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# **Standard Method of Test for Chemical Analysis of Hydraulic Cement**

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**AASHTO Designation: T 105-21<sup>1</sup>**

Technically Revised: 2021

Editorially Revised: 2021

**Technical Subcommittee: 3a, Hydraulic Cement and Lime**

**ASTM Designation: C114-18**



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## 1. SCOPE

1.1. These test methods cover the chemical analyses of hydraulic cements. Any test methods of demonstrated acceptable precision and bias may be used for analysis of hydraulic cements, including analyses for referee and certification purposes, as explained in Section 4. Specific chemical test methods are provided for ease of reference for those desiring to use them. They are grouped as Reference Test Methods and Alternate Test Methods. The reference test methods are long accepted classical chemical test methods, which provide a reasonably well-integrated basic scheme of analysis for hydraulic cements. The alternative test methods generally provide individual determination of specific analytes and may be used alone or as alternates and determinations within the basic scheme at the option of the analyst and as indicated in the individual method. The individual analyst must demonstrate achievement of acceptable precision and bias, as explained in Section 4, when these methods are used.

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**Appendixes**

Appendix X1	Example of Determination of Equivalence Point for the Chloride Determination
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1.3. The values stated in SI units are to be regarded as the standard.

1.4. *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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. See Sections 8.3.2.1 and 16.4.1 for specific caution statements.*

- 1.5. *The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of R 18 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with R 18 alone does not completely assure reliable results. Reliable results depend on many factors; following the suggestions of R 18 or some similar acceptable guideline provides a means of evaluating and controlling some of those factors.*

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## 2. REFERENCED DOCUMENTS

- 2.1. *AASHTO Standard:*
- R 18, Establishing and Implementing a Quality Management System for Construction Materials Testing Laboratories
- 2.2. *ASTM Standards:*
- C25, Standard Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime
  - E29, Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
  - E275, Standard Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers
  - E350, Standard Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron
  - E617, Standard Specification for Laboratory Weights and Precision Mass Standards
  - E832, Standard Specification for Laboratory Filter Papers

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## 3. TERMINOLOGY

- 3.1. *Definition:*
- 3.1.1. *analyte, n*—A substance of interest when performing a quantitative analysis.
- 3.1.1.1. *Discussion*—For the purposes of this test method, analytes are considered to be those items listed in Column 1 of Table 1.
- 3.1.2. *reagent water, n*—water purified by the process of distillation, deionization, reverse osmosis, or any combination of the three processes.
- 3.1.2.1. *distillation, n*—the process of purification by the evaporation and vaporization of water and its subsequent condensation and collection.
- 3.1.2.2. *deionization, n*—the process of purification using the two-step process of converting soluble salts into acids by passing them through a hydrogen exchanger, after which they are removed by an acid absorbent or synthetic resin.
- 3.1.2.3. *reverse osmosis, n*—water purification technology that uses a semipermeable membrane to remove ions, molecules, and larger particles from drinking water.
- 3.1.3. *water (potable), n*—water that is suitable for drinking.

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## 4. DESCRIPTION OF REFEREE ANALYSES

4.1. *Referee Analyses*—When conformance to chemical specification requirements is questioned, perform referee analyses as described in Section 4.1.1. The reference test methods that follow in Sections 7 through 22, or other test methods qualified according to Section 5.4, Performance Requirements for Rapid Test Methods, are required for referee analysis. A cement shall not be rejected for failure to conform to chemical requirements unless all determinations of constituents involved and all necessary separations prior to the determination of any one constituent are made entirely by these methods. When reporting the results of referee analyses, specify which test methods were used.

4.1.1. Referee analyses shall be made in duplicate and on different days. If the two results do not agree within the permissible variation given in Table 1, the determination shall be repeated until two or three results agree within the permissible variation. When two or three results do agree within the permissible variation, their average shall be accepted as the correct value. When an average of either two or three results can be calculated, the calculation shall be based on the three results. For the purpose of comparing analyses and calculating the average of acceptable results, the percentages shall be calculated to the nearest 0.01 (or 0.001 in the case of chloroform-soluble organic substances), although some of the average values are reported to 0.1 as indicated in the test methods. When a blank determination (see Note 1) 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 analyte.

**Note 1**—A blank determination is a procedure that follows all steps of analysis, but in the absence of a sample. It is used for detection and compensation of systematic bias.

**Table 1—Maximum Permissible Variation in Results<sup>a</sup>**

(Column 1) Analyte	(Column 2) Maximum Difference between Duplicates <sup>b</sup>	(Column 3) Maximum Difference of the Average of Duplicates from CRM Certificate Values <sup>b,c,d</sup>
SiO <sub>2</sub> (silicon dioxide)	0.16	±0.20
Al <sub>2</sub> O <sub>3</sub> (aluminum oxide)	0.20	±0.20
Fe <sub>2</sub> O <sub>3</sub> (ferric oxide)	0.10	±0.10
CaO (calcium oxide)	0.20	±0.30
MgO (magnesium oxide)	0.16	±0.20
SO <sub>3</sub> (sulfur trioxide)	0.10	±0.10
LOI (loss on ignition)	0.10	±0.10
Na <sub>2</sub> O (sodium oxide)	0.03	±0.05
K <sub>2</sub> O (potassium oxide)	0.03	±0.05
TiO <sub>2</sub> (titanium dioxide)	0.02	±0.03
P <sub>2</sub> O <sub>5</sub> (phosphorus pentoxide)	0.03	±0.03
ZnO (zinc oxide)	0.03	±0.03
Mn <sub>2</sub> O <sub>3</sub> (manganic oxide)	0.03	±0.03
S (sulfide sulfur)	0.01	<sup>e</sup>
Cl (chloride)	0.003	±0.005 <sup>e</sup>
IR (insoluble residue)	0.10	<sup>e</sup>
Cx (free calcium oxide)	0.20	<sup>e</sup>
CO <sub>2</sub> (carbon dioxide)	0.12	<sup>e, f</sup>
Alk <sub>sol</sub> (water-soluble alkali) <sup>g</sup>	0.75/ <i>w</i> <sup>g</sup>	<sup>e</sup>
Chl <sub>sol</sub> (chloroform-soluble organic substances)	0.004	<sup>e</sup>

<sup>a</sup> When all seven Certified Reference Material (CRM) cements are required, as for demonstrating performance of rapid test methods, at least six of the seven shall be within the prescribed limits, and the seventh shall differ by no more than twice that value. When more than seven CRMs are used, as for demonstrating the performance of rapid test methods, at least 77 percent shall be within the prescribed limits, and the remainder no more than twice the value. When a lesser number of CRM cements are required, all of the values shall be within the prescribed limits.

<sup>b</sup> Where no value appears in Column 3, CRM certificate values do not exist. In such cases, only the requirement for differences between duplicates shall apply.

<sup>c</sup> Interelement corrections may be used for any oxide standardization provided improved accuracy can be demonstrated when correction is applied to all seven CRM cements.

<sup>d</sup> Where a CRM certificate value includes a subscript number, that subscript number shall be treated as a valid significant figure.

<sup>e</sup> Not applicable. No certificate value given.

<sup>f</sup> Demonstrate performance by analysis, in duplicate, of at least one portland cement. Prepare three standards, each in duplicate: Standard A shall be selected portland cement, Standard B shall be Standard A containing 2.00 percent Certified CaCO<sub>3</sub> (e.g., NIST 915a), Standard C shall be Standard A containing 5.00 percent Certified CaCO<sub>3</sub>. Weigh and prepare two separate specimens of each standard. Assign the CO<sub>2</sub> content of Standard A as the average of the two values determined, provided they agree within the required limit of Column 2. Assign CO<sub>2</sub> values to Standards B and C as follows: Multiply the Certified CaCO<sub>3</sub> value (*Y*) for CO<sub>2</sub> (from the certificate value) by the mass fraction of Certified CaCO<sub>3</sub> added to that standard (percentage added divided by 100); multiply the value determined for Standard A by the mass fraction of Standard A in each of the other standards (i.e., 0.98 and 0.95 for Standards B and C, respectively); add the two values for Standards A and B, respectively; call these values *B* and *C*.

Example:

$$B = 0.98A + 0.02Y.$$

$$C = 0.95A + 0.05Y.$$

where for Certified CaCO<sub>3</sub>, if *Y*= 39.9 percent

$$B = 0.98A + 0.80 \text{ percent by mass.}$$

$$C = 0.95A + 2.00 \text{ percent by mass.}$$

Maximum difference between the duplicate CO<sub>2</sub> values for Standards B and C, respectively, shall be 0.17 and 0.24 percent by mass.

Averages of the duplicate values for Standards B and C shall differ from their assigned values (*B* and *C*) by no more than 10 percent of those respective assigned values.

<sup>g</sup> *w* = mass, in grams, of samples used for the test.

## 5. QUALIFICATION FOR DIFFERENT ANALYSES

- 5.1. *Certified Reference Materials*—A Certified Reference Material (CRM) must be used in the qualification of test methods and analysts. Acceptable reference cements are National Institute of Standards and Technology (NIST) CRMs or other reference cements traceable to the NIST CRMs. The reference cement must have an assigned value for the analyte being determined. Traceability consists of documentary evidence that the assigned values of the reference cement are compatible with the certified values of NIST CRMs. To demonstrate traceability for a given analyte, perform a referee analysis (as defined in Section 4.1) on the proposed reference cement, using an NIST CRM for demonstration of precision and accuracy. The reference cement is acceptable if its assigned value agrees with the average referee value within the limits given in Column 3 of Table 1. If the reference cement, as supplied, has no documented guarantee of homogeneity, establish its homogeneity by analyzing at least six randomly selected samples. No result shall deviate from the assigned value by more than the limits given in Column 2 of Table 1. An acceptable reference cement must be accompanied by a document showing the data produced in demonstrating traceability and homogeneity.
- 5.2. *Requirements for Qualification Testing*—Qualified test methods are required whenever testing is performed for the following reasons: (1) Referee analyses, (2) analyses intended for use as a basis for acceptance or rejection of a cement, or (3) manufacturer's certification. When Reference methods are used, qualification testing of the analyst is required as described in Section 5.2.1. When Rapid methods are used, qualification testing of both the analyst and the test method are required as described in Sections 5.2.1 and 5.4. Such demonstration may be made concurrently with analysis of the cement being tested. The requirements for qualification of a test method and analyst are summarized in Table 2.

**Table 2**—Minimum Number of CRMs Required for Qualification of Chemical Testing

	Method Type	
	Reference <sup>a</sup>	Other <sup>b</sup>
Equipment Qualification	None	7
Operator Qualification <sup>c</sup>	1	1

<sup>a</sup> Reference methods are those outlined in Sections 7 through 22.

<sup>b</sup> These may be any test method as described in Section 5.3, Alternative Analyses, or any instrumental or rapid test method, which must be qualified in accordance with Section 5.4, Performance Requirements for Rapid Test Methods.

<sup>c</sup> Each analyst performing acceptance or reference analyses must be qualified in accordance with Section 5.2.1, Performance Requirements for Rapid Test Methods, at a frequency of 2 years. If qualification of the instrument is completed by a single analyst, the analyst has demonstrated individual qualifications per Section 5.2.1.

Initial qualification of the analyst shall be demonstrated by analysis of each constituent of concern in at least one CRM cement in duplicate, no matter what test method is used (Note 2)). Duplicate samples shall be run on different days. The same test methods to be used for analysis of cement being tested shall be used for analysis of the CRM cement. 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. Requalification of the analyst is required every two years.

**Note 2**—When qualifying a rapid method with seven CRMs in accordance with Section 5.4.2, the analyst performing the qualification of the test method may simultaneously qualify for the requirement of Section 5.2.1.

- 5.2.1. The average of the results of acceptable duplicate determinations for each constituent may differ from the CRM assigned value by no more than the value shown in Column 3 of Table 1 after necessary corrections for minor analytes are made.
- 5.2.2. Qualification data demonstrating that the same operator or analyst making the acceptance determination obtained precise and accurate results with CRM cements as per Section 5.4 shall be

made available on request to all parties concerned when there is a question of acceptance of a cement. If the CRM used is not an NIST cement, the traceability documentation of the CRM used shall also be made available on request.

- 5.3. *Alternative Analyses*—The alternative test methods provide, in some cases, procedures that are shorter or more convenient to use for routine determination of certain constituents than are the reference test methods (see Note 3). Longer, more complex procedures, in some instances, have been retained as alternative test methods to permit comparison of results by different procedures or for use when unusual materials are being examined, where unusual interferences may be suspected, or when unusual preparation for analysis is required. Test results from alternative test methods may be used as a basis for acceptance or rejection when it is clear that a cement does or does not meet the specification requirement. Any change in test method procedures from those procedures listed in Sections 7 through 30 requires method qualification in accordance with Section 5.4, Performance Requirements for Rapid Test Methods.

**Note 3**—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.

- 5.3.1. Duplicate analyses and blank determinations are not required when using the alternative test methods. If, however, a blank determination is desired for an alternative test method, one may be used, and it need not have been obtained concurrently with the analysis. The final results, when corrected for blank values, should, in either case, be so designated.

- 5.4. *Performance Requirements for Rapid Test Methods*<sup>2, 3</sup>:

- 5.4.1. *Definition and Scope*—When analytical data obtained in accordance with this test method are required, any test method may be used that meets the requirements of Section 5.4.2, Qualification of a Test Method. 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. See Note 4 for examples of procedures.

**Note 4**—Examples of test methods used successfully by their authors for analysis of hydraulic cement are given in the list of references. Included are test methods using atomic absorption X-ray spectrometry and spectrophotometry-EDTA.

- 5.4.1.1. If more than one instrument, even though substantially identical, is used in a specific laboratory for the same analyses, use of each instrument shall constitute a separate test method and each must be qualified separately.

- 5.4.2. *Qualification of a Test Method*—Prior to use for analysis of hydraulic cement, each test method (see Section 5.4.1) must be qualified individually for such analysis. Qualification data, or if applicable, requalification data, shall be made available pursuant to the Manufacturer's Certification section of the appropriate hydraulic cement specification.

- 5.4.2.1. Using the test method chosen, make single determinations for each analyte under consideration on at least seven CRM samples. Requirements for a CRM are listed in Section 5.1, Certified Reference Materials. Complete two rounds of tests on different days repeating all steps of sample preparations. Calculate the differences between values and averages of the values from the two rounds of tests.

- 5.4.2.2. When seven CRMs are used in the qualification procedure, at least six of the seven differences between duplicates obtained of any single analyte shall not exceed the limits shown in Column 2 of Table 1 and the remaining differences by no more than twice that value. When more than seven CRMs are used, the values for at least 77 percent of the samples shall be within the prescribed limits, while the values for the remainder shall differ by no more than twice that value.