

INCH-POUND

MIL-T-248C
 INT. AMENDMENT 3 (AR)
 18 May 90
 SUPERSEDING
 INT. AMENDMENT 2 (AR)
 16 November 1982

MILITARY SPECIFICATION

TRINITROTOLUENE (TNT)

This Interim Amendment is approved for use within the U.S. Army Armament, Munitions and Chemical Command, with Military Specification MIL-T-248C dated 8 November 1974.

PAGE 2

- 2.1 Standards, Military: Delete "(ABC-STD-105)" which appears at end of Title of document MIL-STD-105.

PAGE 3

- 3.1 Table I: Under "Acidity" delete:

| | |
|-------------|-------------|
| Type I | Type II |
| "0.005 max. | 0.005 max." |

and substitute the following:

| | |
|------------|------------|
| Type I | Type II |
| "0.02 max. | 0.02 max." |

PAGE 4

- 3.5 First Article Testing. Delete in its entirety and substitute the following:

"3.5 Process evaluation. This specification makes provisions for process evaluation testing. Submission of the process evaluation sample by the contractor shall be as specified in the contract."

AMSC N/A

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FSC 1376

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4.1.1 Classification of inspection. Delete "First Article Inspection" and substitute "Process Evaluation Testing (see 4.2)."

4.2 First article inspection. Delete "First Article Inspection" and substitute "Process Evaluation Testing (see 4.2.1.1)."

4.2.1 Submission. Delete in its entirety and substitute the following:

"4.2.1 Submission."

* 4.2.1.1 Continuous process evaluation. Delete in its entirety and substitute the following:

"4.2.1.1 Continuous process evaluation. Prior to initiation of sustained production and after the process has been completely debugged the contractor shall contact the Contracting Officer (see 6.2) for process evaluation testing in accordance with the provisions of 4.3.3.1. The testing shall apply only to TNT that has been produced by the contractor using the same production process, procedures, and equipment that will be used in fulfilling the contract. All materials, except packaging, shall be obtained from the same sources as will be used in regular production. The process evaluation testing shall be witnessed by the Government representative as designated by the Contracting Officer. The process evaluation testing shall also apply wherever a change occurs in the manufacturing process, material used, drawing, specification or source of supply as to significantly affect product uniformity as determined by the Government and whenever there is a lapse in production for a period in excess of 90 days. Prior to submission, the contractor shall inspect the sample to the degree necessary to assure that it conforms to the requirements of the contract and submit a record of this inspection with the sample. A sample known not to conform to the requirements of the contract will not be submitted unless specifically authorized by the Contracting Officer."

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- * 4.2.1.2 Batch process evaluation. Delete in its entirety and substitute the following:

"4.2.1.2 Batch process evaluation. Prior to initiation of sustained production the contractor shall classify an initial production sample as designated by the Contracting Officer (see 6.2) for evaluation in accordance with the provisions of 4.3.3.2. All samples submitted shall have been produced by the contractor using the same production processes, procedures, and equipment as will be used in fulfilling the contract. All materials, except packaging, shall be obtained from the same sources as will be used in regular production. The sample shall be accompanied by certificates of analysis. An initial production quantity, or portion thereof, as directed by the Contracting Officer, shall also be submitted wherever there is a lapse in production for a period in excess of 90 days or wherever a change occurs in the manufacturing process, material used, drawing, specification or source of supply as to significantly affect product uniformity as determined by the Government. Prior to submission, the contractor shall inspect the sample to the degree necessary to assure that it conforms to the requirements of the contract and submit a record of this inspection with the sample. A sample containing known defects will not be submitted unless specifically authorized by the Contracting Officer."

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- * 4.3.1.1.2 Serial lot. Delete in its entirety and substitute the following:

"4.3.1.1.2 Serial lot. A lot shall consist of the maximum amount of TNT loaded from one loading dock in a 24-hour period, manufactured under a continuous set of conditions without a significant process interruption or change during the manufacture of the lot (each transportation unit used will be identified as a subplot). A significant process interruption or change may be defined by the contracting officer. Boxes shall be color-coded to identify TNT by line within each lot. Each box shall be serially marked."

Add a new paragraph 4.3.2.4.1:

CLASSIFICATION OF CHARACTERISTICS

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| PARAGRAPH | TITLE | SHEET 1 OF 1 | | DRAWING NUMBER | |
|-----------------|--|----------------------|-----------------------|-----------------------------|-----------------------------|
| 4.3.2.4.1 | Fiberboard Box (Single Piece) Reusable | | | 9257923 | NEXT HIGHER ASSEMBLY N/A |
| CLASSIFICATION | EXAMINATION OR TEST | CONFORMANCE CRITERIA | REQUIREMENT PARAGRAPH | INSPECTION METHOD REFERENCE | |
| <u>Critical</u> | None defined | | | | |
| <u>Major B</u> | | | | | |
| 131 | Tears longer than 1 inch along score lines if not at open edge. | 0.40% | dwg. 7548645 | Gage | |
| 132 | Tears or cuts longer than 1/2 inch if at open edge. | 0.40% | dwg. 9257923 | Gage | |
| 133 | Tears, cuts or holes which would expose bag liner to view. | 0.40% | dwg. 9257923 | Visual | |
| 134 | Noticable weakening from exposure to moisture or weather. | 0.40% | dwg. 9257923 | Visual | |
| 135 | Contamination from explosive material, oil or grease on interior or exterior (8). | 0.40% | dwg. 7548644 | Visual | |
| 136 | Defacing interfering with legibility of printed matter making further marking impracticable. | 0.40% | | Visual | |
| 137 | Failure of staples, unless replaced. | 0.40% | 5.2 | Visual | |
| 138 | A slight amount of explosive dust on the interior may be permitted to the extent that it does not create a safety hazard or result in classification of empty boxes as dangerous material thus causing higher freight rates. | 0.40% | 5.3 | Visual | |
| 139 | More than 50 percent failure of any glued joint. | 0.40% | 5.2.1 4.3.3.3 | Visual Visual/Manual | |
| NOTES: | | | | | |

AWSMC Form 1570b, 1 Jul 89

Replaces 1570, 1 Feb 85, which may not be used.

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4.3.3 Testing. Delete in its entirety and substitute the following:

"4.3.3 Testing. The product shall be submitted for inspection to determine compliance with all the requirements in section 3. Failure of a sample to comply with any of the requirements shall be considered a major defect and will result in rejection of the lot."

4.3.3.1 Continuous nitration process sampling. Delete in its entirety and substitute the following:

"4.3.3.1 Continuous nitration process sampling.

4.3.3.1.1 Pre-production sampling. After the debugging process has been completed samples shall be selected every hour for the first 8 hours production from each line and subjected to the test specified in 4.4. If any sample fails to comply with the test requirements, the production shall be rejected and the contractor shall go through the debugging process again to bring the process with in specification."

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* 4.3.3.1.2 Delete in its entirety and substitute the following:

"4.3.3.1.2 Samples for regular production (lot acceptance). After the testing for the Pre-production has been completed, samples that represent a sub-lot (a transportation unit regardless of its size) shall be selected from each line and subjected to the solidification test specified in 4.4.2 and tests specified in 4.4.1 and 4.4.3 through 4.4.9. If any samples fail to comply with the test requirements, the sub-lot shall be rejected. The sub-lot data will be recorded on lot acceptance sheet of each lot."

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4.4.2 Determination of solidification point. Delete in its entirety and substitute the following:

"4.4.2 Determination of solidification point. The solidification point shall be determined in accordance with Method 210.1 of MIL-STD-650 except that a National Bureau of Standard Thermometer with a range of 79-82 degrees Centigrade shall be used."

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4.4.4 Acidity. Delete in its entirety and substitute the following:

"4.4.4 Acidity. Transfer an accurately weighed portion of 10.0 grams of sample to 250 mL glass-stoppered iodine flask or equivalent. Add 40 mL methylene chloride from a graduated cylinder to the sample and also to an empty flask which will serve as a blank. Stopper the flask. Swirl the sample flask until dissolution is complete. Put 0.75 mL (approx. 20 drops) of bromothymol blue indicator in a 100 mL graduated cylinder and dilute to the mark with CO_2 - free distilled water. Stopper the flask containing the CO_2 - free water. Transfer the 100 mL of water containing the indicator to the blank flask and replace stopper. Repeat this procedure with the sample flask. Swirl both sample and blank flasks vigorously for 10-20 seconds to ensure interaction of methylene chloride and water layers.

NOTE: Too vigorous swirling or shaking must be avoided or an emulsion will be produced which may take hours to disperse.

Titrate the blank solution first. If the lower (aqueous) layer is blue, add a measured amount of 0.01 N H_2SO_4 dropwise until it turns green or yellow and add an equal amount to the sample. If the solution is green or yellow at the start, begin to titrate with 0.01 N NaOH. Add the NaOH dropwise, stoppering the flask after each addition and swirling vigorously for 5-10 seconds (see note above). The end point is taken as a blue color which persists for 2 minutes after the methylene chloride and water have separated into distinct layers and which persists after one additional 5-10 second swirling.

NOTE: The blue color may fade somewhat or acquire a trace of green coloring after the final swirl but this is acceptable.

The sample is now titrated in a manner similar to that of the blank titration. The end point is a persistent blue color as described in the blank determination but care must be taken to look through the aqueous layer horizontally against a white or colorless background since transmitted or reflected light from the yellow methylene chloride solution will cause a green coloration. In addition, incomplete separation of methylene chloride and water may result in a cloudiness which may also impart a slight green cast to the aqueous layer.

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Experience with the method should allow a determination of the end point to within ± 0.05 mL NaOH.

$$\% \text{ Acidity (as H}_2\text{SO}_4) = \frac{4.9 (A-B) N}{W}$$

Where:

A = mL NaOH for sample
B = mL NaOH for blank
N = Normality of NaOH
W = Sample wt., grams"

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4.4.5 Alkalinity. Delete in its entirety and substitute the following:

"4.4.5 Alkalinity. The specimen shall be considered unsatisfactory with respect to alkalinity when B is greater than A in the calculations under "Acidity", para. 4.4.4."

4.4.6 Insoluble matter. Delete the use of "benzene" as the solvent in the determination of insoluble matter and substitute "methylene chloride".

4.4.7.1.2 Reagents. Delete all references to "benzene" and substitute "dimethylformamide (DMF)".

4.4.7.1.3 Preparation of standard solutions. Delete this title in its entirety and substitute "Preparation of standard sodium solutions".

4.4.7.1.3.1 Add a new paragraph and number it 4.4.7.1.3.1 as follows:

"4.4.7.1.3.1 Stock solution (sodium chloride solution). Preparation: Accurately weigh 127 mg of reagent grade sodium chloride (or equivalent of other suitable sodium standard) to the nearest 0.1 mg on an analytical balance and transfer to a clean dry 1000 mL volumetric flask. Dissolve in sodium-free distilled water and make up to the mark. This solution contains approximately 50 parts per million of sodium. Then transfer a 10 mL aliquot from the 1000 mL volumetric flask into another 1000 mL volumetric flask and dilute to the mark with sodium-free distilled water."

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4.4.7.1.3.2 Standard solutions. Delete in its entirety and substitute the following:

"4.4.7.1.3.2 Standard sodium solutions. Preparation: Only sodium-free distilled water shall be used in these solutions. Transfer 2 mL, 4 mL, 6 mL and 8 mL aliquots of the stock solution into 100 mL volumetric flasks and dilute to volume with distilled water. These solutions will contain approximately 0.01, 0.02, 0.03, and 0.04 ppm sodium, respectively. The concentration of the standards may be adjusted to cover the ranges experienced in the test samples. Calculate the exact concentration according to the following:

a. The following steps form the basis for the calculation of concentration:

$$\begin{array}{l} \text{mg of Na} \\ \text{in sample} \end{array} = \text{mg NaCl used} \times \frac{\text{AW of Na}}{\text{MW of NaCl}}$$

where:

AW = atomic weight
MW = molecular weight

Initial ppm may be expressed as:

$$\text{ppm Na} = \frac{\text{mg of Na}}{\text{total dilution volume}} \times 1000$$

b. The second dilution may be expressed as follows:

$$\begin{array}{l} \text{ppm Na after} \\ \text{second dilution} \end{array} = \frac{\text{VA(1)}}{1000 \text{ mL}} \times \text{initial ppm}$$

where VA(1) = volume to aliquot of stock solution

c. The exact concentration of sodium is then:

$$\begin{array}{l} \text{ppm Na after} \\ \text{final dilution} \end{array} = \frac{\text{VA(2)}}{100 \text{ mL}} \times \text{ppm found in (b) above}$$

where VA(2) = volume of each aliquot taken in
4.4.7.1.3.2."

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4.4.7.1.3.3 Add a new paragraph and number it 4.4.7.1.3.3 as follows:

"4.4.7.1.3.3 Standard solutions (dimethylformamide). Preparation: Transfer by buret 1/2 mL, 1 mL, and 2 mL aliquots of the stock solution into 10 mL volumetric flasks. Add 9.5 mL, 9.0 mL and 8.0 mL of distilled water to the respective flasks and dilute each flask to volume with DMF. The concentration of the standards may be adjusted to cover the ranges experienced in the test samples. These solutions will contain approximately 0.025, 0.050, and 0.10 ppm sodium. Prepare a blank using 10.0 mL of distilled water diluted to the mark in a 100 mL volumetric flask with DMF. Use the same source of water and DMF as used for the standard solution."

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4.4.7.1.5 Calibration of apparatus with standard solutions. Delete in its entirety and substitute the following:

"4.4.7.1.5 Calibration of apparatus with standard solutions.

4.4.7.1.5.1 Sodium-free distilled water. Place sodium-free distilled water in aspirator cup and aspirate into the flame. Record absorbance. Repeat with the nominal 0.01, 0.02, 0.03, and 0.04 ppm standard solutions and record absorbance. Prepare a graph plotting absorbance versus exact concentration."

4.4.7.1.5.2 Dimethylformamide. Place the DMF/distilled water blank in aspirator cup and aspirate into the flame. Record absorbance. Repeat with the nominal 0.025, 0.050, and 0.10 ppm standard solutions and record absorbance. Prepare a graph plotting absorbance versus exact concentration."

4.4.7.1.6 Test procedures. Delete the first sentence and substitute the following:

"4.4.7.1.6 Test procedures. Prepare and test a sample of TNT by one of the following methods:"

4.4.7.1.6.2 Delete in its entirety and substitute the following:

"4.4.7.1.6.2 Sodium by atomic absorption. Prepare the sample by placing 5 mg of the TNT sample, weighed to within 0.2 mg, into a 100 mL volumetric flask and add 10 mL of sodium-free water. Add DMF and shake until the sample is dissolved. Adjust to volume with DMF in a constant temperature bath (20 ± 1 degrees Centigrade). Place the standards and the samples

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in aspirator cup. Using an atomic absorption spectrometer, measure the absorbance of the specimen solution. Determine the ppm sodium in the specimen solution from the calibration curve. To convert the weight of the TNT sample from mg (as measured) to g (as shown in the formula below divide weight by 1000). Then calculate the percent sodium in the TNT as follows:

$$\text{Percent Sodium} = \frac{\text{ppm sodium in specimen solution}}{\text{TNT weight (g)} \times 100}$$

4.4.7.2 Alternate method, colorimetric test. Delete the first sentence in its entirety.

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Add the following paragraphs:

* 4.4.7.3 Alternate method, sodium from TNT dissolution in dimethylformamide (DMF) and atomic absorption spectrophotometry.

* 4.4.7.3.1 Preparation of washed TNT. Add approximately 300 g flake TNT to a 2 L beaker and cover with approximately 1 L of sodium-free distilled water. Heat on a steam bath until the TNT has melted. Stir the suspension for 4 to 5 minutes with a teflon rod. Decant the water. Repeat twice more.

* 4.4.7.3.2 Preparation of sodium solution standards. A 1000 mg/L sodium solution is prepared by weighing 2.54 g (to ± 0.1 mg) dried reagent grade NaCl and quantitatively transferring it to a 1000 mL volumetric flask. Dissolve in sodium-free distilled water and make up to volume with sodium-free distilled water. A 10 mg/L sodium solution is prepared by pipetting 10 mL of the 1000 mg/L solution to a 1000 mL volumetric flask and diluting to volume with sodium-free distilled water. The 0, 0.2, 0.5, and 1.0 mg/L sodium solutions are prepared by first adding 85 mL of DMF and 5.00 g of washed TNT from 4.4.7.3.1 to each of four 100 mL volumetric flasks. Next add by pipet 0, 2.0, 5.0 and 10.0 mL of the 10 mg/L solution to the separate flasks prepared above. Dilute each to volume with sodium-free distilled water.

$$[\text{Na}] \text{ (mg/L)} = \frac{W \text{ (g)} \times 23 \times 1000 \times V \text{ (mL)}}{58.5 \times 100 \times 100} = W \times V \times 0.0393$$

where: Na = Concentration of sodium in standard solutions for calibration in mg/L.

W = Weight of NaCl in grams.

V = Volume of 10 mg/L sodium solution used to prepare the 0, 0.2, 0.5, and 1.0 mg/L solutions in mL.

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4.4.7.3.3 Sample preparation and sodium determination. Weigh 5.00 g (to nearest mg) of TNT sample and transfer quantitatively to a 100 mL volumetric flask. Add 85 ml reagent grade DMF and agitate until completely dissolved. Complete to volume with sodium-free distilled water. Set up and adjust the atomic absorption spectrophotometer according to the manufacturer's instructions for sodium determination. Zero the instrument with the blank solution ("0" prepared in 4.4.7.3.2 above). Measure the standard solutions and sample. Construct a calibration curve with the standard readings and determine the concentration in the sample from the reading obtained. Calculate % Na in sample as follows:

$$\% \text{ Na} = \frac{C \text{ (mg/L)} \times 100 \times 100}{1000 \times 1000 \times W \text{ (g)}} = \frac{C}{100 \times W}$$

where C = Sodium concentration in sample from calibration curve in mg/L.

W = Weight of TNT sample added to 100 ml flask in grams."

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6.2c Provision for submission of first article samples. Delete "first article samples" and substitute "Process evaluation samples".

6.4 Beckman flame spectrophotometer. Delete in its entirety.

6.6 Flake thickness. Delete "first article testing" and substitute "Process evaluation testing".

The margins of this amendment are marked with an asterisk or vertical lines to indicate where changes (additions, modifications, corrections, deletions) from the previous amendment 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 amendment.

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