



Designation: C471M – 20a^{ε1}



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.

^{ε1} NOTE—The title of Table 2 was editorially corrected in May 2021.

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 These test methods appear in the following order:

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1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in these test methods.

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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1.4 These text of this test method 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.

1.5 *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, health, and 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.*

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

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.

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:

3.1.1 For definitions of terms used in these test methods, refer to Terminology **C11**.

3.2 Definitions of Terms Specific to This Standard:

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 $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ 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 ± 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 Wood-fibered Plaster*—Screen the dried sample through a 150 μm (No. 100) sieve (see **Note 1**) 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.

NOTE 1—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 (see **Note 1**) 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.⁴ 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* (NH₄Cl).

6.1.2 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).

6.1.3 *Ammonium Nitrate* (25 g/L)—Dissolve 25 g of ammonium nitrate (NH₄NO₃) in water and dilute to 1 L.

6.1.4 *Ammonium Oxalate* ((NH₄)₂C₂O₄).

6.1.5 *Barium Chloride* (100 g/L)—Dissolve 100 g of barium chloride (BaCl₂·2H₂O) in water and dilute to 1 L.

6.1.6 *Calcium Chloride* (CaCl₂)—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 (HNO₃).

6.1.11 *Potassium Chromate Solution* (100 g/L)—Dissolve 5 g of potassium chromate (K₂CrO₄) in 50 mL of water, mix, add ten 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 one volume of H₂SO₄ (sp gr 1.84) with six 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 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

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 until constant mass has been obtained. The samples are to be cooled in a desiccator prior to each weighing. 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 Test Methods C471M in 2020. Each of ten volunteer 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. C11-2000.⁵

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.

⁴ *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.

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C11-2000. Contact ASTM Customer Service at service@astm.org.

(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.

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 278 results, from ten 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 5 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:

8.5.1 The precision of this test method is based on an interlaboratory study of Test Methods C471M in 2020. Each of ten volunteer 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. C11-2000.⁵

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

TABLE 1 Free Water (in Accordance With Section 7)

Material	Number of Laboratories	Average ^A	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	n	\bar{x}	s_r	s_R	r	R
Natural Gypsum	10	1.906	0.287	0.287	0.803	0.803
Flue Gas Desulphogypsum (FGD)	10	9.909	0.281	0.514	0.786	1.440

^A The average of the laboratories' calculated averages.

TABLE 2 Combined Water (in Accordance with Section 8)[†]

Material	Number of Laboratories	Average ^A	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	n	\bar{x}	s_r	s_R	r	R
Natural Gypsum	9	19.291	0.129	0.129	0.363	0.363
Flue Gas Desulphogypsum (FGD)	9	19.364	0.195	0.279	0.547	0.782

[†] Editorially corrected.

^A The average of the laboratories' calculated averages.

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.

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 278 results, from ten 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 CO₂ is passed through a series of scrubbers to remove water and sulfides. The CO₂ is absorbed with Ascarite, a special sodium hydroxide absorbent, 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—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 Anhydron 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 H₂SO₄ and trap I, to remove any SO₃ 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.

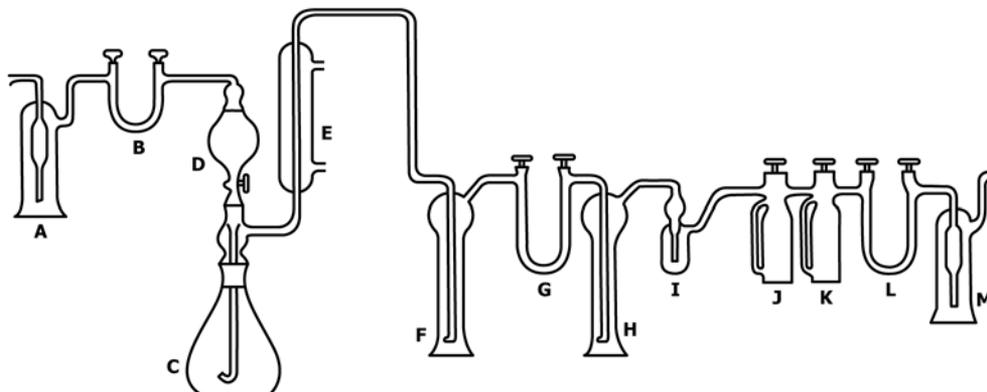


FIG. 1 Apparatus for Carbon Dioxide Analysis

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 H₂SO₄.

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 CO₂-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 CO₂-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 CO₂. 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 CO₂ to the dried sample as follows:

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

where:

- A = mass of absorption bulb + CO₂ 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 CO₂ to the sample as received as follows:

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

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. 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 (AgNO₃) 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 (SiO₂) 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 (Fe₂O₃ + Al₂O₃) 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 (HNO₃), and boil to ensure oxidation of the iron. Add 2 g of ammonium chloride (NH₄Cl) previously dissolved in water. Make alkaline with ammonium hydroxide (NH₄OH). 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 Fe₂O₃ + Al₂O₃. 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 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 ($\text{NaNH}_4\text{HPO}_4$) 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 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 .

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 AgNO₃ 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 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 (SO₃). Calculate the SO₃ 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 HNO₃ 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 K₂CrO₄ solution and titrate with AgNO₃ 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 K₂CrO₄ solution. Titrate to the same color as obtained with the sample.

15.3 *Calculation*—Subtract the volume of AgNO₃ 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 ₂ O ₃ + Al ₂ O ₃)	...
Lime (CaO)	...
Magnesium oxide (MgO)	...
Sulfur trioxide (SO ₃)	...
Sodium chloride (NaCl)	...
Total	100.00 ±

NOTE 4—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 CaSO₄ · ½ H₂O 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 SO₃ combined as gypsum.

(3) Subtract the result obtained in (2) from the total SO₃ found by analysis to obtain the excess SO₃.

(4) Multiply the excess SO₃ by 1.700 to obtain the percentage anhydrite, CaSO₄.

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

Gypsum (CaSO ₄ ·2H ₂ O)	%
Anhydrite (CaSO ₄ natural and manufactured) (Note 4)	...
Silicon dioxide and insoluble (SiO ₂ + Ins.)	...
Iron and aluminum oxide (R ₂ O ₃)	...
Calcium carbonate (CaCO ₃)	...
Magnesium carbonate (MgCO ₃)	...
Magnesium oxide (MgO)	...
Sulfur trioxide (SO ₃)	...
Sodium chloride (NaCl)	...
Total	100.00 ±

NOTE 6—The presence of the different forms of CaSO₄ 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 861.⁶

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.

17.2.1 *Equipment Setup*—Implement a test program for “free water” that takes a sample of 5 to 8 g from an initial temperature to 45 °C at the maximum rate of temperature rise and holds the temperature at 45 °C for up to two hours. The initial temperature shall be defined as a temperature of 20 to 30 °C.

17.3 *Procedure:*

17.3.1 Prior to beginning the test, both the moisture balance and sample temperature must be less than 30 °C. Note that some phases of gypsum are metastable in humidity and as such, samples should be stored to minimize changes due to environmental conditions.

17.3.2 Weigh and evenly distribute 5 to 8 g of a sample of the material as received in a clean tared pan in the moisture balance. Run the described free water test program until a constant mass is reached or two hours of time at 45 °C has elapsed. Constant mass is considered reached if the percent moisture change per minute is 0.01 % or less and 45 °C has been achieved.

17.4 *Calculation and Report*—Report the free water as the percentage loss in weight at the end of the test. This can be calculated by the following formula:

$$\% \text{ free water} = \text{mass of water evaporated} / \text{initial sample mass} \times 100 \tag{3}$$

17.5 *Precision and Bias:*

17.5.1 The precision of this test method is based on an interlaboratory study of Test Methods C471M 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. C11-1003.⁷

17.5.1.1 *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 3.

17.5.1.2 *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 3.

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

17.5.1.4 Any judgment in accordance with statements 17.5.1.1 and 17.5.1.2 would have an approximate 95 % probability of being correct.

17.5.2 *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.

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

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

⁷ 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 3 Free Water in Gypsum in Accordance With Moisture Balance Method (%)

Material	Average ^A	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	\bar{x}	S_r	S_R	r	R
natural gypsum	0.0524	0.0096	0.0361	0.0270	0.1009
flue gas desulphogypsum (FGD)	9.1226	0.2037	0.2911	0.5702	0.8151

^A The average of the laboratories' calculated averages.

18. Alternative Procedure for Analysis of Combined Water in Gypsum Using Moisture Balance

18.1 *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. Note that this test reports the combined water result on a dry basis and assumes that any sample measured has had any free water removed before testing. See Section 17 for a description and test for free water.

18.2 *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.

18.3 *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.

18.3.1 *Equipment Setup*—Implement a test program for “combined water” that takes a sample of 5 to 8 g from an initial temperature to a temperature of 200 °C at the maximum rate of temperature rise and holds the temperature for up to two hours. The initial temperature shall be defined as temperature of 20 to 45 °C.

18.4 *Procedure:*

18.4.1 Prior to beginning the test, both the moisture balance and sample temperature must be 45 °C or less. Note that some phases of gypsum are metastable in humidity and as such, samples should be stored to minimize changes due to environmental conditions.

18.4.2 Weigh and evenly distribute 5 to 8 g of a sample of the material as previously dried to remove free water in a clean tared pan. Run the described combined water test program until a constant mass is reached or two hours of time at 200 °C has elapsed. Constant mass is considered reached if the percent moisture change per minute is 0.01% or less and 200 °C has been achieved.

18.5 *Calculation and Report*—Report the combined water as the percentage loss in weight at the end of the test. This can be calculated by the following formula:

$$\% \text{ combined water} = \frac{\text{mass of water evaporated}}{\text{dry sample mass}} \times 100 \tag{4}$$

18.6 *Precision and Bias:*

18.6.1 The precision of this test method is based on an interlaboratory study of Test Methods C471M 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. C11-1003.⁷

18.6.1.1 *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 4.

18.6.1.2 *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 4.

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

18.6.1.4 Any judgment in accordance with statements 18.6.1.1 and 18.6.1.2 would have an approximate 95 % probability of being correct.

18.6.2 *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.

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

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

19. Alternative Procedure for Analysis of Organic Material and Carbon Dioxide in Gypsum by High Temperature Weight Loss

19.1 *Significance and Use*—As an alternative to Section 9 which specifically measures CO₂, high temperature weight loss

TABLE 4 Combined Water in Gypsum in Accordance With Moisture Balance Method (%)

Material	Average ^A	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	\bar{x}	S_r	S_R	r	R
natural gypsum	19.1313	0.0229	0.0813	0.0641	0.2278
flue gas desulphogypsum (FGD)	19.3761	0.0392	0.1859	0.1098	0.5206

^A The average of the laboratories' calculated averages.

can also be used to determine CO₂. Depending on the temperatures chosen for exposure, measured weight loss is due to a release of free water, combined water, volatile pyrolytic products of organic material and CO₂ that may be present in the sample.

19.2 *Summary of Test Method*—Samples measured by this test method must have known combined water (Section 8 or 18) composition. Dry specimens of known weight are heated at 500 °C for 30 minutes while replicate specimens are heated at 1000 °C for 30 minutes. The organic material composition is determined from the weight loss of the 500 °C heated specimens while the CO₂ composition is determined from the weight loss of both the 500 °C and 1000 °C heated specimens.

19.3 *Equipment:*

19.3.1 *Muffle Furnace*—Box furnace with PID control, capable of temperature control of ±20 °C up to and including 1000 °C.

19.4 *Sample Preparation*—Dry 25 g of the as-received sample at 45 °C until constant mass is reached. Sieve the dried sample through a 100 mesh (149 μm) U.S. standard sieve. If needed, the sample should be finely ground further to achieve this particle size.

19.4.1 Label all crucibles with high temperature resistant markings for individual identification.

19.4.2 Pre-fire empty porcelain or platinum crucibles with lids in a muffle furnace at 1000 °C for 30 minutes. Allow the oven to cool to 300 °C before removing the crucibles to store in a desiccator before use.

19.5 *Procedure*—Two temperature firings are performed, each in triplicate for a total of six specimens per sample.

19.5.1 Weigh an individually identified empty porcelain or platinum crucible and lid.

19.5.2 Add approximately 1 g of the prepared sample to the crucible and place lid. Weigh and record to the nearest 0.001 g.

19.5.3 Repeat for all six specimens.

19.6 *500 °C Weight Loss:*

19.6.1 Program the muffle furnace to ramp up to 500 °C at maximum rate and hold for 30 minutes. Place a set of three crucibles inside the muffle furnace and run the program.

19.6.2 Turn off the muffle furnace and allow to cool to 300 °C.

19.6.3 Remove three crucibles, place in a desiccator and allow them to cool to room temperature

19.7 *1000 °C Weight Loss:*

19.7.1 Program the muffle furnace to ramp up to 1000 °C at maximum rate and hold for 30 minutes. Run the program for the remaining three crucibles.

19.7.2 Turn off the muffle furnace and allow to cool to 300 °C.

19.7.3 Remove three crucibles, place in a desiccator and allow them to cool to room temperature

19.8 Weigh all six crucibles with the lids and record to the nearest 0.001 g.

19.9 *Calculations:*

19.9.1 Calculate weight loss for each crucible at 500 or 1000 °C as follows:

$$\% \text{ weight loss} = [(B - A) - (C - A)] / (B - A) \times 100$$

where:

A = mass of empty crucible and lid, g,

B = mass of crucible and lid + dry sample, g, and

C = mass of crucible and lid + dry sample after high temperature exposure, g.

19.9.2 Calculate organic material as follows:

$$\begin{aligned} \% \text{ organic material} &= \% 500 \text{ °C weight loss} \\ &- \% \text{ combined water (Section 8 or 18)} \end{aligned}$$

19.9.3 Calculate CO₂ as follows:

$$\begin{aligned} \% \text{ CO}_2 &= \% 1000 \text{ °C weight loss} - \% \text{ organic material} \\ &- \% \text{ combined water (Section 8 or 18)} \end{aligned}$$

19.10 *Calculation and Report*—Calculate and report the average organic material and CO₂ of the three specimens.

19.11 *Precision and Bias*—Neither the precision nor the bias for this method has been determined.

20. **Alternative Procedure for Analysis for Calcium Sulfate by Ammonium Acetate Method⁸**

20.1 *Significance and Use*—This test method covers the determination of calcium sulfate in gypsum and gypsum products by extraction with ammonium acetate solution.

20.2 *Reagents and Materials:*

20.2.1 *Ammonium Acetate Solution*—Dissolve 454 g of ammonium acetate in 2 L of water. Add sufficient NH₄OH to make the solution distinctly ammoniacal, using phenolphthalein as the indicator.

20.2.2 *Ammonium Hydroxide Wash Solution*—Dilute 100 mL of concentrated ammonium hydroxide (NH₄OH, sp gr 0.90) to 1 L with water.

20.2.3 *Filter Aid*—Diatomaceous silica, analytical grade.

20.2.4 *Phenolphthalein Indicator Solution.*

20.3 *Procedure Using Gooch Crucible:*

20.3.1 Weigh rapidly approximately 4 g of the well-mixed sample and transfer to a 600 mL beaker. Make all weighings to 0.001 g, except weigh the crucibles and their contents to 0.0001 g.

20.3.2 Without delay, weigh approximately 1 g of the well-mixed sample in a tared weighing bottle having a ground-glass stopper. Dry the sample and weighing bottle to constant weight at 45 °C. Stopper weighing bottles immediately upon removal from the oven in order to prevent absorption of moisture from the air upon cooling.

20.3.3 If the percentage by weight of combined water held by the calcium sulfate is required, heat the sample and weighing bottle to constant weight at 220 °C.

20.3.4 To the contents of the 600 mL beaker (20.3.1), add 350 mL of the ammonium acetate solution, and stir the mixture thoroughly to loosen all of the solid matter from the bottom of the beaker. Add 0.2000 g of redried diatomaceous silica to the mixture. Heat the beaker and contents to 70 °C on a steam or

⁸ This procedure was developed by L. S. Wells and W. F. Clarke, National Bureau of Standards, and modified by B. E. Kester, United States Gypsum Co.

hot water bath, and maintain at that temperature for 30 min, while stirring frequently. During heating, keep the solvent ammoniacal by additions of NH₄OH and phenolphthalein, if indicated. Meanwhile, heat a supply of the ammonium acetate solution to 70 °C, keeping it also distinctly ammoniacal. Filter the mixture, with suction, through a tared Gooch crucible, stirring frequently during filtration to keep the diatomaceous earth suspended in the liquid. Wash the Gooch crucible containing the residue with five 10 mL portions of the warm acetate solution, draining thoroughly after each washing. Wash in the same manner with eight 10 mL portions of the NH₄OH wash solution. Take care to wash the upper walls of the Gooch crucible. Drain the crucible dry with suction, place in an oven at 70 °C, and dry to constant weight (Note 7). Allow the crucible to cool in a desiccator before weighing.

NOTE 7—Avoid overheating in all oven drying of ammonium acetate residues; that is, place crucibles well away from the heating elements. This is of particular importance for samples high in impurities, as these impurities often have water of hydration that is lost on local overheating.

20.4 Procedure Using Tared Filter Papers:

NOTE 8—This procedure is suggested where several samples are to be analyzed at once. It has been found that gravity filtration on six samples will proceed as rapidly as it is possible to handle the samples.

20.4.1 Dry a quarter-folded, 110 mm quantitative filter paper overnight at 70 °C in a wideform, glass-stoppered, 30 by 60 mm weighing bottle. After drying, cool the weighing bottle and paper in a desiccator, and weigh.

20.4.2 Treat the sample exactly as described in 20.3.1, 20.3.2, and 20.3.4 prior to the filtration. Filter the mixture by gravity through a 70 mm glass funnel, stirring frequently during filtration to keep the diatomaceous silica suspended in the liquid. Wash the filter paper and residue with five 10 mL portions of warm acetate solution, draining thoroughly after each washing. Wash in the same manner with eight 10 mL portions of the NH₄OH wash solution. After final draining, replace the paper and residue in the weighing bottle, and dry at 70 °C to constant weight. Cool the weighing bottle, paper, and residue in a freshly prepared desiccator before weighing; this is essential, due to the hygroscopic character of paper.

20.5 Calculation:

20.5.1 Calculate the percentage of loss in weight at 45 °C (free water) as follows:

$$\text{loss in weight at 45°C, \%} = [(A - B)/C] \times 100 \quad (5)$$

where:

- A = original weight of sample and weighing bottle,
- B = weight of sample and weighing bottle dried to constant weight at 45 °C, and
- C = original weight of sample.

Calculate the weight of the 4 g sample (20.3.1), corrected for loss on heating to constant weight at 45 °C.

20.5.2 Calculate the percentage of combined water as follows:

$$\text{combined water, \%} = [(B - D)/(B - E)] \times 100 \quad (6)$$

where:

- B = weight of sample and weighing bottle dried to constant weight at 45 °C,
- D = weight of sample and weighing bottle dried to constant weight at 220 °C, and
- E = weight of weighing bottle.

20.5.3 Calculate the percentage of CaSO₄ · nH₂O on the basis of the sample dried to constant weight at 45 °C as follows:

$$\text{CaSO}_4 \cdot n\text{H}_2\text{O, \%} = [F - (G - H)/F] \times 100 \quad (7)$$

where:

- F = weight of sample, corrected for loss on heating to constant weight at 45 °C,
- G = weight of dried crucible and contents (20.3.4) or weight of weighing bottle and contents (20.4.2), and
- H = weight of crucible plus diatomaceous silica used as filter aid (20.3.4), or weight of weighing bottle, diatomaceous silica used as a filter aid and the weight of filter paper (20.4.2).

20.6 Precision and Bias—Neither the precision nor the bias for the analysis of calcium sulfate by the ammonium acetate method has been determined.

21. Alternative Procedure for Analysis for Sodium Chloride by Coulometric Method⁹

21.1 Significance and Use—This test method covers the determination of sodium chloride in gypsum and gypsum products by the coulometric method.

21.2 Interferences—The presence of sulfide, sulfhydryl, or other silver reactive substances will lead to high results. Such interfering substances are removed by alkaline oxidation with hydrogen peroxide.

21.3 Apparatus:

21.3.1 Chloride Meter:

21.3.1.1 The instrument shall be equipped to measure the concentration of dissolved chloride in aqueous solutions by the coulometric method.

21.3.1.2 The instrument shall be capable of measuring chloride concentrations in the range from 10 to 260 mg/L with a repeatability of ±1 mg/L.

21.4 Reagents:

21.4.1 Acid Buffer Solution—Dissolve 100 mL of 99.5 % acetic acid (HC₂H₃O₂) and 5.5 mL of concentrated nitric acid (sp gr 1.42) in approximately 200 mL of water and dilute to 500 mL.

21.4.2 Diluted Standard Solution (100 mg Cl/L)—Dilute 5.00 mL of stock standard solution to 500 mL.

21.4.3 Gelatin Solution—Add 2.5 g of gelatin and 0.5 g of thymol blue to 250 mL of water and dissolve by stirring continuously while bringing to a boil. With the solution just boiling, continue stirring until all the thymol blue is dissolved. Add 0.5 g of thymol, cool, and dilute the solution to 500 mL.

NOTE 9—The gelatin solution holds the precipitated silver chloride

⁹ This procedure was developed by Westroc Industries Limited.

(AgCl) in suspension and also indicates the presence of the acid buffer. The solution will keep for three months at room temperature or longer if refrigerated. Warm the refrigerated solution to room temperature before use.

21.4.4 *Stock Standard Solution* (10 g Cl/L)—Dissolve 8.240 g of dried sodium chloride (NaCl) in water and dilute to 500 mL.

21.5 *Procedure:*

21.5.1 Weigh 20.0 g of the well-mixed sample and transfer to a 150 mL beaker.

21.5.2 Add 50 mL of water, boil, allow the solid material to settle, and filter off the solution. Add an additional 50 mL of water to the solids, boil, and pour the contents of the beaker into the filter. Wash the residue with 100 mL of hot water, adding the washing to the filtrate. Cool and dilute with water to 250 mL.

21.5.3 Switch on the chloride meter and allow a period of 25 min before use. Set the counter to zero.

21.5.4 Place a magnetic stirring bar in the test beaker, add 10 mL of diluted standard solution, 3 mL of acid buffer solution, and five drops of gelatin solution. Place the test beaker on the platform and lower the electrodes into the solution. Press the “start” button until the pilot light is extinguished. The counter will begin to register after a few seconds. Do not remove the electrodes from the sample until the pilot light comes on. Read the chloride content from the counter. If a reading of 100 ± 1 mg Cl/L is not obtained, refer to the manufacturer’s instruction manual. Reset the counter to zero.

21.5.5 Repeat the procedure used in 21.5.4 using 10 mL of the sample solution instead of the diluted standard solution. Read the result as milligrams of chlorine per litre. When all tests are completed, lower the electrodes into reagent water.

21.6 *Calculation*—Calculate the amount of NaCl as a percentage of the sample as received or dried sample as follows:

$$\text{NaCl, \%} = 0.00206 \times A \quad (8)$$

where:

A = chloride meter reading, mg Cl/L.

21.7 *Precision and Bias*—Neither the precision nor the bias for the sodium chloride analysis by the coulometric method has been determined.

22. Determination of Sand in Set Plaster

22.1 *Summary of Test Methods*—This test method for the determination of the sand content of set gypsum plaster requires for accurate results the following determinations:

22.1.1 Determination of the percentage of insoluble matter in the sand used with the plaster,

22.1.2 Determination of the percentage of insoluble matter in the gypsum neat plaster, and

22.1.3 Determination of the percentage of insoluble matter in the sanded calcined plaster.

NOTE 10—If samples of the original gypsum neat plaster and the sand are not available, an approximation of the insoluble matter may be obtained by the use of this method on plaster and sand from the same sources as those from which the plaster to be analyzed was originally prepared.

22.2 *Significance and Use*—This test method is used for determining the sand content of samples of aggregated plaster taken from job sites to determine compliance with Specification C842.

22.3 *Reagents:*

22.3.1 *Ammonium Acetate* (250 g/L)—Dissolve 250 g of ammonium acetate ($\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$) in water and dilute to 1 L.

22.3.2 *Ammonium Hydroxide* (1 + 59)—Mix one volume of concentrated ammonium hydroxide (NH_4OH) (sp gr 0.90) with 59 volumes of water.

22.4 *Sampling*—Where plaster to be tested is part of a two-coat or three-coat plastering operation, take the sample for analysis from that portion of the entire plaster sheet that comprises the single coat being tested. Separate succeeding coats of plaster by use of a stiff putty knife or similar implement. Not less than 500 g shall be taken as a sample, the sample preferably being obtained from different sections of the wall or ceiling under examination.

22.5 *Procedure:*

22.5.1 In a clean porcelain mortar, grind the set plaster sample to the size of the largest sand particles present, or smaller, so that approximately 100 % of the sample will pass a 2.36 mm (No. 8) sieve. Fine grinding makes solution of the gypsum faster. Place approximately 200 g of the ground sample in a porcelain casserole or evaporating dish, and calcine on a sand bath. Stir the sample continuously with a thermometer during the heating, and adjust the rate of heating so that 20 to 30 min will be required to raise the temperature of the sample to 160 ± 5 °C. Cool the sample to room temperature in a desiccator.

22.5.2 After cooling, weigh accurately 20 ± 0.05 g of the calcined sample into a 600 mL beaker. Add 300 to 350 mL of $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ solution. If acidic to litmus paper, add a few millilitres of NH_4OH (1 + 59) to the stock $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$ solution to render it slightly alkaline prior to the addition to the test sample.

22.5.3 Warm the suspension to a temperature of 70 ± 5 °C and stir continuously for 20 to 30 min. Filter the warm suspension with the aid of suction through a small Büchner funnel or Gooch crucible in which filter paper has previously been placed. Refilter the first 100 mL of the filtrate. Wash the sand remaining in the beaker onto the filter with an additional 100 mL of warm ammonium acetate solution. Wash the beaker and residue with 200 to 300 mL of water, dry the funnel and sand at 100 °C to constant weight. The weight of the residue is the weight of insoluble matter.

22.5.4 *Insoluble Matter in Sand*—Determine the weight of insoluble matter in sand as described in 22.5.1 – 22.5.3, except that no grinding of the sample is necessary.

22.5.5 *Insoluble Matter in Gypsum Neat Plaster*—Determine the weight of insoluble matter in the gypsum neat plaster as described in 22.5.1 – 22.5.3, except that only a 5 g sample is required and no grinding of the sample is necessary.

22.6 *Calculation:*

22.6.1 *Insoluble Matter in Plaster*—Multiply by 5 the weight of the insoluble matter obtained as described in 22.5.3 to obtain the percentage of insoluble matter in sanded plaster.

22.6.2 *Insoluble Matter in Sand*—Multiply by 5 the weight of the insoluble matter in sand obtained as described in 22.5.4 to obtain the percentage of insoluble matter in sand.

22.6.3 *Insoluble Matter in Gypsum Neat Plaster*—Multiply by 20 the weight of the insoluble matter in gypsum neat plaster obtained as described in 22.5.5 to obtain the percentage of insoluble matter in gypsum neat plaster.

22.6.4 Calculate the percentage of sand in the sanded plaster as follows:

$$X = [(C - B)/(A - B)] \times 100 \quad (9)$$

where:

- X = % of sand in sanded plaster,
- A = % of insoluble matter in the sand,
- B = % of insoluble matter in the gypsum neat plaster, and
- C = % of insoluble matter in the sanded plaster.

22.6.5 To express the results as a ratio of the parts of sand per part of plaster by weight, use the following equation:

$$\text{ratio of sand to plaster} = X/(100 - X) \quad (10)$$

NOTE 11—The results obtained by the above procedure indicate the amount of sand originally mixed with the gypsum neat plaster before it had been gauged with water or set.

22.7 *Precision and Bias*—Neither the precision nor the bias for the analysis of sand in set plaster has been determined.

23. Wood-fiber Content in Wood-fiber Gypsum Plaster

23.1 *Significance and Use*—This test method is used to determine the weight of wood fiber in wood-fibered plaster.

23.2 Procedure:

23.2.1 Place a 100 g sample of wood-fiber plaster, prepared as described in Section 4 on a 600 μm (No. 30) sieve nested over a 150 μm (No. 100) sieve (see Note 1). Wash the plaster on the 600 μm sieve with a stream of cold water, removing the 600 μm sieve when the fiber on it is practically or entirely free of plaster. Next, wash the material on the 150 μm sieve until the bulk of the plaster has been washed through the sieve and the residue is mainly fiber. Transfer the material retained on the 150 μm sieve to a 300 mL, vitreous enamel, lipped pan, adding the charge on the 600 μm sieve if the fiber contains any adhering particles of plaster. Elutriate the material in the pan (purify by washing and straining, effecting as clean a separation of fiber from plaster as is practical), catching the elutriated fibers on a 150 μm sieve. To avoid loss of the fine particles of fiber, make the transfer from the pan to the 150 μm sieve by several stages of washing, stirring the charge, and quickly pouring upon the sieve the fiber flotations, repeating the elutriation procedure several times. Examine the fiber collected on the 150 μm sieve and repeat the elutriation if necessary.

23.2.2 Dry the sieves (or sieve) and the residue contained thereon overnight in an oven maintained at a temperature of 45 °C. Carefully invert the sieves, or sieve, over a piece of white paper, and transfer the residual material to the paper by brushing the bottom of the inverted sieve. Examine the transferred material visually, noting whether the separation of fibers from plaster has been complete. Then transfer the material to a weighed platinum crucible and dry to constant weight at a temperature of 45 °C. If the previous visual examination of the charge on the white paper showed that the

fiber was practically free of particles of plaster, report as the percentage of fiber the weight of the fiber dried at 45 °C, divided by 100. If, on the other hand, the visual examination revealed the presence of an appreciable quantity of plaster associated with the fiber, carefully ignite the contents of the crucible to constant weight. In this case, report as the percentage of fiber the loss on ignition, divided by 100.

23.3 *Precision and Bias*—Neither the precision nor the bias for the analysis of wood-fiber content in wood-fiber gypsum plaster has been determined.

24. Optional Procedure for Analysis for Sodium by Atomic Absorption Method

24.1 *Significance and Use*—This test method covers the determination of sodium in gypsum and gypsum products by the atomic absorption method.

24.2 *Interferences*—Sodium is partially ionized in the air-acetylene flame. The effects of ionization will be significantly overcome by the addition of 1 to 2 g/L of another alkali to blanks, standards, and samples. Alternatively, use the air-hydrogen flame, as it produces less ionization and less visible emission than the air-acetylene flame.

24.3 Apparatus:

24.3.1 Atomic Absorption Spectrophotometer:

24.3.1.1 The instrument shall be equipped to measure the concentration of dissolved sodium in aqueous solutions using either the air-acetylene or air-hydrogen flame.

24.3.1.2 The instrument shall be capable of measuring sodium concentrations within the optimum analytical range of 0.1 to 0.5 absorbance units while providing a coefficient of variation of approximately 0.5 to 2 %.

24.4 Reagents:

24.4.1 *Solvent*—Use deionized water to prepare all solutions. If an alkali is to be included for the purpose of suppressing sodium ionization, it is most convenient to add it to the solvent at the start. In this way a constant concentration of alkali in blank, standards, and sample solution is ensured.

24.4.2 *Stock Standard Solution* (1.000 g Na/L)—Dissolve 2.5418 g of dried sodium chloride (NaCl) in water and dilute to 1 L with water.

24.4.3 *Dilute Standard Solutions*—Prepare dilute standard solutions bracketing the absorbance range of the dilute sample solution, using the stock standard solution. (Solutions having a concentration less than approximately 0.500 g/L are unstable for periods of more than day.)

24.5 Procedure:

24.5.1 Take 18 g of the well-mixed sample and transfer to a 150 mL beaker.

24.5.2 Add 50 mL of water, boil, allow the solids to settle, and decant the supernatant liquid into a filter. Add an additional 50 mL of water to the solids, boil, and pour the contents of the beaker into the filter. Wash the residue with 100 mL of hot water, adding the washing to the filtrate. Cool the filtrate to room temperature and dilute to 500 mL in a volumetric flask to make the stock sample solution. Take 10 mL of the solution and make up to 500 mL in a second volumetric flask, to make the dilute sample solution.

24.5.3 Determine the absorbance readings on the dilute standard solutions and the solvent blank at a wavelength of 589.0 to 589.6 nm, following the manufacturer's instruction manual. Subtract the absorbance value for the blank from the absorbance values for the dilute standard solutions and prepare a curve relating sodium concentration in milligrams per litre to absorbance values.

NOTE 12—If the absorbance of the dilute sample solution is known to lie within the linear range, that is, the sodium concentration is below approximately 1 mg/L only one standard and the solvent blank are needed to prepare the curve.

24.5.4 Determine absorbance readings on the dilute sample solution using the same technique, followed with the dilute standard solutions. The concentration of sodium in the dilute sample solution in milligrams per litre is then found by consulting the standard curve.

24.6 *Calculation*—Calculate the amount of NaCl as a percentage of the sample received or the dried sample as required as follows:

$$\text{sodium calculated as \% NaCl} = A \times 6.3553/S \quad (11)$$

where:

A = concentration of dilute sample solution, mg/L, and
S = weight of sample, g.

24.7 *Precision and Bias*—Neither the precision nor the bias for the analysis of sodium by the atomic absorption method has been determined.

25. Optional Procedure for Analysis for Sodium by Flame Photometry

25.1 *Significance and Use*—This test method covers the determination of sodium in gypsum and gypsum products by flame photometry. This test method is based on Test Method D1428.

25.2 Interferences:

25.2.1 Radiation interferences caused by elements other than that being determined are the greatest contributor to error in flame photometry. Some effects are positive and others negative. Of the elements encountered in this analysis, the greatest effect is that of one alkali metal on another. The foreign-element effects cannot be entirely compensated for without employing calibration standards closely duplicating the composition of the sample. However, the effects are minimized by operating at the lowest practical sodium concentration range or by removal of the interfering elements. For example, aluminum has a depressing effect on alkali-metal emission, which is of serious consequence. Remove aluminum from the extraction liquid prior to flame photometry if its concentration has been found, by preliminary tests, to exceed that of the sodium.

25.2.2 Self-absorption causes the curve of intensity versus concentration to decrease its slope at higher concentrations, tending to reduce accuracy. Bracketing the unknown by known standard solutions tends to minimize this interference.

25.3 Apparatus and Materials:

25.3.1 *Flame Photometer*—The instrument shall consist of an atomizer and burner; suitable pressure-regulating devices

and gauges for fuel and air or oxygen; an optical system, consisting of suitable light-dispersing or filtering devices capable of preventing excessive interference from light of wavelengths other than that being measured; and a photosensitive indicating device.

25.3.2 *Supply of Fuel and Air or Oxygen*—The supplies of fuel and air or oxygen shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument.

25.4 Reagents:

25.4.1 Prepare the following stock solutions from reagents that have been dried to constant weight at 105 °C. Store the stock solutions in polyethylene or equally alkali-metal-free containers.

25.4.2 *Sodium Chloride Solution* (2.5418 g/L)—Dissolve 2.5418 g of sodium chloride (NaCl) in water and dilute to 1 L with water. This stock standard solution contains 1.000 g/L of sodium ion.

25.4.3 *Dilute Standard Solutions*—Prepare dilute standard solutions from stock standard solution, bracketing the expected sodium concentration range of the diluted sample extraction liquid. For example, if the expected range of the sample extraction liquid is between 0 and 0.010 g/L, prepare eleven equally spaced standards in tenths of the maximum.

25.5 Calibration of Instrument:

25.5.1 Select the proper photocell; the blue-sensitive phototube having a range from 320 to 620 nm is required for sodium determination. Open the slit width to approximately one fourth of the maximum opening, set the instrument to the maximum sensitivity range, and balance the meter to obtain electrical equilibrium.

25.5.2 Feed fuel and air or oxygen to the burner and ignite the emitted mixture. Adjust fuel and air or oxygen pressures and follow the procedures for warm-up time prescribed by the instrument manufacturer. Set the scale-reading dial at approximately 95 % of full scale, introduce a solution containing the maximum amount of sodium in the range to be covered, and allow the emitted light to strike the photocell.

25.5.3 Select the proper filter, if a filter-type instrument is used. For instruments employing spectral dispersing devices, turn the wavelength dial back and forth slowly and carefully in the vicinity of 589 nm until the galvanometer reaches a maximum deflection. This wavelength setting produces maximum sensitivity. Do not disturb the wavelength dial during the test.

25.5.4 Continue to atomize the maximum standard of the range to be covered, and set the scale-reading of the dial at exactly full scale (100 or 1000) (Note 13). Adjust the gain so as to balance the galvanometer needle.

NOTE 13—For instruments equipped with a variable slit, carry out the procedure described above with the slit width between fully closed and one fourth open for the preliminary test. In determining the proper slit width for optimum instrument performance, consideration must be given to the fact that the intensity of the emission line is approximately proportional to the slit width, whereas continuous background intensity increases as the square of the slit width. A decrease in slit width results in decreased illumination of the phototube for a given concentration and is compensated for by increasing the gain of the instrument. The most favorable operating conditions are obtained with the smallest slit width

that does not result in instability of the galvanometer needle when it is set to give full-scale reading with the maximum standard in the range to be covered. When altering the slit width, determine the background by atomizing a zero standard, and check for sensitivity setting and instrument stability with the maximum standard in the range to be covered. Lower ranges require wider slit widths. Determine and record the optimum slit width for each range and element to be covered. Use these values in all subsequent tests.

25.5.5 Determine the emission intensity of all standards.

25.5.6 Plot emission intensity (scale reading) versus concentration on linear graph paper. For the lower ranges, the curve thus prepared approximates a straight line but sometimes will not intersect zero because of background intensity. At higher ranges, the curves show a decrease in slope with increasing concentration. Record on graphs all data in regard to slit width, fuel pressure, and air or oxygen pressure.

25.6 Procedure:

25.6.1 Weigh 25 g of the well-mixed sample and transfer to a 150 mL beaker.

25.6.2 Add 50 mL of water, boil, allow the solid material to settle, and decant the supernatant liquid into a filter. Add an additional 50 mL of water to the solids, boil and pour the contents of the beaker into the filter. Wash the residue with 75 mL of hot water, adding the washing to the filtrate. Cool and dilute with water to 200 mL in a volumetric flask to make the stock sample solution.

25.6.3 Take 5 mL of the stock sample solution and make up to 100 mL in a volumetric flask to make the dilute sample solution.

NOTE 14—If the concentration of sodium in the sample is found to be greater than the maximum standard, further dilute sample solution with water to bring the concentration within the range. If the concentration of sodium in the sample is less than one tenth of the value of the maximum standard, prepare a new dilute sample solution from the stock sample solution to bring the concentration within the range.

25.6.4 Turn the instrument on and feed fuel and air or oxygen to the burner. Ignite the gas mixture. For instruments with an adjustment slit, set the width to the value determined as outlined in **Note 13**.

25.6.5 Place the scale-reading dial at maximum. Atomize the sample and allow its emitted light to strike the photocell. Set the wavelength to 589 nm as described and adjust the gain to balance the galvanometer. Determine the emission intensity of the sample.

25.6.6 Refer to the standard curve prepared above and read off the concentration of sodium ion in the dilute sample solution in milligrams per litre.

25.7 Calculation—Calculate the amount of NaCl as follows:

$$\text{sodium, calculated as NaCl, \%} = A \times 0.04067 \quad (12)$$

where:

A = concentration of sodium, mg/L read off the standard curve.

25.8 Precision and Bias—Neither the precision nor bias of the analysis for sodium by flame photometry has been determined.

26. Determination of Orthorhombic Cyclooctasulfur (S₈) in Gypsum Panel Products

26.1 Significance and Use:

26.1.1 This test method covers the determination of orthorhombic cyclooctasulfur (S₈) in the core of the gypsum panel product.

26.1.2 Three test methods are available: gas chromatograph equipped with a mass spectrometer (GC/MS), gas chromatograph equipped with an electron capture detector (GC/ECD), and high-performance liquid chromatograph equipped with an ultraviolet detector (HPLC/UV).

26.1.3 This test method 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 test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

26.2 Orthorhombic Cyclooctasulfur (S₈) Standard for GC and HPLC:

26.2.1 Toluene, spectral quality or chromatographic grade.

26.2.2 Orthorhombic Cyclooctasulfur (S₈), to be used as a calibration standard.

26.2.3 OTTAWA Sand, ASTM 20-30 sand (Specification **C778**).

26.2.4 Standard Orthorhombic Cyclooctasulfur (S₈) Solutions:

26.2.4.1 Dissolve 2400 mg of S₈ in 1 L of toluene.

26.2.4.2 Prepare five calibration standards of S₈ in toluene at 1.0, 5.0, 25.0, 50.0, and 100.0 ppm.

26.2.4.3 To evaluate extraction efficiency, add 50 µL of each S₈ prepared standard to 1.0 g of clean OTTAWA sand, and follow steps for sample preparation in each method (GC or HPLC).

27. Determination of Orthorhombic Cyclooctasulfur (S₈) in Gypsum Panel Products by Gas Chromatograph Equipped with Mass Spectrometer (GC/MS)

27.1 Apparatus—Gas chromatograph, equipped with a mass spectrometer detector.

27.1.1 Software must be capable of integrating peak areas.

27.2 Reagents and Materials:

27.2.1 Carrier Gas—Helium, chromatographic grade.

27.2.2 Internal Standard (ISTD)—4,4'-dibromobiphenyl, 5 µg/mL in xylene.

27.2.3 Surrogate Standard—Hexabromobenzene, 2.5 mg/mL hexabromobenzene in xylene.

27.2.4 Xylene—Spectral quality or chromatographic grade.

27.2.5 Decafluorotriphenylphosphine (DFTPP)—Spectral quality or chromatographic grade

27.2.6 Specimen vials should be VOC capable.

27.3 Sample Preparation—Three specimens of the gypsum panel product should be available for testing. Remove surfacing material and crush a piece of each specimen with a mortar and pestle to a powder.

27.3.1 GC Specimen Preparation:

27.3.1.1 Collect 1.0 g of each crushed specimen and transfer to a separate, sealable 10 mL to 20 mL vial.

27.3.1.2 Add 50 μL of surrogate standard to each specimen and mix well using a glass rod and allow sample to air dry.

27.3.1.3 Add 5 mL of toluene to each vial, while rinsing particles from the glass rod. Shake vigorously or otherwise agitate (for example, ultrasonicate) sample for a minimum of 2 min. Allow to settle for 5 min or filter through a fast filter paper.

27.3.1.4 For each specimen, transfer a clear 1.0 mL aliquot of toluene solution to a GC vial. Add 20 μL of ISTD to each vial.

27.4 Preparation of Apparatus:

NOTE 15—Instrument should be tuned daily using decafluorotriphenylphosphine (DFTPP).

27.4.1 *Column*—Fused silica capillary column, 30 m by 0.32 mm; 0.5 μm film thickness; type DB-1 or DB-5, glass gooseneck inlet liner without glass wool.

27.4.2 *Carrier Gas*—Helium.

27.4.3 *Injection Port*—250 °C; splitless.

27.4.4 *Temperature Program*—120 °C for 1 min; 9 °C/min to 285 °C; hold for 10 min.

27.4.5 *Mass Spectrometer*—Full scan (30 to 600 m/z range) in scan mode.

27.4.6 *Sample Injection Amount*—1.0 μL .

27.5 *Identification*— S_8 is identified by mass spectrum (64 m/z will be the most abundant ion). Confirm by retention time matching to a calibration standard. The internal standard will yield ions at 152 and 312 m/z. The surrogate's most abundant ions will be found at 552 m/z.

27.6 Calculation:

27.6.1 Integrate the S_8 and ISTD areas for each calibration standard. Calculate the peak area ratio by dividing the S_8 peak area by the internal standard peak area. Build a calibration curve based on the peak area ratios for each standard.

27.6.2 Calculate the peak area ratio for each specimen. Use the calibration curve to calculate the S_8 concentration in solution.

27.6.3 Calculate the concentration of S_8 in the original sample in mg/kg using the sample weight, final extract volume, and concentration value obtained in 27.6.2.

27.6.4 Calculate the percent recovery of the surrogate compound to evaluate the extraction efficiency.

27.7 *Precision and Bias*—An interlaboratory study of this test method is being conducted and a complete precision statement is expected to be available on or before 2019.

27.8 *Report*—Report the quantity of orthorhombic cyclooctasulfur (S_8) calculated in 27.6, determined to the nearest 1 mg/kg.

28. Determination of Orthorhombic Cyclooctasulfur (S_8) in Gypsum Panel Products by Gas Chromatograph Equipped with Electron Capture Detector (GC/ECD)

28.1 *Apparatus*—Gas chromatograph equipped with an electron capture detector.

28.1.1 Software must be capable of integrating peak areas.

28.2 Reagents and Materials:

28.2.1 *Carrier Gas*—Helium, chromatographic grade.

28.2.2 *Internal Standard (ISTD)*—4,4'-dibromobiphenyl, 5 $\mu\text{g}/\text{mL}$ in xylene.

28.2.3 *Surrogate Standard*—Hexabromobenzene, 2.5 mg/mL hexabromobenzene in xylene.

28.2.4 *Xylene*—Spectral quality or chromatographic grade.

28.2.5 *Specimen Vials* should be VOC capable.

28.3 *Sample Preparation*—Three specimens of the gypsum panel product should be available for testing. Remove surfacing material and crush a piece of each specimen with a mortar and pestle to a powder.

28.3.1 GC Specimen Preparation:

28.3.1.1 Collect 1.0 g of each crushed specimen and transfer to a separate, sealable 10 mL vial.

28.3.1.2 Spike each specimen with 50 μL of surrogate standard. Mix well using a glass rod and allow sample to air dry.

28.3.1.3 Add 5 mL of toluene to each vial, rinsing any particles off of the glass rod. Shake vigorously or otherwise agitate (for example, ultrasonicate) sample for a minimum of 2 min. Allow to settle for 5 min or filter through a fast filter paper.

28.3.1.4 For each specimen, transfer a clear 1.0 mL aliquot of toluene solution to a GC vial. Add 20 μL of internal standard to each vial.

28.4 Preparation of Apparatus:

28.4.1 *Column*—Fused silica capillary column, 30 m by 0.32 mm; 0.5 μm film thickness; type DB-1 or DB-5, glass gooseneck inlet liner without glass wool.

28.4.2 *Carrier Gas*—Helium

28.4.3 *Injection Port*—250 °C; splitless.

28.4.4 *Temperature Program*—120 °C for 1 min; 9 °C/min to 285°; hold for 10 min.

28.4.5 *Sample Injection Amount*—1.0 μL .

28.5 *Identification*—Identification can only be done by retention time matching to a standard.

28.6 Calculation:

28.6.1 Integrate the S_8 and internal standard peak areas for each calibration standard. Calculate the peak area ratio by dividing the S_8 peak area by the internal standard peak area. Build a calibration curve based on the peak area ratios for each standard.

28.6.2 Calculate the peak area ratio for each specimen. Use the calibration curve to calculate the S_8 concentration in solution.

28.6.3 Calculate the concentration of S_8 in the original sample in mg/kg using the sample weight, final extract volume, and concentration value obtained in 28.6.2.

28.6.4 Calculate the percent recovery of the surrogate compound to evaluate the extraction efficiency.

28.7 *Precision and Bias*—An interlaboratory study of this test method is being , and a complete precision statement is expected to be available on or before 2019.

28.8 *Report*—Report the quantity of orthorhombic cyclooctasulfur (S_8) calculated in 28.6.3 determined to the nearest 1 mg/kg.

29. Determination of Orthorhombic Cyclooctasulfur (S₈) in Gypsum Panel Products by High-Performance Liquid Chromatograph Equipped with Ultraviolet Detector (HPLC/UV)

29.1 *Apparatus*—High-performance liquid chromatograph (HPLC) equipped with an ultraviolet (UV) detector.

29.1.1 Software must be capable of integrating peak areas.

29.2 *Reagents and Materials:*

29.2.1 *HPLC Methodology.*

29.2.2 *Tetrachloroethylene*—HPLC grade.

29.2.3 *Methanol*—HPLC grade.

29.2.4 *Deionized Water*—HPLC grade.

29.2.5 *Specimen Vials* should be VOC capable.

29.3 *Sample Preparation*—Three specimens of the gypsum panel product should be available for testing. Remove surfacing material and crush a piece of each specimen with a mortar and pestle to a powder.

29.3.1 *HPLC Sample Preparation:*

29.3.1.1 Collect 1.0 g of each crushed specimen and transfer to a separate, sealable 4.0 mL extraction vial.

29.3.1.2 Add 5 mL of tetrachloroethylene to each vial, rinsing any particles remaining on the glass rod or in the vial. Seal and shake vigorously or otherwise agitate (for example, ultrasonicate) the sample for a minimum of 30 min.

29.3.1.3 Allow each sample to settle for 5 min and then transfer each sample to a separate appropriate vial.

29.3.1.4 Centrifuge each extract for 5 min and then transfer the top layer of each extract to a separate HPLC crimp top vial for analysis.

29.4 *Preparation of HPLC/UV:*

29.4.1 *Column*—5 μm C₁₈ reverse phase column (4.6 by 250 mm).

29.4.2 *UV Wavelength*—250 nm.

29.4.3 *Flow Rate*—1 mL/min.

29.4.4 *Eluent*—95/5 methanol/water.

29.4.5 *Run Time*—15 min.

29.5 *Identification*—Identification of S₈ can only be done by retention time matching to a standard.

29.6 *Calculation:*

29.6.1 Calculate the peak area ratio for each specimen. Use the calibration curve to calculate the S₈ concentration in solution.

29.6.2 Calculate the concentration of S₈ in the original sample in mg/kg using the sample weight, final extract volume, and concentration value obtained in 29.6.1.

29.6.3 Calculate the percent recovery of the orthorhombic cyclooctasulfur (S₈) standard matrix to evaluate the extraction efficiency for the extraction batch.

29.7 *Precision and Bias*—An interlaboratory study of this test method is being , and a complete precision statement is expected to be available on or before 2019.

29.8 *Report*—Report the quantity of orthorhombic cyclooctasulfur (S₈) calculated in Section 29.6.3 determined to the nearest 1 mg/kg.

30. Keywords

30.1 ammonium acetate method; atomic absorption; chemical analysis; coulometric method; flame photometry; gas chromatography/electron capture detector; gas chromatography/mass spectrometry; gypsum; gypsum board; gypsum concrete; gypsum products; high-performance liquid chromatography/UV detector; plaster; sand in set plaster; sulfur (S₈); wood-fiber plaster

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