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Standard Test Method for Examination and Analysis of Hardened Masonry Mortar¹

This standard is issued under the fixed designation C1324; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 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:

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| Petrographic examination | 8 |
| Chemical analysis | 9 |
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~~1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.~~

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

2. Referenced Documents

2.1 ASTM Standards: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 Guide for Petrographic Examination of Aggregates for Concrete

C457 Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete

C823 Practice for Examination and Sampling of Hardened Concrete in Constructions

C856 Practice for Petrographic Examination of Hardened Concrete

C1084 Test Method for Portland-Cement Content of Hardened Hydraulic-Cement Concrete

D1193 Specification for Reagent Water

3. Terminology

3.1 Terms used in this test method are defined in Terminology C125 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

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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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.

masonry mortar. These components may include portland cement, hydrated calcitic or dolomitic lime, masonry cement, aggregates, and air.

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, Test Method C457, and Guide C295, 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, Test Method C457, and Practice C856.

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 $\text{mg}/\text{m}^3(1 + 3)$,

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 $\text{mg}/\text{m}^3(1+4)$.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.

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. Petrographic Examination

8.1 *General:*

8.1.1 Petrographic examination refers to methods in Practice C856 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 and Practice C856.

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. 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, 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. 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.

8.6 *Secondary Products*—Examine the mortar, using methods of Practice C856, 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 23—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 34—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 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:

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

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

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;

⁶Crushers and pulverizers may be obtained from many laboratory supply houses, and include chipmunk type jaw crushers, disc pulverizers, and rotating puck devices.

⁵The sole source of supply of the Stir-Kool Model SK-12 known to the committee at this time is Thermolectrics 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, which you may attend.