INCH-POUND

MIL-E-46495A (AR)
AMENDMENT 4
08 February 1990
SUPERSEDING
AMENDMENT 3
2 NOVEMBER 1982

MILITARY SPECIFICATION

EXPLOSIVE COMPOSITION HTA-3

This Amendment forms a part of Military Specification MIL-E-46495A (MU), dated 30 November 1964, and is approved for use by the US Army Armament Munitions Chemical Command, Department of the Army and is available for use by all Departments and Agencies of the Department of Defense.

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3.2 Composition. Delete Table II in its entirety and substitute the following:

"TABLE II

Material	Percent Type I	Percent Type II	Applicable Paragraphs
TNT	29.0 ± 2.0	28.65 ± 2.0	4.3.1.1
HMX	49.0 ± 2.0	49.0 ± 2.0	4.3.1.2
Aluminum	22.0 ± 2.0	22.0 ± 2.0	4.3.1.4
Calcium Silicate	---	0.35 ± 0.15	4.3.1.3"

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4.2.3 Testing. Add a new paragraph as follows:

"4.2.3 Testing:

PRECAUTION WARNING

This specification covers sampling and testing of toxic or hazardous materials. Accordingly it is emphasized that all applicable safety rules, regulations and procedures must be followed in handling and processing these materials."

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4.3.1.1 TNT. Delete in its entirety and substitute the following:

"4.3.1.1 TNT. Grind the sample to a fine powder using remote grinding equipment and observing proper safety precautions. Store ground sample in a conductive rubber container. Transfer an accurately weighed portion of approximately 2.5 grams into a tared 50 mL high form medium porosity filtering crucible (weigh to the nearest 0.0001 gram). Extract with 30-40 mL portions of toluene saturated with HMX until a total volume of at least 250 mL has been used. Allow each portion to soak the crucible contents for approximately five (5) minutes. (The completeness of the TNT extraction may be checked by testing the filtrate with Webster's Reagent (2 percent potassium hydroxide in ethanol). A dark pink color indicates TNT is present. Draw air through the crucible until it appears dry. Dry the crucible and contents in an oven maintained at 100 + 5 degrees Centigrade for one (1) hour. Cool the crucible and contents in a desiccator and weigh. Retain the crucible and contents for the determinations which follow.

Calculate the TNT content of the sample as follows:

$$\text{Percent TNT} = \frac{100\,((A-B) - MW)}{W - (MW)}$$

Where: A = Weight of crucible and sample, g
B = Weight of crucible and residue after toluene extraction, g
W = Weight of sample, g
M = Moisture content in the sample expressed as a decimal (4.3.2)"

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4.3.1.2 HMX. Delete in its entirety and substitute:

"4.3.1.2 HMX. Place the crucible and content retained from the TNT determination in a beaker or crystalizing dish and add acetone to both the crucibles and the container so that the crucibles are about one third full. Heat the container and crucibles on a steam bath or steam table for about five minutes. Remove the crucibles from the steam bath and while still hot draw the acetone through the crucibles by vacuum filtration and wash with more acetone to remove any HMX crystallized on the inside walls of the crucible. Wash until the HMX is no longer visible on the crucible walls. Repeat the heating and filtration cycle three (3) times. Dry the crucibles and contents for thirty (30) minutes in an oven at 100 ± 5 Degrees Centigrade. Cool in a desiccator and weigh.

Calculate the HMX content of the sample as follows:

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

Where: A = Weight of residue and crucible after the toluene extraction, retained from 4.3.1.1, g
B = Weight of residue and crucible after the acetone extraction, g
W = Weight of sample, g
M = Moisture content, expressed as a decimal (4.3.2)"

4.3.1.3 Calcium silicate. Delete in its entirety and substitute:

"4.3.1.3 Calcium silicate. During the analysis for the calcium silicate content of HTA-3 explosive, blank determinations shall be run on both the calcium silicate and the aluminum. For this purpose calcium silicate and aluminum identical to that used in the manufacture of the sample being analyzed shall be used. A quantity of 0.55 ± 0.05g of aluminum accurately weighed to the nearest 0.0001g shall be placed in a tared, filtered crucible. A quantity of 0.0088 ± 0.0002g of calcium silicate accurately weighed to the nearest 0.0001g shall be placed in a second tared, filtered crucible. Each of the blank samples shall be subjected to the same, analytical procedures as the residue of the explosive sample remaining after the HMX extraction. Wash the residue remaining in the filtering crucible after the HMX determination into a 250 mL teflon or heat-resistant plastic

beaker with an aqueous 15 percent sodium hydroxide solution (a plastic squeeze bottle works best). Cover the beaker with a teflon (or plastic) cover.

Add 10 mL of 15 percent sodium hydroxide solution to the crucible and allow the solution to react with aluminum remaining on the fritted glass and inside wall of the crucible. During the reaction, manipulate the crucible by slightly tilting and rotating to allow the solution to come in contact with the residue adhering to the inside wall of the crucible. Set the crucible in a glass or plastic container during the reaction and repeat the tilting and rotation procedure as often as is necessary to be sure aluminum residue remains.

After the reaction is complete, transfer the crucible contents into the teflon beaker with 15 percent sodium hydroxide solution. Immediately aspirate the crucible with vacuum and wash several times with hot distilled water. After rinsing, set the crucible aside until it is needed for the final calcium silicate filtration.

After the reaction of the crucible contents in the teflon beaker subsides (about 2 - 5 minutes), heat the beaker and contents in a steam bath for five (5) minutes.

After heating, add enough distilled water to triple the volume of solution in the teflon beaker. Allow the solution to stand until an orange precipitate forms (30 minutes to an hour).

Quantitatively transfer the contents of the beaker to the filtering crucible reserved for this purpose using a stream of distilled water. Aspirate the crucible with vacuum to remove the water and alternately rinse with distilled water and aspirate contents of the crucible three more times. Rinse with acetone and aspirate with vacuum. Repeat this acetone rinse and aspiration procedures two more times. After the final rinse aspirate the contents with vacuum until the crucible and contents appear to be dry. Dry the crucible and contents in an oven maintained at 100 ± 5 Degrees Centigrade for one half hour, cool in a desiccator and weigh to the nearest 0.0001g. Backflush the crucible in the following steps:

1. Two (2) times with reagent grade sulfuric acid.
2. Four (4) times with lab grade water.
3. Two (2) times with 5% sodium hydroxide solution.

4. Four (4) times with lab grade water.
5. Two (2) times with soap solution (preferably 30% FL 70 solution).
6. Seven (7) times with hot (85 +/- 10 Degrees Centigrade) lab grade water.
7. Two (2) times with acetone.

Rinse several times with acetone aspirate to dryness with the aid of vacuum. Dry the crucible in an oven at 100 ± 5 Degrees Centigrade for thirty minutes, cool in a desiccator and weigh. (This weight is required in order to determine if the tare of the crucible has changed due to sodium hydroxide etching.)

Calculate the calcium silicate content of the sample as follows:

Calculate aluminum residue and calcium silicate loss factors from the following relations:

$$F' = \text{Aluminum factor} = \frac{W1' - W2'}{W3'}$$

Where: W1' = Weight of aluminum blank crucible and residue after the sodium hydroxide extraction, g

W2' = Weight of aluminum blank crucible after cleaning with solution, g

W3' = Net weight of aluminum blank, g

F" = Calcium Silicate factor

Where: W1" = Weight of calcium silicate blank crucible and residue after the sodium hydroxide extraction, g

W2" = Weight of calcium silicate blank crucible after cleaning with cleaning solution, g

W3" = Net weight of calcium silicate blank, g

The aluminum residue from the explosive sample remaining on the crucible after sodium hydroxide extraction is expressed as:

$$Alr = AlF' = \frac{F' \quad W3 - W1 + W2 - Wo}{1 - F'}$$

Where: Wo = Original tare weight of crucible, g

W1 = Weight of crucible and residue after sodium hydroxide extraction, g

W2 = Weight of crucible after cleaning with cleaning solution, g

W3 = Weight of crucible and residue after HMX extraction

A1 = Weight of aluminum in sample

The calcium silicate weight, corrected for solubility, crucible tare change, and aluminum residue, can then be expressed as:

C = Calcium silicate content = (W1-W2-A1r)F"

and percent calcium silicate is

$$\%C = \frac{100\ C}{W(1-M)}$$

Where: W = Weight of sample

M = Moisture content of sample expressed as a decimal."

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 the notations. Bidders and contractors are cautioned to evaluate the requirements of this document based on the entire contents irrespective of the marginal notations and relationship to the last previous amendment.

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