INCH-POUND

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DETAIL SPECIFICATION

NITROGLYCERIN

This specification is approved for use by U.S Army Armament Research, Development and Engineering Center (ARDEC), and is available for use by all Departments and Agencies of the Department of Defense.

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 l — Using grade B glycerin.

Type 11— 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.

Comments, suggestions, or questions on this document should be addressed to: Commander, US Army ARDEC, ATTN: AMSRD-AAR-QES-E, Picatinny, New Jersey 07806-5000 or email to ardecstdzn@conus.army.mil. Since contact information can change, you may want to verify the currency of this address information using the ASSIST online database at http://assist.daps.dla.mil.

AMSC N/A FSC 1376

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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 Superintendent of Documents, Government Printing Office, Washington 25, D.C. Orders for the above publication should cite "49 CFR 71-90" (Intest 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 previsions. 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 dispute 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 contactor. Verification shall be in accordance with a or b us 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 actions:

(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 sha11 include, but is not limited to —

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

(b) Observation of sampling procedures end 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 inspected by the contractor and found to meet the quality assurance provisions of this specification. The inspection bv 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 processes 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.
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. Book 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.
103. Marking incorrect, incomplete or illegible.
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 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 appropriate titration vessel. 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 toluene. Transfer the solution to a 250-ml separatory funnel and wash twice with 50-ml portions of neutral distilled water. Separate the layers 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 extract of the same volume of toluene 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 :

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

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.

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

Where:

V = ml. sulfuric acid solution required for sample.
v = sulfuric acid solution required for blank.
N = normality of sulfuric acid solution.
W = weight of sample.

4.3.3 Determination of nitrogen. This method is used to determine the nitrogen content of nitroglycerin (NG), Diethyleneglycol Dinitrate (DEGDN) and other liquid nitrate esters. The nitrate in the sample is released by strong sulfuric acid, forming nitric acid, which is then titrated with ferrous sulfate ($FeSO_4$). The titration is monitored by glass and platinum electrodes which read the change in millivolt (mv) output from a range of 500-600 mv to a preselected mv endpoint. The nitrogen content is detemined by the volume of $FeSO_4$ necessary to produce this change.

4.3.3.1 Standardization. Initial standardization shall be in duplicate, done daily and conducted prior to any sample analyses being made. Additional standardization(s) may take place during the typical method operating period.

4.3.3.2 Specimen.

(a.) For KNO_3 standard, 0.5000 ± .050 g sample

(b.) For NG sample. 0.3750 ± .025 g sample

(c.) For DEGDN sample, 0.4000 ± .050 g sample

4.3.3.3 Apparatus.

(a.) A titration system which can titrate to a fixed end point (or locate the end point) in a potentiometric titration shall be used.

(b.) Platinum Electrode. Fisher Cat. No. 13-639-115 or equivalent.

(c.) Glass Electrode. Fisher Cat. No. 13-639-4 or equivalent.

(d.) Magnetic stirrer base with stirring bar.

(e.) Cooling bath. Ice bath or other mechanical cooling system.

(f.) Carboy (painted black for $FeS0_4$). Alternatively, titrant may be stored and used from a dark glass bottle of a size appropriate for expected usage.

(g.) Timer (seconds).

(h.) Recorder for automatic titration (optional).

(i.) Desiccator with drying agent (with color indicator).

(j.) Weighing bottles 1 oz.

(k.) 135° ± 5° C drying oven.

(l.) 100° ± 5° C drying oven.

(m.) 45 ± 2° C drying oven.

(n.) 250 ml beaker(s).

4.3.3.4 Materials.

(a.) Sulfuric acid (H_2S04) ACS reagent grade > 95 %

(b.) Potassium nitrate (Purity 99.99% minimum [metals basis]. NIST or other certification preferred.)

(c.) Ferrous sulfate titrant ($FeSO4 \cdot 7H_2O$) crystals 350 g FeS04 dissolved in 800 ml H_2O. Add 1000 ml of 1:1 H_2SO_4:H_20

(d.) Distilled water

(e.) Tissue paper

4.3.3.5 Safety.

(a.) If acid is spilled on the hands or clothing, flush immediately with plenty of water.

(b.) Keep sulfuric acid contaminated tissues under water.

(c.) To prevent acid bum, always wipe the electrodes free of excess acid before retrieving the spent sample beaker or the rinse acid beaker. Wipe with a dabbing motion to prevent static buildup on the electrodes.

(d.) Always handle liquid nitrate esters with caution due to their sensitivity to heat and impact.

4.3.3.6 Operations.

4.3.3.6.1 Standardization of $FeS0_4$ Titrant Using KNO_3.

(a.) Weigh 0.5 ±0.05 g of KNO_3, that has been pulverized to about the grain consistency of table salt, in a 1 oz. weighing bottle. Dry in a 135° ± 5°C oven for 4 hours, place in a desiccator to cool. If the KNO_3 is out of the oven for more than 3 hours, re-dry in a 100 ± 5 °C oven for a minimum 2 hour.

(b.) Place the stirring bar in a dry 250 ml beaker and add 150 ml of $H2SO_4$. Use only clean beakers or beakers that have not been used in a previous titration. Beakers that have been used in other analyses have contaminants in the scratches of the beaker(s).

(c.) Place the beaker in the cooling bath and on the stirring base. Start the stirrer.

(d.) Weigh the weighing bottle containing the KNO_3 on an analytical balance with 0.1 mg resolution.

(e.) Pour the KNO_3 into the swirl of the acid and set the timer for 8 minutes. If KNO_3 is not dissolved in 8 minutes, dissolving a longer time and/or raising the temperature will aid in complete dissolving.

NOTE: The swirl should be as fast as possible without causing a vortex. A vortex will introduce air into the solution and cause oxidation of the $FeS0_4$.

(f.) Reweigh bottle to obtain weight of sample by difference to the nearest 0.1 mg.

(g.) Lower the electrodes into the acid.

NOTE: The electrodes should have a minimum of 4 hours conditioning time to clean H_2SO_4 before using.

(h.) After 8 minutes, start titrating. Do not titrate so fast that heat builds up in the sample and causes localized oxidation of the sample. Overall titration time should be approximately 6 - 8 minutes.

(i.) As the end point is neared, slow the titration to smaller volume increments and allow adequate time for each increment to completely disperse before adding the next one.

(j.) When the mv reading has reached the preselected end point, stop the titration and record the volume of $FeS0_4$.

(k.) Raise the electrodes. Wipe excessive acid from the electrodes with a tissue.

(l.) Rinse the electrodes with a beaker of clean H_2SO_4 and allow excessive H_2SO_4 to drain into the beaker while preparing tor the next sample. Wipe the electrodes with a tissue before retrieving beaker.

(m.) Calculate the nitrogen equivalent factor (F) for the titrant by the following formula:

$$F = \frac{g\ N}{ml\ FeS0_4\ solution} = \frac{(wt\ of\ KNO_3\ in\ gms) \times (0.13855)}{ml\ FeS0_4\ solution}$$

(n.) Place sufficient nitrate ester sample for the determinations to be made into a weighing bottle and record weight to nearest 0.1 mg. Place a portion equivalent to a 0.35 to 0.40 g sample for nitroglycerin (for DEGN the sample size should be from 0.35 to 0.45 g) into a dry 250 ml titration beaker.

(o.) Before adding the stirring bar, pour 150 ml of H_2SO_4 into the beaker. Then, carefully slide the stirring bar down the side of the beaker.

(p.) Place the beaker in the cooling bath and on the stirring unit and start the stirrer.

(q.) Reweigh the weighing bottle to find the weight of the NG sample by difference to the nearest 0.1 mg.

(r.) Lower the electrodes into the beaker containing the nitrate ester sample.

(s.) Since the sample is a liquid, there is no dissolving time and the titration may begin immediately. The rate of addition at $FeSO_4$ titrant should be controlled to give an overall titration time of 6 to 8 minutes.

(t.) As the end point is neared, slow the titration to the lowest volume increment practical and allow time for each increment to completely disperse before adding the next one.

(u.) When the mv reading has reached the preselected end point. Record the volume of FeSO4 (in ml) required for the titration.

(v.) Raise the electrodes; wipe excessive acid from the electrodes with a tissue.

(w.) Rinse the electrodes with a beaker of clean H_2SO_4 and allow excessive H_2SO_4 to drain into the beaker while preparing for the next sample. Wipe the electrodes with a tissue before retrieving the beaker.

(x.) Calculate the percent Nitrogen in the sample. Multiple determinations for percentage nitrogen may be performed and averaged to provide the final outcome for a manufacturing lot or unit of use.

(y.) Calculation:

$$\%\ N = \frac{(ml\ FeSO_4) \times (F) \times (100.0)}{wt\ of\ sample\ in\ gms}$$

(z.) When the electrodes are not in use, they will be kept submerged in a beaker of clean H_2SO_4, or distilled water, but not the titrant dispensing tip. The end of the dispensing tip will be capped to prevent evaporation of the solution. This evaporation will cause crystallization of the $FeSO_4$ and clog the tip.

4.3.4 Determination of stability. The stability shall be determined as follows: Filter a portion of the nitroglycerin sample through two thicknesses "S & 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 or stainless steel 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 3/8 inch wide. Moisten a horizontal section in the upper 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 or equivalent. 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 any of 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.

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.

6.3 Changes from previous issue. The margins of this specification are marked with vertical lines to indicate where changes from the previous issue were made. This was done as a convenience only and the Government assumes no liability whatsoever for any inaccuracies in these notations. Bidders and contractors are cautioned to evaluate the requirements of this document based on the entire content irrespective of the marginal notations and relationship to the last previous issue.

6.4 Subject term (key word) listing.

Propellants
Polymerized glycerin
Nitrate ester

Preparing activity:
Army – AR
(Project 1376-2009-006)

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at http://assist.daps.dla.mil.