



Designation: C1324 – 20a

Standard Test Method for Examination and Analysis of Hardened Masonry Mortar¹

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1. Scope*

1.1 This test method covers procedures for petrographic examination and chemical analysis of samples of masonry mortars. Based upon such examination and analysis, proportions of components in masonry mortars can be determined.

NOTE 1—This method is also applicable to hydraulic cement-based stucco and plaster. Some historic mortars may contain non-resolvable constituents that may interfere. However, significant information may be obtained by petrographic examinations.

1.2 Interpretations and calculations of chemical results are dependent upon results of the petrographic examination. The use of the chemical results alone is contrary to the requirements of this test method.

1.3 Procedures for sampling, petrographic examination, chemical analysis, and calculations of component proportions are given in the following sections:

	Section
Sampling	7
Petrographic examination	8
Chemical analysis	9
Mortar proportion calculations	10
Report	11

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 The text of this standard references notes and footnotes that provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

1.6 *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.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the*

¹ This test method is under the jurisdiction of ASTM Committee C12 on Mortars and Grouts for Unit Masonry and is the direct responsibility of Subcommittee C12.02 on Research and Methods of Test.

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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

- C114 Test Methods for Chemical Analysis of Hydraulic Cement
- C125 Terminology Relating to Concrete and Concrete Aggregates
- C144 Specification for Aggregate for Masonry Mortar
- C270 Specification for Mortar for Unit Masonry
- C295/C295M Guide for Petrographic Examination of Aggregates for Concrete
- C457/C457M Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete
- C823/C823M Practice for Examination and Sampling of Hardened Concrete in Constructions
- C856/C856M Practice for Petrographic Examination of Hardened Concrete
- C926 Specification for Application of Portland Cement-Based Plaster
- C1084 Test Method for Portland-Cement Content of Hardened Hydraulic-Cement Concrete
- C1180 Terminology of Mortar and Grout for Unit Masonry
- D1193 Specification for Reagent Water

3. Terminology

3.1 Terms used in this test method are defined in Terminology standards C125, C1180, or the other referenced ASTM standards.

4. Significance and Use

4.1 This test method provides procedures for petrographic examination and chemical analysis of mortar for components of masonry mortar. These components may include portland cement, hydrated calcitic or dolomitic lime, masonry cement, aggregates, and air.

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.

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

4.2 The test method consists of procedures and sub-procedures, each requiring a substantial degree of petrographic and chemical skills and relatively elaborate instrumentation.

4.3 The chemical data considered together with results of petrographic examination of a mortar provide for calculation of component proportions and thus allow a determination of mortar composition as represented by Types M, N, S, and O in Table 1 (Proportion Specification Requirements) of Specification **C270**.

4.4 Failure of a mortar to have the composition of any type as defined in Table 1 of Specification **C270** does not necessarily mean that the mortar does not meet the requirements of Specification **C270**. The mortar may meet the alternative requirements of Table 2 (Property Specification Requirements) of Specification **C270**.

4.5 The maleic acid method of analysis is not applicable for the analysis of mortar because it is greatly influenced by carbonation and does not provide for the determination of calcium.

5. Qualifications of Petrographer and Chemist

5.1 Petrographer:

5.1.1 The petrographic examination requires the skill of a petrographer well versed in the petrographic methods of Practice **C856/C856M**, Test Method **C457/C457M**, and Guide **C295/C295M**, and in the evaluations of portland cement-containing materials and of masonry mortars.

5.1.2 The interpretation and evaluation of the petrographic data requires detailed knowledge of the requirements of Specification **C270**.³

5.2 Chemist:

5.2.1 The chemical analysis requires the skills of a chemist well versed in methods of chemical analysis, such as of Test Methods **C114** and Test Method **C1084**, and in the analytical procedures of these standards.

5.2.2 The interpretation and evaluation of the results of the chemical analysis requires detailed knowledge of the requirements of Specification **C270**.⁴

6. Apparatus, Reagents, and Materials

6.1 Apparatus used for the petrographic examination and preparation of specimens are given in Guide **C295/C295M**, Test Method **C457/C457M**, and Practice **C856/C856M**.

6.2 The following apparatus for the chemical analysis shall be chosen from applicable items given in Test Methods **C114** and **C1084** and from the following:

6.2.1 Crushers and pulverizers,

NOTE 2—Crushers and pulverizers may be obtained from many laboratory supply houses, and include chipmunk type jaw crushers, disc pulverizers, and rotating puck devices.

6.2.2 Disk pulverizer,

- 6.2.3 Rotary mill (rotating puck),
- 6.2.4 Sieve, 300 μm (No. 50),
- 6.2.5 Ice bath or electric cooling apparatus,
- 6.2.6 Hot plate,
- 6.2.7 Buchner porcelain funnel,
- 6.2.8 Filter paper,
- 6.2.9 Beakers, 250-mL and 400-mL,
- 6.2.10 Platinum crucibles, and
- 6.2.11 Porcelain crucibles.

6.3 Reagents and Materials:

6.3.1 Soluble silica sub-procedure:

6.3.1.1 Hydrochloric acid, reagent grade, density 1.19 mg/m^3 (1 + 3),

6.3.1.2 Sodium hydroxide, (10 g/L).

6.3.2 Calcium Oxide Sub-procedure—Use reagents as required in Test Methods **C114**.

6.3.3 Insoluble Residue Procedure—Hydrochloric acid, reagent grade, density 1.19 mg/m^3 (1 + 4).

6.3.4 Water—All references to water shall be understood to mean reagent water Type I through IV of Specification **D1193**.

7. Sampling

7.1 The mortar sample should include intact pieces for petrographic analysis. A 10-g sample is usually sufficient for each composite petrographic examination and chemical analysis. The sampling should provide for differentiating between pointing and other mortars from the original mortar. For certain purposes, sampling may be done using procedures in Practice **C823/C823M**.

8. Petrographic Examination

8.1 General:

8.1.1 Petrographic examination refers to methods in Practice **C856/C856M** and principally to light microscopy and to use of a petrographic microscope and a stereoscopic low power microscope. Ancillary techniques include X-ray diffractometry and scanning electron microscopy.

8.1.2 X-ray diffraction analysis can be used to identify phases present in mortar. This information, in conjunction with the petrographic microscopy information, is a basis for determining, for the chemical analysis, which calcium oxide or magnesium oxide sub-procedure is necessary.

8.1.3 The petrographic and X-ray diffraction information is necessary for interpretations in calculating mortar composition.

8.2 Mortar:

8.2.1 Observe the mortar as appropriate, focusing attention on overall mortar appearance, characteristics of tooled surfaces, fracture surfaces and surfaces contacting masonry units, and secondary substances (efflorescence and internal secondary products). Describe and report the observations.

8.2.2 If X-ray diffraction methods are used, analyze for crystalline components in accordance with individual XRD equipment techniques.

8.3 Aggregate:

8.3.1 Examine the aggregate component of the mortar for rock and mineral composition using methods of Guide **C295/C295M** and Practice **C856/C856M**.

³ Erlin, Bernard and Hime, William G., "Evaluating Mortar Deterioration," Association for Preservation Technology, 1987.

⁴ Hime, William G., and Erlin, Bernard, "Methods for Analyzing Mortar," Proceedings of the Third North American Masonry Conference, Masonry Society, 1985.

8.3.2 If components of the aggregate are acid-insoluble, gently break the mortar, and dissolve the paste matrix using dilute hydrochloric acid, and thoroughly wash and filter the residue. Dry the aggregate residue, and examine it microscopically. The residue may also be sorted into particle sizes using the sieving method of Specification **C144**. Aggregate particles broken during the mortar breakdown will skew the gradation analysis results.

8.4 *Paste*—Examine the paste fraction of the mortar using methods of Practice **C856/C856M**. Components of the paste can include residual (partially hydrated portland cement particles), hydration products of the portland cement and their carbonated equivalents, hydrated lime (both calcitic and dolomitic) and carbonated hydrated lime, and finely ground mineral components such as limestone, dolomite, slag, fly ash, clay, and pigments.

8.5 *Air*:

8.5.1 Examine the mortar for air voids and their configurations and distribution. Characterize the air voids as entrained or entrapped as defined in Terminology **C125** and Test Method **C457/C457M**, or both. Characterize air voids by locations, disposition, and relative size.

8.5.2 Estimate the volume of air relative to the volume of the mortar, and report the estimate as percent air.

8.5.3 Measurements of characteristics of the air-void system can be made using Test Method **C457/C457M**. Usually, special impregnation techniques will be necessary to provide a specimen suitable for the processing needed for this examination. An impregnation technique is given in Test Method **C457/C457M**.

8.6 *Secondary Products*—Examine the mortar, using methods of Practice **C856/C856M**, for secondary products in voids and on tooled, fractured, and other surfaces. Describe, identify, and characterize products present.

8.7 *Evaluation of Petrographic Data*:

8.7.1 Mineralogical characteristics of the aggregate should be evaluated relative to components that could interfere in any subsequent chemical analyses, any chemical or physical instability relative to alkali-silica and alkali-carbonate reactions, any degradation due to freezing and thawing, and stability when exposed to moisture.

8.7.2 The paste should be evaluated so the original cementitious components used for preparing the mortar can be identified.

8.7.3 The air-void data should be evaluated so that the mortar can be classed as non-air-entrained or air-entrained.

8.7.4 The individual observations of aggregate, paste, air, secondary products, and tooled and other surfaces should be integrated to establish overall compositional and textural characteristics of the mortar.

8.8 *Miscellaneous*—A report of the results of the petrographic examination should be prepared as a section of the overall report, or as a separate report, depending upon the purpose of the study. The report should include details about each item in 8.2 through 8.7.

9. Chemical Analysis

9.1 *Sample Preparation*—Crush or grind a representative portion of the sample so that all the material passes a 300 μm (No. 50) sieve. To minimize the production of very fine material, use several passes of the sample through the grinding equipment. Remove the portion passing the sieve before regrinding the remainder of the sample. Thoroughly mix by coning from one paper to another ten times.

9.2 *Soluble Silica Sub-procedure (variation of method used in Test Method C1084)*:

9.2.1 Into two 250-mL beakers introduce 100 mL 1 + 4 HCl. Cool within the range of 3 to 5°C (38 to 41°F) using an ice bath or electric cooling apparatus.⁵

9.2.2 Slowly, over a 1-min period, introduce a 2.00-g sample of crushed mortar. Maintain the 3 to 5°C temperature for a 5-min period, and stir the mixture either continuously or at least several times during this period.

NOTE 3—Observation of the solution during the introduction of the sample may provide useful information. Rapid effervescence indicates a substantial amount of calcite or carbonated paste. Slow effervescence suggests a dolomite aggregate.

9.2.3 Decant through a Buchner porcelain funnel fitted snugly with two disks of a quantitative filter paper for fine precipitates, Type II, Class G filter paper. Once the filtration has begun, take care so the mat and accumulated residue do not dry completely until the filtration process is complete. Regulate the suction to maintain a rapid rate of filtration during the greater part of the filtration process. Retain as much of the residue in the beaker as possible. Wash twice by decantation with hot water. Save the filtrate. Carefully transfer the filter paper from the funnel to the beaker containing the balance of the residue so that no residue is lost. Add 75 mL of hot sodium hydroxide solution (10 g/L) to the residue while stirring, macerate the filter paper, and digest for 15 min. During the digestion, occasionally stir the mixture. Decant as before, and wash twice with hot water until the filtrate is neutral to litmus. Combine the filtrates in a 400-mL beaker.

9.2.4 Proceed to analyze as provided in 9.2.5 and 9.2.6, or analyze the soluble silica by any instrumental method found acceptable for cement analysis in accordance with the performance requirement for rapid methods of Test Methods **C114**, provided it can be applied to the filtrate.

NOTE 4—Suitable instrumental techniques may include atomic absorption or inductively coupled plasma spectroscopy.

9.2.5 Evaporate to dryness taking great care to minimize spattering, bake at not over 120°C (248°F) for 1 h, moisten with hydrochloric acid (density 1.19 g/mL), evaporate and bake again. Take up filtration in 75 mL of hydrochloric acid (1 + 3). Heat to boiling, filter through an ashless filter paper, and wash the residue with 50 mL of hot hydrochloric acid (1 + 9) and then with hot water until the washings are free of

⁵ The sole source of supply of the Stir-Kool Model SK-12 known to the committee at this time is Thermoelectrics Unlimited, Wilmington, DE. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

chlorides. Transfer the filter paper and residue to a weighed platinum crucible, dry, and ignite, at first slowly until the carbon of the paper is completely consumed without inflaming, and finally at 1100 to 1200°C for 1 h. Cool in a desiccator and weigh. Reignite to constant weight. Treat the SiO₂ thus obtained, which will contain small amounts of impurities, in the crucible with 1 or 2 mL of water, 2 drops of H₂SO₄ (1 + 1), and about 10 mL of HF, and evaporate cautiously to dryness. Finally, heat the small residue at 1050 to 1100°C for 5 min, cool in a desiccator, and weigh. The difference between this weight and the weight previously obtained represents the weight of SiO₂.

9.2.6 Calculate the silica percentage as:

$$\text{mass of silica}/2 \times 100 \quad (1)$$

9.3 *Calcium Oxide and Magnesium Oxide Sub-procedure (this procedure should be omitted if the aggregate contains significant amounts of calcareous components):*

9.3.1 The following procedure is suitable for mortars that do not contain dolomite: Using the filtrate from the removal of silica, separate the ammonium-hydroxide group and then determine calcium oxide and magnesium oxide in accordance with Test Methods C114, or proceed as described in 9.3.2.

9.3.2 Any calcium or magnesium method found acceptable for cement analysis in accordance with the performance requirements for rapid methods of Test Methods C114 may be used, provided it can be done on the solution of 9.2.4 or 9.3.3.

9.3.3 The following procedure is suitable for mortars that contain dolomite: Add 75 mL of HCl (1 + 3) to duplicate 2.00-g samples in 250-mL beakers. Heat the covered mixture to near boiling on a high-temperature hot plate. Then digest the covered mixture for 15 min at a temperature just below boiling. Filter the solution and proceed as in 9.3.2.

9.4 *Insoluble Residue Procedure:*

9.4.1 *Summary of Test Method*—The insoluble residue is determined by digestion of the sample in hydrochloric acid. The resulting residue is considered to be the siliceous components of the aggregate used in the mortar.

9.4.2 Add 50 mL HCl (1 + 3) to 1.00 g of the sample. Heat the covered mixture rapidly to near boiling on a high-temperature hot plate. Then digest the covered mixture for 15 min at a temperature just below boiling. Filter the solution through a dried and tared cellulose-nitrate membrane filter, washing the beaker, paper, and residue thoroughly with hot water. Dry the filter paper at 110°C for 10 min, cool in a desiccator to room temperature and determine mass.

9.4.3 *Calculation*—Calculate the percentage of the insoluble residue to the nearest 0.01 by multiplying the mass, in grams, of the residue by 100.

9.5 *Loss on Ignition:*

9.5.1 *Summary of Test Method*—The sample is dried at 110°C; this loss is assumed to represent free moisture. The sample is then ignited at 550°C; the loss from 110°C to 550°C is assumed to represent combined water. The sample is then ignited at 950°C; the loss from 550°C to 950°C is assumed to represent carbonates and carbonation.

9.5.2 Place 1.00 g of the sample in a tared porcelain crucible. Dry at 110 ± 5°C for a minimum of 30 minutes, cool

in a desiccator to room temperature and determine mass. Ignite at 550 ± 25°C for 15 ± 2 minutes, cool in a desiccator to room temperature and determine mass. Ignite at 950 ± 25°C for 15 ± 2 minutes, cool in a desiccator to room temperature and determine mass.

9.5.3 *Calculation*—Calculate the percentage of losses at the designated temperatures to the nearest 0.01 by multiplying the loss in mass in grams by 100.

9.6 *Magnesium Hydroxide Procedure:*

9.6.1 *Summary of Procedure*—If magnesium hydroxide (Mg(OH)₂) is detected by petrographic or XRD studies, the sample shall be analyzed by differential thermal analysis (DTA) or Thermal Gravimetric Analysis (TGA) to quantify the amount of magnesium hydroxide present. This information is used in determining the amount of dolomitic lime used in the mortar.

9.6.2 Analyze the sample quantitatively by methods suitable for the individual DTA equipment used.

10. Mortar Proportion Calculations

10.1 For typical mortar systems, based on the petrographic, X-ray diffraction and, where applicable, the differential thermal analysis data, determine the materials making up the mortar (for example, portland cement, masonry cement, calcitic hydrated lime, dolomitic hydrated lime, and siliceous, calcareous-siliceous, or calcareous aggregate).

10.1.1 *For Portland Cement-Calcitic Lime Mortars Containing Siliceous Aggregate:*

10.1.1.1 Portland cement is calculated from the soluble silica data (9.2.6) assuming 21.0 % soluble silica assignable to the portland cement. Hydrated lime is calculated from the calcium oxide data after correcting for the calcium oxide in the portland cement (assuming 63.5 % calcium oxide assignable to the portland cement). Sand is assumed to be the insoluble residue (9.4.3).

10.1.1.2 The mortar proportions are calculated assuming the portland cement mass as 94 lb/ft³ (1506 kg/m³), the lime as 40 lb/ft³ (641 kg/m³), and the sand as 80 lb/ft³ (1281 kg/m³). The as-received chemical analysis data can be used since the proportion of oxides in the mortar remain the same regardless of free moisture and combined moisture contents or carbonation of the sample.

10.1.1.3 If the original materials are available, calculations should be based on the actual oxide values.

10.1.1.4 *Example of Calculation*—Based upon the petrographic examination, the aggregate used in the mortar was a natural siliceous sand containing quartz as a major component and feldspar, orthoquartzite, and granite as minor to trace components. The mortar paste contained portland cement and hydrated lime, and the mortar had an estimated 3 % entrapped air content. Major amounts of quartz and minor to trace amounts of feldspar, calcium carbonate (CaCO₃), and calcium hydroxide (Ca(OH)₂) were detected by X-ray diffraction. (Note that the calcium carbonate detected in the mortar can be either fragmental limestone, carbonated portland cement hydration products, or carbonated hydrated lime. The chemical and petrographic analyses indicated that the calcium carbonate is carbonated hydrated lime.)