



Designation: C 471M – 01

METRIC

Standard Test Methods for Chemical Analysis of Gypsum and Gypsum Products [Metric]¹

This standard is issued under the fixed designation C 471M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope *

1.1 These test methods cover the chemical analysis of gypsum and gypsum products, including gypsum ready-mixed plaster, gypsum wood-fibered plaster and gypsum concrete.

NOTE 1—Gypsum reference standard materials, prepared by Domtar, Inc are available through Brammer Standards Company, Inc.

1.2 The test methods appear in the following order:

	Sections
Alternative Procedure for Analysis for Calcium Sulfate by Ammonium Acetate Method	17-22
Alternative Procedure for Analysis for Sodium Chloride by the Coulometric Method	23-29
Complete Procedure	5-16
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Optional Procedure for Analysis for Sodium by Flame Photometry	47-54
Optional Procedure for Analysis for Sodium by the Atomic Absorption Method	40-46
Preparation of Sample	4
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1.3 The text of this standard references notes and footnotes that provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

C 11 Terminology Relating to Gypsum and Related Gypsum Building Materials and Systems²

¹ These test methods are under the jurisdiction of ASTM Committee C11 on Gypsum and Related Building Materials and Systems and are the direct responsibility of Subcommittee C11.01 on Specifications and Test Methods for Gypsum Products.

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² Annual Book of ASTM Standards, Vol 04.01.

C 22/C 22M Specification for Gypsum²
C 28 Specification for Gypsum Plasters²
C 59 Specification for Gypsum Casting and Molding Plaster²
C 61 Specification for Gypsum Keene's Cement²
C 317 Specification for Gypsum Concrete²
C 842 Specification for Application of Interior Gypsum Plaster²
D 1193 Specification for Reagent Water³
D 1428 Methods of Test for Sodium and Potassium in Water and Water-Formed Deposits by Flame Photometry³
D 2013 Method of Preparing Coal Samples for Analysis⁴
E 11 Specification for Wire-Cloth and Sieves for Testing Purposes⁵

3. Terminology

3.1 *Definitions*—Definitions shall be in accordance with Terminology C 11.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *sample as received, n*—a representative portion of raw gypsum or gypsum product in the state received by the testing laboratory, including aggregates, impurities and water content.

3.2.2 *dried sample, n*—a sample devoid of free water.

3.2.3 *riffle, n*—a hand feed sample divider device that divides the sample into parts of approximately the same weight. (D 2013)

4. Preparation of Sample

4.1 *General Procedures*—Details of sample preparation will vary according to the type of material being tested.

4.1.1 *Sample As Received*—Use a sufficient amount of sample such that, after sieving, not less than 50 g of sample will remain for testing. Weigh the entire sample immediately after opening the container in which the material was received. This will become the weight of the sample as received.

4.1.2 *Drying*—Dry the sample in accordance with Section 7. This will be the weight of the dried sample.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 05.05.

⁵ Annual Book of ASTM Standards, Vol 14.02.

*A Summary of Changes section appears at the end of this standard.

4.1.3 *Crushing and Grinding*—Crush and grind the sample by hand with a mortar and pestle or by mechanical crushing and grinding equipment to pass a 250- μm (No. 60) sieve. Take care, particularly with mechanical equipment, not to expose the sample to temperatures of more than 52°C. Clean the equipment thoroughly between samples. Thoroughly remix the ground sample and store it in an airtight container to avoid contamination.

4.1.4 *Rehydrating*—Thoroughly blend and rehydrate samples which contain calcium sulfate in forms other than $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ and natural anhydrite. Place the sample in distilled water and keep it wet for not less than 48 h. Dry the hydrated sample in an oven at $45 \pm 3^\circ\text{C}$ to constant weight and recrush or grind it in accordance with 4.1.3.

4.1.5 *Sample Reduction*—Thoroughly mix and reduce large samples as required by quartering or by the use of a riffle to obtain a specimen of approximately 50 g.

4.2 *Gypsum (C 22/C 22M)*—Gypsum samples will be received in the form of rocks, powder or both. If necessary crush and reduce the entire dried sample in accordance with 4.1.3 and 4.1.5.

4.3 *Gypsum Plaster, (C 28).*

4.3.1 *Gypsum Ready-Mixed Plaster or Gypsum WoodFibered Plaster*—Screen the dried sample through a 150- μm (No. 100) sieve⁶ and discard the residue retained on the sieve. Reweigh the remaining sample and calculate the percentage of the dried sample. Reduce the sample in accordance with 4.1.5. Thoroughly blend and rehydrate the specimen in accordance with 4.1.4.

4.3.2 *Gypsum Neat Plaster or Gypsum Gauging Plaster*—Reduce the dried sample in accordance with 4.1.5. Thoroughly blend and rehydrate the specimen in accordance with 4.1.4.

4.4 *Gypsum Casting and Molding Plaster, (C 59)*—Reduce the dried sample in accordance with 4.1.5. Thoroughly blend and rehydrate the specimen in accordance with 4.1.4.

4.5 *Gypsum Keene's Cement, (C 61)*—Reduce the dried sample in accordance with 4.1.5. Blend in no more than 1 % molding plaster or K_2SO_4 and rehydrate the specimen in accordance with 4.1.4.

4.6 *Gypsum Concrete, (C 317)*—Screen the dried sample through a 150- μm (No. 100) sieve⁶ and discard the residue retained on the sieve. Reweigh the remaining sample and calculate the percentage of the dried sample. Reduce the sample in accordance with 4.1.5. Thoroughly blend and rehydrate the specimen in accordance with 4.1.4.

4.7 *Gypsum Board*—Cut or break the dried sample into small pieces. Using a mortar and pestle, strike the pieces of the sample to loosen the paper face. Remove the pieces of paper by hand as they are separated from the core of the gypsum board. Carefully scrape any remaining powder from the paper. When all the paper has been removed from the pieces of the sample, reduce the sample in accordance with 4.1.5. Thoroughly blend and rehydrate the specimen in accordance with 4.1.4.

5. Apparatus

5.1 *Analytical Balance*—Capable of weighing the weighing bottles, lids and samples.

5.2 *Balance*—Capable of weighing not less than 100 g at a precision of 0.001 g.

5.3 *Drying Oven*—A mechanical convection oven set at $45 \pm 3^\circ\text{C}$.

5.4 *Desiccator*—Capable of being tightly sealed and containing calcium chloride or equivalent desiccant.

5.5 *Calcining Oven or Furnace*—Capable of achieving and maintaining temperatures to not less than 1000°C.

5.6 *Weighing Bottles*—Borosilicate glass or ceramic containers with tightly sealable lids.

5.7 *Hot Plate*—A controllable hot plate capable of heating casseroles to approximately 120°C.

5.8 *Porcelain Casseroles*—With a capacity of 50 to 100 mL.

5.9 *Filtering Funnels.*

5.10 *Filter Paper*—Ashless filter paper Whatman #42 or equivalent.⁷

5.11 *Porcelain Crucibles.*

5.12 *Mortar and Pestle.*

5.13 *Mechanical Jaw Crusher*—Capable of crushing gypsum rocks up to 50 mm diameter.

5.14 *Mechanical Grinder*—Burr mill or equivalent capable of grinding the granular output of the jaw crusher specified in 5.13.

6. Reagents

6.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, use reagents that conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁸ If it is necessary to use other grades first ascertain that the reagent is of sufficiently high purity so that its use will not lessen the accuracy of the determination.

6.1.1 *Ammonium Chloride* (NH_4Cl).

6.1.2 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

6.1.3 *Ammonium Nitrate* (25 g/L)—Dissolve 25 g of ammonium nitrate (NH_4NO_3) in water and dilute to 1 L.

6.1.4 *Ammonium Oxalate* ($(\text{NH}_4)_2\text{C}_2\text{O}_4$).

6.1.5 *Barium Chloride* (100 g/L)—Dissolve 100 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 L.

6.1.6 *Calcium Chloride* (CaCl_2)—Anhydrous Calcium Chloride with a combined water of not more than 5 %.

6.1.7 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

6.1.8 *Hydrochloric Acid* (1 + 4)—Mix 1 volume of HCl (sp gr 1.19) with 4 volumes of water.

⁷ Whatman No. 42 or an equivalent filter paper has been found suitable for this purpose.

⁸ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁶ Detailed requirements for this sieve are given in Specification E 11.

6.1.9 *Hydrochloric Acid* (1 + 5)—Mix 1 volume of HCl (sp gr 1.19) with 5 volumes of water.

6.1.10 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO₃).

6.1.11 *Potassium Chromate Solution* (100 g/L)—Dissolve 5 g of potassium chromate (K₂CrO₄) in 50 mL of water, mix, add 10 drops of 0.05 N silver nitrate (AgNO₃) solution, allow to stand for 5 min, and filter.

6.1.12 *Potassium Permanganate* (5.6339 g/L)—Dissolve 5.6339 g of potassium permanganate (KMnO₄) in water and dilute to 1 L.

6.1.13 *Silver Nitrate, Standard Solution* (0.05 N)—Prepare and standardize a 0.05 N silver nitrate (AgNO₃) solution.

6.1.14 *Sodium Ammonium Phosphate*—(NaNH₄HPO₄).

6.1.15 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H₂SO₄).

6.1.16 *Sulfuric Acid* (1 + 6)—Carefully mix 1 volume of H₂SO₄ (sp gr 1.84) with 6 volumes of water.

6.1.17 *Nitric Acid* (0.1 N)—Mix 1.4 mL of HNO₃ (sp gr 1.42) with 200 mL of water.

6.1.18 *Phenolphthalein Indicator Solution*—Dissolve 0.25 g of phenolphthalein in 30 mL of methanol and dilute to 50 mL with water.

6.1.19 *Sodium Hydroxide Solution* (0.1 N)—Dissolve 1 g of sodium hydroxide (NaOH) in 250 mL of water.

6.1.20 *Water*—Reagent water shall be in accordance with Specification D 1193, type II. Specification D 1193 gives the following values for type II grade water.

Electrical conductivity, max, μ S/cm at 298 K (25-C)	1.0
Electrical resistivity, min, M Ω -cm at 298 K (25-C)	1.0
Total organic carbon (TOC), max, μ g/L	50.0
Sodium, max, μ g/L	5.0
Chlorides max, μ g/L	5.0
Total silica, max, μ g/L	5.0

7. Free Water

7.1 *Significance and Use*—The free water analysis determines the amount of free water contained in the sample as opposed to chemically combined water, and prepares the sample for further analysis.

7.2 Procedure:

7.2.1 Weigh a sample of the material as received or not less than 50 g to a precision of 0.001 g and spread it out in a thin layer in a suitable vessel. Place in an oven and dry at $45 \pm 3^\circ\text{C}$ for 2 h, then cool in a desiccator and weigh again. The loss of weight corresponds to the free water.

7.2.2 Retain the sample in a sealed container or in the desiccator for further analysis.

7.3 *Calculation and Report*—Calculate and report loss in weight as a percentage of the sample as received or of the dried sample as required.

7.4 *Precision and Bias*—Neither the precision nor the bias for the free water analysis has been determined.

8. Combined Water

8.1 *Significance and Use*—The combined water analysis determines the percent of chemically combined water and is used to calculate the purity of gypsum or the amount of gypsum or gypsum plaster in gypsum products.

8.2 *Interferences*—Some materials, such as organic and hydrated compounds that decompose within the same temperature range as gypsum, will cause high results. When the maximum temperature is exceeded, some carbonates undergo decomposition, which will result in high results.

8.3 Procedure:

8.3.1 For each sample, place three weighing bottles with lids in the preheated calcining oven or furnace and heat for 2 h at 215 to 230°C. Place in the desiccator and allow to cool to room temperature. Weigh the bottles and lids to the nearest 0.0001 g and record the tare weights.

8.3.2 Weigh out three specimens of approximately 1 g each of the sample as prepared in Section 4 and dried in Section 7 to a precision of 0.0001 g in the previously tared weighing bottles and record the total weight with lids.

8.3.3 Place the specimens in the calcining oven with the lids placed loosely on each bottle or crucible for 2 h or until constant weight has been obtained.

8.3.4 Place the lids tightly on the weighing bottles, remove from the oven, and place in the desiccator to cool to room temperature.

8.3.5 Weigh each specimen to a precision of 0.0001 g and record the weights.

8.3.6 Retain the residues for carbon dioxide analysis.

8.4 *Calculation and Report*—Calculate and report the average loss in weight of the three specimens as a percentage of the sample as received or of the dried sample, as required, to the nearest 0.001 g and record the tare weights.

8.5 *Precision and Bias*—Neither the precision nor the bias for the combined water analysis has been determined.

9. Carbon Dioxide

9.1 *Summary of Test Method*—The sample is decomposed with HCl and the liberated CO₂ is passed through a series of scrubbers to remove water and sulfides. The CO₂ is absorbed with Ascarite, a special sodium hydroxide absorbent,⁹ and the gain in weight of the absorption tube is determined and calculated as percent CO₂.

9.2 *Significance and Use*—The carbon dioxide analysis is useful in estimating carbonates and organic carbon for chemical balance.

9.3 Special Reagents:

9.3.1 *Magnesium Perchlorate Desiccant*¹⁰—for drying.

9.3.2 *Sodium Hydroxide Absorbent*⁹—a coarse sodium hydroxide coated silica.

9.4 *Special Apparatus*—The apparatus illustrated in Fig. 1 consists of the following:

9.4.1 *Purifying Jar A*, Fleming, containing sulfuric acid.

9.4.2 *Drying Tube B*, U-shaped with side arms and glass-stoppers. Side arms are shaped to hold rubber tubing. Contains Anhydron on left side and Ascarite on right side.

9.4.3 *Erlenmeyer Flask C*, 250 mL, 24/20 ground-glass joint.

⁹ Ascarite, manufactured by Arthur H. Thomas has been found satisfactory for this purpose.

¹⁰ Anhydron, manufactured by J. T. Baker Inc. has been found satisfactory for this purpose.

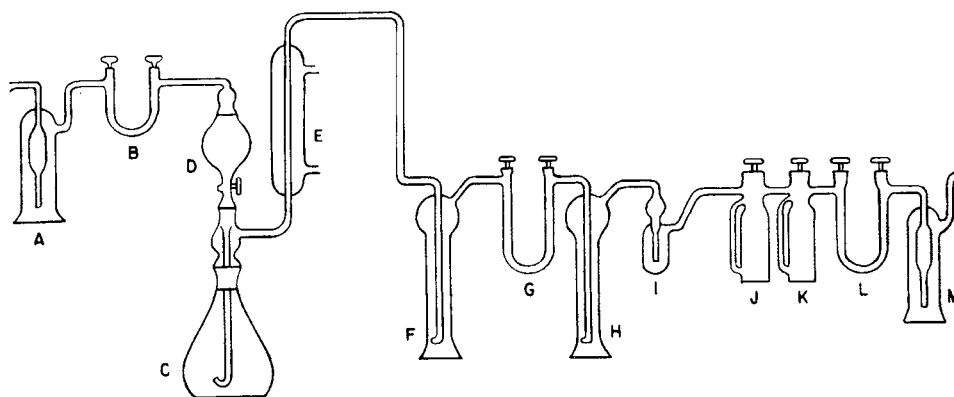


FIG. 1 Apparatus for Carbon Dioxide Analysis

9.4.4 *Separatory Funnel D*, with ground glass stopper and interchangeable hollow ground-glass joint. A delivery tube bent at the end extends into the sample flask approximately 15 mm from the bottom and is used to introduce acid into flask.

9.4.5 *Condenser E*.

9.4.6 *Gas-Washing Bottle F*, 250 mL, with fitted disk containing distilled water to retain most of the acid volatilized from the alkalimeter.

9.4.7 *U-Tube G*, containing mossy zinc to remove the last traces of HCl.

9.4.8 *Gas Washing Bottle H*, 250 mL, with fritted disk, containing concentrated H_2SO_4 and trap *I*, to remove any SO_3 mist that is carried over.

9.4.9 *Absorption Bulb J*, containing Anhydron to remove last traces of water vapor.

9.4.10 *CO₂ Absorption Bulb*, containing Ascarite filled as follows: On the bottom of the bulb, place a layer of glass wool extending above the bottom outlet and on top of this a layer of Anhydron approximately 10 mm thick; immediately above this place another layer of glass wool, then add Ascarite to almost fill the bulb. Place a top layer of Anhydron approximately 10 mm thick on top of the Ascarite and top it off with a covering of glass wool.

9.4.11 *U-Guard Tube L*, filled with Anhydron in left side and Ascarite in right side.

9.4.12 *Purifying Jar M*, Fleming, containing H_2SO_4 .

9.5 Procedure:

9.5.1 After drying as described in Section 8 place the residue obtained in the 250 mL Erlenmeyer flask (C). Connect the flask to the apparatus as shown in Fig. 1. Purge the system free of carbon dioxide by passing a current of CO_2 -free air through the apparatus for 10 to 15 min.

9.5.2 Weigh the absorption bulb to 0.0001 g and attach it to the train. Remove the glass stopper from the separatory funnel, place 50 mL of dilute HCl (1 + 1) in the separatory funnel (D) and replace the stopper with the interchangeable hollow ground-glass joint through which passes a tube for admitting purified air. Open the stopcock of the separatory funnel and admit air through the top of the funnel to force the hydrochloric acid into the Erlenmeyer flask (C).

9.5.3 Start cold water circulating through the condenser (E) and, with CO_2 -free air passing at a moderate rate through the absorption train, place a small hot plate or gas burner under the

sample flask and boil for approximately 2 min. Remove the hot plate and continue the flow of purified air at approximately three bubbles per second for 10 min to sweep the apparatus free of CO_2 . Close the absorption bulb, disconnect it from the train and weigh, opening the stopper momentarily to equalize the pressure. Use a second absorption bulb as counterpoise in all weighings unless a single pan balance is used.

9.6 *Calculation*—Calculate the percent CO_2 to the dried sample as follows:

$$\text{Percent CO}_2 = ((A - B)/C \times 100)(1 - D) \quad (1)$$

where:

A = mass of absorption bulb + CO_2 g,

B = mass of absorption bulb before the run, g,

C = mass of specimen, g, and

D = percent combined water as determined in Section 8 as a decimal.

Calculate the percent CO_2 to the sample as received as follows:

$$\text{Percent CO}_2 = E(1 - F) \quad (2)$$

where:

E = result of Eq 1, and

F = percent free water as determined in Section 7 as a decimal.

9.7 *Precision and Bias*—Neither the precision nor the bias for the carbon dioxide analysis has been determined.

10. Silicon Dioxide and Other Acid Insoluble Matter

10.1 *Summary of Test Method*—The gypsum and other acid soluble components of the sample are dissolved in dilute hydrochloric acid (HCl). The residue is weighed and calculated as silicon dioxide and other acid insoluble matter.

10.2 *Significance and Use*—The silicon dioxide and other acid insoluble matter analysis determines and is used to report the percentage of one of the inert impurities in gypsum and gypsum products.

10.3 *Procedure*—Perform in triplicate.

10.3.1 Weigh approximately 1 g of the specimen prepared in Section 4 to the nearest 0.0001 g.

10.3.2 Place the specimen in a porcelain casserole. Add approximately 50 mL of 1 + 5 hydrochloric acid. Evaporate slowly and carefully to apparent dryness on a hot plate. Take

not less than 20 min to do the evaporation. Make a blank determination with one casserole in parallel. Cool to room temperature.

10.3.3 Add enough hydrochloric acid (sp gr 1.19) to wet the solid residue. Add 20 mL of water, boil and filter through filter paper.⁷ Wash the filter paper thoroughly using not less than 100 mL of room temperature water to render the precipitate chloride free. The most effective washing technique is to use many small quantities of wash water rather than fill the funnel to the brim two or three times. Test the filtrate for chloride by collecting a small amount and adding a few drops of 0.1 normal silver nitrate (AgNO_3) solution. A white precipitate indicates more washing is needed. Discard this test solution.

10.3.4 Place all the filtrate back in the same casserole. Evaporate to dryness and heat to 120°C for 1 h and cool. To the cooled casserole add enough HCl (sp gr 1.19) to wet the solid residue. Add 50 mL of water and boil.

10.3.5 Wash the second contents of the casserole through another filter⁷ paper. Thoroughly wash the residue in the filter paper until chloride free as in 10.3.3. Retain the filtrate for the iron and aluminum oxide analysis.

10.3.6 Dry sufficient crucibles by placing in a cold muffle furnace during warm up or by placing in a drying oven for 15 to 20 min, then placing in a 900°C muffle furnace. Cool crucibles to room temperature in a desiccator.

10.3.7 Transfer both filter papers to a tared crucible and char slowly without flaming. Burn off all the carbon and ignite in a muffle furnace at 900°C for 15 min.

10.3.8 Cool the crucibles in a desiccator and weigh to the nearest 0.0001 g.

10.4 *Calculation and Report*—Calculate the average weight of the three precipitates and report as silicon dioxide (SiO_2) and other insoluble matter to the percentage of sample as received or to the dried sample as required.

10.5 *Precision and Bias*—Neither the precision nor the bias for the silicon dioxide and other acid insoluble matter has been determined.

11. Iron and Aluminum Oxides

11.1 *Significance and Use*—The iron and aluminum oxides ($\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$) analysis is used to determine the quantity of these metal oxides in gypsum or gypsum products.

11.2 *Procedure*—To the filtrate, obtained as described in Section 10, add a few drops of nitric acid (HNO_3), and boil to ensure oxidation of the iron. Add 2 g of ammonium chloride (NH_4Cl) previously dissolved in water. Make alkaline with ammonium hydroxide (NH_4OH). Digest hot for a few minutes until the precipitate coagulates. Filter, wash, ignite the precipitate at 1000°C for 30 min or to constant weight in a muffle furnace and weigh as $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$. Save the filtrate for the CaO analysis.

NOTE 2—The addition of a pinch of ashless filter paper pulp will aid in the filtration of the precipitate.

11.3 *Calculation*—Calculate $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$ to the percentage of sample as received or the dried sample as required. This precipitate may be further treated to separate the two oxides, but this is generally unnecessary.

11.4 *Precision and Bias*—Neither the precision nor the bias for the iron and aluminum oxides analysis has been determined.

12. Calcium Oxide

12.1 *Significance and Use*—The calcium oxide (CaO) analysis is used to determine the amount of CaO and calculate the amount of calcium carbonate (CaCO_3) in gypsum and gypsum products.

12.2 *Procedure*:

12.2.1 To the filtrate obtained as described in Section 11 add 5 g of ammonium oxalate ($(\text{NH}_4)_2\text{C}_2\text{O}_4$) dissolved in water. Digest hot for 30 min, making sure that the solution is always alkaline with NH_4OH . Filter, wash, and ignite the precipitate at 1000°C for 2 h to constant weight in a platinum crucible in a muffle furnace.

12.2.2 *Alternative Method*—To the filtrate obtained as described in Section 11, add 5 g of $(\text{NH}_4)_2\text{C}_2\text{O}_4$ dissolved in water. Digest hot for 30 min, making sure that the solution is always alkaline with NH_4OH . Filter and wash, transfer the precipitate to a beaker, and wash the filter paper with hot $\text{H}_2\text{SO}_4(1 + 6)$, catching the washing in the same beaker. Heat gently to complete solution, adding more H_2SO_4 if necessary. While still warm, titrate with potassium permanganate (KMnO_4) solution (5.6339 g/L) until the pink color persists.

12.3 *Calculation*—The number of milliliters of KMnO_4 solution used gives directly the percentage of lime in the dried sample. Calculate the CaO to the percentage of sample as received or the dried sample as required.

12.4 *Precision and Bias*—Neither the precision nor the bias for the calcium oxide analysis has been determined.

13. Magnesium Oxide

13.1 *Significance and Use*—The magnesium oxide (MgO) analysis is used to determine the amount of MgO and calculate the amount of magnesium carbonate MgCO_3 in gypsum and gypsum products.

13.2 *Procedure*—To the filtrate obtained as described in 12.2.1 or 12.2.2, add enough water to give a total volume of approximately 600 mL. Cool, and add 10 mL of NH_4OH and 5 g of sodium ammonium phosphate ($\text{NaNH}_4\text{HPO}_4$) dissolved in water. Stir vigorously until a precipitate begins to form. Let stand overnight. Filter, using a Gooch crucible, and wash with NH_4NO_3 solution. Ignite at 1000°C for 2 h to constant weight in a muffle furnace.

13.3 *Calculation*—Multiply this weight by 0.36207 to find the weight of magnesium oxide (MgO). Calculate the MgO to the percentage of sample as received or to the dried sample as required.

13.4 *Precision and Bias*—Neither the precision nor the bias for the magnesium oxide analysis has been determined.

14. Sulfur Trioxide

14.1 *Summary of Test Method*—In this test method, sulfate is precipitated from an acid solution of the gypsum with barium chloride (BaCl_2). The precipitate is filtered and weighed as barium sulfate (BaSO_4) and the sulfur trioxide (SO_3) equivalent is calculated.

14.2 *Significance and Use*—The specification for gypsum and some gypsum products specifies the amount of calcium sulfate (CaSO_4) required, either in the dihydrate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) or hemihydrate ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$) form. This procedure assumes that an insignificant amount of sulfate other than calcium sulfate is present. This test method is used to determine compliance to the gypsum and gypsum product specifications. It is also commonly used in quality control work.

14.3 *Interference*—This test method has been developed for natural gypsum and for impurities generally found associated with natural gypsum. Synthetic gypsum will sometimes have an additional number of interfering elements and compounds, and if so, this procedure will not give accurate results. This test method has a number of interferences that theoretically affect the results. Co-precipitation and occlusion are problems if the solution is either too acidic or too basic. Calculations using SO_3 analysis are most accurate on samples that are known to be completely hydrated or completely dehydrated.

14.4 *Procedure*:

14.4.1 Having properly selected and prepared the samples as specified in Section 4, weigh a representative specimen of approximately 0.5 g, to the nearest 0.0001 g.

14.4.2 Place the weighed sample into a 400-mL beaker. Add 50 mL of HCl (1 + 5). Boil and disperse with the flattened end of a glass rod while stirring until the sample is completely broken down. Add approximately 100 mL boiling water and continue boiling for 15 min, with this step to be extended as required, so the combined boiling time is not less than 1 h.

14.4.3 Using filter paper⁷, filter into a clean 600-mL flask and rinse the 400-mL beaker thoroughly with hot distilled water. Carefully wash the sides of the 400-mL beaker while wiping the insides with a rubber-tipped glass rod making sure all splatters and insoluble are washed into the filter paper. Dry and burn off the filter paper leaving the residue to be dried and weighed for insoluble matter, if this test method is not otherwise conducted.

14.4.4 Dilute the filtrate to 400 to 500 mL. Add 1 to 2 drops of 0.1 % methyl red indicator. Prepare a 400 to 500-mL sample of 0.05 to 0.1 NHCl. Add 1 to 2 drops of 0.1 % methyl red indicator. Compare the color of this solution to the color of the filtrate. Dilute the filtrate or add HCl (1 + 5) solution as necessary to match the pH of the 0.05 to 0.1 NHCl solution.

14.4.5 Boil the filtrate solution and add 20 mL of near-boiling 10 % barium chloride solution, preferably with the help of a pipette, drop by drop while stirring. The barium chloride solution should be prepared not less than one day before use. Continue boiling the solution for 10 to 15 min and digest hot for 3 h or until the precipitate settles.

14.4.6 Filter⁷ and wash with approximately 125 to 150 mL of hot water to render the precipitate chloride free. Test the filtrate for chloride by collecting a small amount and adding a few drops of 0.1 N AgNO_3 solution. A white precipitate indicates more washing is needed. Alternately, use filtering crucibles¹¹ for quick filtering if the particular crucibles to be

used are tested prior to use by refiltering the filtrate from the crucibles with filter paper, and no more than 2 mg is collected on the filter paper.

14.4.7 Ignite the precipitate and paper in a tared crucible, and slowly char the paper without inflaming. Burn off all the carbon and ignite in a muffle furnace at 800 to 900°C or using bright red heat over a Bunsen burner for 15 to 20 min. Dry the filtering crucibles by placing in a cold muffle furnace during warm-up or in a drying oven prior to igniting in a muffle furnace at 800 to 900°C for 15 to 20 min.

NOTE 3—Thoroughly cleans crucibles before each use and heat in a furnace at 800 to 900°C and cool in a desiccator before taring.

14.4.8 Cool all crucibles in a desiccator and weigh to the nearest 0.0001 g.

14.5 *Calculation*—Multiply the weight of the precipitate by 0.343 to determine the weight of sulfur trioxide (SO_3). Calculate the SO_3 to the percentage of sample as received or to the dried sample as required.

14.6 *Precision and Bias*—Neither the precision nor the bias for the sulfur trioxide analysis has been determined.

15. Chlorides

15.1 *Significance and Use*—Small amounts of chlorides in gypsum or gypsum products often have a detrimental effect on their use. This procedure is used to measure the amount of chlorides present and report it as sodium chloride.

15.2 *Procedure*:

15.2.1 Weigh approximately 20.0 g of sample as prepared in Section 4 to 0.001 g and transfer to a 400-mL beaker. Add 150 mL of water, stir, and heat to just below the boiling point. Cover with a watch glass and maintain at just below boiling (not less than 80°C) for 1 h with occasional stirring. Filter with suction on a Buchner funnel fitted with a medium filter paper. Wash the residue with four 20-mL portions of hot water.

15.2.2 Add 2 drops of phenolphthalein indicator solution to the filtrate. If the filtrate fails to turn pink, add 0.1 N NaOH solution dropwise with stirring until a faint pink color develops. Add 0.1 NHNO_3 dropwise until the pink color just disappears.

15.2.3 If the chloride content is very low, transfer the entire filtrate quantitatively to a 400-mL beaker and proceed as described in 15.2.4. If larger amounts of chloride are expected, transfer the filtrate quantitatively to a 250-mL volumetric flask, cool to room temperature, and dilute to 250 mL. Take a suitable aliquot, transfer to a 400-mL beaker, and dilute to a volume of 100 to 250 mL.

15.2.4 Place the beaker containing the sample on a white surface, add 0.5 mL (10 drops) of K_2CrO_4 solution and titrate with AgNO_3 solution using a micro buret having a 10-mL capacity and graduated in divisions of 0.02 mL. Titrate until a faint but definite orange color is visible.

15.2.5 Perform a blank titration using the same volume of water as the sample volume and the same amount of K_2CrO_4 solution. Titrate to the same color as obtained with the sample.

15.3 *Calculation*—Subtract the volume of AgNO_3 solution used for the blank titration from the volume used for the sample to give the net titration. A 1-mL net titration is

¹¹ Gooch or Coors filtering crucibles have been found suitable for this purpose.