INCH-POUND
MIL-E-82756(OS)
AMENDMENT 1
14 March 1995

MILITARY SPECIFICATION

EXPLOSIVE, PLASTIC-BONDED, CAST PBXN-103

This Amendment forms a part of MIL-E-82756(OS), dated 4 April 1994, and is approved for use by the Department of the Navy and is available for use by all Departments and Agencies of the Department of Defense.

PAGE 13

After paragraph 4.6.2.6 and before 4.6.3 add:

"4.6.2.7 Alternate test method for cured PBXN-103 mixed plasticizer analysis using High Performance Liquid Chromatography (HPLC).

4.6.2.7.1 Equipment. The following equipment or equivalent shall be used:

a. High performance liquid chromatograph equipped with ultraviolet (UV) detector, auto sampler, column oven and data handling system

b. HPLC C-18 reverse phase column, 25 cm long, 4.6 mm inner diameter

c. Volumetric flasks; 25 and 100 mL

d. Pipets, volumetric, Class A, 1 and 100 mL

e. Auto sampler vials with cap and PTFE faced septa

f. Disposable syringe filters, 25 mm diameter, PTFE membrane, 0.45 micron porosity.

4.6.2.7.2 Reagents. The following reagents shall be used:

a. Tetrahydrofuran (THF), HPLC grade

b. Acetonitrile (ACN), HPLC grade

c. Water, suitable for HPLC

d. Resorcinol, analytical grade

e. Trimethylol Ethane Trinitrate (TMETN), production grade

f. Triethylene Glycol Dinitrate (TEGDN), production grade

g. Ethyl centralite (EC), analytical grade

h. 2, 6-Diethylaniline, analytical grade, 99.5 percent purity.

4.6.2.7.3 Standard preparation. Prepare standard solutions as follows:

a. Resorcinol stock solution: accurately weigh 0.3 grams of resorcinol to ± 0.1 mg into a 100-mL volumetric flask. Bring to volume with ACN and mix well.

b. Internal standard stock solution: accurately weigh 5.0 grams of 2, 6-diethylaniline to ± 0.1 mg into a 100-mL volumetric flask. Dissolve, bring to volume with ACN, and mix well.

c. Mixed plasticizer stock solution: accurately weigh 0.025 grams TEGDN, 0.23 grams TMETN and 0.015 grams EC to ± 0.1 mg into a 100 mL volumetric flask. Pipet 1 mL of the resorcinol stock solution into this flask. Bring to volume with ACN and mix well. Accurately pipet 1 mL of the internal standard stock solution into the 100-mL volumetric flask containing the mixed plasticizer ingredients. Total volume should be 101 mL.

d. Analysis standard: accurately pipet 1 mL of the mixed plasticizer stock solution into a 25-mL volumetric flask and bring to volume with ACN.

4.6.2.7.4 Sample preparation. Prepare sample as follows. Samples should be prepared in duplicate.

a. Cut the sample into small pieces.

b. Accurately weigh a 1-gram sample to ± 0.1 mg into a 100-mL stoppered flask.

c. Accurately pipet 100 mL of tetrahydrofuran (THF) into the flask. Accurately pipet 1 mL of the internal standard solution into the same flask and stopper flask tightly. Allow the mixed plasticizer and nitrocellulose ingredients to dissolve. Shake the sample to thoroughly mix the dissolved ingredients.

d. Accurately pipet 1 mL of this solution into a 25-mL volumetric flask and bring to volume with ACN.

e. Filter sample solution through a 0.45 micron syringe filter before analysis.

4.6.2.7.5 Instrument set up. Set up the instrument in accordance with the manufacturer's manual and as follows:

a. Instrument linearity should be checked by using three different standard concentration levels to cover the expected sample concentration range.

b. The mobile phase composition should be 65% ACN and 35% water. Degas mobile phase before use. The mobile phase composition may be adjusted by the operator to optimize chromatographic system and to insure baseline separation of all peaks.

c. The UV detector is set at 215 nm.

d. Column oven temperature recommended setting is 40°C. The oven temperature setting may be changed by the operator in accordance with instrument instructions. The selected temperature shall be constant throughout the analysis to insure baseline separation of all peaks.

e. **Mobile phase flow rate is 1.0 mL/min.**

f. **All injections are 10 microliters.**

g. Obtain a chromatograph of the standard at the beginning of the analysis and another after all samples are tested. The two results shall be within 2 percent. No more than five samples are to be chromatographed between standard injections.

h. Perform duplicate runs on all standards and samples. Ensure that the duplicate runs agree within 2 percent.

4.6.2.7.6 Calculations. Calculate weight percent of each analyte in sample as follows:

$$Percent\ analyte = \frac{(W_a)(A_1)(A_s)(D)}{(W_s)(A_a)(A_2)}$$

where:

W_a = Weight of analyte, g, from 4.6.2.7.3 (a) for resorcinol and 4.6.2.7.3 (c) for TMETN, TEGDN, and EC
W_s = Sample weight, g, from 4.6.2.7.4
A_1 = Average area of 2, 6-diethylaniline peak in standard
A_2 = Average area of 2, 6-diethylaniline peak in sample
A_a = Average area of analyte peak in standard
A_s = Average area of analyte peak in sample
D = Dilution factor for each analyte

(D = 1 for resorcinol: D = 100 for EC, TMETN and TEGDN)"

Preparing Activity:
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