Designation: ~~C471M – 17~~ C471M – 17$^{\varepsilon 1}$

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

This standard is issued under the fixed designation C471M; 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.

ε^1 NOTE—Corrected equation reference in 9.6 and restored item *(6)* to list in 16.1 editorially in August 2017.

1. Scope*

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

1.2 The test methods appear in the following order:

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~~ safety, health and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C11 Terminology Relating to Gypsum and Related Building Materials and Systems
C22/C22M Specification for Gypsum
C28/C28M Specification for Gypsum Plasters
C59 Specification for Gypsum Casting Plaster and Gypsum Molding Plaster
C61 Specification for Gypsum Keene's Cement
C317/C317M Specification for Gypsum Concrete
C778 Specification for Standard Sand
C842 Specification for Application of Interior Gypsum Plaster
D1193 Specification for Reagent Water
D1428 Test Method for Test for Sodium and Potassium In Water and Water-Formed Deposits by Flame Photometry (Withdrawn 1989)[3]
D2013 Practice for Preparing Coal Samples for Analysis
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—Definitions shall be in accordance with Terminology C11.

3.2 *Definitions:*

3.2.1 *calibration standard, n*—a chemical mixture containing a known quantity of the analyte used to relate the measured analytical signal to the concentration of the analyte.

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

3.2.3 *internal standard, n*—a chemical used in the quantification of S_8 by monitoring and adjusting for minor variances in instrument performance.

3.2.4 *riffle, n*—a hand feed sample divider device that divides the sample into parts of approximately the same weight. (**D2013**)

3.2.5 *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.

3.2.6 *surrogate standard, n*—a chemical used to account for extraction efficiency of S_8.

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.

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 $CaSO_4 \cdot 2H_2O$ 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 ± 3°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* (Specification C22/C22M)—Gypsum samples will be received in the form of rocks or 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,* (Specification C28/C28M).

4.3.1 *Gypsum Ready-Mixed Plaster or Gypsum WoodFibered Plaster*—Screen the dried sample through a 150-µm (No. 100) sieve[4] 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

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Detailed requirements for this sieve are given in Specification E11.

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*, (Specification C59)—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*, (Specification C61)—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*, (Specification C317/C317M)—Screen the dried sample through a 150-µm (No. 100) sieve[4] 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 Panel Products*—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.

COMPLETE PROCEDURE

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 ± 3°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.*

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.[5] 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* ($(NH_4)_2C_2O_4$).

6.1.5 *Barium Chloride* (100 g/L)—Dissolve 100 g of barium chloride ($BaCl_2 \cdot 2H_2O$) 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 one volume of HCl (sp gr 1.19) with four volumes of water.

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

6.1.10 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

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[5] *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.

6.1.11 *Potassium Chromate Solution* (100 g/L)—Dissolve 5 g of potassium chromate (K_2CrO_4) in 50 mL of water, mix, add ten drops of 0.05 N silver nitrate ($AgNO_3$) 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 ($KMnO_4$) 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 ($AgNO_3$) solution.

6.1.14 *Sodium Ammonium Phosphate*—($NaNH_4HPO_4$).

6.1.15 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

6.1.16 *Sulfuric Acid* (1 + 6)—Carefully mix one volume of H_2SO_4 (sp gr 1.84) with six volumes of water.

6.1.17 *Nitric Acid* (0.1 N)—Mix 1.4 mL of HNO_3 (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 D1193, type II. Specification D1193 gives the following values for type II grade water.

~~Electrical conductivity, max, μS/cm at 298 K (25-C)~~	~~1.0~~
~~Electrical resistivity, min, MΩ·cm at 298 K (25-C)~~	~~1.0~~
~~Total organic carbon (TOC), max, μg/L~~	~~50.0~~
~~Sodium, max, μg/L~~	~~5.0~~
~~Chlorides max, μg/L~~	~~5.0~~
~~Total silica, max, μg/L~~	~~5.0~~

Electrical conductivity, max, μS/cm at 298 K (25-C)	1.0
Electrical resistivity, min, M·cm at 298 K (25-C)	1.0
Total organic carbon (TOC), max, μg/L	50.0
Sodium, max, μg/L	50.0 5.0
Chlorides max, μg/L	50.0 5.0
Total silica, max, μg/L	50.0 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 of 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 ± 3°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:*

7.4.1 The precision of this test method is based on an interlaboratory study of ASTM C471M, Standard Test Methods for Chemical Analysis of Gypsum and Gypsum Products, conducted in 2016. Each of 17 laboratories analyzed two different gypsum sample types. Every "test result" represents an individual determination, and all participants reported five test results per material. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. C11-1003[6]

7.4.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

(1) Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

(2) Repeatability limits are listed in Table 1.

7.4.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

(1) Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

(2) Reproducibility limits are listed in Table 1.

7.4.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C11-1003. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Free Water (in Accordance with Section 7)

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	S_r	S_R	r	R
natural gypsum	0.0482	0.0102	0.0217	0.0286	0.0607
flue gas desulphogypsum (FGD)	8.1699	0.7639	1.9213	2.1388	5.3797

[A] The average of the laboratories' calculated averages.

7.4.1.4 Any judgment in accordance with statements 7.4.1.1 and 7.4.1.2 would have an approximate 95 % probability of being correct.

7.4.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

7.4.3 The precision statement was determined through statistical examination of 660 results, from 15 participating laboratories, on two types of gypsum materials.

7.4.4 To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

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 4and 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:*

8.5.1 The precision of this test method is based on an interlaboratory study of ASTM C471M, Standard Test Methods for Chemical Analysis of Gypsum and Gypsum Products, conducted in 2016. Each of 17 laboratories analyzed two different gypsum sample types. Every "test result" represents an individual determination, and all participants reported five test results per material. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. C11-1003.[6]

8.5.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

(1) Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

(2) Repeatability limits are listed in Table 2.

8.5.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

(1) Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

(2) Reproducibility limits are listed in Table 2.

8.5.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

TABLE 2 Combined Water in Accordance with Section 8

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	S_r	S_R	r	R
natural gypsum	18.7931	0.4718	1.4501	1.3211	4.0604
flue gas desulphogypsum (FGD)	19.3946	0.5518	1.3044	1.5449	3.6523

[A] The average of the laboratories' calculated averages.

8.5.1.4 Any judgment in accordance with statements 8.5.1.1 and 8.5.1.2 would have an approximate 95 % probability of being correct.

8.5.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

8.5.3 The precision statement was determined through statistical examination of 660 results, from 15 participating laboratories, on two types of gypsum materials.

8.5.4 To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

9. Carbon Dioxide

9.1 *Summary of Test Method*—The sample is decomposed with HCl and the liberated CO_2 is passed through a series of scrubbers to remove water and sulfides. The CO_2 is absorbed with Ascarite, a special sodium hydroxide absorbent, and the gain in weight of the absorption tube is determined and calculated as percent CO_2.

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*—For drying.

9.3.2 *Sodium Hydroxide Absorbent*—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 Anhydrone on left side and Ascarite on right side.

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

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 Anhydrone to remove last traces of water vapor.

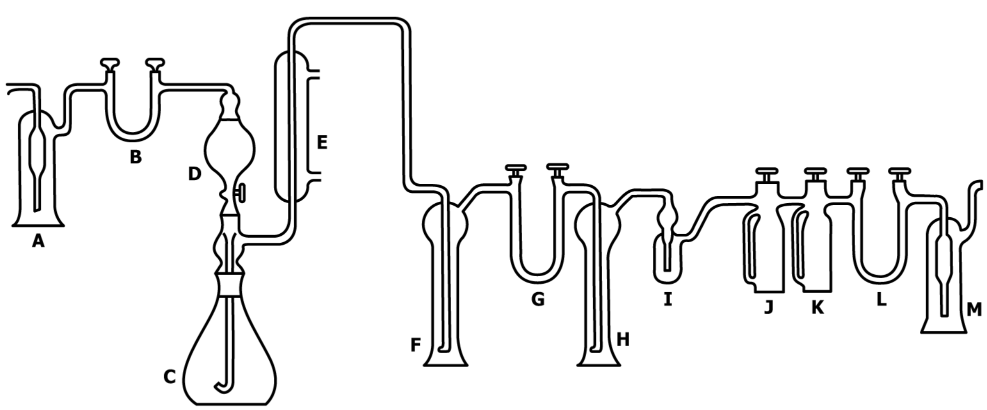

FIG. 1 Apparatus for Carbon Dioxide Analysis

9.4.10 *CO_2 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 Anhydrone 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 Anhydrone 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 Anhydrone 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 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:

$$\text{Percent } CO_2 = \left(\left(A - B\right)/C \times 100\right)\left(1 - D\right) \quad (1)$$

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:

$$\text{Percent } CO_2 = E\left(1 - F\right) \quad (2)$$

where:

~~E = result of Eq 3, and~~
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. 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 filter 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 ($Fe_2O_3 + Al_2O_3$) 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 $Fe_2O_3 + Al_2O_3$. Save the filtrate for the CaO analysis.

NOTE 1—The addition of a pinch of ashless filter paper pulp will aid in the filtration of the precipitate.

11.3 *Calculation*—Calculate $Fe_2O_3 + Al_2O_3$ 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 ($(NH_4)_2C_2O_4$) 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 $(NH_4)_2C_2O_4$ 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 H_2SO_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 ($NaNH_4HPO_4$) 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 ($CaSO_4 \cdot 2H_2O$) or hemihydrate ($CaSO_4 \cdot \frac{1}{2} H_2O$) 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 paper, 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 one to two drops of 0.1 % methyl red indicator. Prepare a 400 to 500-mL sample of 0.05 to 0.1 N HCl. Add one to two 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 N HCl 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 Filter 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 crucibles 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 2—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 two 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 N HNO_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 (ten 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 equivalent to 0.002923 g of sodium chloride (NaCl). Calculate the NaCl as a percentage of the sample as received or the dried sample as required.

15.4 *Precision and Bias*—Neither the precision nor the bias for the chloride analysis has been determined.

16. Report

16.1 Report the results obtained in the analysis as follows:

	%
Free water	...
Combined water	...
Carbon dioxide (CO_2)	...
Silicon dioxide (SiO_2) and insoluble matter	...
Iron and aluminum oxides (Fe_2O_3 + Al_2O_3)	...
Lime (CaO)	...
Magnesium oxide (MgO)	...
Sulfur trioxide (SO_3)	...
Sodium chloride (NaCl)	...
Total	100.00±

NOTE 3—Since it is frequently advisable to recalculate the results obtained in the chemical analysis in order that they may be more enlightening, the following is submitted for consideration:

~~(1) Multiply the percentage of combined water by 4.778 to obtain purity or percentage gypsum. To calculate the percentage of $CaSO_4 \cdot ½ H_2O$ in plasters, multiply the percentage of gypsum by 0.8430.~~

~~(2) Multiply the percentage of combined water by 2.222 to obtain the amount of SO_3 combined as gypsum.~~

~~(3) Subtract the result obtained in (2) from the total SO_3 found by analysis to obtain the excess SO_3.~~

~~(4) Multiply the excess SO_3 by 1.700 to obtain the percentage anhydrite, $CaSO_4$.~~

~~(5) Multiply the percentage of gypsum found in (1) by 0.3257 to obtain the percentage of CaO combined as gypsum.~~

~~(7) Add (5) and (6) together. Then subtract this result from the total CaO percentage found by analysis.~~

~~(8) Multiply the excess CaO percentage by 1.785 to obtain the percentage of calcium carbonate.~~

~~(9) Multiply the percentage of MgO by 2.091 to obtain the percentage of magnesium carbonate.~~

(*1*) Multiply the percentage of combined water by 4.778 to obtain purity or percentage gypsum. To calculate the percentage of $CaSO_4 \cdot ½ H_2O$ in plasters, multiply the percentage of gypsum by 0.8430.

(*2*) Multiply the percentage of combined water by 2.222 to obtain the amount of SO_3 combined as gypsum.

(*3*) Subtract the result obtained in (*2*) from the total SO_3 found by analysis to obtain the excess SO_3.

(*4*) Multiply the excess SO_3 by 1.700 to obtain the percentage anhydrite, $CaSO_4$.

(*5*) Multiply the percentage of gypsum found in (*1*) by 0.3257 to obtain the percentage of CaO combined as gypsum.

(*6*) Multiply the percentage of anhydrite found in (*4*) by 0.4119 to obtain the percentage of CaO combined as anhydrite.

(*7*) Add (*5*) and (*6*) together. Then subtract this result from the total CaO percentage found by analysis.

(*8*) Multiply the excess CaO percentage by 1.785 to obtain the percentage of calcium carbonate.

(*9*) Multiply the percentage of MgO by 2.091 to obtain the percentage of magnesium carbonate.

NOTE 4—Having made the calculations in Note 3, the results may be reported as follows:

	%
Gypsum ($CaSO_4 \cdot 2H_2O$)	...
Anhydrite ($CaSO_4$ natural and manufactured) (Note 3)	...
Silicon dioxide and insoluble (SiO_2 + Ins.)	...
Iron and aluminum oxide (R_2O_3)	...
Calcium carbonate ($CaCO_3$)	...
Magnesium carbonate ($MgCO_3$)	...
Magnesium oxide (MgO)	...
Sulfur trioxide (SO_3)	...
Sodium chloride (NaCl)	...
Total	100.00±

NOTE 5—The presence of the different forms of $CaSO_4$ may be determined by a microscopic examination. A paper titled "Gypsum Analysis with the Polarizing Microscope" containing suggested methods can be found in ASTM STP 861.[7]

17. Alternative Procedure for Analysis of Free Water in Gypsum Using Moisture Balance

17.1 *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.

17.2 *Equipment*—A programmable moisture balance, capable of temperature control of ±1°C to at least 200°C. The moisture balance must be capable of measuring a minimum of 0.01 % loss in weight and be able to bring the temperature of an empty tray from ambient conditions up to 200°C.

[7] Green, George W., "Gypsum Analysis with the Polarizing Microscope," *The Chemistry and Technology of Gypsum, ASTM STP 861*, ASTM, 1984, pp. 22–47.