MIL-STD-1234
NOTICE 1
18 December 1965

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 supersede the pages listed:

New Page	Date	Superseded Page	Date
ii	18 December 1965	ii	22 June 1962
iv	18 December 1965	iv	22 June 1962
v	18 December 1965	v	22 June 1962

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

Revised Method	Date	Superseded Method	Date
101.1.1	18 December 1965	101.1	22 June 1965
101.2.1	18 December 1965	101.2	22 June 1965
101.8.1	18 December 1965	101.3	22 June 1962
101.4.1	18 December 1965	101.4	22 June 1962
102.1.1	18 December 1965	102.1	22 June 1962
102.2.1	18 December 1965	102.2	22 June 1962

3. The following method has been added:

Method No.	Title	Date
101.5	Moisture (Kari Fischer Distilation Method)	18 December 1965

4. Retain this notice and insert before the Table of Contents.

FSC 1370

Project No. 1370-0205

DEPARTMENT OF DEFENSE

Washington 25, D.C.

Pyrotechnics: Sampling, Inspection and Testing

MIL-STD-1234

    1. This Military Standard has been approved by the Department of Defense and is mandatory for use by all Department and Agencies of the Department of Defense.

    2. Recommended corrections, additions, or deletions should be addressed to Commanding Officer, Picatinny Arsenal, Dover, New Jersey 07801, ATTN: SMUPA-DC7.

Supersedes Page ii of 22 June 1962

ALPHABETICAL INDEX OF TEST METHODS–Continued

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NUMERICAL INDEX OF TEST METHODS

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Method 101.1.1

MOISTURE (DESICCATION METHOD)

1. SCOPE

1.1 This method is used for determining the moisture content of pyrotechnic compositions on a basis of loss of weight in a desiccated atmosphere. This method is used on compositions when it is undesirable to use heat due to the sensitivity and volatility of the material being tested.

2. SPECIMEN

2.1 The specimen shall consist of approximately 2 gm of the pyrotechnic weighed to within 0.2 mg or as the applicable specification specifies.

3. APPARATUS

3.1 Weighing dish with tight fitting cover and of suitable diameter so that the specimen can be spread out in a thin layer.

3.2 Desiccator containing a suitable desiccant such as anhydrous calcium sulfate, or as specified in applicable specifications,

4. PROCEDURE

4.1 Place the specimen in the weighing dish and weigh the dish, contents and cover.

4.2 Place the dish with cover removed in the desiccator maintained at 25 degrees plus or minus 5 degrees C.

Note. Vacuum may be applied to the desiccator to shorten the period of drying. The desiccator is evacuated to a pressure of 380 mm plus or minus 20 mm of mercury and maintained at a temperature of 25 degrees plus or minus 5 degrees C. Results obtained in this manner are usually reported as loss on vacuum desiccation, percent.

4.3 Weigh the stoppered dish and contents at intervals until the loss in weight between weighings does not exceed 0.5 mg.

4.4 Determine the loss in weight and calculate the loss as the percentage moisture in the sample.

4.5 Calculation

$$\text{Percent moisture} = \frac{A100}{W}$$

where:

A = loss in weight, gm.
W = weight of sample, gm.

Method 101.2.1

MOISTURE (KARL FISCHER METHOD)

1. SCOPE

1.1 This is a general method for determining the moisture in pyrotechnics which do not react with the Karl Fischer reagent and from which the moisture is easily extractable.

Note. Although the application of the Karl Fischer reagent for determining the moisture content of pyrotechnics is general in scope, its chemical activity with materials other than moisture imposes certain limitations that must be taken into account by modification of the method for the purpose of adaption to specific applications. Detailed specifications should be consulted for adaptations to materials that require modifications of the procedure listed herein.

2. SPECIMEN

2.1 The specimen shall consist of approximately 8 to 10 gm of the pyrotechnic.

Note. The specimen shall contain not more than 0.01 gm of water, The specimen weight should be adjusted after a preliminary determination, so that the water will not exceed 0.01 gm.

3. APPARATUS

The arrangement now used at Picatinny Arsenal will be described although numerous modifications, which give equally satisfactory results, may be used. There are also available automatic titrimeters and aquameters especially designed for use with the Karl Fischer dead-spot end point method, which are compact and satisfactory.

3.1 Titration assembly consisting essentially of:

(a) Burets, automatic, Machlett autoburet, 50 ml capacity, 2 required, one left and one right.

(b) Titrator stand.

(c) Magnetic stirrer with Teflon coated stirring bar.

3.2 Titration flask (fig. 2) consisting essentially of:

(a) Beaker, Berzelius, 200 or 300 ml without spout, at least 4 required.

(b) Rubber stopper to fit beaker, prepared as shown in (fig. 3).

(c) Glass stopper for sample porthole of rubber stopper (fig. 3).

(d) Iron ring support (fig. 2).

(e) Drying tube, calcium chloride, 3 required.

(f) Weighing bottles with cap-style ground covers, 2 required.

3.3 Direct dead-stop circuit assembly (fig. 1) consisting essentially of:

(a) Dry cell battery, 1.5 volts.

(b) Rheostat, 250,000 ohms.

(c) Micro-ammeter, Weston Model 440, resistance 164 ohms, or equal.

(d) Platinum electrodes, 2 required.

3.4 Over

3.5 Assemble the apparatus as follows:

(a) Attach each buret to the titrator stand.

(b) Clamp the iron ring support to the titrator stand so that it holds the rubber stopper firmly in place, allowing the buret tips to pass through the designated holes (see fig. 3) in the stopper.

(c) Pass the electrodes through the holes in the rubber stopper and connect them to the dead stop circuit with electrical wire as shown in (fig. 1).

(d) Insert a drying tube in the hole in the robber stopper and attach the other two drying tubes to the vents of the burets.

(e) Place the glass stopper in the sample port hole.

(f) Place the magnetic stirrer so that when the rubber stopper is firmly fitted in the mouth of the titration beaker, the beaker will rest on the magnetic stirrer.

(g) Place the required number of beakers in the oven maintained at 100 degrees plus or minus 2 degrees C. to dry.

(h) Fill the reservoirs of the burets with the required reagents.

4. MATERIALS

4.1 Karl Fischer reagent, stabilized single solution, placed in the reservoir of a buret.

4.2 Standard water-in-methanol, 1 mg of water per 1 ml of solution, placed in the reservoir of the buret.

Note. The Karl Fischer reagent and standard water-in-methanol solution are commercially available as standard stock items. If it is desirous to prepare the reagents, their preparation can be found in Mitchell, John Jr. and Smith, Donald Milton, "Aquametry", Interscience Publishers, inc., New York, 1948, Chapter IV and ASTM Method E203-62T.

4.3 Sodium tartrate dihydrate, primary standard for Karl Fischer reagents containing 15.66 plus or minus 0.05 wt percent water.

4.4 Special solvent as indicated in the applicable pyrotechnic specification.

Note. The special solvent or advents employed for each material will vary depending on the nature of the ingredients. Some of the solvents commonly employed are methanol ethanol, dioxane, acetic acid, dimethyl formamide and pyridine: methanol (1 vol: 3 Vol.).

Method 101.2.1

5. PROCEDURE

5.1 Reach a preliminary end point as follows :

(a) Dry the beakers in an oven maintained at 100 degrees plus or minus 5 degrees C.

(b) Remove one of the dried beakers and attach it, while still hot, firmly to the rubber stopper and allow it to cool to room temperature.

(c) Transfer 100 ml of the special solvent through the sample port-hole of the rubber stopper into the dry beaker. Add the magnetic stirring bar and stopper immediately. The platinum electrodes should be immersed in solution and should never be exposed throughout the determination.

(d) Start the magnetic stirrer and titrate the water in the solvent by adding a slight excess of the Karl Fischer reagent to produce a reddish-brown color which persists for at least half a minute.

(e) Adjust the variable resistance at this point, so that with excess Karl Fischer reagent present, the needle of the micro-ammeter will be at the upper end of the scale.

(f) Titrate the solution slowly with the water-in-methanol solution until the end point is reached. The end point is defined as the point at which one drop of the standard water-in-methanol solution causes the micro-ammeter needle to waver and slowly drift from the upper end of the scale toward the lower end in not less than 15 seconds.

5.2 Determine the milliliters of standard

2

water-in-methanol solution per milliliter of Karl Fisher reagent, designating the ratio as "R" as follows:

(a) Reach a preliminary end point as specified in 5.1.

(b) Immediately fill the burets and add an accurately measured portion of approximately 10 ml of the Karl Fischer reagent.

(c) Immediately titrate the solution with standard water-in-methanol solution.

Note. The second end point may be used as the preliminary end point for a check determination.

(d) Calculate the ratio "R" as follows:

$$R = \frac{A}{B}$$

where:

A = standard water-in-methanol reagent, ml.

B = Karl Fischer reagent, ml.

Note. Standardization shall be made at least once a day.

5.3 Determine the grams of water per milliliter of standard water-in-methanol solution designating the Factor as "F" as follows:

(a) Place approximately 1 gm of the sodium tartrate dihydrate in a weighing bottle, close tightly with a ground glass, cap-style cover and weigh.

(b) Reach a preliminary end point as specified in 5.1.

(c) Quickly introduce, from the weighing bottle, about 0.5 gm of the sodium tartrate dihydrate through the sample porthole. Quickly stopper the porthole and recover the weighing bottle. The bottle and contents are re-

weighed at the end of the titration and the difference in weighings (5.3a) is taken as the weight of sodium tartrate dihydrate used.

(d) Add excess of Karl Fischer reagent and titrate immediately after reaching the preliminary end point.

(e) Upon reaching the second end point, repeat the determination, take the buret readings and refill them. Add a slight excess of Karl Fischer reagent; and titrate with standard water-in-methanol solution to an end point.

(f) Calculate the factor "F" for each determination as follows:

$$F = \frac{0.1566W}{VR\text{-}S}$$

where :

W = sodium tartrate dihydrate, added, gm.

V = Karl Fischer reagent, ml.

R = ratio as determined in para. 5.2

S = standard water-in-methanol solution, ml.

(g) Determine "F" from the average of the two determinations. Duplicate results should agree within 0.01.

5.4 Determine the percent moisture of the specimen as follows:

(a) Place the specimen in a weighing bottle, cover tightly with a cap style ground glass cover and weigh.

(b) Reach a preliminary end point as specified in paragraph 5.1.

(c) Immediately upon reading the preliminary end point, fill the burets.

Method 101.2.1

(d) Introduce the specimen from the weighing bottle through the sample porthole and recover the weighing bottle. Close the porthole and add an excess of Karl Fischer reagent. The bottle and contents are reweighed at the end of the titration and the difference in Weighings (5.4a) is taken as the sample weight.

Note. Lose of 0.05 gm. of the specimen during the transfer to the titration flask has no appreciable effect on the accuracy of the determination.

(e) Allow the contents of the beaker to to stir for approximately 1.5 min. or until the sample is dissolved.

(f) Titrate the solution with standard water-in-methanol solution. The volume of the back-titration with the standard water-in-methanol solution shall be less than 1 ml.

(g) Calculate the percent water as follows:

$$\text{Percent water} = \frac{F(VR\text{-}S)100}{W}$$

where:

F = factor determined as specified in para. 5.3

R = ratio determined as specified in para. 5.2

V = Karl Fischer reagent, ml.

S = standard water-in-methanol, ml.

W = weight of specimen, gm.

FIGURE 1.

FIGURE 2.
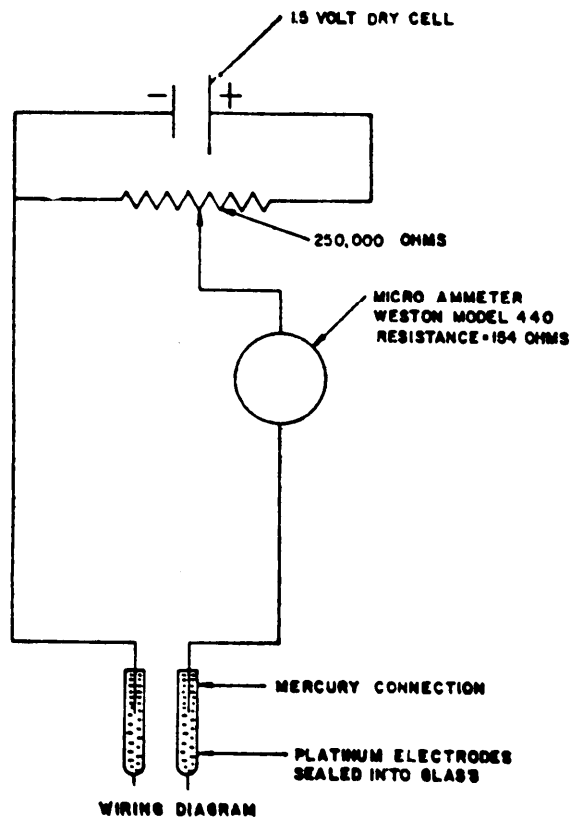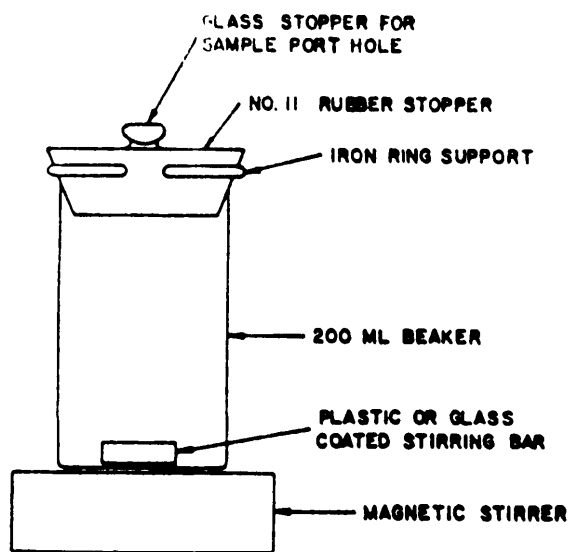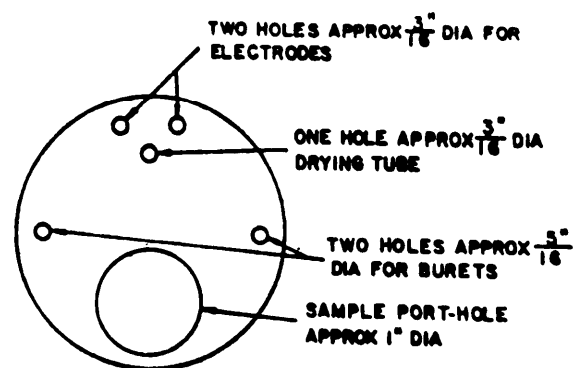

FIGURE 3.

Method 101.2.1

Method 101.3.1

MOISTURE (MODIFIED KARL FISCHER METHOD)

1. SCOPE

1.1 This is a general method for determining the moisture in pyrotechnics which do not react with the Karl Fischer reagent and from which the moisture is slowly extractable.

Note. Although the application of the Karl Fischer reagent for determining the moisture content of pyrotechnics is general in scope, its chemical reactivity with materials other than moisture imposes certain limitations that must be taken into account by modification of the method for the purpose of adaptation to specific applications. Detailed specifications should be consulted for adaptations to materials that require modifications of the procedure listed herein.

2. SPECIMEN

2.1 The specimen shall consist of approximately 8 to 10 gm of the pyrotechnic.

Note. The specimen should contain not more than 0.01 gm of water. The specimen weight should be adjusted after a preliminary determination so as to exceed the 0.01 gm maximum.

3. APPARATUS

3.1 The apparatus used in this method is identical with that used in Method 101.2.1.

4. MATERIALS

4.1 The materials used in this method are identical with those used in Method 101.2.1.

5. PROCEDURE

5.1 Follow the procedure described in Method 101.2.1 from paragraph 5.1 to 5.3.

5.2 Determine the percent moisture in the specimen as follows:

(a) Place the specimen in a weighing bottle, cover tightly with a cap style ground glass cover and weigh.

(b) Reach a preliminary end point as specified in Method 101.2.1 paragraph 5.1.

(c) Immediatily after reaching the preliminary end point, fill the burets.

(d) Introduce the specimen from the weighing bottle through the sample porthole. Quickly restopper the porthole and recover the weighing bottle. The bottle and contents are reweighed at the completion of the titration and the difference in weighings (5.2.a) is taken as the sample weight.

Note. Loss of 0.05 gm of the sample during the transfer to the titration flask has no appreciable effect on the accuracy of the determination.

(e) Allow the contents of the beaker to stir for approximately 15 min.

(f) After the stirring period, add a slight excess of Karl Fischer reagent.

(g) Titrate the solution with standard water-in-methanol solution. The volume of the back titration should be less than 1 ml.

(h) Conduct a blank determination using 100 ml. of the special solvent; titrating to a preliminary end point, stirring for 15 min; and then titrating to an end point specified for the specimen.

(i) Calculate the percent water as follows :

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$$\text{Percent water} = \frac{F \ [(VR\text{–}S) \ — \ (V'R\text{–}S')] \ 100}{W}$$

where:

F = factor determined as specified in Method 101.2.1, para. 5.3

R = ratio determined as specified in Method 101.2.1, para. 5.2

V = Karl Fischer reagent used in specimen, ml.

V' = Karl Fischer reagent used in blank, ml.

S   = standard water-in-methanol solution used in sample, ml.

S' = standard water-in-methanol solution used in blank, ml.

W = weight of sample, gm.

Method 101.3.1

2

Method 101.4.1

MOISTURE (KARL FISCHER EXTRACTION METHOD)

1. SCOPE

1.1 This is a general method for determining the moisture in pyrotechnics which react with the Karl Fischer reagent and from which the moisture is difficult to extract.

2. SPECIMEN

2.1 The specimen shall consist of approximately 8 to 10 gm. of the pyrotechnic weighed to within 1 mg.

3. APPARATUS

3.1 The apparatus used in this method is identical with that used in Method 101.2.1 except that two 50 ml capacity Schuster dropping bottles, having a curved neck tapered to a tip which is stoppered with a rubber policeman and a side tabulation fitted with a rubber stopper are also employed. Alternatively, 50 ml. volumetric flasks may be used in place of the Schuster bottles.

4. MATERIALS

4.1 The materials used in this method are identical with those used in Method 101.2.1 except that an extraction solvent specified in the applicable pyrotechnic specification is employed.

5. PROCEDURE

5.1 Place the specimen in a Schuster dropping bottle and fit a rubber policeman over the tip and a rubber stopper in the side tabulation and weigh the assembly. If a volumetric flask is used, transfer the weighed specimen directly to the flask.

5.2 Add approximately 35 ml of the extraction solvent into the Schuster dropping

bottle through the side tabulation, replace the rubber stopper and weigh the assembly. The difference between this weight and the weight of the Schuster assembly obtained in paragraph 5.1 is taken as the weight of extraction solvent added. If a volumetric flask is used, simply dilute to volume with the extraction solvent.

Note. Care should be taken to exclude atmospheric moisture.

5.3 Prepare a blank as follows:

    (a) *With Schuster bottle.* Tare a second Schuster bottle that is fitted with a rubber policeman and a rubber stopper. Add approximately 35 ml of the extraction solvent through the side tabulation, replace the rubber stopper and weigh the assembly. The difference between this weight and that obtained in 5.2 is taken as the weight of extraction solvent added.

    (h) *With volumetric flask.* Fill a second volumetric flask to the mark with the extraction solvent.

5.4 Allow the two bottles to stand for at least 17 hours or for the time required in the applicable specification.

5.5 After the required extraction period, determine the "R' and "F" as described in Method 101.2.1 paragraph 5.1 to 5.3.

5.6 Determine the percent moisture in the specimen as follows:

    (a) Reach a preliminary end point as specified in Method 101.2.1 paragraph 5.1.

    (b) Immediately upon reaching the preliminary end point, fill the burets.

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(c) Add rapidly, through the sample porthole, a 20 to 25 gm. portion of the supernatant solution through the tapered tip of the Schuster dropping bottle containing the specimen. Quickly stopper the porthole and replace the rubber policeman on the Schuster bottle. The Schuster bottle is weighed at the conclusion of the titration and the difference between this weight and that obtained from the Schuster assembly after the addition of the extraction solvent as in paragraph 5.2 is taken as the weight of extraction solvent added to the titration beaker. If a vol-
umetric flask is used, take a 25 ml aliquot.

(d) Allow the contents of the beaker to stir for 30 seconds.

(e) After the stirring period, add a slight excess of Karl Fischer reagent.

(f) Titrate the solution with standard water-in-methanol solution.

(g) Repeat the determnination of moisture of the prepared blank in the same manner used to determine moisture in the specimen.

(h) Calculate the percent moisture as follows:

Schuster bottle:

$$\text{Percent water} = \frac{FT \left[\dfrac{VR-S}{G} - \dfrac{V'R-S'}{G'} \right] 100}{W}$$

Volumetric flask:

$$\text{Percent water} = \frac{F[(RV-S)-(RV'S')]}{W'}$$

where:

F = factor determined as specified in Method 101.2.1, para. 5.3

R = ratio determined as specified in Method 101.2.1, para. 5.2

V = Karl Fischer reagent used for sample, ml.

V' = Karl Fischer reagent used for blank ml.

S = standard water-in-methanol solution used for sample, ml.

S' = standard water-in-methanol solution used for blank, ml.

G = weight of decanted portion of extraction solvent used to extract sample, gm.

G' = weight of decanted portion of extraction solvent used for blank, gm.

T = total weight of extraction solvent used to extract the sample, gm.

W = weight of pyrotechnic, gm.

W' = weight of pyrotechnic represented by aliquot, gm.

Method 101.4.1   **2**

Method 101.5

MOISTURE (KARL FISCHER DISTILLATION METHOD)

1. SCOPE

1.1 This method is used for determining the moisture content of pyrotechnic compositions which contain materials which react with the Karl Fischer reagent and which cannot be separated by extraction.

2. SPECIMEN

2.1 The specimen shall consist of approximately 25 grams (gm) of the mixture weighed to within 1 milligram (mg). In the event that the sample has begun to harden before weighing, it should be rubbed gently through a 20-mesh screen with a rubber stopper.

3. APPARATUS

3.1 Allihn condensers, 300 millimeter (mm) jacket with standard taper 24/40 inner-member joint, such as Kimax No. 18260, protected by drying tubes containing a suitable desiccant

3.2 Automatic burets-two, 25 milliliter (ml), such as Kimax No. 17138F,

3.3 Boiling flasks, 250 ml, flat-bottomed, with standard taper 24/40 joint, such as Kimax No. 25055.

3.4 Dead-stop indication circuit consisting of platinum electrodes in series with micro-ammeter and source of direct current (de) voltage variable from 0 to 1½ volts such as shown in figure 1 of MIL-STD-1234, Method 101.2.1; the apparatus described in Method 101.2.1 may be used.

3.5 Distillation receiver, similar to figure 1, of borosilicate glass. This item may be conveniently fabricated by attaching a standard taper No. 2 stop-cock such as Kimax No. 41004, to a crankcase dilution receiver ASTM standard D-322 such as Kimax No. 22009, and drawing-out and curving the outward stem to serve as a draw-off spigot, as shown in the figure. Alternatively, a receiver such as Scientific Glass App. Co. No. JD 8100 may be used with the spigot attached to the bottom of the graduated portion (figure 2).

3.6 Electric hot plate.

3.7 Oven, 70 degrees Centigrade (C), suitably heated.

3.8 Tall-form beakers, 300 ml., such as Kimax No. 14030, or other suitable titration flask.

3.9 Volumetric flasks, 50 ml, with standard taper stopper, such as Kimax No. 28015.

Note. The boiling flasks, receivers, beakers or titration flasks, and volumetric flasks must be dry.

4. MATERIALS

4.1 Karl Fischer reagent, stabilized, Fisher Scientific Company No. SO–K–3 or equivalent, stored in an automatic buret the air-inlet of which is protected by a drying tube containing a suitable desiccant.

4.2 Methanol, anhydrous, suitable for Karl Fischer analyses.

4.3 Sodium tartrate dihydrate containing 15.66 plus or minus 0.05 percent of water as determined by the loss of weight when dried at 160 degrees C. for 3 hours.

4.4 Trichlorethylene, Fisher certified grade, Catalog No. T–341, or equivalent, prepared by shaking with water in a separa-

tory funnel at least two minutes and left to stand in contact with water at least over-night.

4.5 Water-in-methanol standard solution, 1 mg of water per ml of anhydrous methanol, stored in an automatic buret, the air-inlet of which is protected by a drying tube containing a suitable desiccant.

5. PROCEDURE

5.1 The specimen is weighed and transferred to the dried boiling-flask which is immediately re-stoppered. Another such flask without sample is subjected to the same subsequent treatment and serves as a blank. Just previous to use the condenser and receiver are rinsed with anhydrous methanol which is discarded. (A small residue of methanol in the condenser and receiver does no harm.) The stopper is now removed from the flask, the receiver (its stopcock closed) is inserted in its place, and a 100 ml portion of water-saturated tichlorethylene pipetted into the receiver so that Column A (Fig. 1) remains substantially filled. The assembly is now connected to the condenser. (In the event that these operations are interrupted, the receiver should be stoppered). The temperature of the heat source is adjusted so that the reflux from the condenser is about 2 drops per second. The distillation is continued for 2 hours, at the end of which time the flask and contents are allowed to cool. When condensate no longer drips from the condenser into the receiver, the contents of the receiver are drawn off through the stopcock into a drid 50-ml volumetric flask. The drying tube is removed and the condenser and receiver washed with a few portions of anhydrous methanol which are drained into the volumetric flask. The boiling-flask and receiver are now detached from the condenser and the receiver is further washed with successive portions of methanol which should be sufficient in volume to substantially fill the capillary E (fig. 1) without causing

it to overflow, until the volumetric flask is filled to its mark. The flask is stoppered and mixed by shaking and may be set aside until titrated.

5.2 Transfer a 20 ml aliquot from the volumetric flask into a dried titration vessel containing a stirrer, and sufficient neutralized solution to cover the electrodes. The vessel is quickly covered, stirring commenced, and Karl Fischer reagent added, 1 ml at a time, until the titrated solution remains a distinct red-brown color (not light tan) for at least a minute. The solution is now back-titrated with standard water-in-methanol solution, which is added in a steady stream until the brown color is replaced by yellow, then drop-wise until the microammeter reading commences to fall as described in Method 101.2.1, paragraph 5.1. The percentage of water is calculated as follows:

$$\text{Percent water} = F\ [R\ (V_a - V_b) - (S_a - S_b)\ ]$$

where:

F = gm weight of water per ml of standard water-in-methanol solution determined as follows:

V_a and V_b = ml Karl Fischer reagent added to specimen and blank respectively.

R = ml of standard water-in-methanol solution per ml of Karl Fischer reagent determined as shown in paragraph 5.3.

S_a and S_b = ml of standard water-in-methanol solution for back-titration of specimen and blank respectively.

W = weight of specimen in aliquot, gm = 10 gm (as directed).

5.3 To determine R, a 100 ml portion of anhydrous methanol in a dried titration vessel is "neutralized" by titration with Karl

Fischer reagent and back-titration with water-in-methanol solution in the manner already described (These volumes need not be recorded). An accurately measured volume (approximately 5 ml.) of Karl Fischer reagent is added to the neutralized methanol and titrated back to the endpoint with standard water-in-methanol solution. R is then calculated as follows:

$$R = \frac{Sr}{Vr}$$

where:

Vr = ml. of Karl Fischer reagent added (5, as directed).

Sr = ml. of standard water-in-methanol solution required to neutralize V_r.

Note. R. should be the average of at least two determinations which do not differ by more than 0.03.

5.4 To determine F, and accurately weighed 0.3 plus or minus 0.1 gram portion of sodium tartrate dihydrate is added to a 100 ml volume of anhydrous methanol which has previously been neutralized as described in the foregoing paragraph. When dissolved, the solution is titrated with Karl Fischer reagent and back-titrated with standard water-in-methanol solution in the same man-

ner as the methanol solution resulting from distillation of the sample. F is calculated in the following manner:

$$F = \frac{15.66 \ W_f}{V_f R - S_f}$$

where:

W_f = grams of sodium tartrate dihydrate.

V_f = ml. of Karl Fischer reagent added to sodium tartrate dihydrate solution.

S_f = ml. of standard water-in-methano solution for back titration of sodium tartrate dihydrate solution.

Note. F should be the average of at least two determinations which do not differ by more than 0.003.

5.5 For production control, where it is impractical to perform a blank determination for each sample, the blank value should be determined daily by a minimum of two titrations and should be rechecked more frequently whenever new reagents are used.

Note. The range of values obtained for the blank should not change the value of the moisture content by more than 0.02 percent.

Method 101.5
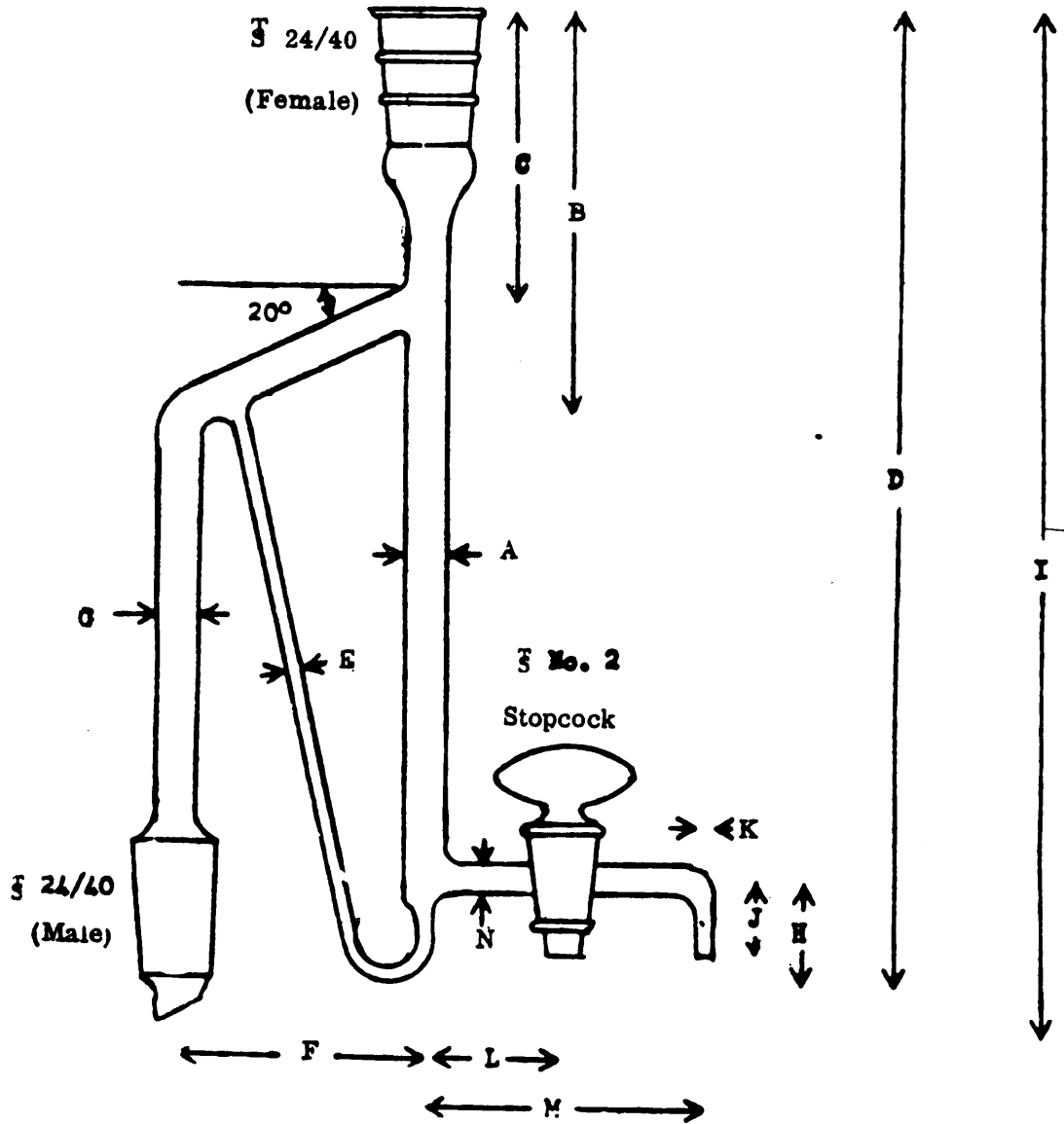
A...10.7-11.3 mm   D...280-300 mm   H...22-26 mm   L...35-45mm
B...122-127 mm   E...4-5 mm I. D.   I...300-320 mm   M...75-85 mm
C...87-92 mm   F...65-75 mm   J...20-25 mm   N...7-9 mm O.D.
                    G...11-15 mm I.D. K...1.0-1.4 mm

FIGURE 1. *Distillation receiver*

GRADUATED FROM 0 TO 5ML. IN
0.1 ML. DIVISIONS.
REFERENCE: SCIENTIFIC GLASS
APPARATUS CO., CAT. #JD-8100,
PAGE 1328
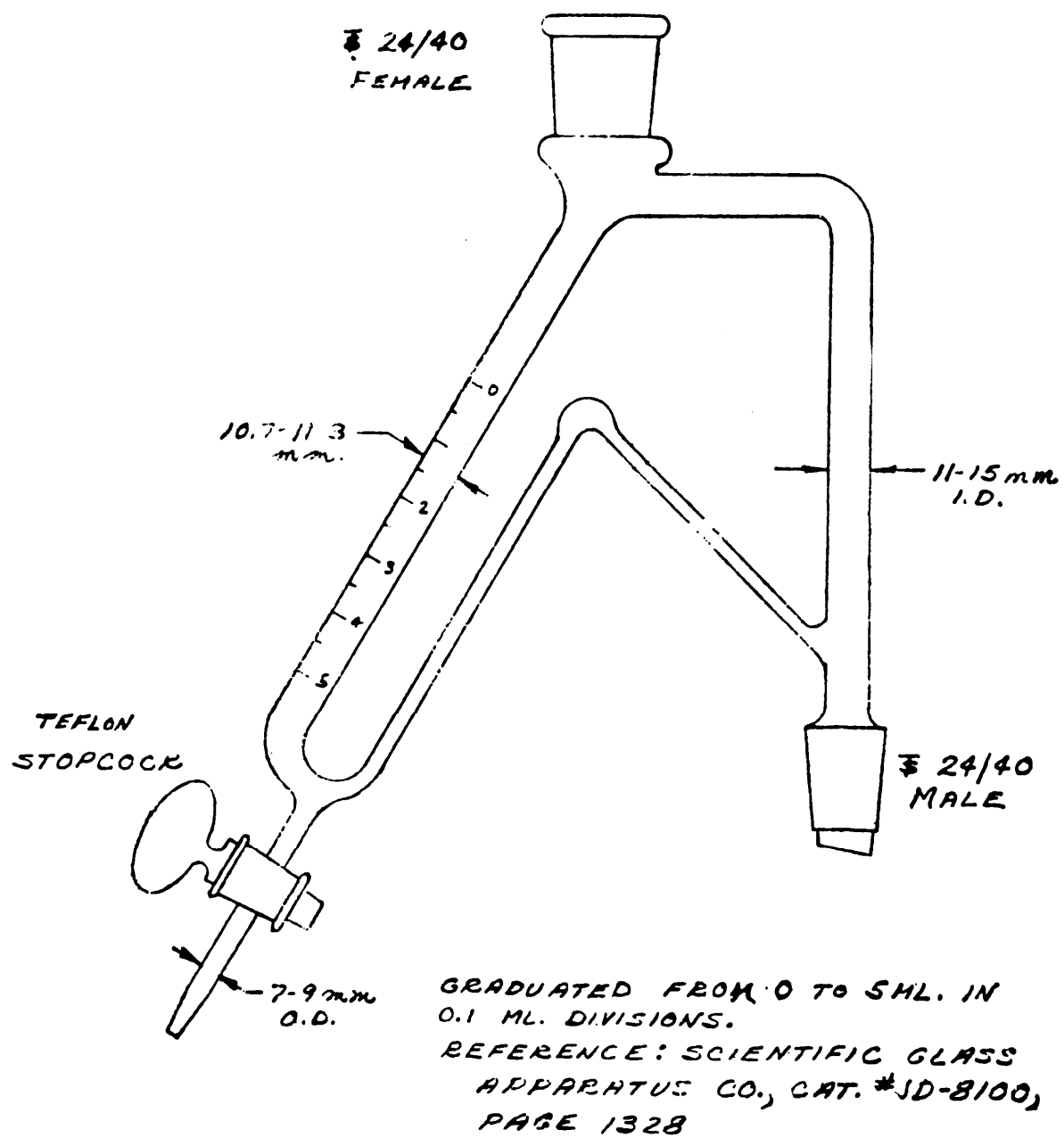
FIGURE 2. *Distillation receiver.*

Method 101.5

Method 102.1.1

VOLATILES (OVEN METHOD)

1. SCOPE

1.1 This method is used for determining the volatiles content of pyrotechnics on the basis of loss in weight on heating at atmospheric pressure, at a specified temperature and for a specified time.

Note. This method is applicable to a variety of heating conditions and is not intended to restrict or limit such factors as temperature or time of drying. For specific instructions refer to the applicable pyrotechnic specification.

2. SPECIMEN

2.1 The specimen shall consist of approximately 2 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Weighing dish of suitable diameter so that the specimen can be spread out in a thin layer.

Note. Samples that exhibit hygroscopicity require a glass receptacle with a ground glass, cap style type of cover.

3.2 Oven.

3.3 Desiccator containing a suitable desiccant such as anhydrous calcium sulfate.

4. PROCEDURE

4.1 Place the specimen in a tared stoppered weighing dish.

4.2 Heat the dish and contents with the cover removed at 100 degrees plus or minus 5 degrees C., and at atmospheric pressure for 4 hours or as specified in the applicable specification.

4.3 Cover the dish and cool to room temperature in a desiccator.

4.4 Determine the loss in weight, and calculate the percentage of volatiles in the sample.

4.5 Calculations.

$$\text{Percent volatiles} = \frac{A100}{W}$$

where:

A = loss in weight, gm.

W = weight of sample, gm.

Method 102.2.1

VOLATILES (VACUUM OVEN METHOD)

1. SCOPE

1.1 This method is used for determining the moisture and volatile solvent content of pyrotechnics on the basis of loss of weight on heating at 55 degrees C. under vacuum.

2. SPECIMEN

2.1 The specimen shall consist of approximately 2 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Weighing dish: diameter, 60 mm; depth, 30 mm, or of suitable size to allow the specimen to be spread out in a thin layer.

3.2 Vacuum oven.

3.3 Desicator containing a suitable desiccant such as anhydrous calcium sulfate.

4. PROCEDURE

4.1 Place the specimen in the tared weighing dish, and heat it for 4 hours, or until constant weight is obtained, in the vacuum oven at a temperature of 55 degrees plus or minus 2 degrees C. and a pressure (absolute) of 80 plus or minus 10 mm of mercury.

4.2 Cool the specimen to room temperature in the desiccator.

4.3 Determine the loss in weight, and calculate (by weight) the percentage of volatiles in the sample as follows:

$$\text{Percent volatiles} = \frac{A100}{W}$$

where:

A = loss in weight, gm.

W = weight of sample, gm.

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