Designation: C471M – 20a$^{\varepsilon 1}$

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

This standard is issued under the fixed designation C471M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—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:

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.

1.4 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D1428 Test Method for Test for Sodium and Potassium In Water and Water-Formed Deposits by Flame Photometry (Withdrawn 1989)[3]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

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

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[2] 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 $CaSO_4 \cdot 2H_2O$ and natural anhydrite. Place the sample in distilled water and keep it wet for not less than 48 h. Dry the hydrated sample in an oven at 45 ± 3 °C to constant weight and recrush or grind it in accordance with 4.1.3.

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

4.2 *Gypsum* (Specification C22/C22M)—Gypsum samples will be received in the form of rocks or powder, or both. If necessary crush and reduce the entire dried sample in accordance with 4.1.3 and 4.1.5.

4.3 *Gypsum Plaster* (Specification C28/C28M):

4.3.1 *Gypsum Ready-mixed Plaster or Gypsum 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* (NH_4Cl).

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

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

6.1.4 *Ammonium Oxalate* ($(NH_4)_2C_2O_4$).

6.1.5 *Barium Chloride* (100 g/L)—Dissolve 100 g of barium chloride ($BaCl_2{\cdot}2H_2O$) in water and dilute to 1 L.

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

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

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

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

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

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

6.1.12 *Potassium Permanganate* (5.6339 g/L)—Dissolve 5.6339 g of potassium permanganate ($KMnO_4$) in water and dilute to 1 L.

6.1.13 *Silver Nitrate, Standard Solution* (0.05 N)—Prepare and standardize a 0.05 N silver nitrate ($AgNO_3$) solution.

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

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

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

6.1.17 *Nitric Acid* (0.1 N)—Mix 1.4 mL of HNO_3 (sp gr 1.42) with 200 mL of water.

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

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

6.1.20 *Water*—Reagent water shall be in accordance with Specification D1193, type II. Specification D1193 gives the following values for type II grade water.

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

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.