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MILITARY STANDARD

PYROTECHNICS: SAMPLING, INSPECTION AND TESTING



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Pyrotechnics: Sampling,
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1. This standard has been approved by the Department of Defense and is mandatory for use by the Departments of the Army, the Navy, and the Air Force, effective 22 June 1962.

2. Recommended corrections, additions, or deletion should be addressed to the Standardization Division, Armed Forces Support Center, Washington 25, D.C.

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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 for bids form a part of this standard to the extent specified herein:

- O-A-51 — Acetone.
- O-C-141 — Carbon tetrachloride; technical grade.
- RR-S-366 — Sieves, standard for testing purposes.
- JAN-E-199 -- Ether, diethyl.
- MIL-E-463A — Alcohol, ethyl (for Ordnance use).

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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.

SECTION 3

TESTING

1. SCOPE

1.1 This section contains the physical and chemical tests for pyrotechnics. It also includes directions for preparing standard and indicator solutions to be used in the determinations.

1.2 Each test, as well as each procedure for preparing the special solutions, is considered as a separate method, and is assigned an individual method number.

2. NUMBERING SYSTEM

2.1 Test method groups. Methods are arranged in seven groups according to category of test or type of solution—that is, general, physical or chemical, and standard or indicator solution. These groups are identified numerically by hundreds. Tests of a general nature are in the 100 group; physical tests are in the 200 group; sample preparation in the 300 group, and so on.

2.2 Basic numbers. Method numbers in each group are arranged in subgroups, according to the ingredient or property to be determined. Differences in the method of determination are differentiated by the addition of tenths decimals to the subgroup numbers to form basic numbers, such as 101.1, 101.2, and 101.3. (These are the basic numbers for three different methods for determining the moisture content of the pyrotechnic.) The basic number of a test method is its official designation, and should be used without revision number whenever it is referenced. It always refers to the same method of determination, the same accuracy, and the same end result, regardless of revision status.

2.3 Revision numbers. Revision numbers appear as second tenths decimals to the subgroup numbers. (For example, "Method 101.2" would denote the second revision of Method 101.1.2.) Revision numbers are assigned to basic numbers when minor changes are made in the method to clarify it or to give additional details that will increase the reproducibility of the test results. Major changes in a method, which would significantly alter the method of testing, or affect the accuracy or end result of the test, are considered grounds for assigning a new basic number.

2.4 Suffix (T). The suffix (T) is used to denote a tentative status of the test. This tentative status is applied to newly developed tests, and is retained until the method has been adopted as standard. A method adopted without change retains its basic number only; a method adopted with change retains its basic number only; a method adopted with changes is assigned a revision number.

METHOD 101.1

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 3 gm of the pyrotechnic weighed to within 0.2 mg or as the applicable specification specifies.

3. APPARATUS

3.1 Weighing dish (aluminum or glass) ; with tight fitting cover.

3.2 Desiccator containing a suitable desiccant such as calcium chloride, 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 $25^{\circ} \pm 5^{\circ}\text{C}$.

Note. Vacuum may be applied to the desiccator to shorten the period of drying. The desiccator is evacuated to a pressure of $380 \text{ mm} \pm 20 \text{ mm}$ of mercury and maintained at a temperature of $26^{\circ} \pm 6^{\circ}\text{C}$. 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 1 mg.

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

4.5 Calculation.

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

where:

A = loss in weight, gm.

B = weight of sample, gm.

METHOD 101.2

MOISTURE (KARL FISCHER METHOD)

1. SCOPE

1.1 This is a general method for determining moisture of pyrotechnics that do not react with the Karl Fischer reagent used and which dissolve rapidly in the reagents or 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 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 shall contain not more than 0.01 gm of water. The specimen weight should be adjusted after a preliminary determination, so as not to exceed the 0.01 gm. maximum.

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-stop end point method, which are compact and satisfactory.

3.1 Titration assembly consisting essentially of —

- (a) Burets, automatic, Machlett auto-buret, 50 ml capacity, 2 required, one left and one right.
- (b) Titrator stand.
- (c) Magnetic stirrer with Teflon coated stirring bar.
- (d) Timer.

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

- (a) Beaker, Berzelius, 200 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. 3).
- (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.

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- (b) Rheostat, 250,000 ohms.
- (c). Micro-ammeter, Weston Model 440, resistance 154 ohms, or equal.
- (d) Platinum electrodes, 2 required.

3.4 Oven

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 rubber 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 $100^{\circ} \pm 2^{\circ}\text{C}$. 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 desired 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.

4.3 Sodium tartrate dehydrate, primary standard for Karl Fischer reagents. Extract water assay is shown on each unit.

4.4 Special solvent as indicated in the applicable pyrotechnic specification.

Note. The special solvent or solvents employed for each material will vary depending on the nature of the ingredients. Some of the solvents commonly employed are methanol, ethanol, dioxane, diethyl-ether, acetic acid and dimethyl-formamide.

5. PROCEDURE

Method 101.2

5.1 Reach a preliminary end point as follows:

- (a) Dry the beakers in an oven maintained at $100^{\circ} \pm 5^{\circ}\text{C}$.
- (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 porthole 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 and 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 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 water-in-methanol solution per milliliter of Karl Fischer reagent, designating the ratio as "R" as follows:

- (a) Reach a preliminary end point as specified in 5.1.
- (b) Immediately upon reaching the preliminary end point, start the timer and fill the burets.
- (c) Add an accurately measured portion of approximately 10 ml of the Karl Fischer reagent.
- (d) Titrate the solution with standard water-in-methanol solution, controlling all operations so as to reach an end point within 3.5 min. \pm 15 sec. after reaching the preliminary end point.

Note. The second end point may be used as the preliminary end point for a check determination. In this case, the timer is returned to zero and started as soon as the second end point has been reached; the buret readings taken and the buret refilled; and the titration to the end point completed within 3.5 min. \pm 15 sec. after the previous end point.

- (e) Calculate the ratio "R" as follows:

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

where:

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

B = Karl Fischer reagent, ml.

Note. Because the ratio is constantly changing the standardization shall be made at least once a day, more often for more accurate work.

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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 dehydrate 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) Immediately upon reaching the end point start the timer and fill the burets.
- (d) Quickly introduce, from the weighing bottle, about 0.5 gm. of the sodium tartrate dehydrate through the sample porthole. Quickly stopper the porthole and recover the weighing bottle. The bottle and contents are reweighed at the end of the titration and the difference in weighings (5.3a) is taken as the weight of sodium tartrate dehydrate used.
- (e) Titrate the solution, controlling all operations so as to reach an end point within 3.5 min. \pm 15 sec. after reaching the preliminary end point.
- (f) Immediately upon reaching the second end point, repeat the determination by quickly returning the timer to zero and starting it; taking the buret readings and refilling them; adding a slight excess of Karl Fischer reagent; and titrating with standard water-in-methanol solution to an end point within 3.5 min. \pm 15 sec. after the previous end point.
- (g) Calculate the factor "F" for each determination as follows:

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

where:

W = sodium tartrate dehydrate added, gm.

V = Karl Fischer reagent, ml.

R = ratio as determined in Para. 5.2

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

- (h) 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 start the timer and fill the burets.
- (d) Introduce the specimen from the weighing bottle through the sample porthole and recover the weighing bottle. 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. Loss 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 stir for approximately 1.5 min.

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- (f) Titrate the solution with standard water-in-methanol solution to reach an end point within 3.5 min. \pm 16 sec. after reaching the preliminary end point. 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:

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

where:

- F = factor determined as specified in para. 5.3
R = ratio determined as specified in para. 6.2
V = Karl Fischer reagent ml.
S = standard water-in-methanol, ml.
w = weight of specimen, gm.

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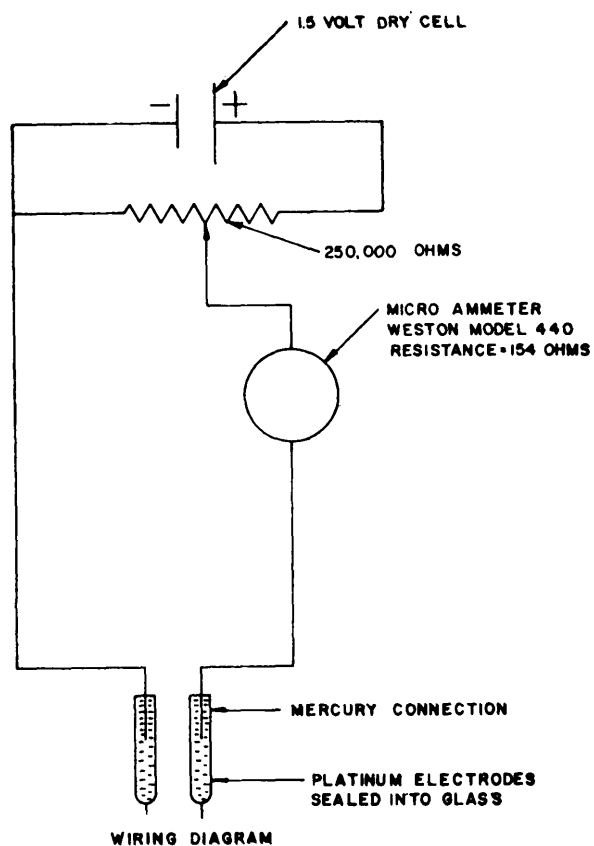


FIGURE 1.

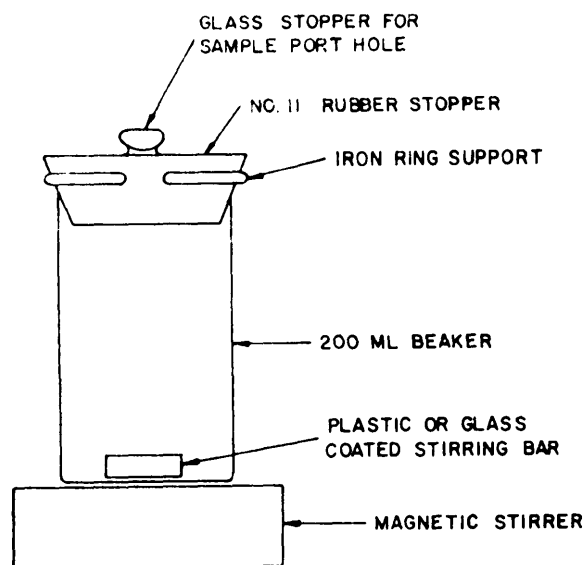


FIGURE 2.

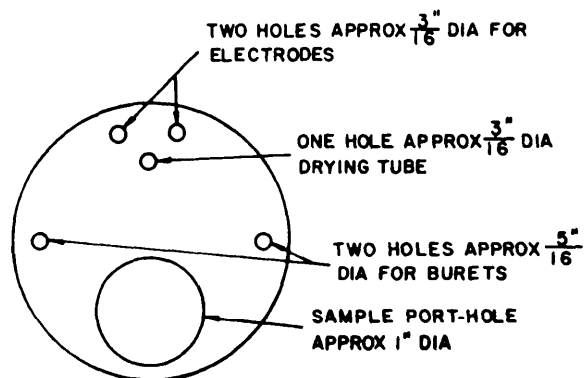


FIGURE 3.

METHOD 101.3

MOISTURE (MODIFIED KARL FISCHER METHOD)

1. SCOPE

1.1 This is a general method for determining moisture of pyrotechnics that do not react with the Karl Fischer reagent used and which dissolve slowly in the reagents or 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 not 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.

4. MATERIALS

4.1 The materials used in this method are identical with those used in Method 101.2.

5. PROCEDURE

5.1 Follow the procedure described in Method 101.2 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 paragraph 5.1.
- (c) Immediately upon reaching the preliminary end point start the timer and 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 (5.2a) 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.

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- (f) After the stirring period add a slight excess of Karl Fischer reagent.
- (g) Titrate the solution with standard water in methanol solution to reach an end point within 18.5 ± 0.25 min. after reaching the preliminary end point. 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 within 18.5 ± 0.25 min. after the preliminary end point as specified for the specimen.
- (g) Calculate the percent water as follows:

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

where:

- F = factor determined as specified in Method 101.2, Paragraph 5.3.
- R = ratio determined as specified in Method 101.2, Paragraph 6.2.
- 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 101.4

MOISTURE (KARL FISCHER EXTRACTION METHOD)

1. SCOPE

1.1 This is a general method for determining moisture of pyrotechnics that react with the Karl Fischer reagents used and which do not dissolve in the reagents or 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 0.2 mg.

3. APPARATUS

3.1 The apparatus used in this method is identical with that used in Method 101.2 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.

4. MATERIALS

4.1 The materials used in this method are identical with those used in Method 101.2 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.

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.

Note. Care should be taken to exclude atmospheric moisture.

5.3 Prepare a blank as follows:

- (a) Tare a second Schuster bottle that is fitted with a rubber policeman and a rubber stopper.
- (b) 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 5.3 is taken as the weight of extraction solvent added.

5.4 Allow the two bottles to stand for at least 17 hours.

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5.5 After the 17-hour period determine the "R" and "F" as described in Method 101.2 paragraph 5.1 to 5.3.

6.6 Determine the percent moisture in the specimen as follows:

- (a) Reach a preliminary end point as specified in Method 101.2 Paragraph 5.1.
- (b) Immediately upon reaching the preliminary end point start the timer and fill the burets.
- (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.
- (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, controlling all operations so as to reach an end point within 3.5 min. \pm 15 sec. after reaching the preliminary end point.
- (g) Repeat the determination of moisture using 20 to 26 ml of the prepared blank determination in the same manner as that used to determine moisture in the specimen.
- (h) Calculate the percent moisture as follows:

$$\text{Percent water} = \frac{\frac{FT \text{ VR-S} - V'R-S'}{G} \times 100}{W}$$

where:

F = factor determined as specified in Method 101.2, Paragraph 5.3.

R = ratio determined as specified in Method 101.2, Paragraph 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.

METHOD 102.1

VOLATILES (OVEN METHOD)

1. SCOPE

1.1 This method is used for determining the volatiles of pyrotechnics on the basis of loss of weight at atmospheric pressure on heating at $100^{\circ} \pm 6^{\circ}\text{C}$.

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 (aluminum or glass).

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 an indicating desiccant.

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 $100^{\circ} \pm 5^{\circ}\text{C}$., and at atmospheric pressure for 4 hours or until constant weight is attained.

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.

$$\text{Percent volatiles} = \frac{A}{B} \times 100$$

where:

A = loss in weight, gm.

B = weight of sample, gm.

METHOD 102.2

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°C. under vacuum.

2. SPECIMEN

2.1 The specimen shall consist of approximately 5 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Weighing dish (aluminum or glass): diameter, 60 mm; depth, 30 mm.

3.2 Vacuum oven.

3.3 Desiccator containing an indicating desiccant.

4. PROCEDURE

4.1 Place the specimen in the tared weighing dish, and heat it for 6 hours in the vacuum oven at a temperature of $55^{\circ} \pm 2^{\circ}\text{C}$. and a pressure (absolute) of 80 ± 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:

$$\text{Percent volatiles} = \frac{A}{W} \times 100$$

where:

A = loss in weight, gm.

W = weight of sample, gm.

METHOD 201.1

GRANULATION

1. SCOPE

1.1 This method is used for determining the percentages of the various size granules comprising a sample of a pyrotechnic composition.

2. SPECIMEN

2.1 The specimen shall consist of a weighed portion of approximately 10 gm of composition.

3. APPARATUS

3.1 Set of U. S. Standard sieves with receiving pan and lid conforming to specification RR-S-366.

3.2 Mechanical shaker, geared to produce 300 ± 15 gyrations and $150 \pm$ taps of a striker per minute.

4. PROCEDURE

4.1 Superimpose the sieves of the meshes required by the applicable pyrotechnic specification and attach the receiving pan to the bottom.

4.2 Place the 10 gm specimen in the top sieve, mount the assembly on the shaker and operate the shaker for 10 minutes.

4.3 At the end of 10 minutes weigh the material retained on each sieve and in the pan and calculate the percentage passing through each sieve.

4.4 Calculation.

$$\text{Percent retained} = \frac{A + B}{W} 100$$

$$\text{Percent through} = \frac{W - (A + B)}{W} 100$$

where:

A = weight retained on designated sieve, gm.

B = weight retained on sieves nested above designated sieve, gm.

C = weight of sample, gm.

METHOD 202.1

AVERAGE PARTICLE SIZE (FISHER SUBSIEVE SIZER)

1. SCOPE

1.1 This method is used for determining the average particle diameter of the various size granules comprising the pyrotechnic in the 2 micron to 50 micron range.

2. SPECIMEN

2.1 The specimen shall consist of an accurately weighed portion (to within 0.01 gm.) of sample equal in grams to the density of the sample.

3. APPARATUS

3.1 Fisher subsizer. Composed of an air pump, an air pressure regulator, a precision bore sample tube, a standardized double range air flowmeter and a calculator chart, along with accessory equipment necessary to correlate these parts in a unit (see fig. 1 and 2), or equal.

3.2 Calibrator. Fisher Laboratory Appliances, Catalog No. 14-313-7 or equal.

4. PROCEDURE

4.1 Standardize the apparatus as follows:

- (a) Check the water in the standpipe of the pressure regulator. The water level, as observed through the round window located in the upper lefthand side of the front panel (fig. 2), should coincide exactly with the calibration mark on the standpipe. If necessary, adjust the water level by adding or removing water through the glass intake arm extending through the top of the instrument cabinet. The intake arm should be stoppered except when adjustments are being made. The water level should be checked only when the sample tube has been removed from the instrument.
- (b) Check the condition of the drying agent in the drying tube through the small window in the left side of the cabinet. Use an indicating type drying agent so that the condition of the drying agent may be determined visually at any time.

4.2 Check the adjustment of the sample packing assembly as follows:

- (a) Screw one of the porous plugs (see fig. 1) to the plug manipulator.
- (b) Lay a paper disk over one end of the sample tube and push the plug into the sample tube for a distance of approximately 0.5 inch, with the perforated surface of the plug against the surface of the paper disk, forcing the paper to crimp around the edges and precede the plug into the sample tube.
- (c) Remove the plug manipulator and place the sample tube in a vertical position with the paper side of the plug up.

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- (d) Lay a second paper disk over the top of the sample tube, attach the second porous plug to the manipulator, and force the plug and paper disk down into the sample tube until contact is made with the lower paper disk and plug and remove the manipulator.
- (e) Place the sample tube on the brass post beneath the rack and pinion (fig. 2) with the lower plug in contact with the upper end of the brass post, and lower the rack until the flat bottom end comes in firm contact with the upper plug.
- (f) The tip of the pointer, carried on the crossbar attached to the upper end of rack, should coincide exactly with the base line on the calculator chart. If necessary, loosen the setscrew holding the lower brass post in its mount and adjust the height of the post until the tip of the pointer does coincide exactly with the base line.

4.3 Calibrate the apparatus as follows:

- (a) Set the calculator chart to indicate the porosity value stamped on the calibrator.
- (b) Check the level of the liquid meniscus in the manometer in the usual manner.
- (c) Mount the calibrator, without porous plug or filter paper, between the rubber cushioned supports just to the right of the brass post. Screw the upper cap down onto the calibrator until an airtight seal is obtained.
- (d) Plug the line cord into a 115 volt, 60 cycle alternating current line. Throw the electrical switch to the "ON" position.
- (e) Adjust the pressure control knob until the bubbles rise in the standpipe at the rate of two to three bubbles per second.
- (f) Allow the liquid in the manometer tube to rise until the maximum rise is obtained.
- (g) Without disturbing the calculation chart, the rack is turned up until the upper edge of the crossbar coincides with the liquid meniscus in the manometer. The particle size is indicated by the location of the tip of the pointer with relation to the curves on the chart.
- (h) The reading stamped "High" on the calibrator tube can be obtained with the range control set to the 0.2 to 20.0 micron range and the reading stamped "Low" should be obtained with the range control set to 20 to 50 micron range. In the event the readings obtained with the calibrator do not agree within 0.05 microns of the value. Stamped on the calibration the instrument should be adjusted.
- (i) Remove the back panel and turn the instrument to bring the right side forward. There are two glass capillary tubes mounted on the right rear side of the front panel and connected into the system with rubber tubing. The instrument readings are made to coincide with the calibrator readings by moving the fine wires in or out of the capillary tubes.

- (j) Set the range control to the 0.2 to 20 micron range and loosen the set screw holding the fine wire in position in the capillary tubes.
- (k) To raise the meniscus in the manometer, the wire in the capillary nearest to the right side of the case should be moved further up into the capillary tube. Conversely to lower the meniscus, the wire should be withdrawn. At least 30 seconds should elapse between moving the wire and taking a reading to allow the meniscus to stabilize.
- (l) When this wire is adjusted so that the meniscus reads the high value stamped on the calibrator, the set screw holding the wire is tightened down, the range switch is put into the 20 to 50 micron position, and the same procedure followed with the second wire when this is finished the back is replaced the instrument is ready for use.

Note. Under normal conditions of cleanliness, there is not much chance of any dirt clogging the ruby orifice of the calibrator. However, the tube can be cleaned by flushing with acetone and drying with vacuum. The orifice should not be cleaned with a wire, since the ruby can be chipped.

The calibrator value was determined at a temperature of 77 °F. and a slight variation in readings occur both above and below this temperature. For a temperature change of plus or minus 10°F. the calibration value change is about plus or minus 0.1 micron and should be allowed for whenever the readings obtained disagree with the values given, on the tube.

When the above calibrating instructions have been followed, a sample will give a high-low ratio of 2-1; however, this is not true of the calibrator.

4.4 Determine the average particle size as follows:

- (a) Screw one of the porous plugs to the plug manipulator (see fig. 1).
- (b) Lay a paper disk over one end of the sample tube and push the plug into the sample tube for a distance of approximately 0.5 inch, with the perforated surface of the plug against the surface of the paper disk, forcing the paper to crimp around the edges and precede the plug into the sample tube.
- (c) Place the sample tube in a verticle position with the paper side of the plug up.
- (d) Transfer the specimen into the sample tube with the aid of a small funnel and tap the side of the tube to settle the powder.
- (e) Lay a second paper disk over the top of the sample tube, attach the second porous plug to the manipulator, and force the plug and paper disk approximately 2 inches down into the sample tube. Remove the manipulator.
- (f) Lower the rack until the pointer lies on the base line.
- (g) Shift the calculator chart until a porosity value of 0.80 is indicated by the pointer on the porosity scale located along the bottom edge of the calculator chart.

Note. The porosity value is a measure of the degree of packing of the sampling.

- (h) Do not move the chart after making this setting until the detemination has been completed.

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- (i) Raise the rack and place the sample tube on the brass post beneath the rack and pinion (see fig. 2) with the lower plug in contact with the upper end of the brass post.
- (j) Lower the rack until the flat bottom comes in contact with the upper plug. Continue lowering the rack by turning the pinion knob manually until the tip of the pointer coincides with a point on the sample height curve on the chart.
- (k) The sample has now been packed to a porosity value of 0.80. Raise the rack and remove the sample tubes.
- (l) Adjust the initial level of the water meniscus in the manometer tube, located over the calculator chart, by means of the manometer control knob so that the meniscus coincides with the upper edge of the metal crossbar attached to the rack when the tip of the pointer coincides exactly with the baseline on the calculator chart. If adjustment of the water meniscus can not be made because of too little water in the manometer tube, water may be added through the top, of the manometer tube.
- (m) Turn the range control knob, located at the extreme upper right of the front panel, to the "LO" position.
- (n) Mount the sample tube, without disturbing the sample in any way, between the rubber-cushioned supports of the sample tube holder.
- (o) Screw the upper cap down onto the sample tube until an airtight seal is obtained at both ends.
- (p) Plug the line cord into a 110-volt, 60-cycle alternating current line and throw the electrical switch at the lower right-hand corner of the front panel to the "ON" position. This turns on the air pump as well as the pilot lamp which illuminates the tip of the bubbler tube in the pressure regulator standpipe, as observed through the round window in the lower left hand corner of the front panel and the level of water in the standpipe as observed through the upper window.
- (q) Adjust the pressure control knob until the bubbles rise in the standpipe at the rate of 2 to 3 per second.
- (r) Allow the water level in the manometer tube to rise to a maximum level. Without disturbing the chart, turn the rack up until the upper edge of the crossbar coincides with the water meniscus in the manometer.
- (s) From the location of the tip of the pointer, read the average particle diameter in microns from the chart. With the range control in the "LO" position, the particle diameters within the range of 0.2 to 20.0 microns maybe read directly from the chart. With the range control in the "HI" position, the particle diameter is obtained by doubling the reading.
- (t) Repeat the above determination at lower porosities taking care to read just the chart as required.
- (u) Continue until the bed is very tightly compressed, making all of these determinations on the same sample.

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- (v) Plot the porosities against the corresponding values of average particle diameter and record the value of the average particle diameter in the range of porosity within which a change in porosity does not appreciably affect the determined value for the average particle diameter.
- (w) Record the porosity range used to obtain the average particle diameter.
- (x) For materials with porosities below 0.4 (sample height below 1.4 cm) use the supplementary chart (fig. 3) for particle size determinations.

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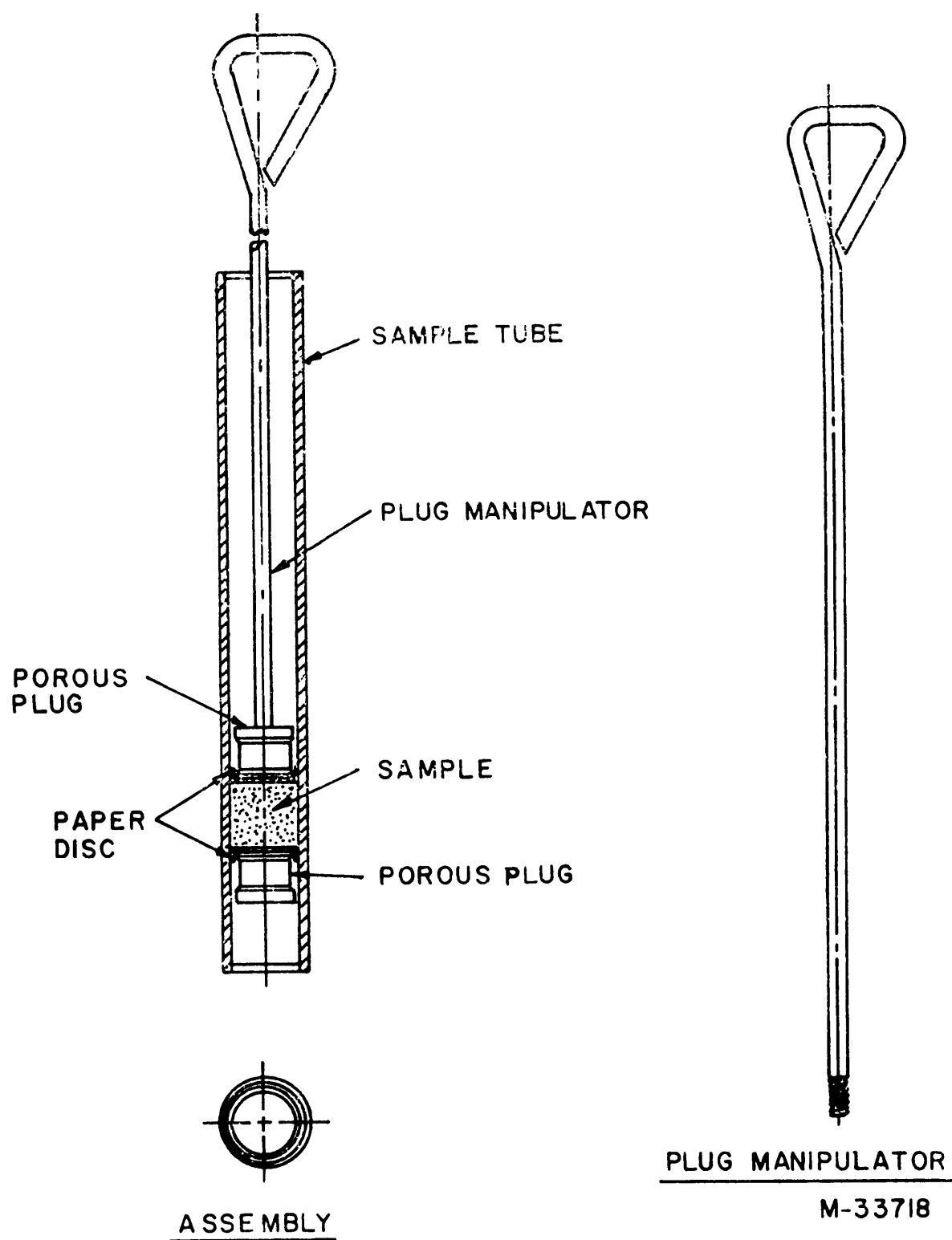
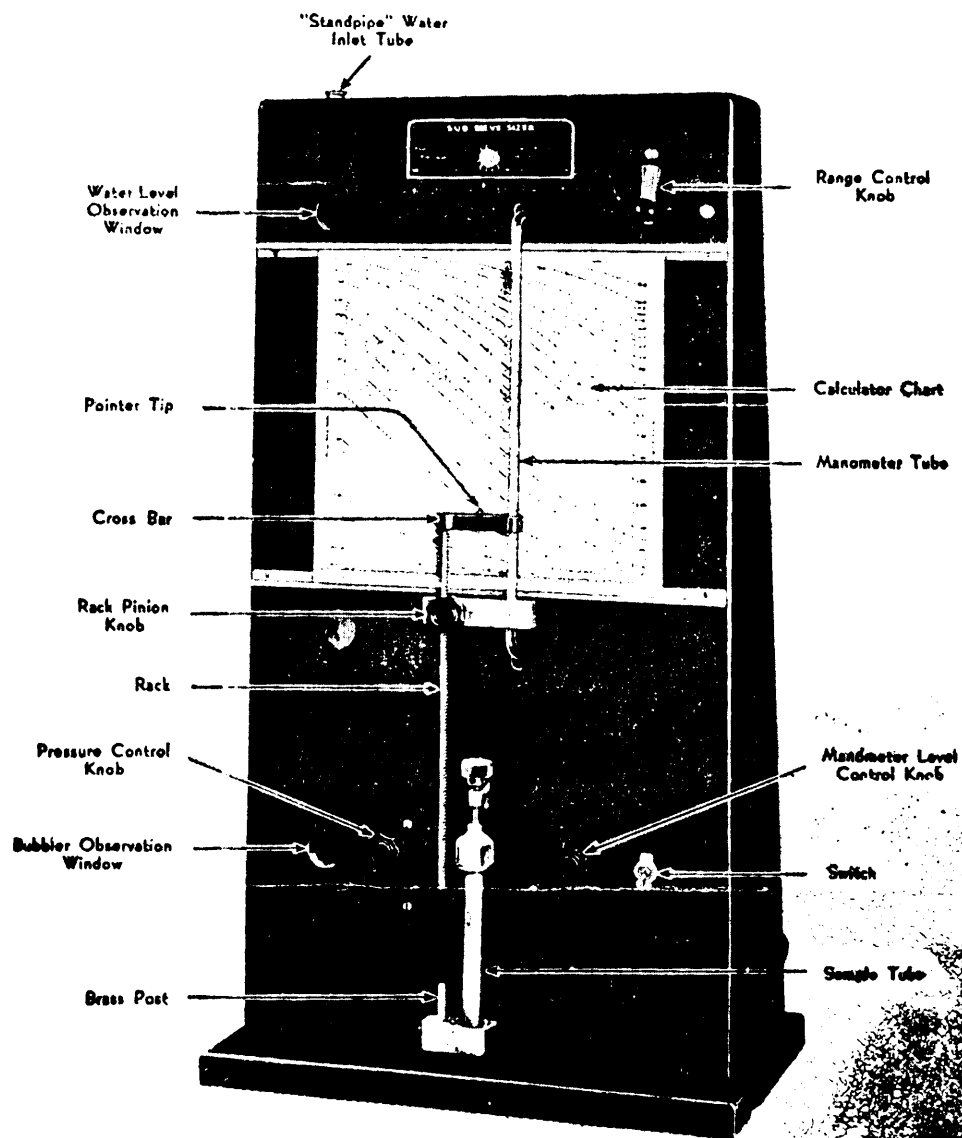


FIGURE 1.

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The Fisher Sub Sieve Sizer

FIGURE 2.

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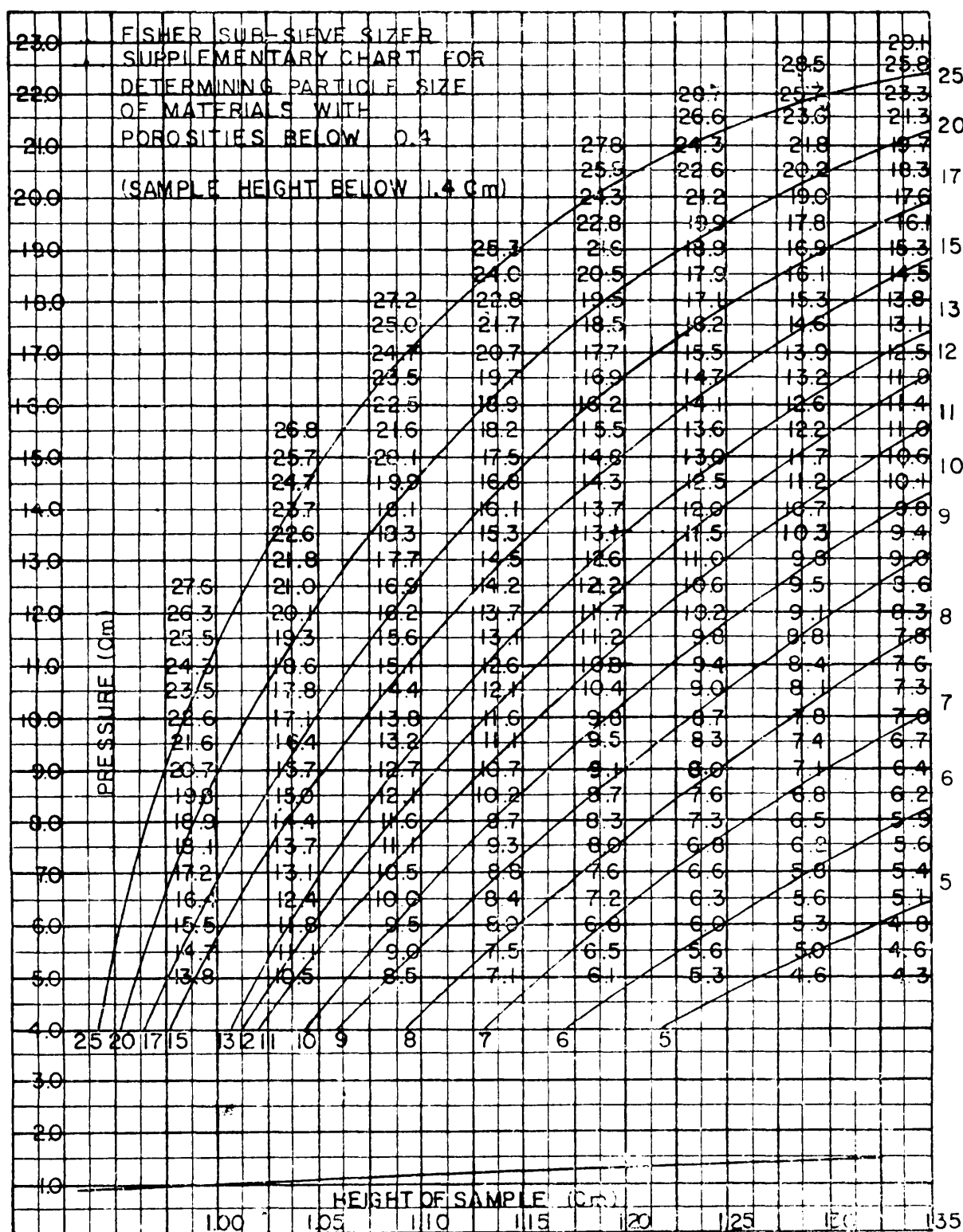


FIGURE 3.

Method 202.1

METHOD 203.1

HYGROSCOPICITY (EQUILIBRIUM METHOD)

1. SCOPE

1.1 This test is used for determining the hygroscopicity of pyrotechnics by measuring the weight gain of the composition after reaching equilibrium with known humidity conditions at 30°C.

2. SPECIMEN

2.1 The specimen shall consist of approximately 5 grams of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Weighing bottle (glass), with cap style ground glass cover; diameter 60 mm, depth 30 mm.

3.2 Desiccator, 10 liter capacity, containing 1 liter of sulfuric acid solution for producing a specified relative humidity at 30°C, as indicated in table I.

TABLE I. Percentages of sulfuric acid required at 30°C for producing definite relative humidities.

Relative Humidity percent	Sulfuric acid percent \pm 0.5
10	65.2
20	59.2
25	56.2
35	51.2
50	43.7
60	39.5
65	36.2
70	33.4
75	30.6
90	18.6

3.3 Oven.

3.4 Desiccator containing an indicating desiccant.

4. PROCEDURE

4.1 Transfer the specimen to a tared weighing bottle.

4.2 Dry the specimen to constant weight by a method suitable for the material being tested. Record the weight of the dry specimen.

4.3 Place the bottle and contents, with cover removed, in a desiccator containing the desired humidity producing solution.

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4.4 Place the desiccator in an oven maintained at a constant temperature of $30^{\circ} \pm 2^{\circ}\text{C}$.

4.5 Remove the bottle from the desiccator after 24 hours, cover each bottle with its ground glass cover, cool to room temperature in a desiccator containing an indicating desiccant and weigh.

4.6 Return the bottle and contents, with the cover removed to the relative humidity desiccator and store at $30^{\circ} \pm 2^{\circ}\text{C}$. for another 24-hour period. Again remove the bottle from the desiccator, cover each bottle with its ground glass cover, cool to room temperature in a desiccator containing an indicating desiccant and weigh.

4.7 Repeat the storing and weighing procedure until the change in weight between successive 24-hour intervals is no greater than 0.2 mg.

4.8 Calculate the gain in weight to percent hygroscopicity at the conditions specified for relative humidity and temperature as follows:

$$\text{Percent hygroscopicity} = \frac{100(A - W)}{W}$$

where:

A = weight of specimen after conditioning at the specified condition, gm.

W = weight of dry sample, gm.

METHOD 204.1

SELECTIVE SOLVENT EXTRACTION (LEACHING METHOD)

1. SCOPE

1.1 This method is used for determining a component in a pyrotechnic composition by the solvent extraction method.

Note. This method is applicable to a variety of extraction conditions, and is not intended to restrict or limit such factors as the solvent or apparatus used, or the time of extraction. For specific instructions, refer to the applicable pyrotechnic specification.

2. SPECIMEN

2.1 The specimen shall consist of an accurately weighed portion of the pyrotechnic as specified in the applicable method or specification.

3. APPARATUS

3.1 Filtering crucible, Gooch, Sela or pyrex

3.2 Filtering suction apparatus.

3.3 Oven.

4. MATERIALS

4.1 Extracting solvent such as water, organic solvent or acid or alkali solution, as specified in the applicable pyrotechnic specification.

5. PROCEDURE

5.1 Transfer the specimen to a tared filtering crucible.

5.2 Add 5 ml of the suitable solvent to the crucible.

5.3 Allow the solvent to remain in contact with the specimen at least 1 minute before applying suction.

5.4 Repeat the extraction with additional 5 ml portions of the solvent until the ingredient being determined is completely removed (as indicated by a suitable qualitative test) or until a measured volume of suitable solvent has been used (the volume being specified in the applicable pyrotechnic specification).

6.5 Aspirate the crucible and residue until no trace of the extracting solvent can be detected by examination.

5.6 Dry the crucible and residue in an oven at a suitable temperature, cool in a desiccator and weigh.

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5.7 Calculate the percent ingredient as follows:

$$\text{Percent ingredient} = \frac{(W - A) 100}{W}$$

where:

A = weight of residue, gm.

W = weight of specimen, gm.

METHOD 204.2

SELECTIVE SOLVENT EXTRACTION (EXTRACTION METHOD)

1. SCOPE

1.1 This method is used for determining a component in a pyrotechnic composition by the solvent extraction method where a lengthy extraction period between solvent and component is necessary.

Note. This method is applicable to a variety of extraction conditions, and is not intended to restrict or limit such factors as the solvent or apparatus used, or the time of extraction. For specific instructions, refer to the applicable pyrotechnic specification.

2. SPECIMEN

2.1 The specimen shall consist of an accurately weighed portion of the pyrotechnic as specified in the applicable method or specification.

3. APPARATUS

3.1 Beaker.

3.2 Filtering crucible, Gooch, fritted glass, or sels porcelain or equivalent.

3.3 Filtering suction apparatus.

3.4 Oven.

4. MATERIALS

4.1 Extracting solvent, such as water, organic solvent or acid or alkali solution, as specified in the applicable pyrotechnic specification.

5. PROCEDURE

5.1 Transfer the specimen to a beaker.

5.2 Add a sufficient amount of the extraction solvent to dissolve the ingredient being determined.

5.3 Heat the beaker and contents on a steam bath with occasional stirring until all the ingredient being determined is dissolved.

5.4 Allow the solution to cool to room temperature and filter through a tared filtering crucible

5.5 Wash the crucible and residue with the solvent until the ingredient is completely removed (as indicated by a suitable qualitative test) or until a measured volume of suitable

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solvent has been used to wash the residue (as specified in the applicable pyrotechnic specification).

6.6 Calculate the percent ingredient being determined as follows:

$$\text{Percent ingredient} = \frac{(W - A)100}{W}$$

where:

A = weight of residue, gm.

W = weight of specimen, gm.

METHOD 204.3

SELECTIVE SOLVENT EXTRACTION (SOXHLET METHOD)

1. SCOPE

1.1 This method is used for determining a component in a pyrotechnic composition by the solvent extraction method, where a long extraction period is necessary.

Note. This method is applicable to a variety of extraction conditions, and is not intended to restrict or limit such factors as the solvent or apparatus used, or the time of extraction. For specific instructions, refer to the applicable pyrotechnic specification.

2. SPECIMEN

2.1 The specimen shall consist of an accurately weighed portion of the pyrotechnic as specified in the applicable method or specification.

3. APPARATUS

3.1 Extraction thimble.

3.2 Extractor (Soxhlet or equivalent).

3.3 Hotplate (Preferably steam or hot water heated).

3.4 Condenser (Allihn type or equivalent).

3.5 Vacuum desiccator maintained at a pressure (absolute) of approximately 25 mm of mercury over an indicating desiccant.

3.6 Extraction flask.

3.7 Dry air stream.

4. MATERIALS

4.1 Extracting solvent such as water, organic solvent or acid or alkali solution, as specified in the applicable pyrotechnic specification.

5. PROCEDURE

5.1 Transfer the specimen to the extraction thimble and the prescribed solvent to a tared extraction flask.

5.2 Assemble the extraction apparatus on a hotplate and adjust the condenser at the rate of two or three drops per second.

5.3 Continue the extraction to completion as indicated by a suitable qualitative test or for a specified period of time (designated in the applicable pyrotechnic specification.)

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5.4 When the extraction is complete, evaporate the solvent, using a stream of dried air.

5.5 Dry the flask and residue in a vacuum desiccator until the change in weight between the weighings at 2 hour intervals does not exceed 2 mg.

5.6 Calculate the percent ingredient as follows:

$$\text{Percent ingredient} = \frac{A \ 1 \ 0 \ 0}{W}$$

where:

A = weight of residue, gm.

W = weight of sample, gm.

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METHOD 204.4
SELECTIVE SOLVENT EXTRACTION
(INSOLUBLE RESIDUE METHOD)

1. SCOPE

1.1 This method is used for determining a component in pyrotechnic composition by removing all other constituents by the solvent extraction method, leaving the desired component as a residue.

Note. This method is applicable to a variety of extraction conditions, and is not intended to restrict or limit such factors as the solvent or apparatus used, or the time of extraction. For specific instructions, refer to the applicable pyrotechnic specification.

2. SPECIMEN

2.1 The specimen shall consist of an accurately weighed portion of the pyrotechnic as specified in the applicable method or specification.

3. APPARATUS

3.1 The apparatus used in this method is identical with that used in Method 201.1, 201.2, or 201.3 according to the extraction method employed (see par. 5.2).

4. MATERIALS

4.1 The materials used in this method are identical with those used in Method 201.1, 201.2 or 201.3 according to the extraction method employed (see par. 5.2),

Note. The ideal selective solvent should extract out of the composition the materials other than the component being determined. In many cases it may be found that the component being determined as insoluble residue may be slightly soluble in the solvent employed. It is then necessary to saturate the solvent with the ingredient being determined prior to extraction.

5. PROCEDURE

5.1 Transfer the specimen to a beaker, crucible or extraction thimble as specified in the applicable pyrotechnic specification.

5.2 Dissolve, leach or extract out of the specimen all of the ingredients, other than the one being determined, by the use of suitable solvents as specified in Method 201.1, 201.2, or 201.3.

5.3 Dry the residue in an oven at an applicable temperature for the material, cool in a desiccator and weigh.

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5.4 Calculate the percent ingredient as follows:

$$\text{Percent ingredient} = \frac{A 100}{W}$$

where:

A = weight of residue, gm.

W = weight of sample, gm.

METHOD 301.1
SAMPLE PREPARATION (DISSOLUTION)

1. SCOPE

1.1 This method is used to dissolve a pyrotechnic for 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 Steam bath.

3.2 Stirring rod.

3.3 Watchglass.

3.4 Beaker.

4. MATERIALS

4.1 Dissolving solvent, such as water, organic solvent or acid or alkali solutions as specified in the applicable method or explosive specification,

5. PROCEDURE

5.1 Transfer the specimen to a beaker.

5.2 Add sufficient solvent to the beaker to dissolve the specimen. Cover the beaker and heat on a steam bath with occasional stirring to aid the dissolution.

5.3 When the specimen is completely dissolved, remove the covered beaker from the steam bath and allow to cool to room temperature.

5.4 Retain the solution for subsequent determinations.

METHOD 301.2
SAMPLE PREPARATION
(DISSOLUTION — EXTRACTION)

1. SCOPE

1.1 This method is used for extracting water soluble material from pyrotechnics containing binder or bonding material 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 Beakers.

3.2 Steam bath.

3.3 Watchglass.

3.4 Stirring rod.

4. MATERIALS

4.1 Dissolving solvent, such as benzene, toluene, or acetone as specified in the applicable method or pyrotechnic specification.

5. PROCEDURE

5.1 Follow the procedure described in Method 301.1 for the dissolution of the specimen.

5.2 To the solution retained for subsequent determinations, add a volume of distilled water to bring the solvent soluble material out of solution. The volume of water should be at least equal to the volume of solvent used in Method 301.1.

5.3 Allow the solution to stand until all precipitated material has settled and retain the beaker and contents for subsequent determinations.

METHOD 301.3

SAMPLE PREPARATION (EXTRACTION)

1. SCOPE

1.1 This method is used for extracting soluble ingredients from a pyrotechnic for use in subsequent determinations or to prevent it from interfering with subsequent determinations.

2. SPECIMEN

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

3. APPARATUS

3.1 Beakers.

3.2 Watchglass.

3.3 Steam bath.

3.4 Stirring rod.

3.5 Filtering crucible, sintered glass.

3.6 Suction filtering apparatus.

3.7 Oven.

4. MATERIALS

4.1 Extracting solvent, such as water, organic solvent or acid or alkali solution as specified in the applicable method or explosive specification.

5. PROCEDURE

5.1 Transfer the specimen to a beaker.

5.2 Add 100 ml of solvent to the beaker.

5.3 Cover the beaker with a watch glass and place it on a steam bath.

5.4 Heat the beaker with occasional stirring until all soluble material is in solution as shown by the lack of change in the amount of undissolved material in the beaker.

5.5 Decant the hot supernatant liquid through a filtering crucible, collecting the filtrate in a clean beaker.

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5.6 Repeat the extraction with an additional 100 ml of solvent, decanting through the same crucible and adding this filtrate to the filtrate from the first extraction.

5.7 Transfer the residue to the crucible and wash with suitable solvent until free of soluble material, adding the washings to the filtrate solutions.

5.8 Aspirate the residue until no trace of the extraction solvent can be detected by examination.

5.9 Retain the residue and the filtrate for subsequent determinations.

METHOD 301.4

SAMPLE PREPARATION (LEACHING)

1. SCOPE

1.1 This method is used for leaching out a soluble ingredient from a pyrotechnic for use in subsequent determinations or to prevent it from interfering with 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 Filtering crucible, sintered glass.

3.2 Suction filtering apparatus.

3.3 Beaker.

3.4 Oven.

4. MATERIALS

4.1 Leaching solvent, such as water, organic solvent or acid or alkali solutions as specified in the applicable method or explosive specification.

5. PROCEDURE

5.1 Transfer the specimen to a filtering crucible.

5.2 Leach the specimen with successive 10 ml portions of the leaching solvent, allowing each successive portion to remain in contact with the specimen for approximately 2 minutes before applying suction.

5.3 Catch the filtrate in a clean beaker.

5.4 Continue the leaching until all soluble material is removed as indicated by an applicable qualitative test on a drop of filtrate coming through the crucible or achieving constant weight of the crucible and contents.

5.5 Aspirate the residue in the crucible until no trace of the leaching solvent is detected by examination.

5.6 Retain the residue and the filtrate for subsequent determinations.

METHOD 301.5
SAMPLE PREPARATION
(ORGANIC DESTRUCTION AND SAMPLE DISSOLUTION)

1. SCOPE

1.1 This method is used to bring pyrotechnics into solution via destruction of the organic matter.

2. SPECIMEN

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

3. APPARATUS

3.1 Beaker, 400 ml.

3.2 Watchglass, ribbed.

3.3 Hotplate in well-ventilated hood.

4. MATERIALS

5.1 Nitric acid, 70-percent.

4.2 Sulfuric acid, concentrated.

5. PROCEDURE

5.1 Transfer the specimen to a beaker and cover the beaker with a watchglass.

5.2 Cover the specimen with water.

5.3 Add 10 ml of nitric acid and warm to dissolve the specimen.

5.4 Add 10 ml of sulfuric acid.

5.5 Evaporates on a hotplate until fumes of sulfur trioxide are evolved.

5.6 Allow the beaker and contents to cool and then add 5 ml of nitric acid.

5.7 Reheat the beaker and contents until the fumes of sulfur trioxide are evolved.

5.8 Repeat steps 5.6 and 5.7 until all organic matter is destroyed as indicated by the

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sulfuric acid solution in the beaker being colorless. (Make sure all the nitric acid is removed by fuming a little more.)

5.9 Remove the beaker from the hotplate and allow to cool.

5.10 Wash down the watchglass cover and side walls of the beaker, cautiously with a stream of approximately 25 ml of distilled water.

5.11 Cover the beaker with the watchglass and retain the solution for subsequent determinations.

METHOD 301.6

SAMPLE PREPARATION (SOXHLET EXTRACTION)

1. SCOPE

1.1 This method is used for extracting soluble ingredients from a 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, carbon tetrachloride 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 301.7
SAMPLE PREPARATION
(MULTIPLE SOLVENT EXTRACTION)

1. SCOPE

1.1 This method is used for extracting solvent soluble ingredients from pyrotechnics for use in subsequent determination or to prevent them from interfering with 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 Beaker.

3.2 Steam bath.

3.3 Filter stick or filtering crucible.

3.4 Suction filtering apparatus.

4. MATERIALS

4.1 Extracting solvents such as water, organic solvents or acid or alkali solution as specified in the applicable method or pyrotechnic specification.

5. PROCEDURE

5.1 Transfer the specimen to a beaker.

5.2 Add approximately 25 ml of solvent to the beaker.

5.3 Cover the beaker with a watchglass and place it on a steam bath.

5.4 Heat the beaker with occasional swirling until all soluble material is in solution.

5.5 Remove the beaker and contents from the steam bath and allow to cool to room temperature.

5.6 Remove all the solution by means of the filter stick and suction or by decanting the solution through a sintered glass crucible. Wash with the same solvent.

5.7 Add approximately 25 ml of a second solvent to the beaker and repeat step 5.3 to 5.6.

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5.8 Repeat the extraction with additional solvents until only the desired material remains in the beaker.

5.9 Retain the beaker and contents for subsequent determinations.

METHOD 401.1

CHLORIDE (SILVER NITRATE METHOD)

1. SCOPE

1.1 This method is used to determine the chloride content or the chloride compound content of pyrotechnics in the absence of sulfides, cyanides and halogens.

2. SPECIMEN

2.1 The specimen shall consist of a portion of the pyrotechnic weighed to within 0.2 mg, containing approximately 0.1 gm of chlorine.

3. APPARATUS

3.1 Hotplate.

3.2 Gooch crucible or equivalent.

3.3 Suction filtering apparatus.

3.4 Oven.

3.5 Desiccator containing indicating desiccant.

3.6 Test tube.

4. MATERIALS

4.1 Silver nitrate solution (approximately 1.0N). Dissolve approximately 17 gm of silver nitrate in 100 ml of water.

4.2 Nitric acid solution — 35 percent (1:1).

4.3 Wash solution, 1 ml of 70 percent nitric acid per 100 ml of distilled water.

4.4 Hydrochloric acid solution (approximately 0.1N). Dilute 9.5 ml of HCL with water to make 1000 ml.

5. PROCEDURE

5.1 Leach the specimen with suitable solvent to remove the chloride as specified in Method 301.4 retaining the filtrate in a beaker.

5.2 Make the solution just acid with nitric acid and add 5 to 10 ml of nitric acid in excess.

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5.3 If the water solution contains a chloride and a heavy metal which form basic salts (e.g., stannic, ferric, etc. solutions), or which may tend to reduce the silver solution, it is necessary to remove these by precipitation with ammonium hydroxide or potassium carbonate solution as follows:

- (a) To the solution, acidified with acid, and containing an excess of the latter, add sufficient ammonium hydroxide (free from chloride) to precipitate the heavy metals.
- (b) Filter, and wash the residue several times with distilled water catching the filtrate and washings in a beaker.
- (c) Add 5 to 10 ml of nitric acid to the filtrate. Add acid until acid is indicated by litmus.

5.4 To the acidified original solution, if it contained no heavy metals, or to the acidified filtrate from the solution from which heavy metals have been removed, add sufficient silver nitrate reagent, slowly and with constant stirring, to completely precipitate the chlorides present. Test for complete precipitate, allowing the precipitate to settle, adding a few drops of silver nitrate solution to the supernatant liquid and seeing whether further precipitation takes place.

Caution: Carry out the determination in subdued light.

5.5 Heat the suspension nearly to boiling, while stirring constantly, and maintain it at this temperature until the precipitate coagulates and the supernatant liquid is clear (2 to 3 min.).

5.6 Test for completeness of precipitation by adding a few drops of silver nitrate solution to the supernatant liquid.

5.7 If no further precipitate appears, set the beaker aside in the dark, and allow the solution to stand for at least 1 to 2 hours before filtration.

5.8 Decant the solution through a tared filtering crucible.

5.9 Wash the precipitate four times by recantation using 20 ml portions of wash solution.

5.10 Transfer the precipitate to the crucible with the wash solution.

5.11 Wash the precipitate with the wash solution until 3 to 5 ml of the washings, collected in a test tube, show no turbidity with 1 to 2 drops of 0.1N hydrochloric acid.

5.12 Wash thoroughly with distilled water.

5.13 Dry the crucible and precipitate at $135^{\circ} \pm 5^{\circ}\text{C.}$ for 4 hours, cool in a desiccator and weigh.

5.14 Calculate as follows:

$$\text{Percent chloride} = \frac{A \ 24.74}{W}$$

$$\text{Percent substance sought} = \frac{100AB}{nC W}$$

where:

- A = weight of precipitate, gm.
- B = molecular weight of substance sought.
- W = weight of specimen, gm.
- n = number of chlorine atoms in substance sought.
- C = molecular weight of silver chloride.

METHOD 402.1
CHLORATE (FERROUS SULFATE METHOD)

1. SCOPE

1.1 This method is used to determine the chlorate compound content of pyrotechnics in the absence of halides, sulfides, and cyanides.

2. The specimen shall consist of approximately 5 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Volumetric flask, 250 ml.

3.2 Pipet.

3.3 Beaker, 400 ml.

3.4 Watchglass.

4. MATERIALS

4.1 Ferrous sulfate, 10 percent solution.

5. PROCEDURE

5.1 Extract the specimen with a suitable solvent to remove the chlorate compound as specified in Method No. 301.3, collecting the filtrate in a beaker.

5.2 Transfer the filtrate to a 250 ml volumetric flask and dilute to the mark with water.

5.3 Transfer an aliquot containing approximately 0.2 gm of the chlorate to a 400 ml beaker and dilute to approximately 100 ml.

5.4 Add 50 ml of the ferrous sulfate solution. Cover with watchglass.

5.5 Heat to boiling with constant stirring.

5.6 Boil the solution for 15 min.. and allow to cool.

5.7 Add nitric acid to the cooled solution until the deposited basic ferric salt is dissolved.

5.8 Precipitate the chloride as silver chloride as specified in Method 401.1, par. 5.3 to 5.12.

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5.9 Calculate the chlorate compound content as follows:

$$\text{Percent chlorate compound} = \frac{100AB}{nCW}$$

where:

- A = weight of silver chloride precipitate, gm.
- B = molecular weight of chlorate compound.
- C = molecular weight of silver chloride.
- W = weight of specimen, represented by the aliquot, gm.
- n = number of chloride atoms in compound.

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METHOD 403.1

PERCHLORATE (AMMONIUM CHLORIDE METHOD)

1. SCOPE

1.1 This method is used to determine the perchlorate content of pyrotechnics in the absence of cyanides, chlorates, and halogens.

2. SPECIMEN

2.1 The specimen shall consist of approximately 5 gm of the pyrotechnic, weighed to within 0.2 mg.

3. APPARATUS

3.1 Volumetric flask, 250 ml.

3.2 Hotplate.

3.3 Steam bath.

3.4 Evaporating dish, platinum.

3.5 Watchglass.

3.6 Beaker, 400 ml.

4. MATERIALS

4.1 Ammonium chloride, reagent grade.

4.2 Nitric acid, 70-percent.

4.3 Silver nitrate solution, 10-percent.

5. PROCEDURE

5.1 Leach the specimen with a suitable solvent to remove the perchlorate as specified in Method 301.4, retaining the filtrate in a beaker.

5.2 Transfer to a volumetric flask and dilute to the mark with distilled water.

5.3 Transfer an aliquot containing approximately 0.4 gm of the perchlorate to a platinum evaporating dish.

5.4 Carefully evaporate the contents on a hotplate to almost dryness and complete the evaporation to dryness on a steam bath.

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5.5 Intimately mix the residue with 1.5 gm of ammonium chloride.

5.6 Cover the evaporating dish with a watchglass or lid and ignite gently until fuming ceases. Continue the heating for 1 hour at a temperature below the fusing point.

Caution: Do not fuse the resulting chloride as the crucible may be attacked.

5.7 Repeat the ignition with another 1.5 gm of ammonium chloride.

5.8 Allow the dish and contents to cool and quantitatively transfer the residue to a clean 400-ml beaker, with the aid of a stream of hot distilled water.

5.9 Dilute the filtrate to about 200 to 250 ml with distilled water.

5.10 Add 4 ml of nitric acid and precipitate the chloride by the addition of silver nitrate solution as specified in Method 401.1, paragraphs 5.3 to 5.12.

5.11 Calculate the perchlorate compound content as follows:

$$\text{Percent perchlorate compounds} = \frac{100AB}{nCW}$$

where:

A = weight of silver chloride precipitate, gm.

B = molecular weight of perchlorate compound.

C = molecular weight of silver chloride.

W = weight of specimen represented by the aliquot, gm.

n = number of chloride atoms in compound.

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METHOD 404.1
HEXACHLOROBENZENE (PARR BOMB METHOD)

1. SCOPE

1.1 This method is used to determine hexachlorobenzene content of pyrotechnics in the absence of cyanides and halogens.

2. SPECIMEN

2.1 The specimen shall consist of approximately 1 gm of the pyrotechnic, weighed to within 0.2 gm.

3. APPARATUS

3.1 Fitting crucible sintered glass.

3.2 Steam bath.

3.3 Parr bomb with water jacket.

3.4 Steel spatula.

3.5 Beaker, 600 ml.

4. MATERIALS

4.1 Carbon disulfide.

4.2 Benzoic acid, C. P. Grade.

4.3 Sodium peroxide, C. P. Grade.

4.4 Silver nitrate solution, 10 percent.

4.5 Nitric acid, 35 percent (1:1).

5. PROCEDURE

5.1 Leach the specimen with carbon disulfide to remove the hexachlorobenzene as specified in Method No. 301.4.

5.2 Evaporate the extract on a steam bath to a volume of approximately 10 ml and transfer this to the fusion cup of a Parr Bomb.

5.3 Place the cup and contents on a steam bath and allow the solvent to evaporate.

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5.4 Rinse the beaker with carbon disulfide to remove any residual hexachlorobenzene, then transfer the rinsings to the cup and allow the solvent to evaporate.

5.5 Gently loosen the residue adhering to the inner surface of the cup by means of a steel spatula. This step is necessary in order to insure the proper mixing with the other ingredients used in the bomb.

5.6 Mix the residue thoroughly with 0.500 gm of finely powdered benzoic acid.

5.7 Attach the fuse to the bomb head, then quickly transfer a portion of approximately 15 gm of sodium peroxide to the fusion cup and thoroughly mix the peroxide with the other ingredients in the cup.

5.8 Place the bomb under the contact arm in the water jacket and allow a continuous stream of cold water to pass through the jacket.

5.9 Ignite the charge, using a current of approximately 3 amperes.

5.10 Allow the bomb to cool in the water jacket for about 5 min.

5.11 Dismantle the bomb, rinse the cup with hot water, and collect the rinsings in a 600 ml beaker.

5.12 Place the fusion cup on its side in the beaker and add 100 ml of water; cover the beaker with a watchglass and heat until decomposition of sodium peroxide incomplete.

5.13 Remove the cup, rinse it with water, and dilute the solution in the beaker to 300 ml. If there is a precipitate, filter the solution and wash the precipitate with hot water until free of chlorides.

5.14 Render the solution faintly acid by the addition of 35 percent nitric acid solution and precipitate the chloride by the addition of 20 ml of silver nitrate solution as specified in Method 401.1 paragraphs 5.3 to 5.12.

5.15 Calculate the percentage of hexachlorobenzene in the sample as follows:

$$\text{Percent hexachlorobenzene} = \frac{33.12A}{W}$$

where:

A = weight of precipitate, gm.

W = weight of the specimen, gm.

METHOD 405.1

LEAD SULFOCYANATE (SILVER NITRATE METHOD)

1. SCOPE

1.1 This method is used to determine the lead sulfocyanate (lead thiocyanate) content of pyrotechnics in the absence of halides and cyanides.

2. SPECIMEN

2.1 The specimen shall consist of approximately 1 gm. of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Filtering crucible, medium porosity, sintered glass.

3.2 Erlenmeyer flask, 500 ml.

3.3 Suction filtering apparatus.

3.4 Titration apparatus.

4. MATERIALS

4.1 Potassium nitrate solution, 5-percent.

4.2 Nitric acid, 3N solution (Nitric acid shall be water-white and free from oxides of nitrogen).

4.3 Silver nitrate, 0.1N standard solution as specified in Method 607.1.

4.4 Ferric ammonium sulfate indicator solution as specified in Method 705.1.

4.5 Potassium sulfocyanate, 0.1N standard solution as specified in Method 608.1.

4.6 Ferric ammonium sulfate 0.15N standard solution as specified in Method 6031.

5. PROCEDURE

5.1 Transfer the specimen to a tared, porosity sintered glass crucible.

5.2 Extract the contents of the crucible with five 20 ml portions of a 5-percent potassium nitrate solution catching the filtrate in a 500 ml Erlenmeyer flask. The contents of the crucible shall be stirred after the addition of each portion of the potassium nitrate solution has been allowed to remain in contact with the sample for a minimum of 5 min. before suction is applied.

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5.3 Wash the contents of the crucible with 5 ml portions of distilled water, adding these washings to the filtrate, until all the sulfocyanate has been extracted.

5.4 Add exactly 35 ml of standardized 0.1N silver nitrate to the filtrate and washings in the 500 ml Erlenmeyer flask.

5.5 Swirl the flask and contents, and add 10 ml of 3N nitric acid. Continue the swirling of the mixture for 5 min.

5.6 Add five ml of ferric ammonium sulfate indicator and titrate the mixture with standardized 0.1N potassium sulfocyanate solution to the first permanent pale rose coloration.

5.7 Calculate the percentage of lead sulfocyanate in the sample as follows:

$$\text{Percent lead sulfocyanate} = \frac{(35A - BC) 16.17}{W}$$

where:

A = normality of silver nitrate solution.

B = volume in ml of 0.1N potassium sulfocyanate solution.

C = normality of 0.1N potassium sulfocyanate solution.

W = weight of sample.

METHOD 406.1

BARIUM SALTS (SULFATE METHOD)

1. SCOPE

1.1 This method is used to determine the percent of barium compound in a pyrotechnic in the absence of strontium, calcium and lead.

2. SPECIMEN

2.1 The specimen shall consist of approximately 5 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Gooch crucible or equivalent.

3.2 Hotplate.

3.3 Volumetric flask, 500 ml.

3.4 Beaker.

4. MATERIALS

4.1 Sulfuric acid, concentrated.

4.2 Hydrochloric acid, 38 percent.

5. PROCEDURE

5.1 Dissolve the barium salts as specified in Method 301.3, retaining the filtrate in a beaker.

5.2 Transfer the filtrate to a 500 ml volumetric flask and dilute to the mark with distilled water.

5.3 Transfer a 50 ml aliquot of the solution to a 250 ml beaker.

Caution: The aliquot should contain not more than 0.15 gm. of barium. A smaller aliquot should be taken, if necessary, so as not to exceed this maximum.

5.4 Add 5 ml of concentrated sulfuric acid, and evaporate until fumes of sulfur trioxide are evolved.

5.5 Cool the liquid, dilute with 100 ml of distilled water, add 2 ml of concentrated sulfuric acid, and heat to boiling.

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5.6 Allow the hot solution to stand on a steam bath or hotplate until the precipitate of barium sulfate has settled, and filter through a previously ignited and tared Gooch crucible.

5.7 Wash the precipitate ten times with 25 ml portions of hot water, dry in an oven at $100^{\circ} \pm 5^{\circ}\text{C}$. for 1 hour and in a muffle furnace at approximately 750°C . for 30 min., cool in a desiccator and weigh.

5.8 Calculate the percentage of barium salt in the sample as follows:

$$\text{Percent barium compound} = \frac{100AB}{nCW}$$

where:

A = weight of precipitate, gm.

W = weight of sample, gm.

B = molecular weight of barium compound.

C = molecular weight of barium sulfate.

n = number of barium atoms in compound.

METHOD 406.2
BARIUM SALTS (CHROMATE METHOD)

1. SCOPE

1.1 This method is used to determine the barium compound content of pyrotechnics in the presence of strontium and calcium.

2. SPECIMEN

2.1 The specimen shall consist of approximately 5 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Hotplate.

3.2 Gooch crucible or equivalent.

3.3 Volumetric flask, 500 ml.

3.4 Beaker, 400 ml.

3.5 Pipet.

4. MATERIALS

4.1 Ammonium acetate solution, 40 percent.

4.2 Potassium bichromate solution (sulfate free), 10 percent.

4.3 Potassium Bichromate.

4.4 Wash solution urea.

4.5 Hydrochloric acid.

5. PROCEDURE

5.1 Extract the specimen with suitable solvent as specified in Method 301.3 retaining the filtrate in a beaker.

5.2 Transfer the extract to a 500 ml volumetric flask and dilute to the mark with distilled water.

5.3 Pipet a 100 ml aliquot of the solution into a 400 ml beaker and dilute to 250 ml with distilled water.

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Caution: The aliquot shall not contain more than 0.4 gm. of barium, a smaller aliquot should be taken, if necessary, so as not to exceed this maximum.

5.4 Add 7 ml of hydrochloric acid, and stir to dissolve.

5.5 Heat to about 80°C. and add with stirring 10 ml of ammonium acetate solution, 25 ml of potassium dichromate solution, and 10 gm of urea.

5.6 Cover with a watchglass, heat to boiling, boil moderately until a precipitation settles on the bottom of the beaker and then continue boiling moderately for 60 to 65 minutes.

5.7 Midway during the 50 to 65 minute boiling period, wash down the cover lid with water and bring the volume to 225 to 250 ml by adding hot water.

5.8 Filter through a tared sintered glass crucible of medium porosity, transfer the precipitate to the crucible with potassium bichromate wash solution.

5.9 Wash the crucible and contents four times with water.

5.10 Dry the barium chromate precipitate for one hour in a oven at $120^{\circ} \pm 5^{\circ}\text{C}$.

5.11 Cool in a desiccator and weigh. Calculate the increase in weight to percent barium salt as follows:

$$\text{Percent barium compound} = \frac{100AB}{nCW}$$

where:

- A = weight of precipitate, gm.
- B = molecular weight of the barium compound.
- C = molecular weight of barium chromate.
- n = number of barium atoms in compound.
- W = weight of specimen, represented by the aliquot, gm.

METHOD 407.1
ALUMINUM (AMMONIUM HYDROXIDE METHOD)

1. SCOPE

1.1 This method is used to determine aluminum in pyrotechnics, (iron, titanium, nickel, zinc and copper may interfere).

2. SPECIMEN

2.1 The specimen shall consist of approximately 0.5 gm of the pyrotechnic, weighed to within 0.2 mg.

3. APPARATUS

3.1 Beaker, 400 ml (2 required).

3.2 Watchglass, ribbed.

3.3 Hotplate.

3.4 Tongs, rubber tipped.

3.5 Filter paper, 41 Whatman or equivalent.

3.6 Crucible, porcelain or platinum.

3.7 Muffle furnace.

3.8 Desiccator with indicating desiccant.

4. MATERIALS

4.1 Hydrochloric acid solution, 50-percent.

4.2 Ammonium chloride, reagent grade.

4.3 Nitric acid, 70-percent.

4.4 Methyl red indicator solution as specified in Method 702.1.

4.5 Ammonium hydroxide, concentrated.

4.6 Ammonium nitrate solution, 2-percent.

5. PROCEDURE

5.1 Leach the specimen in a suitable solvent to remove interfering material as speci-

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fied in Method 301.4, retaining the residue containing the aluminum in a filtering crucible of medium porosity.

5.2 Transfer the crucible and the residue to a beaker.

5.3 Add carefully 25 ml of hydrochloric acid solution and cover the beaker with a watchglass.

5.4 After the reaction has subsided, place the beaker and contention a hotplate and boil for 5 minutes.

5.5 Remove the beaker from the hotplate and allow to cool.

5.6 Remove the glass crucible with rubber tipped tongs and wash the side and bottom of the crucible several times with water, catching the washings in the beaker.

5.7 Filter the contents of the beaker through the same crucible and wash the beaker and crucible with ten 15 ml portions of water, catching the filtrate and washings in a clean beaker.

5.8 Add 5 gm of ammonium chloride and 5 ml of nitric acid and heat the solution to boiling on a hot plate.

5.9 Remove the beaker from the hot plate and allow to cool.

5.10 Add 2 to 3 drops of methyl red indicator solution and then add dropwise ammonium hydroxide until the indicate change to yellow is obtained.

5.11 Heat the solution to boiling and filter through No. 4 Whatman filter paper.

5.12 Wash the precipitate several times with a 2-percent ammonium nitrate solution.

5.13 Transfer the filter paper and precipitate to a porcelain or platinum crucible.

5.14 Dry the crucible and contents and then ignite in a muffle furnace, gently at first, until the paper is thoroughly charred, and then gradually increase the heat.

5.15 Cover the crucible and heat at 1000° to 1100° for 1 hour.

5.16 Cool in a desiccator and weigh rapidly.

5.17 Calculate the percent aluminum as follows:

$$\text{Percent aluminum} = \frac{62.91A}{W}$$

where:

A = weight of precipitate, gm.

W = weight of sample, gm.

METHOD 407.2
ALUMINUM (8-HYDROXY QUINOLINE METHOD)

1. SCOPE

1.1 This method is used to determine the aluminum content in pyrotechnics, in the presence of magnesium.

2. SPECIMEN

2.1 The specimen shall consist of a portion of the pyrotechnic weighed to within 0.2 mg, containing approximately 0.02 gm. of aluminum.

3. APPARATUS

3.1 Beaker, 100 ml.

3.2 Watchglass.

3.3 Filter crucible medium porosity sintered glass.

3.4 Beaker, 600 ml.

3.5 Hotplate.

3.6 Steam bath.

3.7 Oven.

4. MATERIALS

4.1 Sulfuric acid, concentrated.

4.2 Bromo phenol blue indicator solution as specified in Method 710.1.

4.3 Ammonium hydroxide solution, 50-percent.

4.4 Oxine reagent solution prepared just prior to use by dissolving 6 gm of 8-hydroxy quinoline in 100 ml of 2N acetic acid.

4.5 Ammonium acetate solution, 2 M.

5. PROCEDURE

5.1 Transfer the specimen to a 100 ml beaker and add 25 ml of distilled water.

5.2 Add carefully 25 ml of concentrated sulfuric acid in 1 to 2 ml portions, keeping the

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beaker covered with a watchglass as much as possible during this operation to prevent loss of material by spattering.

5.3 Heat the liquid in the beaker to boiling and allow to boil for 3 to 5 minutes to insure complete solution of the aluminum and magnesium.

5.4 Cool the beaker and contents to room temperature.

5.5 Filter the solution through a filtering crucible and wash the insoluble matter thoroughly with water.

5.6 Transfer the combined filtrate and washings to a 600 ml beaker and dilute to approximately 250 ml with water.

5.7 Add 10 drops of brom phenol blue indicator solution, add ammonium hydroxide solution with stirring until the solution turns blue and then add 2 drops ammonium hydroxide in excess.

5.8 Heat the solution to boiling and add 25 ml of oxine reagent solution. Add the oxine reagent solution in small portions and stir the solution in the beaker after each addition.

5.9 Heat the solution to boiling and add, while stirring, 40 ml of 2 M ammonium acetate solution.

5.10 Place the beaker and contents on a steam bath for about 30 minutes to permit the precipitate to settle.

5.11 Filter through a tared filtering crucible and wash with 10 ml portions of cold distilled water, allowing each portion of water to drain through the crucible before adding another.

5.12 Dry the crucible and contents at 130° to 135°C. for approximately 2.5 hours, cool in a desiccator and weigh.

5.13 Calculate the percentage of aluminum in the sample as follows:

$$\text{Percent of aluminum} = \frac{5.871A}{W}$$

where:

W = weight of sample, gm.

A = weight of precipitate, gm.

METHOD 408.1
TOTAL LEAD (CHROMATE METHOD)

1. SCOPE

1.1 This method is used to determine the percentage of total lead and the percent lead compound of pyrotechnics in the absence of barium.

2. SPECIMEN

2.1 The specimen shall consist of a portion of the pyrotechnic weighed to within 0.2 mg, containing approximately 0.3 gm of the lead salt.

3. APPARATUS

3.1 Beaker.

3.2 Hotplate or steam bath.

3.3 Filtering crucible, fine porosity sintered glass has been found satisfactory.

3.4 Oven.

3.5 Desiccator containing indicating desiccant.

4. MATERIALS

4.1 Acetic acid, glacial (JAN-A-465).

4.2 Potassium bichromate, 10-percent solution.

4.3 Ammonium acetate, reagent grade.

4.4 Nitric acid, 70-percent.

4.5 Sulfuric acid solution, 50 percent.

4.6 Ammonium hydroxide, concentrated.

4.7 Methyl red indicator solution as specified in Method 702.1.

5. PROCEDURE

5.1 Prepare the specimen as specified in Method 301.5, retaining the solution in a beaker.

5.2 Add 20 gm of ammonium acetate and one drop of methyl red indicator solution.

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Neutralize with ammonium hydroxide until the solution just begins to turn yellow.

5.3 Heat to a gentle boil until all lead sulfate is dissolved. If any residue remains, filter the solution on a No. 12 Whatman paper and wash the beaker and residue well with hot distilled water.

5.4 Heat the filtrate to boiling and add 10 ml of 10 percent potassium bichromate solution while stirring.

5.5 Digest at just boiling until the precipitate appears bright orange. (This may require digestion for 10 to 15 min.)

5.6 Filter through a tared filtering crucible, and wash well with warm distilled water.

5.7 Dry in an oven at 110°C. for 1 hour. Let cool to room temperature and weigh.

5.8 Calculate the lead content of the sample as follows:

$$\text{Percent total lead} = \frac{64.11A}{W}$$

$$\text{Percent lead compound} = \frac{100AB}{nCW}$$

where:

A = weight of precipitate, gm.

B = molecular weight of lead compound.

C = molecular weight of lead chromate.

W = weight of specimen.

n = number of lead atoms in compound.

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METHOD 408.2

TOTAL LEAD (SULFATE METHOD)

1. SCOPE

1.1 This method is used for determining the percentage of total lead and the percent lead compounds in pyrotechnics containing no barium, strontium or calcium.

2. SPECIMEN

2.1 The specimen shall consist of a portion of the pyrotechnic weighed to within 0.1 mg, containing approximately 0.3 gm of the lead compound.

3. APPARATUS

3.1 Gooch crucible or equivalent.

3.2 Suction filtering apparatus.

3.3 Muffle furnace.

3.4 Desiccator containing an indicating desiccant.

4. MATERIALS

4.1 Ethyl alcohol solution, 50-percent.

4.2 Ethyl alcohol 95-percent.

5. PROCEDURE

5.1 Prepare the specimen for the determination as specified in Method 301.5, retaining the solution in a beaker.

5.2 Add 25 ml of water and 50 ml of 95-percent ethyl alcohol to the beaker.

5.3 Allow the beaker and contents to stand at least 1 hour.

5.4 Filter the precipitate of lead sulfate on a tared Gooch crucible.

5.5 Wash the crucible and residue thoroughly with 1:1 ethyl alcohol solution.

5.6 Aspirate the crucible for 5 minutes then dry for 15 to 30 minutes at 110°C.

5.7 Ignite the crucible in a muffle furnace for 15 minutes at 500° to 600°C.

5.8 Cool to room temperature in a desiccator and weigh.

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5.9 Calculate the percentage of lead or lead compound as follows:

$$\text{Percent total lead} = \frac{68.32A}{W}$$

$$\text{Percent lead compound} = \frac{100AB}{nC W}$$

where:

A = weight of precipitate, gm.

B = molecular weight of the lead compound, gm.

n = number of lead atoms in compound.

C = molecular weight of lead sulfate.

W = weight of specimen, gm.

Note. Alternate Method. In steps 5.2, 5.3 and 5.7. Omit the use of ethyl alcohol and maintain the sulfuric acid content of the solution to about 15 percent and use 15 percent sulfuric acid solution to transfer and wash the precipitate from the beaker to the filtering crucible. A sintered glass crucible may be used in place of the Gooch type and dried at 110° to 115° C. instead of 500° to 600° C.

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METHOD 409.1

TOTAL SULFUR

1. SCOPE

1.1 This method is used to determine the sulfur content of pyrotechnics when the sulfur impartially or totally insoluble in carbon disulfide.

2. SPECIMEN

2.1 The specimen shall consist of approximately 1 gm of the pyrotechnic, weighed to within 0.2 mg.

3. APPARATUS

3.1 Porcelain crucible.

3.2 Muffle furnace.

3.3 Desiccator with indicating desiccant.

3.4 Beaker, 400 ml.

3.5 Beaker, 800 ml.

3.6 Filter paper, 42 Whatman or equivalent.

3.7 Filtering crucible, Gooch type or equivalent.

4. MATERIALS

4.1 Eschka's Mixture. Mix 2 parts of porous calcined magnesia and 1 part of anhydrous sodium carbonate.

4.2 Sodium carbonate, reagent grade.

4.3 Hydrochloric acid, 38-percent.

4.4 Bromine, reagent grade.

4.5 Barium chloride solution, 10-percent.

5. PROCEDURE

5.1 Mix the specimen intimately with approximately 6 gm of Eschka's mixture in a porcelain crucible.

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5.2 Cover the mixture in the porcelain crucible with approximately 2 gm more of the Eschka's mixture.

5.3 Place the crucible and contents in a muffle furnace at room temperature, and heat the furnace so that it reaches a temperature of approximately $800^{\circ} \pm 25^{\circ}\text{C}$. for an additional $1\frac{1}{2}$ hours.

5.4 Cool the crucible and contents, place in a beaker, and add 100 ml of distilled water and 10 gm of sodium carbonate.

5.5 Boil the mixture for at least 10 minutes, and filter through No. 42 Whatman filter paper into a 800 ml beaker.

5.6 Neutralize the solution with hydrochloric acid and add an excess of 4 ml of hydrochloric acid.

5.7 Dilute the solution to 400 ml with distilled water and add several milliliters of bromine.

5.8 Boil off the excess bromine and add 10 ml of the barium chloride solution slowly to the boiling solution.

5.9 Allow the precipitate to settle on a steam bath for at least 2 hours, and filter through a tared Gooch crucible.

5.10 Wash the precipitate with hot water, dry, and ignite at dull red heat for about $\frac{1}{2}$ hour.

5.11 Cool in a desiccator and weigh.

5.12 Calculate the increase in weight of the crucible to percentage of sulfur in the sample as follows:

$$\text{Percent sulfur} = \frac{13.73A}{W}$$

where:

A = weight of precipitate, gm.

W = weight of sample gm.

METHOD 409.2

SULFUR (URBON DISULFIDE SOLUBLE)

1. SCOPE

1.1 This method is used to determine the carbon disulfide soluble sulfur content of pyrotechnics.

2. SPECIMEN

2.1 The specimen shall consist of approximately 1 gm of the pyrotechnic, weighed to within 0.2 mg.

3. APPARATUS

3.1 Beaker, 400 ml.

3.2 Steam bath.

3.3 Filter paper, No. 42 Whatman or equivalent.

3.4 Hotplate.

3.5 Filtering crucible, Gooch type or equivalent.

3.6 Muffle furnace.

3.7 Oven.

3.8 Desiccator containing an indicating desiccant.

4. MATERIALS

4.1 Carbon disulfide.

4.2 Nitric acid, 70-percent.

4.3 Bromine, reagent grade.

4.4 Barium chromate, 10-percent solution.

5. PROCEDURE

5.1 Leach the specimen with a suitable solvent to remove interfering nitrates as specified in Method 301.4, retaining the residue in a filtering crucible.

5.2 Extract the sulfur by pouring small portions of carbon disulfide into the crucible

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and allow it to pass through without suction, until a total volume of approximately 100 ml of the solvent has been used.

Caution: Low results will be obtained by the above procedure in case a form of sulfur, insoluble in carbon disulfide, is present. Therefore, if a qualitative test of the residue from the carbon disulfide extraction shows sulfur to be present, determine the sulfur content of the composition as specified in Method 409.1.

5.3 Evaporate the carbon disulfide solution to dryness on a steam bath.

5.4 Add 10 ml of concentrated nitric acid and 5 ml of bromine.

5.5 Evaporate the solution to dryness on a steam bath.

5.6 Add 100 ml of distilled water to the residue and stir well.

5.7 Filter the solution through a filter paper catching the filtrate in a clean beaker.

5.8 Heat the filtrate to boiling and slowly add 5 ml of 10-percent barium chloride solution.

5.9 Boil for 10 minutes and digest on a steam bath for 2 hours.

5.10 Filter through a tared Gooch crucible and wash the precipitate thoroughly with hot distilled water.

5.11 Dry the crucible at 135°C. for 1 hour.

5.12 Ignite the crucible in a muffle furnace at dull red heat for 1 hour.

5.13 Remove the crucible, cool in a desiccator and weigh.

5.14 Calculate the percentage sulfur as follows:

$$\text{Percent sulfur} = \frac{13.73A}{W}$$

where:

A = weight of precipitate, gm.

W = weight of sample, gm.

METHOD 410.1

ANTIMONY SULFIDE (PERMANGANATE METHOD)

1. SCOPE

1.1 This method is used to determine antimony sulfide content of pyrotechnics.

2. SPECIMEN

2.1 The specimen shall consist of approximately 1 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Erlenmeyer flask, 800 ml.

3.2 Hotplate.

3.3 Titration assembly.

4. MATERIALS

4.1 Hydrochloric acid, 38-percent.

4.2 Sulfuric acid, concentrated.

4.3 Sodium sulfite, Reagent Grade.

4.4 Potassium permanganate, 0.1N standard solution as specified in Method 606.1.

5. PROCEDURE

5.1 Leach the specimen with a suitable solvent to remove interfering oxidizable material as specified in Method 301.4, retaining the residue in a filtering crucible.

5.2 Transfer the crucible and residue to a 800 ml flask and add approximately 50 ml of hydrochloric acid.

Caution: Caution should be exercised during this addition to prevent foaming of this mixture out of the flask.

5.3 Let stand for about 3 minutes at room temperature.

5.4 Add 15 ml of concentrated sulfuric acid.

5.5 Add cautiously, in small quantities, 1 gm of sodium sulfite.

5.6 Wash the sides with distilled water.

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5.7 Heat gently for about 10 min. to completely expel sulfur dioxide.

5.8 Dilute with 350 ml of water and cool to 10° to 12°C.

5.9 Titrate with 0.1N potassium permanganate solution to a pink color.

5.10 Run a blank titration using the same quantities of reagents and titrate to the same end point.

5.11 Calculate as follows:

$$\text{Percent antimony sulfide} = \frac{8.493 (V - v) N}{W}$$

where:

V = permanganate solution used in titration of sample, ml.

v = permanganate solution used for blank, ml.

N = normality of permanganate solution.

W = weight of sample, gm.

METHOD 411.1
NICKEL (DIMETHYLGLYOXIME METHOD)

1. SCOPE

1.1 This method is used to determine nickel content of pyrotechnics.

2. SPECIMEN

2.1 The specimen shall consist of approximately 1 gm of the pyrotechnic, weighed to within 0.2 mg.

3. APPARATUS

3.1 Whatman filter paper, No. 42 or equivalent.

3.2 Beaker, 250 ml.

3.3 Volumetric flask 250 ml.

3.4 Pipet, 25 ml.

3.5 Beaker, 600 ml,

3.6 Litmus paper.

3.7 Hotplate.

3.8 Pipet, 10 ml.

3.9 Steambath.

3.10 Crucible, sintered glass, medium porosity.

3.11 Oven.

4. MATERIALS

4.1 Ethyl alcohol, 50-percent solution.

4.2 Hydrochloric acid, 50-percent solution.

4.3 Tartaric acid, reagent grade.

4.4 Ammonium chloride solution, 10-percent.

4.5 Ammonium hydroxide, concentrated.

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4.6 Hydrochloric acid, 38-percent.

4.7 Dimethyl glyoxime-in-methanol, 1-percent solution.

5. PROCEDURE

5.1 Leach the specimen with suitable solvent to remove interfering ingredients as specified in Method 301.4, retaining the residue on a filter paper.

5.2 Treat the filter paper and residue, which has been placed in a 250 ml beaker, with 40 ml of the 50-percent hydrochloric acid and allow to remain at room temperature overnight.

5.3 Filter through a No. 42 Whatman filter paper, collecting the filtrate in a 250 ml volumetric flask.

5.4 Wash the residue with approximately 150 ml of distilled water and make up to volume.

5.5 Discard the residue.

5.6 Pipet a 25 ml aliquot into a 600 ml beaker and dilute with water to approximately 20 ml.

5.7 Add 5 grams of tartaric acid and 10 ml of a 10-percent solution of ammonium chloride.

5.8 Add concentrated ammonium hydroxide until the solution is slightly alkaline to litmus.

5.9 If a precipitate forms, add several drops of concentrated hydrochloric acid to clear the solution, and again neutralize with concentrated ammonium hydroxide.

5.10 Transfer the beaker to a hotplate, heat to nearly boiling, and add by means of a pipet; 10 ml of a 1-percent solution of dimethyl glyoxime-in-methanol, with vigorous stirring.

5.11 Add concentrated ammonium hydroxide until the mixture has a distinct ammonium odor.

5.12 Place on a steam bath for approximately ½ hour and filter through a tared, medium porosity, sintered glass crucible.

5.13 Wash with three 20 ml portions of the ethyl alcohol, cool in a desiccator and weigh.

5.14 Calculate as follows:

$$\text{Percent nickel} = \frac{20.32A}{W}$$

where:

A = weight of precipitate, gm.

W = weight of sample represented by aliquot, gm.

METHOD 412.1

MAGNESIUM (EUDIOMETER METHOD)

1. SCOPE

1.1 This method is used for determining the metallic magnesium content of pyrotechnics provided magnesium is the only elemental metal present that will produce hydrogen gas.

2. SPECIMEN

2.1 The specimen shall consist of a portion of the pyrotechnic containing approximately 0.47 gm of magnesium (as determined from the detailed specifications for the pyrotechnic being tested) Weigh the specimen to within 0.2 mg.

3. APPARATUS

3.1 Apparatus consists of following (see fig. I).

- (a) Reaction flask, Erlenmeyer flask fitted with a 2-hole rubber stopper.
- (b) Buret, 25-ml capacity, with tip inserted through one hole of the rubber stopper.
- (c) U-Tube, A drying tube containing Ascarite, connected to the reaction flask through the second hole of the rubber stopper.
- (d) Stopcock, 3-way "T" type, inserted between the drying tube and the gas buret so that the reaction flask can be exposed to atmospheric pressure.
- (e) Gas buret, calibrated from 450 ml, to 500 ml, connected to a water leveling bulb filled with water saturated with Hydrogen. The buret is water jacketed to achieve accurate and rapid temperature control.
- (f) Circulating pumps, water pump that is used to circulate the water through the water jacket.
- (g) Reservoir, pail of water in which the reaction flask is immersed.

4. MATERIALS

4.1 Sulfuric acid solution, 6:19.

4.2 Distilled water saturated with hydrogen gas.

5. PROCEDURE

5.1 Leach the specimen with suitable solvents, to remove interfering materials that react with sulfuric acid to liberate gas, as specified in Method 301.4, retaining the residue on a glass filtering crucible.

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5.2 Transfer the crucible and residue to the 600 ml wide mouth Erlenmeyer flask

5.3 Add 200 ml of distilled water, saturated with hydrogen gas.

5.4 Connect the Erlenmeyer flask to the apparatus.

5.5 Open the 3-way "T" stopcock so that the flask and the gas buret are exposed to atmospheric pressure.

5.6 Adjust the level of the liquid in the gas buret to the zero point with the leveling bulb.

5.7 Turn the 3-way "T" stopcock so that it will only allow gas to flow from the reaction flask to the gas buret.

5.8 Lower the leveling bulb and add 25 ml of the sulfuric acid solution from the 25 ml buret.

5.9 After the addition of the acid, agitate the flask occasionally until the reaction is complete. The reaction shall be considered complete when the volume of generated gas remains within 1 ml for a period of 20 minutes.

5.10 When the reaction is complete, adjust the level on the gas buret to that of the water leveling bulb.

5.11 Read the volume of gas in the gas buret. If the evolved gas is less than is measurable by the gas buret add a known volume of water through the acid buret until a readable volume is reached on the gas buret.

5.12 The determination shall be started and finished at the same temperature (approximately 25°C.).

5.14 Calculate the percentage of magnesium in the sample on a dry basis as follows:

$$\text{Percentage of magnesium} = \frac{(A - B) (C - D) 0.03901}{W(273+t)}$$

where:

A = Volume read on gas buret, ml.

B = Volume of acid or volume of acid plus water added from buret, ml.

C = Atmospheric pressure, mm.

D = Vapor pressure of water at the temperature of the determination, mm.

t = Temperature at which the determination is made, °C.

W = Weight of sample, gm.

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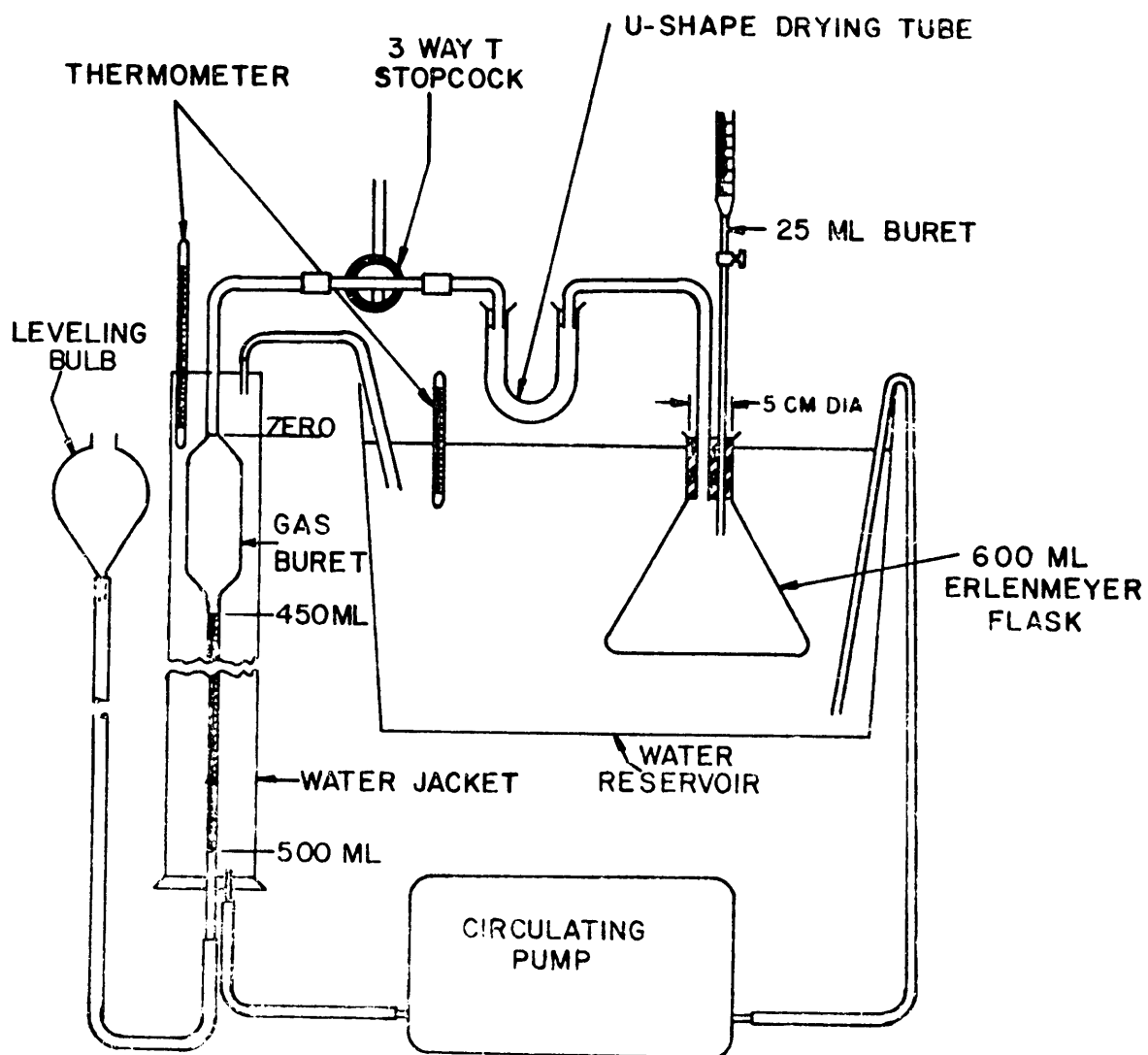


FIGURE 1. *Equipment assembly.*

METHOD 412.2
MAGNESIUM (PYROPHOSPHATE METHOD)

1. SCOPE

1.1 This method is used to determine the magnesium content of pyrotechnics which do not contain sodium or potassium.

2. SPECIMEN

2.1 The specimen shall consist of 5 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Beaker, 250 ml.

3.2 Rubber-tipped tongs.

3.3 Beaker, 600 ml.

3.4 Whatman filter paper, No. 42 or equivalent.

3.5 Muffle furnace.

3.6 Crucible, porcelain.

3.7 Pipet, 50 ml.

3.8 Volumetric flask, 500 ml.

4. MATERIALS

4.1 Hydrochloric acid solution, 10-percent,

4.2 Hydrochloric acid, 38-percent.

4.3 Sodium hydrogen phosphate, Reagent Grade.

4.4 Nitric acid, 70-percent.

4.5 Sulfuric acid, concentrated.

4.6 Ammonium hydroxide, concentrated.

4.7 Ammonium hydroxide solution, 5-percent.

5. PROCEDURE

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5.1 Extract the specimen with a suitable solvent to remove interfering and binder material as specified in Method 301.3, retaining the residue containing the magnesium in a crucible.

5.2 Transfer the crucible and residue to a 250-ml beaker.

5.3 Dissolve the residue in the 10-percent hydrochloric acid solution.

5.4 Remove the crucible with rubber-tipped tongs and wash thoroughly with distilled water, catching the washings in the beaker.

5.5 Transfer the solution to a 500 ml volumetric flask and makeup to that volume with distilled water.

5.6 Transfer a 50 ml aliquot of the solution to a 250 ml beaker and add 5 ml of concentrated sulfuric acid.

5.7 Evaporate by boiling until the fumes of sulfur trioxide are evolved.

5.8 Cool the liquid, dilute with 100 ml of distilled water, add 2 ml of concentrated hydrochloric acid, and heat to boiling.

5.9 Allow the solution to cool and filter.

5.10 Wash the precipitate with hot distilled water, catching the filtrate and washings in a filter flask

5.11 Transfer the filtrate and washings to a 500-ml volumetric flask and make up to volume with distilled water.

5.12 Transfer a 200-ml aliquot to a 600-ml beaker and add 10 ml of a 10-percent solution of sodium hydrogen phosphate.

5.13 Add 100 ml of concentrated ammonium hydroxide, stir the mixture, and allow to stand overnight.

5.14 Filter through Whatman 42 filter paper, and wash with 5-percent ammonium hydroxide solution.

5.15 Place the filter paper in stered porcelain crucible, dry and ignite at about 500°C. to burn off the filter paper and finally ignite at 1000° to 1100° for 30 minutes.

5.16 Cool the crucible in a desiccator, and weigh.

5.17 Calculate as follows:

$$\text{Percent magnesium} = \frac{21.84A}{W}$$

where:

A = weight of precipitate, gm.

W = weight of sample aliquot, gm.

METHOD 413.1
TITANIUM AND TITANIUM DIOXIDE
(JONES REDUCTION METHOD)

1. SCOPE

1.1 This method is used to determine the titanium or titanium dioxide content of pyrotechnics containing no other metals that are reduced in the Jones reductor.

2. SPECIMEN

2.1 The specimen shall consist of a portion of the pyrotechnic, weighed to within 0.2 mg containing approximately 0.1 – 0.2 gm of titanium.

3. APPARATUS

3.1 Jones reductor, prepared as follows:

- (a) Use a reductor tube having a total length of approximately 500 mm. The minimum length of the column available for the reducing column shall be approximately 450 mm and the outside diameter of the reducing column shall be approximately 20 mm.

Note. A drawing of a suitable Jones Reductor can be found on page 698 of "Textbook of Quantitative Inorganic Analysis," by. Kolthoff and Sandell, The MacMillan Co., New York, 1943.

The Jones (Blair) Reductor, Catalogue No. 8596-A, Arthur H. Thomas Co., Philadelphia, Pa., has been found satisfactory.

- (b) Insert a perforated porcelain disc, approximately 16 mm in diameter and 2 mm thick into the tube.

Note. Perforated Porcelain Disc, Catalog No. 4139, size 16 mm in diameter and 2 mm thick, Arthur E Thomas Co., Philadelphia, Pa., has been found suitable.

- (c) Place a loosely packed plug of glass wool approximately 2 cm thick upon the perforated disc and fill the tube with water.
- (d) Insert the delivery tube of the Jones reductor through a rubber stopper of sufficient size to fit a 1000-ml filtering flask. Insert it in such a manner that when the 1000-ml filtering flask is attached, the tube shall reach to within 5 mm of the bottom of the flask. If it is not sufficiently long, attach, with the aid of a piece of rubber tubing, a glass tube of sufficient length and having approximately the same internal diameter and wall thickness as the delivery tube.
- (e) Attach a 1000-ml filtering flask to the reductor tube.
- (f) Add amalgamated zinc (prepared according to par. 4.7) to the reductor tube until the column is completely packed to within approximately 1 cm of the bottom of the upper reservoir.

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- (g) Wash the amalgamated zinc with approximately 500 ml of distilled water with the aid of gentle suction being careful to keep the amalgamated zinc always covered by water.

Caution: Exposure of the amalgamated zinc to the atmosphere results in the formation of basic salts which tend to, clog the zinc column and reduce the efficiency of the reductor.

- (h) Condition the reductor before using by passing 150 ml of 5-percent sulfuric acid solution followed by 50 ml of water through the reducing column with the aid of gentle suction.
- (i) Test the reductor by adding 1 drop of 0.1N potassium permanganate solution to the washings. The pink coloration shall last at least 15 seconds. If it does not last, repeat the conditioning until it does.
- (j) After use, pass distilled water through the reductor until the washings are neutral to litmus paper and then allow enough water to remain in the reductor so that its level is at least 5 cm above the reducing column.

3.2 Suction flask, 1000 ml.

3.3 Filter paper, rapid filtration type.

4. MATERIALS

4.1 Sulfuric acid solution, 5-percent.

4.2 Nitric acid, 70-percent.

4.3 Titanous chloride, 0.2N standard solution as specified in Method 601.1.

4.4 Ferric ammonium sulfate 0.15N standard solution, as specified in Method 603.1.

4.5 Potassium thiocyanate solution, 5-percent.

4.6 Inert gas, carbon dioxide.

4.7 Amalgamated zinc prepared as follows:

- (a) Shake 800 gm of zinc with 400 ml of 2.5-percent mercuric chloride solution in a 1 liter flask for 2 minutes.
- (b) Wash several times with 5-percent sulfuric acid solution.
- (c) Finally wash thoroughly with distilled water.

5. PROCEDURE

5.1 Prepare the specimen for the determination as specified in Method 301.5, retaining the solution in a beaker.

5.2 Dilute the solution cautiously, to approximately 200 ml.

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5.3 Filter the solution through a rapid filtration filter paper and wash the beaker and paper with three 20 ml portions of 5:95, sulfuric acid solution.

5.4 Activate the Jones reductor by passing 100 ml of 5:95 sulfuric acid solution through the reducing column with the aid of gentle suction. Discard this acid.

5.5 Add forty ml of standardized ferric ammonium sulfate solution of approximately 0.15N to a clean 1000 ml suction flask.

5.6 Attach the flask to the reductor in such a way that the delivery tube of the reductor is below the surface of the ferric alum solution.

5.7 Pass the solution through the reductor at a rate not exceeding 100 ml per minute with the aid of gentle suction.

5.8 Follow this with 100 ml of 5:95 sulfuric acid solution and 50 ml of distilled water.

5.9 Disconnect the suction and remove the flask from the reductor.

5.10 Add 5 ml of approximately 5-percent solution of potassium thiocyanate to the flask.

5.11 Titrate the excess ferric alum, until the red color disappears, using 0.2N standardized titanous chloride solution, while passing a stream of carbon dioxide into the filtering flask.

5.12 Run a blank by titrating 40 ml of the 0.15N ferric ammonium sulfate in 300 ml of 5:95 sulfuric acid with 0.2N titanous chloride solution using 5 ml of a 5-percent solution of potassium thiocyanate solution.

5.13 Calculate the percent titanium or titanium dioxide as follows:

$$\text{Percent titanium} = \frac{(A - B)N4.79}{W}$$

$$\text{Percent titanium dioxide} = \frac{(A - B)N7.99}{W}$$

where:

A = ml of titanous chloride required for blank (number of ml of titanous equivalent to 40 ml of the ferric alum).

B = ml of titanous chloride required for sample.

N = normality of titanous solution.

W = weight of sample, gm.

METHOD 414.1

IRON (JONES REDUCTOR METHOD)

1. SCOPE

1.1 This method is used to determine the total iron or iron compound content of pyrotechnics containing no other metals that are reduced in the Jones reductor.

2. SPECIMEN

2.1 The specimen shall consist of a portion of the pyrotechnic, weighed to within 0.2 mg, containing approximately 0.2 gm of iron.

3. APPARATUS

3.1 Jones reductor, prepared as specified in Method 413.1 except 25 cm in length shall be attached to a 500 ml filtering flask.

3.2 Beaker, 250 ml.

3.3 Watchglass.

3.4 Filtering flask, 500 ml.

3.5. Titration apparatus.

4. MATERIALS

4.1 Hydrochloric acid solution, 50-percent.

4.2 Hydrochloric acid solution, 10-percent.

4.3 Potassium bichromate, 0.1N standard solution as specified in Method 605.1.

4.4 Barium diphenylamine sulfonate indicator solution as specified in Method 708.1.

5. PROCEDURE

5.1 Extract the iron in the specimen by gently warming with 10 ml of 50-percent hydrochloric acid as specified in Method 301.3, retaining the filtrate in a beaker.

5.2 Dilute the solution in the beaker with 60 ml of distilled water.

5.3 Pass the solution through the reductor with several 10 ml portions of 10-percent hydrochloric acid solution, catching these washings in the filtering flask.

5.4 Wash the solution through the reductor with several 10 ml portions of 10-percent hydrochloric acid solution, catching these washings in the filtering flask.

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5.5 Finally wash with several 10 ml portions of distilled water adding these washings to the flask.

5.6 Titrate the solution in the filtering flask to a permanent blue-purple color with the potassium bichromate solution using 15 ml of the barium diphenylamine sulfonate indicator solution.

5.7 Calculate the percent iron or iron compound as follows:

$$\text{Percent iron} = \frac{AN5.585}{W}$$

$$\text{Percent iron compound} = \frac{ANB}{10nW}$$

where:

A = potassium bichromate solution used, ml.

B = molecular weight of iron compound.

n = number of iron atoms in compound.

N = normality of potassium bichromate solution.

W = weight of sample, gm.

Note. If the end point is not clean add an excess of potassium dichromate to the specimen and back titrate with ferrous ammonium sulfate.

METHOD 415.1

POTASSIUM SALTS (TETRAPHENYLBORON METHOD)

1. SCOPE

1.1 This method is used for determining the potassium sulfate or potassium nitrate content of pyrotechnics containing no barium nitrate, strontium, and copper.

2. SPECIMEN

2.1 The specimen shall consist of approximately 5 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Steam bath.

3.2 Beaker, 250 ml.

3.3 Filter paper, No. 40 Whatman or equivalent.

3.4 Pipet, 20 ml.

3.5 Filtering crucible, sintered glass, medium porosity.

3.6 Suction filtration apparatus with a three way stopcock in the suction line.

3.7 Oven.

3.8 Desiccator containing an indicating desiccant.

4. MATERIALS

4.1 Aluminum chloride ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$) 0.2M solution (5 gm per 100 ml of distilled water).

Warning: Do not use anhydrous aluminum chloride.

4.2 Sodium tetraphenylboron, 3-percent solution prepared as follows:

- (a) Dissolve 9 gm of sodium tetraphenylboron in 300 ml of distilled water.
- (b) Add 2 ml of 0.2M aluminum chloride solution, stir and allow to stand for 30 minutes.
- (c) Filter into a polyethylene or pyrex bottle.

On prolonged standing, the solution may become turbid. If it does, it should be refiltered before using.

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5. PROCEDURE

5.1 Prepare the specimen for the determination as specified in Method 301.5 retaining the solution in a 250 ml beaker.

5.2 Add approximately 50 ml of distilled water and heat on the steam bath for 16 minutes.

5.3 Filter the solution quantitatively through a No. 40 Whatman filter paper into a 250 ml beaker. Wash the precipitate with several small portions of distilled water and catch the washings in the beaker.

5.4 Dilute the solution in the beaker to approximately 100 ml and add 5 to 6 drops of the 0.2M aluminum chloride solution.

5.5 Using a pipet, add approximately 20 ml of the 3-percent sodium tetraphenylboron solution, stirring constantly.

5.6 Allow the precipitate to settle for 5 minutes.

5.7 Filter the solution through the tared filtering crucible with the aid of a suction filtration apparatus, being careful to keep a continuous flow of solution and subsequent washings (par. 5.8) through the filter until the last washing is complete.

Note. The rate of filtration may be controlled by manipulating a stopcock in the suction line.

5.8 Rinse the beaker with five 20-ml portions of distilled water and pour each through the filtering crucible.

5.9 When the solution and rinsings have passed through the filter, dry the crucible and contents at $120 \pm 2^{\circ}\text{C}$. for 30 minutes, and cool to room temperature in a desiccator.

5.10 Weigh the crucible and calculate the potassium salt content as follows:

$$\text{Percent potassium nitrate} = \frac{28.22A}{W}$$

$$\text{Percent potassium sulfate} = \frac{24.32A}{W}$$

where:

A = weight of precipitate, gm.

W = weight of specimen, gm.

METHOD 416.1
ZIRCONIUM OR ZIRCONIUM HYDRIDE
(CUPFERRON METHOD)

1. SCOPE

1.1 This method is used to determine zirconium and zirconium hydride content in pyrotechnics in the absence of interfering elements such as iron.

2. SPECIMEN

2.3 The specimen shall consist of approximately 1 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Beaker, 250 ml.

3.2 Watchglass.

3.3 Medicine dropper.

3.4 Hotplate.

3.5 Glass rod.

3.6 Whatman filter paper, No. 42 or equivalent.

3.7 Beaker, 400 ml.

3.8 Ice bath.

3.9 Whatman filter paper, No. 40 or equivalent.

3.10 Crucible, porcelain.

3.11 Oven.

3.12 Muffle furnace.

4. MATERIALS

4.1 Nitric acid, 70-percent

4.2 Sulfuric acid, concentrated.

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4.3 Cupferron solution — Dissolve 6 gm cupferron in 100 ml of distilled water and filter.

4.4 Hydrochloric acid solution, approximately 1N.

5. PROCEDURE

5.1 Prepare the specimen for the determination as specified in Method 301.5, retaining the solution in a beaker.

5.2 Add cautiously to the beaker, without removing the watchglass, sufficient distilled water to make a volume of approximately 150 ml. Stir the mixture thoroughly with a glass rod and allow to cool to room temperature.

5.3 Filter through a Whatman No. 42 filter paper, collecting the filtrate in a clean 400-ml beaker.

5.4 Wash the residue on the filter paper with sufficient distilled water to make the total volume in the 400-ml beaker approximately 200 ml. Discard the filter paper.

5.5 Cool the beaker in an ice bath to $10^{\circ} \pm 2^{\circ}\text{C}$. and add, with stirring, 32 ml of freshly prepared cupferron solution.

5.6 Filter the precipitate using a Whatman No. 40 paper and wash with five 20-ml portions of cold hydrochloric acid solution.

5.7 Transfer the precipitate and paper to a tared crucible and dry in an oven at 100° to 105°C . for two hours.

5.8 Place the crucible and contents in a muffle furnace at 300° to 400°C . until complete charring of the paper has occurred, and the rush of gases from decomposition of the organic matter has ceased. Increase the temperature to $900^{\circ} \pm 50^{\circ}\text{C}$. and continue the ignition for $\frac{1}{2}$ hour.

5.9 Cool in a desiccator, weigh and calculate the zirconium oxide to percentage of zirconium or zirconium hydride in the sample as follows:

$$\text{Percent zirconium} = \frac{74.03A}{W}$$

$$\text{Percent zirconium hydride} = \frac{75.65A}{W}$$

where:

A = weight of precipitate, gm.
 W = weight of sample, gm.

METHOD 417.1
SODIUM OXALATE (POTASSIUM PERMANGANATE METHOD)

1. SCOPE

1.1 This method is used to determine the sodium oxalate content of pyrotechnics containing no other material that is oxidized by potassium permanganate.

2. SPECIMEN

2.1 The specimen shall consist of a portion of the pyrotechnic weighed to within 0.2 mg, containing approximately 0.2 gm of the oxalate.

3. APPARATUS

3.1 Beaker, 600 ml.

3.2 Beaker, 400 ml.

3.3 Titration apparatus.

3.4 Filtering crucible.

3.5 Steam bath.

4. MATERIALS

4.1 Potassium permanganate, 0.1N standard solution as specified in Method 606.1.

4.2 Sulfuric acid solution, 25-percent.

5. PROCEDURE

5.1 Transfer the specimen to a 400-ml beaker.

5.2 Add 100 ml of cold sulfuric acid solution; during the addition of the acid keep the beaker covered as much as possible to prevent loss by spattering.

5.3 Warm the beaker and contents on a steam bath for 5 minutes.

5.4 Filter the solution through a crucible and wash the insoluble matter thoroughly with water.

5.5 Transfer the combined filtrate and washings to a 600 ml beaker and dilute to approximately 250 ml with water.

5.6 Heat the solution to approximately 85°C.

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5.7 Titrate with approximately 0.1N potassium permanganate solution until the faint pink color is obtained which persists for approximately 30 seconds. Do not permit the solution to fall below 70°C. during the titration.

6.8 Calculate the percentage of sodium oxalate as follows:

$$\text{Percent sodium oxalate} = \frac{6.701AN}{W}$$

where:

A = volume of potassium permanganate solution used, ml.
N = normality of permanganate solution.
W = weight of the specimen, gm.

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METHOD 418.1

STRONTIUM NITRATE (SULFATE METHOD)

1. SCOPE

1.1 This method is used to determine the strontium nitrate content of pyrotechnics in the absence of barium, lead, calcium, silicon, and heavy metals.

2. SPECIMEN

2.1 The specimen shall consist of a portion of the pyrotechnic, weighed to within 0.2 mg, containing 0.15 to 0.2 gm of strontium nitrate.

3. APPARATUS

3.1 Beaker, 250 ml.

3.2 Hotplate.

3.3 Watchglass.

3.4 Crucible, sintered porcelain (Selas No. 3001).

3.5 Desiccator containing an indicating an indicating desiccant.

3.6 Muffle furnace.

4. MATERIALS

4.1 Sulfuric acid, concentrated.

4.2 Ethyl alcohol, 95-percent.

4.3 Wash solution — 1 ml of conc. sulfuric acid added to a mixture of 250 ml of 95-percent alcohol and 250 ml of water.

5. PROCEDURE

5.1 Extract the strontium nitrate from the sample with water, as specified in Method 801.3, retaining the filtrate in a 250 ml beaker.

5.2 Add 3 ml of sulfuric acid to the filtrate in the beaker and cover with a watch-glass.

5.3 Place the beaker and contents on a hotplate and evaporate to strong fumes of sulfuric acid.

5.4 Remove the beaker and contents from the hotplate and allow to cool.

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5.5 Wash down the watchglass and sides of the beaker with water.

5.6 Again evaporate to strong fumes of sulfuric acid.

5.7 Allow to cool, add 10 ml of water and boil for 2 minutes.

5.8 Add 50 ml of 95-percent alcohol and allow to stand overnight.

5.9 Filter through a tared sintered porcelain crucible and transfer and wash the precipitate with the wash solution.

5.10 Wash the precipitate finally once with 95-percent alcohol and aspirate to dryness.

5.11 Place the sintered crucible in a muffle furnace and ignite for 30 minutes at 700°C. Cool in a desiccator and weigh.

5.12 Calculate the percent strontium nitrate as follows:

$$\text{Percent strontium nitrate} = \frac{115.22A}{W}$$

where:

A = weight of precipitate, gm.

W = weight of sample, gm.

METHOD 419.1

ZINC OXIDE (FORMIC ACID METHOD)

1. SCOPE

1.1 This method is used to determine the zinc oxide content of pyrotechnics.

2. SPECIMEN

2.1 The specimen shall consist of approximately 1 gm of the pyrotechnic weighed to within 0.2 mg.

3. APPARATUS

3.1 Crucible, medium porosity, porcelain.

3.2 Beaker, 400 ml.

3.3 Muffle furnace.

4. MATERIALS

4.1 Acetic acid solution.

4.2 Sodium hydroxide solution, 10-percent.

4.3 Litmus paper.

4.4 Sulfuric acid solution, 5-percent.

4.5 Sulfuric acid solution, 30-percent.

4.6 Hydrogen sulfide, gas.

5. PROCEDURE

5.1 Leach the specimen with suitable solvent to remove interfering and binder materials as specified in Method 301.4, retaining the residue in a medium porosity sintered glass crucible.

5.2 Leach the residue with five 10-ml portions of acetic acid solution, allowing each portion to remain in contact with the residue approximately 1½ minutes before applying vacuum, catching the filtrate in a beaker.

5.3 Wash the residue with distilled water and add these washings to the filtrate.

5.4 Make the filtrate just alkaline with 10-percent sodium hydroxide solution.

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5.5 Make the solution neutral to litmus paper by means of a 30-percent solution of sulfuric acid and then add 3 ml of 5-percent sulfuric acid.

5.6 Dilute to 200 ml and pass a steady stream of hydrogen sulfide through the solution for approximately 40 minutes.

5.7 Allow the precipitate of zinc sulfide to settle for at least 10 minutes.

5.8 Filter the solution through a Gooch crucible and wash with distilled water.

5.9 Ignite the crucible and contents at a bright red heat for approximately 1 hour.

5.10 Cool the crucible and contents in a desiccator and weigh rapidly.

5.11 Calculate the percentage of zinc oxide as follows:

$$\text{Percent zinc oxide} = \frac{A100}{W}$$

where:

A = weight of precipitate, gm.

W = weight of linseed-oil-free sample, gm.

METHOD 420.1

NITRO COMPOUNDS (TITANOUS CHLORIDE METHOD)

1. SCOPE

1.1 This method is used to determine the explosive nitro compound content (such as TNT and tetranitrocarbazole) in pyrotechnics.

2. SPECIMEN

2.1 The specimen shall consist of a portion of the pyrotechnic, weighed to within 0.2 mg, containing approximately 0.15 gm of nitro-nitrogen.

3. APPARATUS

3.1 Storage system for 0.2N titanous chloride and 0.15N ferric ammonium sulfate as specified in Methods 601.1 and 603.1 (fig. 1).

3.2 Titration flask (fig. 2).

3.3 Reflux condenser.

3.4 Hotplate, three heat control.

3.5 Volumetric flask, 250 ml.

4. MATERIALS

4.1 Titanous chloride 0.2N standard solution as specified in Method 601.1

4.2 Ferric ammonium sulfate 0.15N standard solution as specified in Method 603.1.

4.3 Ammonium thiocyanate, 20-percent solution as specified in Method 706.1.

4.4 Hydrochloric acid solution, 15-percent.

4.5 Inert gas (carbon dioxide).

5. PROCEDURE

5.1 Extract the specimen with suitable solvent to remove the organic nitro compound as specified in Method 301.3 retaining the filtrate in a beaker.

5.2 Transfer the filtrate to a 250 ml volumetric flask and dilute to the mark with the suitable solvent.

5.3 Using a 25 ml pipet, transfer a 25 ml aliquot to the titration flask (fig. 2).

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5.4 Evaporate the aliquot to dryness.

5.5 Add 25 ml of glacial acetic acid and carefully heat on a steam bath until complete solution of the residue occurs.

5.6 Allow the solution to cool to room temperature.

5.7 Displace the air in the titration flask by passing in a current of inert gas for 5 minutes.

5.8 Add an accurately measured amount, approximately 45-50 ml of 0.2N titanous chloride to the titration flask.

Note. At least 25 percent excess of reagent is necessary to force the reduction to completion. The amount of reagent required is determined as follows:

$$\text{Volume of titanous chloride required} = \frac{W}{\left\{ \frac{\text{M.W.}}{n \times 6000} \right\} N} \times 1.25$$

where:

W = weight of sample, gm.
 M.W. = molecular weight of nitro compound, gm.
 n = number of "NO₂" groups in nitro compound.
 N = normality of titanous chloride solution.

5.9 Add 25 ml of 15 percent hydrochloric acid solution to the flask and add a few beads to prevent bumping.

5.10 Connect the flask to a reflux condenser; start the flow of cold water through the reflux condenser and boil the solution in the titration flask gently for 20 to 30 minutes on a hotplate.

5.11 Increase the current of inert gas, then cautiously immerse the flask in a cold water bath, and cool to room temperature.

5.12 Disconnect the condenser and add 5 ml of 20-percent ammonium thiocyanate indicator.

5.13 Titrate with standard 0.15N ferric ammonium sulfate solution to the first permanent red color.

5.14 Run a blank determination as follows:

- (a) Sweep the air from the titration flask with inert gas as before.
- (b) Add 25 ml of glacial acetic acid and about 20 ml of 0.2N titanous chloride.
- (c) Connect the flask to a reflux condenser and boil the solution on a hotplate for 30 minutes.
- (d) Cool the contents and disconnect the condenser.
- (e) Add 5 ml of 20-percent ammonium thiocyanate solution and titrate to the

first permanent red color with standard 0.15N ferric ammonium sulfate.

5.15 Calculate the organic nitro compound as follows:

$$\text{Percent nitro compound} = \frac{\left(\frac{\text{M.W.} \times 100}{6n \times 1000} \right) \left((AN - FB) - (CN - DF) \right)}{W}$$

where:

- A = titanous chloride used in sample, ml.
- N = normality of titanous chloride.
- B = ferric ammonium sulfate used in sample, ml.
- F = normality of the ferric ammonium sulfate.
- C = titanous chloride used in blank, ml.
- D = ferric ammonium sulfate used in blank.
- W = weight of sample represented by aliquot taken, gm.
- M.W. = molecular weight of nitro compound being tested, gm.
- n = number of nitro groups in nitro compound being tested.

METHOD 421.1
POTASSIUM AND BARIUM SALTS
(FLAME SPECTROPHOTOMETRIC METHOD)

1. SCOPE

1.1 This method is used for determining the potassium perchlorate and barium nitrate contents of photoflash powder between 20 to 40 percent.

Note. This method maybe used when only one of the salts is present, but new standard solution must be made.

2. SPECIMEN

2.1 For potassium perchlorate the specimen shall consist of exactly 0.5906 gms of the powder.

2.2 For barium nitrate the specimen shall consist of exactly 0.3171 gm of the powder.

3. APPARATUS

3.1 Flame spectrophotometer (Beckman Instruments Inc., Model D U, with flame attachment, Model 9200, and Regulator Model 9200; or equivalent).

3.2 Volumetric flasks 1000 ml.

3.3 Volumetric flasks, 250 ml.

3.4 Buret, 25 ml.

3.5 Funnel.

3.6 Filter paper, Whatman No. 41 or equivalent.

4. MATERIALS

4.1 Master solution of potassium perchlorate containing 1000 ppm of potassium, prepared by transferring 3.5436 gm of dried analytical reagent grade potassium perchlorate to a 1000-ml volumetric flask and diluting to the mark with distilled water.

Note. This master solution is used in preparing solutions of known potassium perchlorate in accordance with table I.

4.2 Master solution of barium nitrate containing 1000 (ppm) of barium, prepared by transferring 1.9029 gm of dried analytical reagent grade barium nitrate to a 1000 ml volumetric flask, and diluting to the mark with distilled water.

Note. This master solution is used in preparing solution of known barium concentration in accordance with table II.

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TABLE I. *Standard concentration of Potassium Perchlorate*

Percentage of Potassium Perchlorate	Master solution of Potassium Perchlorate, ml per 260 ml	Master solution of Barium Nitrate, ml per 250 ml
20	8.50	23.27
25	10.50	23.27
28	11.75	23.27
30	12.50	23.27
32	13.25	23.27
35	14.50	23.27
40	16.50	23.27

TABLE II. *Standard concentration of Barium Nitrate*

Percentage of Barium Nitrate	Master solution of Barium Nitrate, ml per 250 ml	Master solution of Potassium Perchlorate, ml per 260 ml
20	8.50	6.71
25	10.50	6.71
28	11.75	6.71
30	12.50	6.71
32	13.25	6.71
35	14.50	6.71
40	16.50	6.71

5. PROCEDURE

5.1 Place the specimen of potassium perchlorate in a 1000-ml volumetric flask, dilute to the mark with distilled water and shake vigorously.

5.2 Allow the major portion of the aluminum to settle, and then filter approximately 100 ml of the solution through a dry No. 41 filter paper, rejecting the initial 25-30 ml.

5.3 Place the specimen of barium nitrate in a 1000-ml volumetric flask, dilute to the mark with distilled water and shake vigorously.

5.4 Allow the major portion of the aluminum to settle, and then filter approximately 100 ml of the solution through a dry No. 41 filter paper, reject the initial 25-30 ml.

5.5 Adjust the flame spectrophotometer in accordance with Table 3.

TABLE III, *Setting for the Beckman DU spectrophotometer with flame attachment*

Settings	Potassium	Barium
Wavelength (mu).....	768	553.5
Phototube	Red	Blue
Resistor position	No. 3	No. 2
Switch selector	0.1	0.1
Slit width (mm).....	0.065	0.11
Sensitivity	3 turns from extreme	Counter Clockwise
Control.....	Clockwise position	Full
Zero suppression	off	No. 1
Sensitivity	off	No. 4
Burner.....	Medium	Medium
Oxygen, pounds.....	Lo	10
Hydrogen, (lbs.).....	3	3

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5.6 Place the sample beaker containing the powder solution to the sample positioner, and move the beaker under the capillary of the aspirator-burner by rotating the sample positioner knob.

Note. When correctly positioned, the beaker will be tilted, and the capillary tube will project into its lowest corner. The capillary tube should not touch the beaker; if it does, bend the sample positioner to correct the condition.

5.7 Take the transmission dial reading.

5.8 Place the beaker containing the standard solutions in the spectrophotometer and take the reading.

Note. After each reading, aspirate a small quantity of distilled water through the atomizer-burner to prevent clogging.

5.9 Prepare an intensity concentration graph on linear graph paper as follows:

- (a) Plot the percent of potassium or barium against the transmission dial reading obtained for each solution.
- (b) Draw a straight line between the points.

5.10 Using the transmission dial reading obtained for the specimen read on the graph for potassium or barium the percent in the sample.

METHOD 501.1

75°C. INTERNATIONAL TEST

1. SCOPE

1.1 This method is used to determine volatility or decomposition in pyrotechnics by subjecting them to a temperature of 75°C. at atmospheric pressure.

2. SPECIMEN

2.1 The specimen shall consist of approximately 10 gm of the pyrotechnic weighed to within 0.5 mg.

3. APPARATUS

3.1 Oven maintained at $75^{\circ} \pm 0.5^{\circ}\text{C}$.

3.2 Lipless beaker, 35 mm in diameter and 50 mm high with a watchglass cover.

3.3 Desiccator containing an indicating desiccant.

4. PROCEDURE

4.1 Transfer the specimen to the beaker, (par. 3.2), cover with a watchglass and weigh.

4.2 Heat at 75°C. for 48 hours, cool in a desiccator, and weigh.

4.3 Calculate the loss in weight to percent of volatility of the sample.

4.4 Note if the material has undergone decomposition or is markedly volatile as indicated by —

- (a) Discoloration.
- (b) The appearance of colored fumes above the material.
- (c) The development of an odor indicating acidity.
- (d) A loss in weight distinctly greater than the moisture content determined by a method appropriate for the material.

METHOD 502.1

100°C. HEAT TEST

1. SCOPE

1.1 This method is used for determining the stability of pyrotechnics by subjecting them to 100°C. at atmospheric pressure.

2. SPECIMEN

2.1 The specimen shall consist of approximately 0.6 gm of the pyrotechnic as received.

3. APPARATUS

3.1 Oven, maintained at $100^{\circ} \pm 1^{\circ}\text{C}$.

3.2 Test tubes (preferably pyrex glass) approximately 10 mm in diameter and 75 mm in length.

3.3 Perforated pipe bomb.

3.4 Desiccator containing an indicating desiccant.

4. PROCEDURE

4.2 Transfer specimens to each of two tared test tubes (par. 3.2) and to each of two similar, but untared, test tubes. Enclose the four tubes in the perforated pipe bomb and place in an oven maintained at $100^{\circ} \pm 1^{\circ}\text{C}$.

4.3 At the end of 48 hours remove the two tared tubes, cool in a desiccator, and weigh. Replace the tubes in the oven for another 48 hours, cool in a desiccator and reweigh.

4.4 Calculate the percentage loss in weight during each 48-hour period of heating. Subtract the percentage of moisture (4.1) the percentage of loss in weight calculated for the first 48-hour heating period.

4.5 Allow the two untared tubes to remain in the oven for 100 hours of continuous heating and note if ignition or explosion occurs.

METHOD 503.1

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 constituents 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) section 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:

$$B = \frac{W}{13.59L}$$

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).

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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 (fig. 2).

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:

- (a) Length of capillary from heating tube joint to surface of mercury pool in cup (C_1).
- (b) Height of mercury column above the surface of the mercury pool (H_1).
- (c) Barometric pressure in millimeters of mercury (P_1).
- (d) Temperature of room in degrees Centigrade (t_1).

4.8 Immerse the heating tube in the constant temperature bath (see fig. 2), 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 Record the following data:

- (a) Length of capillary from heating tube joint to the surface of the mercury pool in the cup (C).
- (b) Height of mercury column above the surface of the mercury pool (H).
- (c) Barometric pressure in millimeters of mercury (P).
- (d) Temperature of the room in degrees Centigrade (t).

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

$$\begin{aligned} \text{Volume of gas, ml} &= (A + B) (C - H) \frac{273 (P - H)}{760 (273 + t)} \\ &\quad - (A + B) (C_1 - H_1) \frac{273 (P_1 - H_1)}{760 (273 + t)} \end{aligned}$$

where:

A = volume of heating tube (less 5 ml allowance for specimen), ml.

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B = Unit capacity of capillary ml per mm (par. 4.1).

C = Length of capillary from heating tube joint to top of mercury column at end of test, mm (par. 4.10).

c_1 = Length of capillary from heating tube joint to top of mercury column at BEGINNING of test, mm (par. 4.7).

H = Height of mercury column above surface of mercury pool at END of test, mm (par. 4.10).

H_1 = Height of mercury column above surface of mercury pool at BEGINNING of test, mm (par. 4.7).

P = Atmospheric pressure at END of test, mm (par. 4.10).

P_1 = Atmospheric pressure at BEGINNING of test, mm (par. 4.7).

t = Temperature of room at END of test, °C. (par. 4.10).

t_1 = Temperature of room at BEGINNING of test, °C. (par. 4.7).

METHOD 504.1

REACTIVITY TEST

1. SCOPE

1.1 This method is used to determine the reactivity of a pyrotechnic with contact material.

2. SPECIMEN

2.1 The specimen shall consist of 5 gm of the pyrotechnic and 5 gm of the contact material. A 2.5 gm portion of the materials is tested as received except in the case of solvent containing contact materials (paints, adhesives, etc.) which would in normal usage be in the dry state. In this case the materials are air dried on glass plates and removed in the form of films for testing. The remaining portion of the pyrotechnic and contact material are reduced to a practicable fineness for intimacy of contact. Pyrotechnics are pulverized under gentle pressure in an agate mortar; metals are tested as fine milled chips or fillings; films, cloth and paper are cut into 1/8-inch squares; propellants are rasped or milled to a fineness of approximately 12 mesh.

3. APPARATUS

3.1 The apparatus used in this method is identical with that used in Method 503.1.

4. MATERIALS

4.1 Contact materials — as specified in the applicable method or specification.

5. PROCEDURE

5.1 Standardize the vacuum stability measuring apparatus as described in Method 503.1, paragraph 4.1.

5.2 Place weighed portion of 2.5 grams of the pyrotechnic in one heating tube, and 2.5 gm. of content material in 2nd heating tube.

5.3 In a third tube place a 5 gram portion of a 50/50 mixture of the contact material and pyrotechnic.

5.4 Determine the volume of gas evolved as specified in Method 503.1, paragraphs 4.3 to 4.12.

5.5 Determine from the amount of gas produced by the mixture of contact material and the pyrotechnic in excess of the amount of gas evolved by the materials themselves, as follows:

$$\text{Gas due to reactivity, ml} = A - (B + C)$$

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where:

- A = amount of gas evolved by the 50/50 mixture of the contact material and the pyrotechnic.
 B = amount of gas evolved by the pyrotechnic.
 C = amount of gas evolved by the contact material.

5.6 Report the reactivity from the following table:

Gas due to reactivity	Reactivity
Less than 0.0 ml.....	None
From 0.0 ml to 1.0 ml.....	Negligible
From 1.0 ml to 2.0 ml.....	Very slight
From 2.0 ml to 3.0 ml.....	Slight
From 3.0 ml to 5.0 ml.....	Moderate
More than 5.0 ml.....	Excessive

Note. The empirical adjective rating of reactivity was devised as a yardstick for the purpose of reporting and classifying data. With the complete data available, the investigator will evaluate its significance as it applies to his particular problem. In general, the first condition (less than zero) occurs when the amount of gas generated by the controls is in excess of that generated by the mixture. Since the Vacuum Stability Test is sensitive only to the first place, minus values of low order (0.10 - 0.30) may be accepted as essentially indicative of no reactivity. Minus values of higher order may indicate a stabilizing or blanketing effect produced by the contact materials upon the pyrotechnic and should be taken into account by the investigator in evaluation of the data. Since the test is severe, reactivity up to and including slight is considered acceptable. Use of a moderately reactive material should be justified by further testing with modifications of existing procedures to more closely approximate the intended use; or a less reactive substitute material may be sought. This latter action is normally taken upon establishment of excessive reactivity.

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METHOD 505.1
IMPACT SENSITIVITY TEST
(U.S. BUREAU OF MINES APPARATUS)

1. SCOPE

1.1 This method is used for determining the sensitivity to impact of pyrotechnics without confinement.

2. SPECIMEN

2.1 The specimen shall consist of approximately 0.020 gm. of the 50/100 mesh pyrotechnic.

3. APPARATUS

3.1 The apparatus for this test, shown by the attached photographs, 1 and 2 consists essentially of an impact block, a plunger, and a weight which can be dropped from any desired height from 1 to 100 cm. The details of the base block and plunger tip are given in Picatinny Arsenal Drawing No. PB-45147.

3.2 The impact block has an easily replaceable insert of hardened steel having a Rockwell hardness of C-63 \pm 2, and is strongly supported by a metal base imbedded in a large block of reinforced concrete. The hardened steel insert has a very uniform, smooth, mirror-like surface.

3.3 The plunger is located directly over the impact block and is supported by a heavy frame work which is imbedded in the same base as the impact block. The Plunger is equipped with a replaceable hard steel tip 1 cm in diameter having a Rockwell hardness of C-63 \pm 2. The plunger is held in position by a metal sleeve which fits snugly, and the apparatus is so constructed that the plunger can be raised about 2.5 cm above the block and locked in this position by means of a rod which extends out horizontally from the plunger.

3.4 Three weights of 500, 1000, and 2000 gm respectively, are used with the apparatus. Only one of these weights can be used at one time. The weight to be used is inserted between two guide bars extending from the plunger frame to the frame at the top of the apparatus. The weight is raised to the desired height by means of an encased magnetic coil held in place by sleeves attached to the two guide bars. A contact point which breaks the electromagnetic circuit and allows the weight to fall when touched by the contact point can be set at any desired height by means of the screw lift, which is equipped with a dial indicating the height of fall of the weight in centimeters. (The zero point should be checked before using the machine). The magnetic coil assembly is raised by a motor driven chain hoist which is supported by two sprockets located at the top and bottom of the guide bars. The resolving chain engages the magnetic assembly by means of a pin extending out on each side of the chain. After the contact point on the magnetic assembly makes contact with the upper contact point and allows the weight to fall, a rod with a cone shaped tip

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extending down from the frame supporting the upper contact point releases the magnetic assembly from the chain hoist pin by spreading apart the spring held supports extending out from the assembly and allowing the assembly to fall slowly down to the weight.

3.5 The impact block insert and plunger tip should be examined frequently to insure that the plunger tip rests evenly on the impact block insert so that the force of impact is distributed uniformly over the entire area between the plunger tip and impact block insert. This can be determined easily as follows: Place a piece of carbon paper on a piece of plain white paper and place the two sheets on the impact block so that the plain paper is on the bottom. Bring the plunger tip down to rest on the carbon paper and then drop the weight from a convenient height so as to make a visible imprint on the plain paper. The carbon imprint on the plain paper should be uniform in color density. If it is found that the impact block is unsatisfactory, place small pieces of thin metal sheet under the low portion until a satisfactory position is obtained.

3.6 When the apparatus is assembled it should be checked by determining impact test values for standard explosives, such as mercury fulminate, tetryl and TNT, having widely different drop test values. The test values of standard mercury fulminate, tetryl and TNT are 5 mc, 26 cm and 100 + cm, respectively when a 2 kg weight is used.

4. PROCEDURE

4.1 Lock the plunger in the raised position and insert the weight desired between the guide bars.

4.2 Close the magnetic circuit and bring the weight into contact with the magnetic lifter.

4.3 Set the upper contact point so that the weight will be released from such a height that the resulting impact is expected to cause explosion.

4.4 Place the specimen in the center of the impact block, which shall be maintained at a temperature of $25^{\circ} \pm 2^{\circ}\text{C}$.

4.5 Lower the plunger to the impact block, place a centering device around the plunger, hold firmly and raise the plunger to the lock position.

4.6 Weigh the pyrotechnic and place into the centering device, distribute evenly with a very small brush, lower the plunger gently upon the pyrotechnic and remove the centering device. If the sample is not visible around the entire edge of the tip, raise the plunger, lock it into place and recenter the sample.

4.7 Start the motor which operates the chain hoist and as soon as the weight falls, shut off the motor switch.

4.8 If no detonation occurs, as indicated by absence of fire, smoke, or noise, remove the sample from the impact block and plunger tip and repeat 5.6 and 5.7.

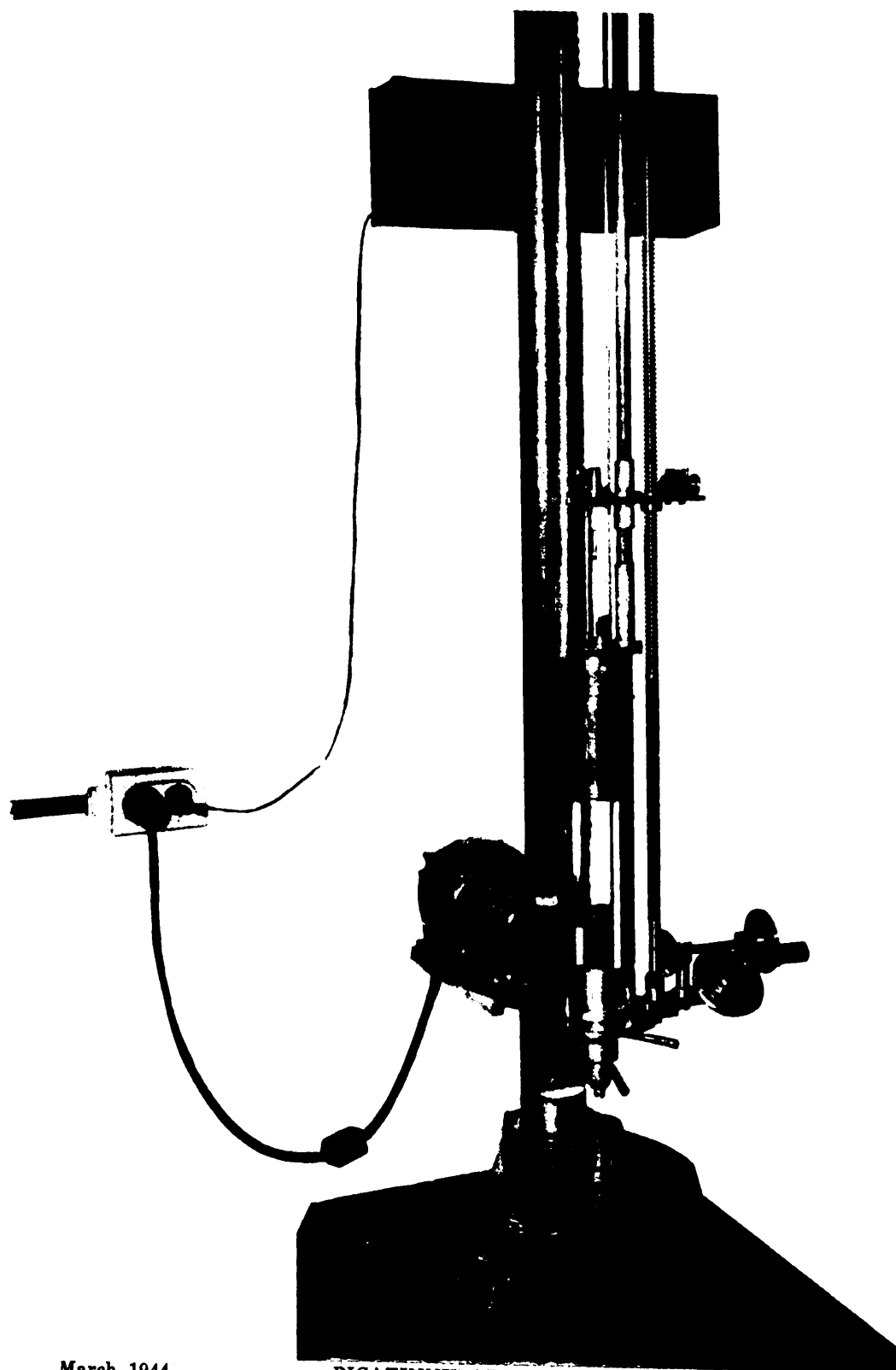
4.9 Drop the weight from greater heights on new portions of the sample until detonation occurs.

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4.10 Record as the test value the minimum height in centimeters from which the falling weight by impact causes detonation of the sample in at least one of ten tests and from one centimeter below which the falling weight causes no explosions in ten tests.

Note. After each detonation, carefully clean the block and plunger tip with fine abrasive paper (a Carborundum or emery abrasive paper of grit No. 0 has been found satisfactory). When cleaning the tip, place the Carborundum paper on the block and allow the tip and plunger to rest on the paper. Then move the paper horizontally between the block and plunger tip so that the surface of the tip does not become rounded at the edges. If either the impact block or plunger tip is dented, cracked, or deformed in any way, replace it before making further tests.

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March 1944

PICATINNY ARSENAL

ORDNANCE DEPARTMENT

Tech. 14)

(L)

FIGURE 1. *Impact Test, Bureau of Mines Apparatus.*

Method 505.1

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March 1944

PICATINNY ARSENAL

ORDNANCE DEPARTMENT
(Tech. 14)

FIGURE 2. *Weights, Plunger Tip, and Base Block for Impact Test, Bureau of Mines Apparatus*
(L)

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METHOD 601.1

TITANOUS CHLORIDE (0.2N STANDARD SOLUTION)

1. SCOPE

1.1 This method is used for the preparation of a 0.2N standard solution of titanous chloride, using as a base either a titanous chloride solution or titanium hydride.

2. APPARATUS

2.1 Storage system (fig 1), consisting essentially of:

- (a) Storage bottle, amber- colored pyrex glass, (or clear pyrex glass painted black), 18-liter.
- (b) Kipp generator for maintaining a protective atmosphere of carbon dioxide.

Note. A cylinder of inert gas may be used. However, a special pressure release valve must be reinstalled so that the pressure on the buret and on the storage bottle is approximately equal to prevent gassing of the solution in the buret.

- (c) Buret, 50-ml.

2.2 Titration flask (fig. 2).

2.3 Filter paper, Whatman No. 41, fluted.

2.4 Hotplate in a well-ventilated hood.

3. MATERIALS

3.1 Titanous chloride, 20-percent solution (or titanium hydride, reagent grade).

3.2 Hydrochloric acid, Reagent grade, 38-percent.

3.3 Inert gas (carbon dioxide or nitrogen), highest purity, oxygen-free.

3.4 Potassium bichromate, National Bureau of Standards Sample No. 136, dried for 2 hours at 100°C.

3.5 Sulfuric acid, 10-percent solution.

3.6 Barium diphenylamine sulfonate indicator solution, as specified in Method 708.1.

Note. Sodium diphenylbenzidine sulfonate indicator solution, specified in Method 707.1, may be used as an alternative, and will produce essentially the same endpoint color in the titration process. However, in case of dispute, use the barium diphenylamine sulfonate solution.

3.7 Ammonium thiocyanate indicator solution, 20-percent, as specified in Method 706.1.

4. PROCEDURE

MIL-STD-1234
22 June 1962**4.1** Prepare the 0.2N solution from 20-percent titanous chloride solution as follows:

Note. The proportions given in the following procedure will yield one liter of solution. Normally 18 liters are prepared at one time.

- (a) Mix 150 ml of the 20-percent titanous chloride solution with 100 ml of 38-percent hydrochloric acid. Dilute to one liter with distilled water.
- (b) Mix the solution thoroughly by passing a current of oxygen-free inert gas through it.
- (c) Filter the solution through No. 40 Whatman filter paper until the filtrate is completely clear.
- (d) Agitate the solution thoroughly with the inert gas for a few minutes and store the solution in the amber storage bottle. Maintain the inert atmosphere during storage, using the apparatus shown in figure 1.

4.2 Prepare the 0.2N solution from titanium hydride as follows:

- (a) For each liter of solution required, warm 100 ml of 38-percent hydrochloric acid to 70° to 80°C. on a hotplate in a well-ventilated hood.
- (b) Add, in small portions, 12 gm of titanium hydride, and cover with a watch-glass.

Note. Remove the watchglass only to add titanium hydride.

- (c) Warm the solution on the hotplate until the evolution of hydrogen has nearly ceased.
- (d) Remove from the hotplate, and cool to room temperature.
- (e) Add approximately 250 ml of oxygen-free distilled water, and mix the solution thoroughly for 5 to 10 minutes by passing a current of oxygen-free inert gas through it.
- (f) Filter the solution through the filter paper until the filtrate is completely clear.
- (g) Add 100 ml of the 38-percent hydrochloric acid, dilute the solution to one liter with oxygen-free distilled water.
- (h) Agitate the solution with the inert gas, and store as described in paragraph 4.1.

4.3 Standardize the solution as follows:

- (a) Attach the source of inert gas to the titration flask, and allow the gas to flow for 5 minutes to displace the air in the flask. Continue the flow of gas through the flask throughout the entire titration procedure.
- (b) Transfer to the titration flask 0.3 to 0.4 gm of potassium bichromate, weighed to within 0.2 mg, and dissolve in 100 ml of distilled water.
- (c) Add 50 ml of 10-percent sulfuric acid solution.

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- (d) Titrate the solution with the 0.2N titanous chloride solution, until near the end point, as shown by the change in color to green.

Note. The change in color is from brownish purple, to purple, and to a distinct green.

- (e) Add five drops of the barium diphenylamine sulfonate indicator solution, and continue the titration until the end point is reached, as shown by the appearance of a light-green color that does not change to a light-blue color for at least 30 seconds.

Note. The light-green color will change to light blue upon standing.

4.4 Make a blank determination to correct for the presence of iron as follows:

- (a) Add 5 ml of the 20-percent ammonium thiocyanate indicator solution to the flask. If iron is present, a ferric thiocyanate complex will form and the solution will change from light blue to deep red.
- (b) Back-titrate the ferric thiocyanate complex with the 0.2N titanous chloride until the light-blue color reappears.

4.5 Determine the milliliters of titanous chloride solution required to complete both the titration and the back-titration. Add the two values and apply buret and temperature corrections.

4.6 Calculate the normality of the titanous chloride solution using the following equation:

$$\text{Normality of titanous chloride} = \frac{A}{0.04904B}$$

where :

A = Potassium bichromate, gm.

B = Titanous chloride (including iron blank, temperature, and buret corrections), ml.

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APPARATUS FOR STORING AND USING TITANOUS CHLORIDE SOLUTION

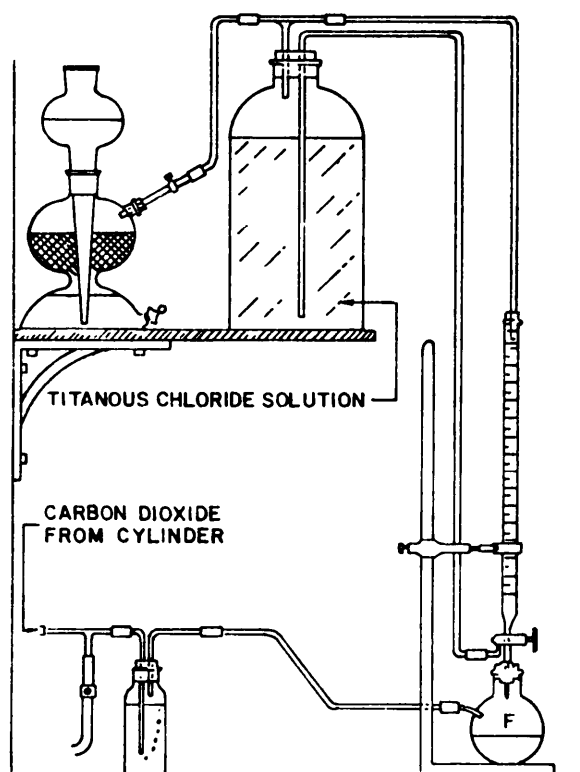
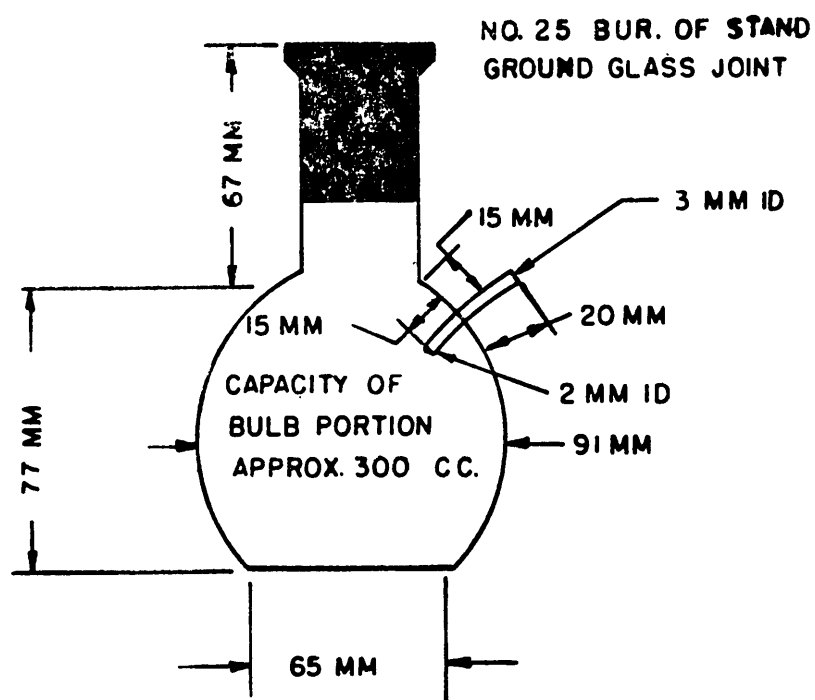


FIGURE 1.

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TITRATION FLASK
PYREX GLASS

All demensions are approximate

FIGURE 2.

METHOD 602.1

SODIUM HYDROXIDE (0.1N STANDARD SOLUTION)

1. SCOPE

1.1 This method is used for the preparation of 0.1N standard solution of sodium hydroxide.

2. APPARATUS

2.1 Beaker, 2 liter.

2.2 Watchglass.

2.3 Storage bottle, clear pyrex glass, 18 liters.

2.4 Storage bottle, clear pyrex glass, rubber stoppered, 2 liters.

2.5 Tube, drying, 150 mm filled with ascarit to protect against CO₂ from atmosphere.

2.6 Beaker, 400 ml.

2.7 Titration apparatus.

3. MATERIALS

3.1 Sodium hydroxide, C. P., pellet form.

3.2 Hydrochloric acid, standard 0.1N solution as specified in Method 604.1.

4. PROCEDURE

4.1 Prepare a supply of 50-percent sodium hydroxide as follows:

- (a) To 1300 ml of water in a 2-liter beaker, add slowly, with stirring, approximately 1350 gm of sodium hydroxide pellets.
- (b) Stirr intermittently until solution is complete. Cover with a watchglass and let cool to room temperature.
- (c) Decant into a bottle, close with a rubber stopper and let stand 2 weeks, or preferably longer, until the solution is perfectly clear.

Notc. One ml of this solution contains approximately 0.75 gm. of sodium hydroxide.

4.2 Prepare the 0.1N standard solution of sodium hydroxide as follows:

- (a) For each liter of solution, add 6 ml of the 50-percent sodium hydroxide to a pyrex bottle containing one liter of CO₂— free water.

Note. Normally 18 liters of the solution are prepared.

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- (b) Mix the solution thoroughly by shaking at hourly intervals during the day, let stand overnight, then shake again.
- (c) Store this solution in a pyrex bottle protected from atmospheric conditions by a drying tube, filled with ascarit.

4.3 Standardize the solutions follows:

- (a) Measure accurately 330 to 40 ml of standard 0.1N hydrochloric acid into a 400 ml beaker and dilute to 100 ml with freshly boiled and cooled distilled water.
- (b) Add the proper indicator.

Note. The choice of an indicator to be used in the standardization will depend upon the character of the subsequent titrations. In general, the same indicator should be used for the standardization as for the titration of the sample.

- (c) Titrate to the end point.
- (d) Calculate the normality of the standard sodium hydroxide solution as follows:

$$\text{Normality of sodium hydroxide} = \frac{AN}{B}$$

where:

- A = volume of hydrochloric acid, ml.
- B = volume of sodium hydroxide, ml.
- N = normality of hydrochloric acid ml.

METHOD 603.1**FERRIC AMMONIUM SULFATE (0.15N STANDARD SOLUTION)****1. SCOPE**

1.1 This method is used for the preparation of a 0.15N standard solution of ferric ammonium sulfate. (The normality of this solution remains constant indefinitely.)

2. APPARATUS

2.1 Beaker, 600-ml.

2.2 Graduate, 1000-ml.

2.3 Filter paper, Whatman No. 1, or equivalent.

2.4 Titration flask (fig. 1).

2.5 Buret.

2.6 Storage bottle, pyrex glass, 18-liter.

3. MATERIALS

3.1 Hydrated ferric ammonium sulfate, $\text{Fe}_2(\text{SO}_4)_3(\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$, CP grade.

3.2 Sulfuric acid, concentrated.

3.3 Hydrochloric acid, 15-percent.

3.4 Ammonium thiocyanate, 20-percent solution as specified in Method 706.1.

3.5 Titanous chloride, standard 0.2N solution as specified in Method 601.1., freshly standardized.

3.6 Inert gas (carbon dioxide or nitrogen), highest purity, oxygen-free.

4. PROCEDURE

4.1 Prepare the solution as follows:

- (a) Pour 400 ml of distilled water into a 600-ml beaker, and add 75 gm of hydrated ferric ammonium sulfate and 25 ml of the concentrated sulfuric acid.
- (b) Transfer the solution to a 1000-ml graduate, and dilute to capacity with distilled water. Mix thoroughly by passing a current of air through the solution.
- (c) Filter the solution through the Whatman No. 1 filter paper.

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- (a) Attach a source of inert gas to the titration flask, and allow the gas to flow gently for 5 minutes to displace the air in the flask. Continue the flow of gas through the flask throughout the titration.
- (b) Transfer an accurately measured portion of 40 to 45 ml of the ferric ammonium sulfate solution to the titration flask.
- (c) Add 15 ml of the 15-percent hydrochloric acid solution.
- (d) Titrate with newly standardized 0.2N titanous chloride solution until near the end point.
- (e) Add 5 ml of the ammonium thiocyanate solution, and continue the titration until the red color just disappears.

4.3 Apply temperature and buret corrections to the observed readings, and calculate the normality of the ferric ammonium sulfate solution, using the following equation:

$$\text{Normality of ferric ammonium sulfate} = \frac{AN}{B}$$

where:

A = titanous chloride solution, ml.

B = ferric ammonium sulfate solution, ml.

N = normality of titanous chloride solution

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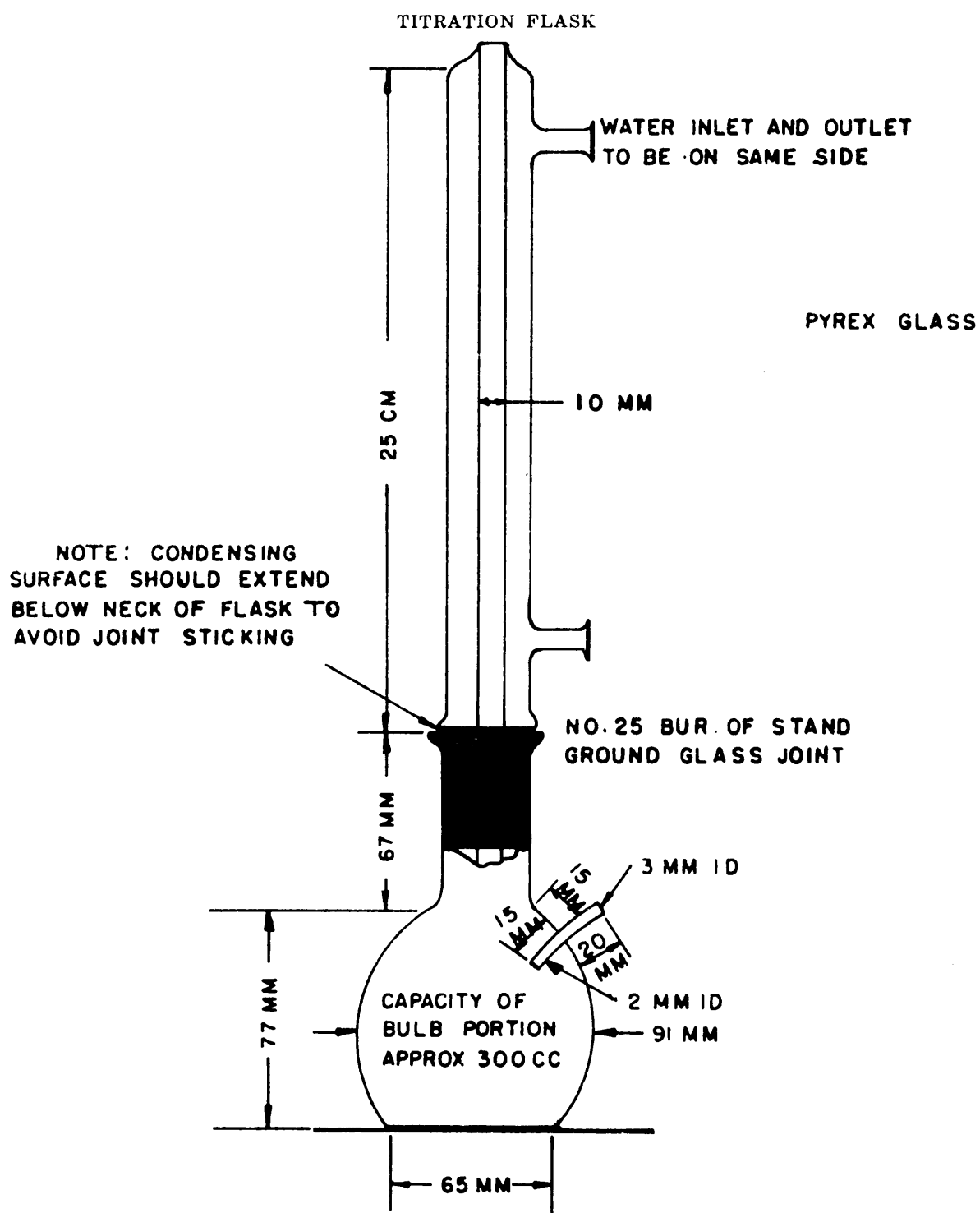


FIGURE 1.

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METHOD 604.1

HYDROCHLORIC ACID (0.1N STANDARD SOLUTION)

1. SCOPE

1.1 This method is used for the preparation of a 0.1N standard solution of hydrochloric acid.

2. APPARATUS

2.1 Storage bottle, clear pyrex glass, 18 liter.

2.2 Drying tube, 150 mm, calcium chloride filled.

2.3 Beaker, 400 ml.

2.4 Cylinder, graduated, 60 ml.

2.5 Watchglass.

2.6 Crucible, medium porosity, fritted glass.

2.7 Rubber policeman.

2.8 Wash bottle.

2.9 Oven, 140°–150°C.

3. MATERIALS

3.1 Hydrochloric acid, concentrated (approximately 37-percent).

3.2 Calcium chloride.

3.3 Silver nitrate solution. Dissolve 24 gm silver nitrate in water. Add 90 ml of 70-percent nitric acid and dilute to one liter. Store in dark bottle.

3.4 Silver nitrate wash solution. 0.050 gm of silver nitrate per liter.

3.5 Sodium chloride solution, 1-percent.

3.6 Nitric acid wash solution, 0.01N. Dilute 7 ml of 70-percent nitric acid to one liter to form an approximately 0.1N solution. Dilute 100 ml of this solution to one liter to form 0.01N solution as needed.

4. PROCEDURE

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4.1 Prepare the solution as follows

- (a) For each liter of solution, add 8.5 ml of hydrochloric acid to 800 ml of distilled water and dilute to one liter with distilled water.

Note. Normally 18 liters of the solution are prepared at one time.

- (b) Shake intermittently for at least — hour.
- (c) Store this solution in a pyrex bottle protected from atmospheric contamination by means of a 150 mm drying tube filled with calcium chloride.

4.2 Standardize the solution as follows:

- (a) Accurately measure 35 to 40 ml of the hydrochloric acid solution into a 400 ml beaker.
- (b) Add 100 to 150 ml of distilled water.
- (c) By means of a graduate, add slowly, during the course of 2-3 minutes, 45 to 50 ml of silver nitrate solution, with moderate stirring. The precipitation must be conducted at room temperature in diffuse light, and in the absence of halogen acid vapors.
- (d) Cover the beaker with a watch glass and place in the dark.
- (e) Allow the precipitate to settle overnight at room temperature.
- (f) Test for completeness of precipitation by adding a few drops of the silver nitrate solution to the supernatant liquid in the beaker.
- (g) Decant the supernatant liquid slowly through a tared fritted glass crucible and wash the precipitate with five 25 to 30 ml portions of silver nitrate wash solution.
- (h) Transfer the precipitate quantitatively to the crucible, using a rubber policeman and a wash bottle containing 0.01N nitric acid.
- (i) Wash the precipitate with the 0.01N nitric acid solution until the filtrate, when tested with 1-percent sodium chloride solution, shows no turbidity.
- (j) Dry the crucible and contents at 140-150°C. for 2 hours, cool in desiccator for 1 hour, then weigh.
- (k) Calculate the normality of the hydrochloric acid as follows:

$$\text{Normality of the hydrochloric acid} = \frac{A6.9766}{B}$$

where:

A = the weight of precipitate, gm.

B = the volume of hydrochloric acid solution used, ml.

Note. If a freshly standardized sodium hydroxide solution is available, hydrochloric acid may be standardized by cross titration as follows:

Measure accurately 30 to 40 ml of the standard acid into a 250 ml Erlenmeyer flask or 400 ml beaker, dilute with 50 to 100 ml of recently boiled and cooled distilled water and add

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the proper indicator. Titrate with the standard sodium hydroxide solution until the indicator changes color. Calculate the normality of the hydrochloric acid as follows:

$$\text{Normality of hydrochloric acid} = \frac{AN}{B}$$

where:

A = the volume of sodium hydroxide, ml.
B = the volume of hydrochloric acid, ml.
N = normality of sodium hydroxide.

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METHOD 605.1
POTASSIUM BICHROMATE (0.1N STANDARD SOLUTION)

1. SCOPE

1.1 This method is used for the preparation of a 0.1N standard solution of potassium bichromate.

2. APPARATUS

2.1 Graduate cylinder, 1000 ml.

2.2 Erlenmeyer flask 300 ml.

2.3 Oven.

2.4 Rubber stopper provided with a Bunsen valve.

3. MATERIALS

3.1 Potassium bichromate, reagent grade.

3.2 Iron, reagent grade.

3.3 Sodium bicarbonate, reagent grade.

3.4 Sulfuric acid, 5N solution.

3.5 Barium diphenylamine sulfate indicator solution as specified in Method 708.1.

3.6 Phosphoric acid solution, 50-percent.

4. PROCEDURE

4.1 Dissolve 5 gm of potassium bichromate, in sufficient water to make exactly 1000 ml.

4.2 Standardize the solution as follows:

- (a) Treat 2 gm of sodium bicarbonate with 20 ml of 5N sulfuric acid in an Erlenmeyer flask in order to displace the air in the flask.
- (b) Add 0.25 gm of Fe (wt to 0.1 mg) and immediately stopper the flask with a rubber stopper equipped with a Bunsen valve.
- (c) When the reaction has ceased add 10 ml of phosphoric acid and titrate with potassium bichromate, using 15 ml of the barium diphenylamine sulfate as an indicator.

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- (d) Titrate slowly near the end point which is indicated by a purplish or blue-violet color.
- (e) Calculate the normality of the potassium bichromate solution as follows:

$$\text{Normality of potassium bichromate} = \frac{W}{0.05585A}$$

where:

W = weight of actual iron used, gm.
A = potassium bichromate used, ml.

METHOD 606.1
POTASSIUM PERMANGANATE (0.1N STANDARD SOLUTION)

1. SCOPE

1.1 This method is used for the preparation of a 0.1N standard solution of potassium permanganate.

2. APPARATUS

2.1 Beaker, 600-ml.

2.2 Hotplate.

2.3 Gooch crucible (ignited).

2.4 Buret, 50-ml.

2.5 Storage bottle, amber-colored pyrex glass (or clear pyrex painted black).

3. MATERIALS

3.1 Potassium permanganate, reagent grade.

3.2 Sodium oxalate, National Bureau of Standards Sample No. 40, dried for two hours at 105°C.

3.3 Sulfuric acid solution prepared by adding 50 ml concentrated sulfuric acid to 950 ml of distilled water, boiling for 10 to 15 minutes, and cooling to room temperature.

4. PROCEDURE

4.1 Prepare the 0.1N solution of potassium permanganate as follows:

- (a) Dissolve 3.2 to 3.3 gm of the potassium permanganate in approximately 300 ml of distilled water for each liter of solution desired.
- (b) Boil the solution vigorously on the hotplate for 30 minutes.
- (c) Filter the solution through the ignited Gooch crucible.
- (d) Dilute the filtrate to the required volume with freshly boiled distilled water.
- (e) Let the solution stand at room temperature for at least 12 hours before standardization or use.

4.2 Standardize the solution as follows:

Note. This procedure is essentially the method described by Fowler and Bright in The Journal of Research, National Bureau of Standards, Vol. 15 (1935), page 493.

- (a) Place 0.3 gm of sodium oxalate weight to 0.1 mg in the 600-ml beaker.

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Add 250 ml of the sulfuric acid solution (par. 3.3), and stir until the oxalate has dissolved.

- (b) Add 39 to 40 ml of the 0.1N potassium permanganate solution to be standardized at a rate of 25 to 35 ml per minute, while stirring slowly.
- (c) Allow the solution to stand until the pink color disappears approximately 45 seconds).
- (d) Heat the solution to 55° to 60°C., and complete the titration by adding potassium permanganate solution drop by drop, allowing each drop to become decolorized before adding the next. The titration may be considered complete when the faint pink coloration persists for 30 seconds.

4.3 Run a blank determination using exactly the same volumes of solutions, omitting the sodium oxalate.

4.4 Calculate the normality of the potassium permanganate solution using the following equation:

$$\text{Normality of potassium permanganate} = \frac{A}{0.0670 \cdot (B-C)}$$

where:

A = sodium oxalate, gm.

B = potassium permanganate required for the determination, ml.

C = potassium permanganate required for the blank, ml.

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METHOD 607.1

SILVER NITRATE (0.1N STANDARD SOLUTION)

1. SCOPE

1.1 This method is used for the preparation of a 0.1N standard solution of silver nitrate.

Note. The normality of the solution will be sufficiently accurate for most analytical requirements. For more precise requirements, the silver nitrate solution may be standardized against CP sodium chloride or CP potassium chloride by the Volhard method described in the Official Methods of Analysis of the Association of Official Agricultural Chemists, Seventh Edition, 1950, Method No. 39.29.

2. APPARATUS

2.1 Volumetric flask, one liter (painted black or covered with tape to exclude light).

3. MATERIALS

3.1 Silver nitrate, CP grade, dried for 2 hours at 120°C.

4. PROCEDURE

4.1 Dissolve 17.006 gm of silver nitrate in distilled water.

4.2 Transfer this solution quantitatively to the one-liter volumetric flask, and dilute to the required volume with freshly boiled distilled water.

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METHOD 608.1
POTASSIUM THIOCYANATE (0.1N STANDARD SOLUTION)

1. SCOPE

1.1 This method is used for the preparation of a 0.1N standard solution of potassium thiocyanate.

2. APPARATUS

2.1 Volumetric flask, 1-liter.

3. MATERIALS

3.1 Potassium thiocyanate, reagent grade.

3.2 Silver nitrate, 0.1N standard solution as specified in Method 607.1.

4. PROCEDURE

4.1 Dissolve 9.720 gm of potassium thiocyanate in distilled water, transfer to the one-liter volumetric flask, and make up to volume with distilled water.

4.2 Determine by blank titration the milliliters 0.1N silver nitrate solution equivalent to one milliliter of the potassium thiocyanate solution.

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METHOD 701.1

STARCH INDICATOR SOLUTION

1. MATERIALS

1.1 Mercuric iodide, CP grade.

1.2 Soluble starch (suitable for iodometry) : 2 gm.

1.3 Water, freshly distilled.

2. PROCEDURE

2.1 Mix gm of the starch and several milligrams of the mercuric iodide (as a preservative) with a little distilled water. Add the mixture slowly to 500 ml of boiling distilled water. Allow the liquid to boil for 6 minutes, then cool to room temperature.

Note. This solution should be stable for several weeks.

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METHOD 702.1

METHYL RED INDICATOR SOLUTION

1. MATERIALS

1.1 Methyl red, reagent grade: 0.1 gm.

1.2 Ethyl alcohol, 95-percent (MIL-E-463, Grade 1) : 100 ml.

2. PROCEDURE

2.1 Dissolve the methyl red in the ethyl alcohol, while stirring.

Note. This solution is quite stable and should be effective for several months. For the average titration involving 200 to 400 ml of solution, 3 to 4 drops of this indicator are used.

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METHOD 703.1

PHENOLPHTHALEIN INDICATOR SOLUTION

1. MATERIALS

1.1 Phenolphthalein, reagent grade: 0.1 gm.

1.2 Ethyl alcohol, 95-percent (MIL-E-463, Grade 1) : 100 ml.

1.3 Sodium hydroxide, 0.5N solution.

1.4 Hydrochloric acid, 0.5N solution.

2. PROCEDURE

2.1 Dissolve the phenolphthalein in the ethyl alcohol.

2.2 Neutralize the acidity of the alcohol by adding 0.5N solution of sodium hydroxide, drop by drop, until a faint-pink color develops, then adding one drop of 0.5N hydrochloric acid solution until the color is just discharged.

Note. If protected from contact with carbon dioxide, this solution will keep indefinitely.

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METHOD 704.1

METHYL ORANGE INDICATOR SOLUTION

1. MATERIALS

1.1 Methyl orange, reagent grade: 0.1 gm.

1.2 Water, freshly distilled: 100 ml.

2. PROCEDURE

2.1 Dissolve the methyl orange in the distilled water.

Note. This solution should be stable for several months.

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METHOD 705.1
FERRIC AMMONIUM SULFATE INDICATOR SOLUTION

1. MATERIALS

1.1 Ferric ammonium sulfate, reagent grade: 200 gm.

1.2 Nitric acid, 70-percent, reagent grade: 25 ml.

1.3 Water, freshly distilled: 300 ml.

2. PROCEDURE

2.1 Dissolve the ferric ammonium sulfate in the distilled water.

2.2 Heat the solution to boiling, then add the nitric acid.

2.3 Filter the solution, and store in a pyrex glass bottle.

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METHOD 706.1
AMMONIUM THIOCYANATE INDICATOR SOLUTION (20-PERCENT)

1. MATERIALS

1.1 Ammonium thiocyanate, reagent grade: 20 gm.

1.2 Water, freshly distilled: 100 ml.

2. PROCEDURE

2.1 Dissolve the ammonium thiocyanate in the distilled water.

Caution: This solution must be colorless for use as an indicator.

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METHOD 707.1
SODIUM DIPHENYLBENZIDINE SULFONATE
INDICATOR SOLUTION

1. MATERIALS

1.1 Sodium diphenylbenzidine sulfonate reagent grade: 0.50 gm.

1.2 Water, freshly distilled: 100 ml.

2. PROCEDURE

2.1 Dissolve the sodium diphenylbenzidine sulfonate in the distilled water.

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METHOD 708.1
BARIUM DIPHENYLAMINE SULFONATE
INDICATOR SOLUTION

1. MATERIALS

1.1 Barium diphenylamine sulfonate, reagent grade: 0.32 gm.

1.2 Water, freshly distilled: 11 ml.

2. PROCEDURE

2.1 Dissolve the barium diphenylamine sulfonate in the distilled water.

Note. Alternate indicator solution. Prepare a diphenylamine sulfonate acid solution from the barium salt and sulfuric acid.

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METHOD 709.1
ERIOCHROME BLACK T INDICATOR SOLUTION

1. MATERIALS

1.1 Eriochrome black T indicator, reagent grade: 0.4 gm.

1.2 Methyl alcohol: 100 ml.

2. PROCEDURE

2.1 Dissolve the eriochrome black T in the methyl alcohol.

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