivalent. Transfer a 2 ml portion of the filtered material, by means of a pipette, to each of three test tubes which are 5.5 inches long, 0.50 inches internal diameter and 0.62 inches outside diameter. (Care should be taken during the transfer not to leave droplets of the nitroglycerin on the sides of the test tubes.) Stopper each tube by means of a new, tightly fitting cork through which passes a tightly fitted glass rod equipped with a platinum holder for a strip of standard potassium iodide starch-indicator paper. Using forceps hang on to the platinum holder a strip of standard potassium iodide starch-indicator paper. The standard potassium iodide starch-indicator test paper shall normally be approximately 1 inch long and ⅜ inch wide. Moisten a horizontal section in the upper half of the standard test paper with a 50 percent solution (by volume) of pure glycerin in distilled water. This is conveniently accomplished by dipping a small diameter glass rod into the glycerin-water solution, and as the rod is withdrawn, contact is made with the side of the container so as to minimize the volume of the solution adhering to the rod. Draw the rod across the paper strip so as to produce a level and distinct line of demarcation on the lower edge of the wet area. Prepare a blank by suspending a moistened strip of the standard test paper in a clean dry tube. Adjust the temperature of the heat tube bath to 82.2 ± 1° C. and insert all four test tubes into the heating solution. The depth of immersion of the test tubes into the heating solution should be approximately 2 inches. The bath should be placed in such a position that the test tubes are viewed against a white background illuminated by bright diffused daylight. Note the time of insertion of the tubes into the bath.

During the test the line of demarcation on the standard test paper should be 3 inches above the level of the nitroglycerin in the test tube. The line of demarcation in the blank tube is regulated at an equivalent height in the tube. Consider the end point of the test to be the first appearance of any discoloration at the line of demarcation between the wet and the dry portion of the test paper in the sample tube which is in excess of the discoloration observed at the same position on the test paper in the blank. Note the time for completion of the test to the nearest minute. Consider the minimum time for the three tubes to represent the heat test value of the sample. After the test rinse all rods and tubes with acetone to remove nitroglycerin, wash with warm soapy water, rinse thoroughly with tap water, then with distilled water and dry in a steam oven at approximately 80° C.

5. PREPARATION FOR DELIVERY

5.1 Packing.

5.1.1 *Level C.* Nitroglycerin is classed as a "Forbidden Explosive" in Tariff Number 13, Code of Federal Regulations, Interstate Commerce Commission Regulations for the transportation of Explosives and Other Articles by Freight. For shipment by public highways see Tariff Number 13, appropriate section of Interstate Commerce Commission Regulations applying to shipments made by Way of Common and Contract Carriers by Public Highway.

5.2 Marking. The marking of interior packages and exterior shipping containers shall be in accordance with instructions contained in Tariff Number 13, Interstate Commerce Commission Regulations, and in accordance with Standard MIL-STD-129.

6. NOTES

6.1 Ordering data. Procurement documents should specify the following:

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

(b) Type required.

6.2 Intended uses. Nitroglycerin is intended for the following uses:

(a) Type I for use in propellant.

(b) Type II for use in propellant.

Custodians:
Army—Ordnance Corps
Navy—Bureau of Naval Weapons
Air Force—WADD

Preparing activity:
Army—Ordnance Corps