



Designation: C25 – 19

Standard Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime¹

This standard is issued under the fixed designation C25; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the chemical analysis of high-calcium and dolomitic limestone, quicklime, and hydrated lime. These test methods are classified as either standard (preferred) or alternative (optional).

1.2 The standard test methods are those that employ classical gravimetric or volumetric analytical procedures and are typically those required for referee analyses where chemical specification requirements are an essential part of contractual agreement between buyer and seller.

1.3 Alternative or optional test methods are provided for those who wish to use procedures shorter or more convenient than the standard methods for the routine determinations of certain constituents. Optional test methods may sometimes be preferred to the standard test methods, but frequently the use of modern and expensive instrumentation is indicated which may not be accessible to everyone. Therefore, the use of these test methods must be left to the discretion of each laboratory.

1.4 The analytical procedures appear in the following order:

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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. For specific precautionary statements, see 9.3, 10.2.1, 18.4.3, 31.6.4.2, X2.3.1, and X5.4.1.1.*

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

¹ These test methods are under the jurisdiction of ASTM Committee C07 on Lime and Limestone and are the direct responsibility of Subcommittee C07.05 on Chemical Tests.

Current edition approved May 1, 2019. Published July 2019. Originally approved in 1919. Last previous edition approved in 2017 as C25 – 17. DOI: 10.1520/C0025-19.

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.

- C50/C50M** Practice for Sampling, Sample Preparation, Packaging, and Marking of Lime and Limestone Products
- C51** Terminology Relating to Lime and Limestone (as used by the Industry)
- C911** Specification for Quicklime, Hydrated Lime, and Limestone for Selected Chemical and Industrial Uses
- D1193** Specification for Reagent Water
- E29** Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E50** Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
- E70** Test Method for pH of Aqueous Solutions With the Glass Electrode
- E173** Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)³
- E177** Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E200** Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis
- E691** Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E832** Specification for Laboratory Filter Papers

3. Terminology

3.1 *Definitions:* Definitions—Unless otherwise specified, for definitions of terms used in these test methods refer to Terminology **C51**.

4. Significance and Use

4.1 These test methods provide accurate and reliable analytical procedures to determine the chemical constituents of limestone, quicklime, and hydrated lime (see **Note 1**). The percentages of specific constituents which determine a material's quality or fitness for use are of significance depending upon the purpose or end use of the material. Results obtained may be used in relation to specification requirements.

4.2 Because quicklime and hydrated lime quickly absorb water and carbon dioxide from the air, precision and bias are extremely dependent upon precautions taken during sample preparation and analysis to minimize excessive exposure to ambient conditions.

NOTE 1—These test methods can be applied to other calcareous materials if provisions are made to compensate for known interferences.

5. General Apparatus and Materials and Reagents

5.1 General Apparatus and Materials:

5.1.1 *Balance*—The balance shall be of an analytical type with a capacity not to exceed 200 g. It may be of conventional design or it may be a constant-load, direct-reading type. It shall

be capable of reproducing weighings within 0.0002 g with an accuracy of ± 0.0002 g. Rapid weighing devices that may be provided such as a chain, damper, or heavy riders shall not increase the basic inaccuracy by more than 0.0001 g at any reading and with any load within the rated capacity of the balance.

5.1.2 *Weights*—Weights used for analysis shall conform to Class S-1 requirements of the National Institute of Standards and Technology as described in NIST Circular 547.⁴ They shall be checked at least once a year or when questioned, and adjusted to within allowable tolerances for Class S-1 weights. All new sets of weights purchased shall have the weights of 1 g and larger made of stainless steel or other corrosion-resistant alloy not requiring protective coating and shall meet the density requirements for Class S.

5.1.3 *Glassware and Laboratory Containers*—Standard volumetric flasks, burets, pipets, dispensers, and so forth, shall be carefully selected precision grade or better and shall be calibrated, if necessary, to meet the requirements of each operation. Standard-type interchangeable ground glass or TFE-fluorocarbon joints are recommended for all volumetric glassware. Polyethylene containers are recommended for all aqueous solutions of alkalis and for standard solutions where the presence of dissolved silica or alkali from the glass would be objectionable.

5.1.4 *Desiccators*—Desiccators shall be provided with a good desiccant such as anhydrous magnesium perchlorate, activated alumina, sulfuric acid, or phosphoric anhydride. Anhydrous calcium sulfate may also be used provided it has been treated with a color-changing indicator to show when the desiccant has lost its effectiveness. Calcium chloride and silica gel are not satisfactory desiccants for this type of analysis.

5.1.5 *Filter Paper*—Filter paper shall conform to the requirements of Specification **E832**, Type II (quantitative). Class E shall be used for coarse and gelatinous precipitates. When medium-textured paper is required, Class F filter paper shall be used. When a retentive paper is needed, Class G shall be used.

Recommendations:

Class	Filter Pore Size (μm)	Filter Speed
E	20 to 25	fast speed
F	8	medium speed
G	2.5	slow speed

5.1.6 *Crucibles*—Platinum crucibles and tight fitting lids should preferably be made of pure unalloyed platinum and be of 25 to 35-mL capacity. Where alloyed platinum is used for greater stiffness or to obviate sticking of fused material to crucible or lid, the alloyed platinum should not decrease in weight by more than 0.2 mg when heated at 1200 °C for 1 h.

5.1.7 *Muffle Furnace*—The electric muffle furnace should be capable of continuous operation up to 1000 °C and be capable of intermittent operation at higher temperatures if required. It should have an indicating pyrometer accurate to ± 25 °C.

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

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 3460, Gaithersburg, MD 20899-3460.

5.2 Reagents:

5.2.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society⁵ where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. In addition to this, it is desirable in many cases for the analyst to ensure the accuracy of his results by running blanks or checking against a comparable sample of known composition.

5.2.2 Purity of Water—Unless otherwise indicated, references to water are understood to mean distilled water or other water of equivalent purity. Water conforming to Specification **D1193** meets these requirements.

5.2.3 Concentration of Reagents:

5.2.3.1 Concentrated Acids and Ammonium Hydroxide—When acids and ammonium hydroxide are specified by name or chemical formula only, it shall be understood that concentrated reagents approximating the following specific gravities or concentrations are intended:

Acetic acid (HC ₂ H ₃ O ₂)	99.5 %
Hydrochloric acid (HCl)	sp gr 1.19
Hydrofluoric acid (HF)	48 %
Nitric acid (HNO ₃)	sp gr 1.42
Perchloric acid (HClO ₄)	70 %
Phosphoric acid (H ₃ PO ₄)	85 %
Sulfuric acid (H ₂ SO ₄)	sp gr 1.84
Ammonium hydroxide (NH ₄ OH)	sp gr 0.90

5.2.3.2 Dilute Reagents—The concentration of dilute acids and NH₄OH except when standardized, are specified as a ratio stating the number of measured volumes of the concentrated reagent to be diluted with a given number of measured volumes of water. In conformance with international practice, new and revised methods will use the “plus” designation instead of the ratio (:) symbol as the specified designation of dilution; for example, H₂SO₄ (5 + 95) means 5 volumes of concentrated H₂SO₄ (sp gr 1.84) diluted with 95 volumes of water.

5.2.3.3 Standard Solutions—Concentrations of standard solutions shall be expressed as normalities (*N*) or as equivalents in grams per millilitre of the component to be determined, for example: 0.1 *N* K₂Cr₂O₇ solution (1 mL = 0.004 g Fe₂O₃). The average of at least three determinations shall be used for all standardizations. The standardization used to determine the strength of the standard solutions is described in the text under each of the appropriate procedures.

6. General Procedures

6.1 Sampling—Samples of lime and limestone for chemical analysis shall be taken and prepared in accordance with the requirements of Practice **C50/C50M** applicable to the material to be tested.

6.2 Tared or Weighed Crucibles—The tare weight of crucibles shall be determined by preheating the empty crucible to constant weight at the same temperature and under the same conditions as shall be used for the final ignition of a residue and cooling in a desiccator for the same period of time used for the crucible containing the residue.

6.3 Constancy of Weight of Ignited Residue—To definitely establish the constancy of weight of the ignited residue, the residue and container shall be ignited at the specified temperature and time, cooled to room temperature in a desiccator, and weighed. The residue and container shall then be reheated for at least 30 min at the same temperature, cooled in a desiccator for the same period of time, and reweighed. Additional ignition periods may be required until two consecutive weights do not differ by more than 0.2 mg, at which time it shall be considered that constant weight has been attained. For ignition loss, each reheating period shall be 5 min.

6.4 Calculation:

6.4.1 The calculations included in the individual procedures sometimes assume that the exact weight specified has been used. Accurately weighed samples which are approximately but not exactly equal to the weight specified may be used provided appropriate corrections are made in the calculation. Unless otherwise stated, weights of all samples and residues should be recorded to the nearest 0.0001 g.

6.4.2 In all mathematical operations on a set of observed values, the equivalent of two more places of figures than in the single observed values shall be retained. For example, if observed values are read or determined to the nearest 0.1 mg, carry numbers to the nearest 0.001 mg in calculation.

6.5 Rounding Figures—Rounding figures to the nearest significant place required in the report should be done after the calculations are completed, in order to keep the final results free from calculation errors. The rounding procedure should follow the principle outlined in Practice **E29**.

7. Performance Requirements for Test Methods

7.1 Referee Analyses—The reference test methods that appear in Sections 8 through 32, or any other test methods qualified in accordance with 7.3, are required for referee analysis in those cases where conformance to the requirements of a chemical specification are questioned. In these cases a limestone, quicklime, or hydrated lime shall not be rejected for failure to conform to chemical requirements unless all sample preparation and analysis of any one constituent is made entirely by reference test methods prescribed in the appropriate sections of this test method or by other qualified test methods. Exception can be made when specific test methods are prescribed in the standard specification for the limestone, quicklime, or hydrated lime in question. The test methods actually used for the analysis shall be designated.

7.1.1 When there is a question regarding acceptance, referee analyses shall be made in duplicate. If the two results do not agree within the permissible variation given in Table 1, the determination including sample preparation shall be repeated in duplicate until the results agree within the permissible variation. When the results agree within the permissible

⁵ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeia Convention, Inc. (USPC), Rockville, MD.

TABLE 1 Maximum Permissible Variations in Results^A

(Column 1)	(Column 2)	(Column 3)
Constituent	Maximum Difference Between Duplicates	Maximum Difference of the Average of Duplicates from CRM Certificate Values ^B
Al as Al ₂ O ₃	0.20	±0.30
Ca as CaO	0.20	±0.30
Mg as MgO	0.20	±0.30
C (lime and hydrated lime)	0.20	±0.30
C (limestone)	0.60	±0.60
Fe as Fe ₂ O ₃	0.10	±0.15
Si as SiO ₂	0.15	±0.30
Mn	0.05	±0.10
P	0.02	±0.05
Sr as SrO	0.05	^C
S	0.03	±0.05

^A For demonstrating the performance of rapid test methods the CRM closest in overall composition to the limestone shall be used (see Table 2). In the case of quicklime or hydrated lime, the CRM closest in overall composition, after heating at 1000 °C for 1 h, to the product composition shall be used, except for C and S determinations (see Note 3).

^B Interlelement corrections may be used for any standardization provided improved accuracy can be demonstrated.

^C No CRM currently available.

variation, their average shall be accepted as the correct value. For the purpose of comparing results, the percentages shall be calculated to one more significant figure than reported as indicated in the test methods. When a blank determination is specified, one shall be made with each individual analysis or with each group of two or more samples analyzed on the same day for a given constituent.

7.1.2 Test results from Referee methods intended for use as a basis for product acceptance or rejection, or for manufacturer's certification, can be used only after demonstration of precise and accurate analyses by meeting the requirements of 7.1.3, or except when demonstrated under 7.3.2.1. Such demonstrations may be made concurrently with analysis of the limestone, quicklime, or hydrated lime product being tested. The demonstration is required only for those constituents being used as a basis for acceptance, rejection, or certification of a limestone, quicklime, or hydrated lime, but may be made for any constituent of limestone, quicklime, or hydrated lime product for which a standard exists. Such demonstrations must be made annually.

7.1.3 Demonstrations shall be made by analysis of each constituent of concern in a CRM limestone, quicklime, or hydrated lime (see Notes 2 and 3). Duplicate samples shall be run on different days. The same test methods to be used for analysis of the limestone, quicklime, or hydrated lime being tested shall be used for analysis of the CRM. If the duplicate results do not agree within the permissible variation given in Table 1, the determinations shall be repeated, following identification and correction of problems or errors, until a set of duplicate results do agree within the permissible variation.

NOTE 2—The term CRM refers to approved Certified Reference Materials listed in Table 2.

NOTE 3—There are no CRMs that are quicklime or hydrated lime as supplied. When analyzing a quicklime or hydrated lime the CRM in carbonate form needs to be converted to closely resemble the matrix of the product being tested. To accomplish this conversion, heat the chosen CRM for 1 h at 1000 °C, immediately prior to analysis and protect it from

hydration or carbonation with sealed containers and desiccation during cooling. Carbon and sulfur may be driven off during heating, rendering the converted CRM unsuitable as a standard for carbon and sulfur determinations. For carbon and sulfur determinations use the appropriate CRM in its normal matrix.

7.1.4 The average of the results of acceptable duplicate determinations for each constituent may differ from the CRM certificate value by no more than the value shown in Column 3 of Table 1. When no CRM certificate value is given, a generally accepted accuracy standard for that constituent has not been identified. In such cases, only the differences between duplicate values as specified in 7.1.3 shall apply and notification of this exception shall be reported.

7.1.5 In questions concerning the acceptance or rejection of a limestone, quicklime, or hydrated lime product, upon request data shall be made available to all parties involved demonstrating that precise and accurate results were obtained with CRM samples by the same analyst making the acceptance determination.

7.2 *Optional Analyses*—The alternative test methods, as opposed to reference methods, provide procedures that are, in some cases, shorter or more convenient to use for routine determination of some constituents (see Note 4). In some instances longer, more complex procedures have been retained as alternative test methods to permit comparison of results by different procedures or for use when unusual materials are being examined, or when unusual preparation for analysis is required. Results from alternative test methods may be used as a basis for acceptance or rejection.

NOTE 4—It is not intended that the use of reference test methods be confined to referee analysis. A reference test method may be used in preference to an alternative test method when so desired. A reference test method must be used where an alternative test method is not provided.

7.2.1 Duplicate analyses and blank determinations are left to the discretion of the analyst when using the alternative test methods. The final results should include the number of determinations performed and whether or not they were corrected for blank values.

7.3 *Performance Requirements for Alternative Test Methods:*

7.3.1 *Definition and Scope*—When analytical data obtained in accordance with this section is required, any test method may be used that meets the requirements of 7.3.2. A test method is considered to consist of the specific procedures, reagents, supplies, equipment, instrument, and so forth, selected and used in a consistent manner by a specific laboratory.

7.3.1.1 If more than one instrument is used for the same analysis, use of each instrument shall constitute a separate test method and each must be qualified separately.

7.3.2 *Qualification of a Test Method*—Prior to use each test method (see 7.3.1) must be qualified for each material that will be tested. Qualification data or, if applicable, requalification data shall be made available.

7.3.2.1 Using the test method chosen, make single determinations for each constituent under consideration on the CRM which in overall composition most closely resembles the limestone, quicklime, or hydrated lime to be tested (see Note 2). Complete two rounds of tests on nonconsecutive days