MILITARY STANDARD

PYROTECHNICS: SAMPLING, INSPECTION AND TESTING

TO ALL HOLDERS OF MIL-STD-1234:

1. The following pages of MIL-STD-1234 have been revised and supercede the pages listed:

NEW PAGE	DATE	SUPERSEDED PAGE	DATE
IV	28 May 1973	IV	30 March 1967
V	28 May 1973	V	30 March 1967
VI	28 May 1973	VI	30 March 1967
I	28 May 1973	I	22 June 1962

2. The following methods have been revised and supersede the methods listed:

REVISED METHOD	DATE	SUPERCEDED METHOD	DATE
301.6.1	28 May 1973	301.6	22 June 1962
503.1.1	28 May 1973	503.1	22 June 1962

3. The following is a cumulative list of earlier changes:

a. Superseded pages.

NEW PAGE	DATE	SUPERCEDED PAGE	DATE
i i	18 Dec 65	i i	22 Jun 62
i v	18 Dec 65	i v	22 Jun 62
v	18 Dec 65	v	22 Jun 62
i i	30 Mar 67	i i	18 Dec 65
i i i	30 Mar 67	i i i	22 Jun 62
i v	30 Mar 67	i v	18 Dec 65
v	30 Mar 67	v	18 Dec 65
v i	30 Mar 67	v i	22 Jun 62

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b. Superseded Methods.

NEW METHOD	DATE	SUPERSEDED METHOD	DATE
101.1.1	18 December 65	101.1	22 June 62
101.2.1	18 December 65	101.2	22 June 62
101.3.1	18 December 65	101.3	22 June 62
101.4.1	18 December 65	101.4	22 June 62
102.1.1	18 December 65	102.1	22 June 62
102.2.1	18 December 65	102.2	22 June 62

c. New methods

METHOD NO.	TITLE	DATE
101.5	Moisture (Karl Fischer Distillation Method)	18 December 65
101.6	Moisture (Electrolytic Hygrometer Method)	30 March 67
506.1	Friction sensitivity (By the Roto-Friction Method)	30 March 67

4. Retain notice and insert before table of contents.

b. Holders of MIL-STD-1234 will verify that page changes and additions indicated have been entered and will destroy the previous notice (notice page only). The latest notice (notice page) will be retained as a check sheet. This issuance, together with appended pages, is a separate publication. Each notice is to be retained by stocking points until the Military Standard is completely revised or cancelled.

CUSTODIANS:
ARMY–MU
NAVY–OS
AIR FORCE–11

REVIEW ACTIVITIES:
ARMY–MU
NAVY–OS
AIR FORCE–70

PREPARING ACTIVITY:
ARMY–MU

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SECTION 1
INTRODUCTION

1. SCOPE

1.1 This standard describes the general methods of sampling, inspecting, and testing pyrotechnics for conformance with the material requirements of the applicable pyrotechnic specification. In the event of conflict between these methods and those in the applicable pyrotechnic specification, the latter shall take precedence.

2. REFERENCED DOCUMENTS

2.1 The issue of the following documents in effect on the date invitation forbids form a part of this standard to the extent specified herein:

O-A-51 — Acetone.

RR-S-366 — Sieves, standard for testing purposes.

JAN-E-199 — Ether, diethyl.

MIL-E-463A — Alcohol, ethyl (for Ordnance use).

SECTION 2

SAMPLING AND INSPECTION

1. SCOPE

1.1 This section specifies the procedures for sampling pyrotechnics.

2. SAMPLING

2.1 Selection of the required test samples from each lot of the pyrotechnic after the pyrotechnic has been packed and sealed for shipment shall be as stated in the applicable specification.

3. PACKING and MARKING

3.1 Packing. Transfer samples to approved air tight containers, and seal the containers immediately. Keep the containers sealed and stored in a safe location at room temperature until ready for testing.

3.2 Marking. Label each pyrotechnic container with the following information:

(a) Pyrotechnic designation.

(b) Lot number.

(c) Lot size.

(d) Manufacturers name and plant designation.

(e) Contract or purchase order number.

4. TEST SPECIMEN

4.1 Ballistic samples. Select a sample as specified in the applicable pyrotechnic specification.

4.2 Chemical and physical test samples. Select an 8-ounce sample from each lot of pyrotechnic unless otherwise specified in the applicable pyrotechnic specification.

4.3 Surveillance test samples. Select a sample as specified in the applicable pyrotechnic specification.

5. INSPECTION

5.1 Before testing the pyrotechnic inspect the sample container to see that it is not broken, unstoppered, or otherwise damaged. Also check that it has been labeled correctly. Discard the contents of damaged or improperly labeled container, and report condition to the (Government inspector (or other proper official) at the plant.

METHOD 301.6.1

SAMPLE PREPARATION (SOXHLET EXTRACTION)

1. SCOPE

1.1 This method is used for extracting soluble ingredients from a pyrotechnic for use pyrotechnic for use in subsequent determinations.

2. SPECIMEN

2.1 The specimen shall consist of an accurately weighed portion of the pyrotechnic, suitable for the subsequent determinations.

3. APPARATUS

3.1 Soxhlet or equivalent extractor.

3.2 Extraction thimble.

3.3 Steam or hot water bath.

4. MATERIALS

4.1 Extraction solvent, such as ether, or methylene chloride as specified in the applicable method or pyrotechnic specification.

5. PROCEDURE

5.1 Transfer the specimen to an extraction thimble.

5.2 Place the thimble in a Soxhlet extraction apparatus.

5.3 Place 200 ml of the extraction solvent in the flask of the extraction apparatus.

5.4 Place the extraction apparatus on a steam or hot water bath and adjust the temperature of the bath so that the solvent drips from the end of the condenser at the rate of 2 to 3 drops per second.

5.5 Continue the extraction for at least 16 hours.

5.6 Test for completeness of extraction in the following manner:

(a) Allow the extraction to become almost full of the solvent.

(b) Separate the extractor from the flask.

(c) Draw one or two drops of the solvent from the extractor.

(d) Transfer the drops to a clean spot plate.

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(e) Qualitatively test the drops to determine if they contain any of the solvent soluble material.

5.7 Continue the extraction until all solvent soluble material has been removed.

5.8 When extraction is complete, dismantle the extractor and retain the extract and the residue for subsequent determinations.

METHOD 503.1.1
100°C. VACUUM STABILITY TEST

1. SCOPE

1.1 This method is used for comparing the resistance to decomposition by heat of any pyrotechnic with that of another; to indicate the presence or absence of unstable impurities in a pyrotechnic which is stable at 100°C.

2. SPECIMEN

2.1 The specimen shall consist of 5 gm of the dried pyrotechnic.

3. APPARATUS

3.1 Constant temperature bath.

Note. A bath consisting of a solution of glycerin and water (specific gravity 1.05 for the 100°C. test has been found satisfactory). Check the temperature of the bath by inserting a thermometer to the bottom of the empty heating tube (fig. 1) immersed in the bath. Adjust the temperature of the bath by adding one or the other of the constituent of the solution.

3.2 Vacuum stability measuring apparatus (fig. 1), heating tube.

3.3 Vacuum pump.

4. PROCEDURE

4.1 Standardize the vacuum stability measuring apparatus (fig. 1) as follows:

(a) Determine the volume of the heating tube by filling it with mercury from a buret until the mercury reaches the level at which it will contact the ground glass joint of the capillary tube.

(b) Determine the unit capacity of the capillary by placing exactly 10 gm of mercury in its cup, and manipulating the tube so that all the mercury passes into the long (85-cm) sect ion of the capillary. Be sure that the mercury remains as a continuous column. Measure the length of the mercury column at three positions in the long section of the capillary, and average the three measurements. Calculate the unit capacity of the capillary, using the following formula:

$$B = \frac{W}{13.54L}$$

where:

B = unit capacity of capillary, ml per mm.
W = weight of mercury, gm.
L = average length of mercury column, mm.

4.2 Place the dried specimen in the heating tube (fig. 1).

4.3 Coat the ground glass joint of the capillary tube with a light film of petroleum jelly, and make an airtight connection between the heating tube and the capillary by pressing the tube up against the capillary with a twisting motion.

4.4 Mount the apparatus on a rack so that the long section of the capillary is nearly vertical, and the cup at the bottom rests on a solid support.

4.5 Fill the cup with 7.0 ml of mercury and connect a vacuum line to the mouth of the cup.

4.6 Evacuate the capillary to a pressure of approximately 5 mm of mercury (absolute).

Note. Evacuation will be facilitated by tilting the apparatus until the capillary opening in the bottom of the cup is free of mercury.

4.7 When the pressure has been reduced to 5 mm of mercury, remove the vacuum line and allow the mercury to enter the capillary. Record the following data:

The total length in mm of the capillary tube minus the vertical height of the column of mercury in the cup before heating (E_1).

b. The height of mercury column above surface of mercury pool at beginning of test (H_1).

c. The temperature of room at brginning of test (t_1).

d. The barometric pressure in rnillimeters of mercury at beginning of test (P_1).

4.8 Immerse the heating tube in the constant temperature bath (see fig.1), being careful not to loosen the connection between the heating tube and the capillary. Heat tube for 40 hours.

4.9 Remove the tube from the constant temperature bath and allow it to cool to room temperature.

4.10 Reward the following data:

a. The total length of the capillary tube minus vertical height of mercury column in the cup after heating (B).

b. The height of mercury column above the surface of mercury pool at the end of test (H).

c. The temperature of room at end of test (t).

d. The barometric pressure in millimeters of mercury at end of test (P) .

4.11 Calculate the volume of gas (at standard temperature and pressure) liberated during test, as follows:

$$\text{cc gas} = [A + C(B-H)] \frac{273P}{760\,(273 + t)} - [A + C(B_1-H_1)] \frac{273P_1}{760\,(273 + t_1)}$$

Were:

A = is the volume of the heating tube minus 5.00 cc or 1.00 cc (volume of explosive)

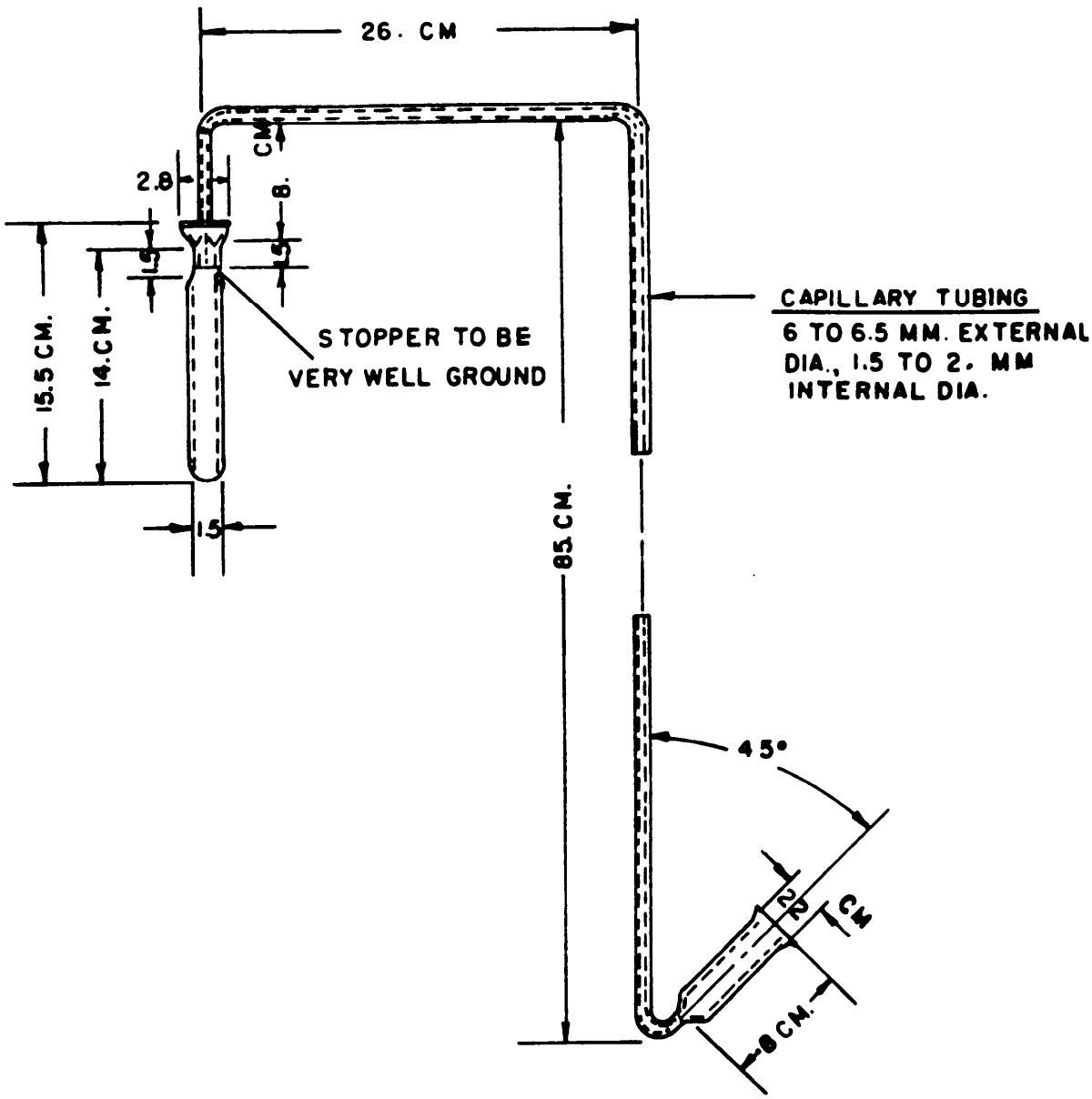
B = is the total length in mm of the capillary tube minus the vertical. height of the column in mercury in the cup at the end of the test

B_1 = is the total length in mm of the capillary tube minus the vertical height of the column of mercury in in the cup before heating

C = is the determined unit capacity of the capillary tubing in cc per mm

P = is the barometer reading at the end of the test minus H

P_1 = is the barometer reading when the test is started minus H_1



APPARATUS FOR
120°C. VACUUM STABILITY TEST

FIGURE 1.

* U.S. GOVERNMENT PRINTING OFFICE: 1974-603-109/1280