
Standard Method of Test for Chemical Analysis of Hydraulic Cement

AASHTO Designation: T 105-22¹

Technically Revised: 2022

Technical Subcommittee: 3a, Cement, Lime, and Concrete Materials

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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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.*

2. REFERENCED DOCUMENTS

2.1. *AASHTO Standard:*

- M 339M/M 339, Thermometers Used in the Testing of Construction Materials
- 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
- E1, Standard Specification for ASTM Liquid-in-Glass Thermometers
- E29, Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E230/E230M, Standard Specification for Temperature-Electromotive Force (emf) Tables for Standardized Thermocouples
- 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
- E879, Standard Specification for Thermistor Sensors for General Purpose and Laboratory Temperature Measurements
- E2877, Standard Guide for Digital Contact Thermometers

2.3. *International Electrotechnical Commission Standard:*

- IEC 60584-1:2013 Thermocouples - Part 1: EMF Specifications and Tolerances

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.

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^a

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

^a 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.

^b 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.

^c Interlement corrections may be used for any oxide standardization provided improved accuracy can be demonstrated when correction is applied to all seven CRM cements.

^d Where a CRM certificate value includes a subscript number, that subscript number shall be treated as a valid significant figure.

^e Not applicable. No certificate value given.

^f 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 CaCO₃ (e.g., NIST 915a), Standard C shall be Standard A containing 5.00 percent Certified CaCO₃. Weigh and prepare two separate specimens of each standard. Assign the CO₂ content of Standard A as the average of the two values determined, provided they agree within the required limit of Column 2. Assign CO₂ values to Standards B and C as follows: Multiply the Certified CaCO₃ value (*Y*) for CO₂ (from the certificate value) by the mass fraction of Certified CaCO₃ 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 CaCO₃, 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 CO₂ 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.

^g *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 ^a	Other ^b
Equipment Qualification	None	7
Operator Qualification ^c	1	1

^a Reference methods are those outlined in Sections 7 through 22.

^b 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.

^c 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*^{2, 3}:

- 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.

- 5.4.2.3. For each analyte and each CRM, the average obtained shall be compared to the certified concentrations. Where a certificate value includes a subscript number, that subscript shall be assumed to be a significant number. When seven CRMs are used in the qualification procedure, at least six of the seven averages for each analyte shall not differ from the certified concentrations by more than the value shown in Column 3 of Table 1, and the remaining average by more than twice that value. When more than seven CRMs are used in the qualification procedure, at least 77 percent of the averages for each analyte shall not differ from the certified concentrations by more than the value shown in Column 3 of Table 1, and the remaining average(s) by more than twice that value.
- 5.4.2.4. The standardization, if needed, used for qualification and analysis of each constituent shall be determined by valid curve-fitting procedures. A point-to-point, sawtooth curve that is artificially made to fit a set of data points does not constitute a valid curve-fitting procedure. A complex polynomial drawn through the points is similarly not valid. For the same reason, empirical interelement corrections may be used only if $\leq (N - 3)/2$ is employed, where N is the number of different standards used. The qualification testing shall be conducted with specimens newly prepared from scratch, including all the preparation stages applicable for analysis of an unknown sample, and employing the reagents currently in use for unknown analyses.
- 5.4.3. *Partial Results*—Test methods that provide acceptable results for some analytes but not for others may be used only for those analytes for which acceptable results are obtained.
- 5.4.4. *Report of Results*—When performing chemical analysis and reporting results for Manufacturer's Certification, the type of method (Reference or Rapid) and the test method used, along with any supporting qualification testing, shall be available on request.
- 5.4.5. *Rejection of Material*—See Section 4.1, Referee Analyses, and Section 5.3, Alternative Analyses.
- 5.4.6. *Requalification of a Test Method:*
- 5.4.6.1. Requalification of a test method shall be required upon receipt of substantial evidence that the test method may not be providing data in accordance with Table 1 for one or more constituents. Such requalification may be limited to those constituents indicated to be in error and shall be carried out prior to further use of the method for analysis of those constituents.
- 5.4.6.2. Substantial evidence that a test method may not be providing data in accordance with Table 1 shall be considered to have been received when a laboratory is informed that analysis of the same material by Reference Test Methods run in accordance with Section 4.1.1, the final average of a Cement and Concrete Reference Laboratory (CCRL) sample, a certificate value of an NIST CRM, the assigned value of an alternate CRM, or an accepted value of a known secondary standard differs from the value obtained by the test method in question by more than twice the value shown in Column 2 of Table 1 for one or more constituents. When indirect test methods are involved, as when a value is obtained by difference, corrections shall be made for minor constituents to put analyses on a comparable basis prior to determining the differences. For any constituents affected, a test method also shall be requalified after any substantial repair or replacement of one or more critical analytes of an instrument essential to the test method.
- 5.4.6.3. If an instrument or piece of equipment is replaced, even if by one of identical make or model, or is significantly modified, a previously qualified test method using such new or modified instrument or equipment shall be considered a new method and must be qualified in accordance with Section 5.4.2.
- 5.4.7. *Precision and Bias*—Different analytical test methods are subject to individual limits of precision and bias. It is the responsibility of the user to demonstrate that the test methods used at least meet the limits of precision and bias shown in Table 1.

6. GENERAL

6.1. *Interferences and Limitations:*

- 6.1.1. These test methods were developed primarily for the analysis of portland cements. However, except for limitations noted in the procedure for specific constituents, the reference test methods provide for accurate analyses of other hydraulic cements that are completely decomposed by hydrochloric acid, or where a preliminary sodium carbonate fusion is made to ensure complete solubility. Some of the alternative test methods may not always provide accurate results because of interferences from elements that are not removed during the procedure.

Note 5—Instrumental analyses can usually detect only the element sought. Therefore, to avoid controversy, the actual procedure used for the elemental analyses should be noted when actual differences with reference procedures can exist. For example, P_2O_5 and TiO_2 are included with Al_2O_3 in the usual wet test method and sulfide sulfur is included in most instrumental procedures with SO_3 .

- 6.1.2. When using a test method that determines total sulfur, such as most instrumental test methods, sulfide sulfur will be determined with sulfate and included as such. In most hydraulic cements, the difference resulting from such inclusion will be insignificant, less than 0.05 weight percent. In some cases, notably slags and slag-containing cements but sometimes other cements as well, significant levels of sulfide may be present. In such cases, especially if there is a question of meeting or not meeting a specification limit or when the most accurate results are desired, analytical test methods shall be chosen so that sulfate and sulfide can be reported separately.

Where desired, when using instrumental test methods for sulfate determination, if sulfide has been determined separately, correct the total sulfur results (expressed as an oxide) in accordance with the following calculation:

$$SO_3 = S_{\text{total}} - (2.5S^-) \quad (1)$$

where:

SO_3 = sulfur trioxide excluding sulfur,

S_{total} = total sulfur in the sample, expressed as the oxide, from instrumental results,

2.5 = molecular ratio of SO_3/S^- to express sulfur as SO_3 , and

S^- = sulfide sulfur present.

6.2. *Apparatus and Materials:*

- 6.2.1. *Balance*—The analytical balance used in the chemical determinations shall conform to the following requirements:

- 6.2.1.1. The balance shall be capable of reproducing results within 0.0002 g, with an accuracy of ± 0.0002 g. Direct-reading balances shall have a sensitivity not exceeding 0.0001 g (see Note 6). Conventional two-pan balances shall have a maximum sensibility reciprocal of 0.0003 g. Any rapid weighing device that may be provided, such as a chain, damped motion, 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.

Note 6—The sensitivity of a direct-reading balance is the weight required to change the reading one graduation. The sensibility reciprocal for a conventional balance is defined as the change in weight required on either pan to change the position of equilibrium one division on the pointer scale at capacity or at any lesser load.

- 6.2.2. *Weights*—Weights used for analysis shall conform to Types I or II, Grades S or O, Classes 1, 2, or 3, as described in ASTM E617. They shall be checked at least once a year, or when questioned, and adjusted at least to within allowable tolerances for Class 3 weights (see Note 7). For this purpose, each laboratory shall also maintain, or have available for use, a reference set of standard

weights from 50 g to 10 mg, which shall conform at least to Class 3 requirements and be calibrated at intervals not exceeding 5 years by the NIST. After initial calibration, recalibration by the NIST may be waived, provided it can be shown by documented data obtained within the time interval specified that a weight comparison between summations of smaller weights and a single larger weight nominally equal to that summation establishes that the allowable tolerances have not been exceeded. All new sets of weights purchased shall have the weights of 1 g and larger made of stainless steel or other corrosion-resisting alloy not requiring protective coating, and shall meet the density requirements for Grades S or O.

Note 7—The scientific supply houses do not presently list weights as meeting ASTM E617. They list weights as meeting NIST or International Organization of Legal Metrology (OIML) standards. The situation with regard to weights is in a state of flux because of the trend toward internationalization. Hopefully, this will soon be resolved. NIST Classes S and S-1 and OIML Class F₁ weights meet the requirements of this standard.

- 6.2.3. *Glassware and Laboratory Containers*—Standard volumetric flasks, burets, and pipets should be of precision grade or better. Standard-taper, interchangeable, ground-glass joints are recommended for all volumetric glassware and distilling apparatus, when available. Wherever applicable, the use of special types of glassware (e.g., colored glass for the protection of solutions against light, alkali-resistant glass, and high-silica glass having exceptional resistance to thermal shock) is recommended. 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. Such containers shall be made of high-density polyethylene having a wall thickness of at least 1 mm.
- 6.2.4. *Desiccators*—Desiccators shall be provided with a good desiccant such as magnesium perchlorate, activated alumina, or sulfuric acid. Anhydrous calcium sulfate may also be used, provided it has been treated with a color-change indicator to show when it has lost its effectiveness. Calcium chloride is not a satisfactory desiccant for this type of analysis.
- 6.2.5. *Filter Paper*—Filter paper shall conform to the requirements of ASTM E832, Type II, Quantitative. When coarse-textured paper is required, Class E paper shall be used; when medium-textured paper is required, Class F paper shall be used; and when retentive paper is required, Class G paper shall be used.
- 6.2.6. *Crucibles*:
- 6.2.6.1. *Platinum Crucibles*—For ordinary chemical analysis, platinum crucibles should preferably be made of pure unalloyed platinum and be of 15 to 30 mL capacity. Where alloyed platinum is used for greater stiffness or to obviate sticking of crucible and lid, the alloyed platinum shall not decrease in weight by more than 0.2 mg when heated at 1200°C for 1 h.
- 6.2.6.2. *Porcelain Crucibles*—Should be glazed inside and out, except for the outside bottom and rim, and be of 5–15 mL capacity.
- 6.2.7. *Muffle Furnace(s)*—The muffle furnace(s) shall be capable of operation at the temperatures required, between 800 and 1200°C, and shall have an indicating pyrometer accurate within $\pm 25^\circ\text{C}$, as corrected, if necessary, by calibration. More than one furnace may be used, provided each is used within its proper operating temperature range. When measuring temperature during use, the thermometer for measuring the temperature of materials shall meet the requirements of M 339M/M 339 with a temperature range of at least 800 to 1200°C, and an accuracy of $\pm 12.5^\circ\text{C}$ (see Note 8).

Note 8—Thermometer types suitable for use include ASTM E1 mercury thermometers; IEC 60584 thermocouple thermometer Type K, any Class; or ASTM E230/E230M thermocouple thermometer, Type K, any Class.

6.2.8. *Oven(s)*—The ovens(s) shall be capable of operation at the temperatures required, between 55 to 200°C, within $\pm 5^\circ\text{C}$, as corrected, if necessary, by calibration. More than one oven may be used, provided each is used within its proper operating temperature range. The thermometer for measuring the temperature of materials shall meet the requirements of M 339M/M 339 with a temperature range of at least 55 to 200°C, and an accuracy of $\pm 2.2^\circ\text{C}$ (see Note 9).

Note 9—Thermometer types suitable for use include ASTM E1 mercury thermometers; IEC 60584 thermocouple thermometer Type K, Class 2; or ASTM E230/E230M thermocouple thermometer, Type K Standard.

6.2.9. *Hot Plate(s)*—Hot plates shall be used to heat, boil, steam bath, digest, or dry samples at temperatures between 50 to 200°C, as defined in this standard. Unless otherwise specified, the thermometer for measuring the temperature of materials shall meet the requirements of M 339M/M 339 with a temperature range of at least 55 to 200°C, and an accuracy of $\pm 2.2^\circ\text{C}$ (see Note 10).

Note 10—Thermometer types suitable for use include ASTM E1 mercury thermometers; IEC 60584 thermocouple thermometer Type K, Class 2; or ASTM E230/E230M thermocouple thermometer, Type K Standard.

6.3. *Reagents:*

6.3.1. *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 Society, where such specifications are available.⁴ 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.

6.3.2. Use reagent water as defined in Section 3.1.2 for all tests.

6.3.3. *Concentration of Reagents:*

6.3.3.1. *Prepackaged Reagents*—Commercial prepackaged standard solutions or diluted prepackaged concentrations of a reagent may be used whenever that reagent is called for in the procedures, provided the purity and concentrations are as specified. Verify purity and concentration of such reagents by suitable tests.

6.3.3.2. *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 of the following specific gravities or concentrations by weight are intended:

Acetic acid ($\text{HC}_2\text{H}_3\text{O}_2$)	99.5 percent
Hydrochloric acid (HCl)	sp gr 1.19
Hydrofluoric acid (HF)	48 percent
Nitric acid (HNO_3)	sp gr 1.42
Phosphoric acid (H_3PO_4)	85 percent
Sulfuric acid (H_2SO_4)	sp gr 1.84
Ammonium hydroxide (NH_4OH)	sp gr 0.90

6.3.3.3. The desired specific gravities or concentrations of all other concentrated acids shall be stated whenever they are specified.

6.3.4. *Diluted Acids and Ammonium Hydroxide*—Concentrations of diluted acids and ammonium hydroxide, except when standardized, are specified as a ratio stating the number of volumes of the concentrated reagent to be added to a given number of volumes of water (e.g., HCl (1+99) means 1 volume of concentrated HCl (sp gr 1.19) added to 99 volumes of water).

- 6.3.5. *Standard Solutions*—Concentrations of standard solutions shall be expressed as normalities (*N*) or as equivalents in grams per milliliter of the analyte to be determined (e.g., 0.1 *N* Na₂S₂O₃ solution or K₂Cr₂O₇ (1 mL = 0.004 g Fe₂O₃)). The average of at least three determinations shall be used for all standardizations. When a material is used as a primary standard, reference has generally been made to the standard furnished by NIST. However, when primary standard grade materials are otherwise available, they may be used, or the purity of a salt may be determined by suitable tests.
- 6.3.6. *Nonstandardized Solutions*—Concentrations of nonstandardized solutions prepared by dissolving a given weight of the solid reagent in a solvent shall be specified in grams of the reagent per liter of solution, and it shall be understood that water is the solvent unless otherwise specified (e.g., NaOH solution (10 g/L) means 10 g of NaOH dissolved in water and diluted with water to 1 L). Other nonstandardized solutions may be specified by name only, and the concentration of such solutions will be governed by the instructions for their preparation.
- 6.3.7. *Indicator Solutions:*
- 6.3.7.1. *Methyl Red*—Prepare the solution on the basis of 2 g of methyl red/L of 95 percent ethyl alcohol.
- 6.3.7.2. *Phenolphthalein*—Prepare the solution on the basis of 1 g of phenolphthalein/L of 95 percent ethyl alcohol.
- 6.4. *Sample Preparation:*
- 6.4.1. Before testing, pass representative portions of each sample through a No. 20 (850 μm) sieve or any other sieve having approximately 20 openings per 1 in. to mix the sample, break up lumps, and remove foreign materials. Discard the foreign materials and hardened lumps that do not break up on sieving or brushing.
- 6.4.2. By means of a sample splitter or by quartering, the representative sample shall be reduced to a laboratory sample of at least 50 g. Where larger quantities are required for additional determinations such as water-soluble alkali, chloride, and duplicate testing, and so forth, prepare a sample of at least 100 g.
- 6.4.3. Pass the laboratory sample through a No. 100 (150-μm) sieve. Further grind the sieve residue so that it also passes the No. 100 sieve. Homogenize the entire sample by again passing it through the sieve.
- 6.4.4. Transfer the sample to a clean, dry, glass container with an airtight lid, and further mix the sample thoroughly.
- 6.4.5. Expedite the above procedure so that the sample is exposed to the atmosphere for a minimum time.
- 6.5. *General Procedures:*
- 6.5.1. *Weighing*—The calculations included in the individual test methods assume that the exact weight specified has been used. Accurately weighed samples, that are approximately but not exactly equal to the weight specified, may be used provided appropriate corrections are made in the calculations. Unless otherwise stated, weights of all samples and residues should be recorded to the nearest 0.0001 g.
- 6.5.2. *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.

- 6.5.3. *Constancy of Weight of Ignited Residues*—To definitely establish the constancy of weight of an ignited residue for referee purposes, the residue shall be ignited at the specified temperature and for the specified time, cooled to room temperature in a desiccator, and weighed. The residue shall then be reheated for at least 30 min, cooled to room temperature in a desiccator, and reweighed. If the two weights do not differ by more than 0.2 mg, constant weight is considered to have been attained. If the difference in weights is greater than 0.2 mg, additional ignition periods are required until two consecutive weights agree within the specified limits. For ignition loss, each reheating period shall be 5 min.
- 6.5.4. *Volatilization of Platinum*—The possibility of volatilization of platinum or alloying constituents from the crucibles must be considered. On reheating, if the crucible and residue lose the same weight (within 0.2 mg) as the crucible containing the blank, constant weight can be assumed. Crucibles of the same size, composition, and history shall be used for both the sample and the blank.
- 6.5.5. *Calculation*—In all operations on a set of observed values such as manual multiplication or division, retain the equivalent of at least two more places of figures than in the single observed values. 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. When using electronic calculators or computers for calculations, perform no rounding, except in the final reported value.
- 6.5.6. *Rounding Figures*—Rounding of figures to the number of significant places required in the report should be done after calculations are completed to keep the final results substantially free of calculation errors. The rounding procedure should follow the principle outlined in ASTM E29.⁵ In assessing analyst and method qualification in accordance with Section 5, the individual duplicate results, the difference between them, the average of duplicates on CRMs, and the difference of this average from the certificate value shall be left unrounded for comparison with the required limits. (See Note 11.) Round results for reporting as shown in Table 3.
- Note 11**—The rounding procedure referred to in Section 6.5.6, in effect, drops all digits beyond the number of places to be retained if the next figure is less than 5. If it is more than 5 or equal to 5 and subsequent places contain a digit other than 0, then the last retained digit is increased by one. When the next digit is equal to 5 and all other subsequent digits are 0, the last digit to be retained is unchanged when it is even and increased by one when it is odd. For example, 3.96 (50) remains 3.96, but 3.95 (50) becomes 3.96.
- 6.6. *Recommended Order for Reporting Analyses*—The following order is recommended for reporting the results of chemical analysis of hydraulic cement:
- SiO₂ (silicon dioxide)
 - Al₂O₃ (aluminum oxide)
 - Fe₂O₃ (ferric oxide)
 - CaO (calcium oxide)
 - MgO (magnesium oxide)
 - SO₃ (sulfur trioxide)
 - Na₂O (sodium oxide)
 - K₂O (potassium oxide)
 - TiO₂ (titanium dioxide)
 - P₂O₅ (phosphorus pentoxide)
 - ZnO (zinc oxide)
 - Mn₂O₃ (manganic oxide)
 - Insoluble residue
 - Free calcium oxide

- CO₂ (carbon dioxide)
- Water-soluble alkali
- Chloroform-soluble organic substances

Table 3—Rounding of Reported Results

Analyte	Decimal Places
SiO ₂ (silicon dioxide)	1
Al ₂ O ₃ (aluminum oxide)	1
Fe ₂ O ₃ (ferric oxide)	2
CaO (calcium oxide)	1
MgO (magnesium oxide)	1
SO ₃ (sulfur trioxide)	2
LOI (loss on ignition)	1
Na ₂ O (sodium oxide)	2
K ₂ O (potassium oxide)	2
SrO (strontium oxide)	2
TiO ₂ (titanium dioxide)	2
P ₂ O ₅ (phosphorous pentoxide)	2
ZnO (zinc oxide)	2
Mn ₂ O ₃ (manganic oxide)	3
S (sulfide sulfur)	2
Cl (chloride)	3
IR (insoluble residue)	2
Cx (free calcium oxide)	1
CO ₂ (carbon dioxide)	1
Alk _{sol} (water-soluble alkali)	2
Chl _{sol} (chloroform-soluble organic substances)	3

REFERENCE TEST METHODS

7. INSOLUBLE RESIDUE (REFERENCE TEST METHOD)

7.1. *Summary of Test Method:*

7.1.1. In this test method, insoluble residue of a cement is determined by digestion of the sample in hydrochloric acid followed, after filtration, by further digestion in sodium hydroxide. The resulting residue is ignited and weighed (see Note 12).

Note 12—This test method, or any other test method designed for the estimation of an acid-insoluble substance in any type of cement, is empirical because the amount obtained depends on the reagents and the time and temperature of digestion. If the amount is large, there may be a little variation in duplicate determinations. The procedure should be followed closely to reduce the variation to a minimum.

7.1.2. When this test method is used on blended cement, the decomposition in acid is considered to be complete when the portland cement clinker is decomposed completely. An ammonium nitrate solution is used in the final washing to prevent finely ground insoluble material from passing through the filter paper.

7.2. *Reagents:*

7.2.1. Ammonium Nitrate Solution (20 g NH₄NO₃/L).

7.2.2. Sodium Hydroxide Solution (10 g NaOH/L).

7.3. *Procedure:*

7.3.1. To 1 g of the sample (see Note 13), add 25 mL of cold water. Disperse the cement in the water and while swirling the mixture, quickly add 5 mL of HCl. If necessary, warm the solution gently and grind the material with the flattened end of a glass rod for a few minutes until it is evident that decomposition of the cement is complete (see Note 14). Dilute the solution to 50 mL with hot water (nearly boiling) and heat the covered mixture rapidly to near boiling by means of a high-temperature hot plate. Then digest the covered mixture for 15 min at a temperature just below boiling (see Note 15). Filter the solution through a medium-textured paper into a 400mL beaker; wash the beaker, paper, and residue thoroughly with hot water; and reserve the filtrate for the sulfur trioxide determination, if desired (see Note 16). Transfer the filter paper and contents to the original beaker, add a small portion from a total of 100 mL of hot (near boiling) NaOH solution (10 g/L). Macerate the filter paper using a glass rod. After complete maceration, add remaining NaOH solution and proceed to digest covered mixture at a temperature just below boiling for 15 min. During the digestion, occasionally stir the mixture. Acidify the solution with HCl using methyl red as the indicator and add an excess of 4 or 5 drops of HCl. Filter through medium-textured paper and wash the residue at least 14 times with hot NH_4NO_3 solution (20 g/L), making sure to wash the entire filter paper and contents during each washing. Ignite the residue in a weighed platinum or porcelain crucible at 900 to 1000°C, cool in a desiccator, and weigh.

Note 13—If sulfur trioxide is to be determined by turbidimetry, it is permissible to determine the insoluble residue on a 0.5-g sample. In this event, the percentage of insoluble residue should be calculated to the nearest 0.01 by multiplying the weight of residue obtained by 200. However, the cement should not be rejected for failure to meet the insoluble residue requirement unless a 1-g sample has been used.

Note 14—If a sample of portland cement contains an appreciable amount of manganic oxide, there may be brown compounds of manganese that dissolve slowly in cold diluted HCl but rapidly in hot HCl in the specified strength. In all cases, dilute the solution as soon as decomposition is complete.

Note 15—To keep the solutions closer to the boiling temperature, it is recommended that these digestions be carried out on an electric hot plate rather than in a steam bath.

Note 16—Continue with the sulfur trioxide determination (see Sections 17.1.2.1 through 17.1.3) by diluting to 250 mL or 200 mL, as required by the appropriate section.

7.3.2. *Blank*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

7.4. *Calculation*—Calculate the percentage of the insoluble residue to the nearest 0.01 by multiplying the weight in grams of the residue (corrected for the blank) by 100.

8. SILICON DIOXIDE (REFERENCE TEST METHOD)

8.1. *Selection of Test Method*—For cements other than portland and for which the insoluble residue is unknown, determine the insoluble residue in accordance with Section 7 of these test methods. For portland cements and other cements having an insoluble residue less than 1 percent, proceed in accordance with Section 8.2. For cements having an insoluble residue greater than 1 percent, proceed in accordance with Section 8.3.

8.2. *Silicon Dioxide in Portland Cements and Cements with Low Insoluble Residue:*

8.2.1. *Summary of Test Method*—In this test method, silicon dioxide (SiO_2) is determined gravimetrically. Ammonium chloride is added, and the solution is not evaporated to dryness. This test method was developed primarily for hydraulic cements that are almost completely

decomposed by hydrochloric acid, and should not be used for hydraulic cements that contain large amounts of acid-insoluble material and require a preliminary sodium carbonate fusion. For such cements, or if prescribed in the standard specification for the cement being analyzed, the more lengthy procedure in Section 8.3 shall be used.

8.2.2. *Reagent*—Ammonium chloride (NH_4Cl).

8.2.3. *Procedure*:

8.2.3.1. Mix thoroughly 0.5 g of the sample and about 0.5 g of NH_4Cl in a 50mL beaker, cover the beaker with a watch glass, and add cautiously 5 mL of HCl , allowing the acid to run down the lip of the covered beaker. After the chemical action has subsided, lift the cover, add 1 or 2 drops of HNO_3 , stir the mixture with a glass rod, replace the cover, and set the beaker on a steam bath for 30 min (see Note 17). During this time of digestion, stir the contents occasionally and break up any remaining lumps to facilitate the complete decomposition of the cement. Fit a medium-textured filter paper to a funnel, transfer the jellylike mass of silicic acid to the filter as completely as possible without dilution, and allow the solution to drain through. Scrub the beaker with a policeman, and rinse the beaker and policeman with hot HCl (1+99). Wash the filter two or three times with hot HCl (1+99) and then with 10 or 12 small portions of hot water, allowing each portion to drain through completely. Reserve the filtrate and washings for the determination of the ammonium hydroxide group (see Note 18).

Note 17—A hot plate may be used instead of a steam bath if the heat is so regulated as to approximate that of a steam bath. Under conditions where water boils at a lower temperature than at sea level, such as at higher elevations, 30 min may not be sufficient to recover all of the silica. In such cases, increase the time of digestion as necessary to get complete recovery of the silica. In no case should this time exceed 60 min.

Note 18—Determine the ammonium hydroxide group in accordance with the procedure described in Sections 9.1 through 9.3.

8.2.3.2. 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 flaming, and finally at 1100 to 1200°C for 1 h. Cool in a desiccator and weigh. Reignite to constant weight. Treat the SiO_2 thus obtained, which will contain small amounts of impurities, in the crucible with 1 or 2 mL of water, 2 drops of H_2SO_4 (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 SiO_2 . Consider the weighed residue remaining after the volatilization of SiO_2 as combined aluminum and ferric oxides, and add it to the result obtained in the determination of the ammonium hydroxide group.

8.2.3.3. If the HF residue exceeds 0.0020 g, the silica determination shall be repeated, steps should be taken to ensure complete decomposition of the sample before a silica separation is attempted and the balance of the analysis (ammonium hydroxide group, CaO , and MgO) determined on the new silica filtrate, provided the new silica determination has an HF residue of 0.0020 g or less, except as provided in Sections 8.2.3.4 and 8.2.3.5.

8.2.3.4. If two or three repeated determinations of a sample of portland cement consistently show HF residues higher than 0.0020 g, this is evidence that contamination has occurred in sampling or the cement has not been burned properly during manufacture. In such a case, do not fuse the large HF residue with pyrosulfate for subsequent addition to the filtrate from the silica separation. Instead, report the value obtained for the HF residue. Do not ignite the ammonium hydroxide group in the crucible containing this abnormally large HF residue.

8.2.3.5. In the analysis of cements other than portland, it may not always be possible to obtain HF residues under 0.0020 g. In such cases, add 0.5 g of sodium or potassium pyrosulfate ($\text{Na}_2\text{S}_2\text{O}_7$ or $\text{K}_2\text{S}_2\text{O}_7$) to the crucible and heat below red heat until the small residue of impurities is dissolved in the melt

(see Note 19). Cool, dissolve the fused mass in water, and add it to the filtrate and washings reserved for the determination of the ammonium hydroxide group.

Note 19—A supply of nonspattering pyrosulfate may be prepared by heating some pyrosulfate in a platinum vessel below red heat until the foaming and spattering cease, cooling, and crushing the fused mass.

8.2.3.6. *Blank*—Make a blank determination, following the same procedure and using the same amounts of reagents, and correct the results obtained in the analysis accordingly.

8.2.4. *Calculation*—Calculate the percentage of SiO_2 by multiplying the mass in grams of SiO_2 by 200 (100 divided by the mass (see Section 8.2.3.1) or equivalent mass (see Section 8.3.2.1) of the sample used (0.5 g)). Round in accordance with Table 3.

8.3. *Silicon Dioxide in Cements with Insoluble Residue Greater Than 1 Percent:*

8.3.1. *Summary of Test Method*—This test method is based on the sodium carbonate fusion followed by double evaporation to dryness of the hydrochloric acid solution of the fusion product to convert silicon dioxide (SiO_2) to the insoluble form. The solution is filtered, and the insoluble siliceous residue is ignited and weighed. Silicon dioxide is volatilized by hydrofluoric acid, and the loss of weight is reported as pure SiO_2 .

8.3.2. *Procedure:*

8.3.2.1. Weigh a quantity of the ignited sample equivalent to 0.5 g of the as-received sample calculated as follows:

$$W = [(0.5(100.00 - I))/100] \quad (2)$$

where:

W = weight of ignited sample, g, and

I = loss of ignition, percent.

The ignited material from the loss on ignition determination may be used for the sample.

Thoroughly mix the sample with 4 to 6 g of Na_2CO_3 by grinding in an agate mortar. Place a thin layer of Na_2CO_3 on the bottom of a platinum crucible of 20- to 30-mL capacity, add the cement- Na_2CO_3 mixture, and cover the mixture with a thin layer of Na_2CO_3 . Place the covered crucible over a moderately low flame, increase the flame gradually to a maximum (approximately 1100°C), and maintain this temperature until the mass is quiescent (about 45 min). Remove the burner, lay aside the cover of the crucible, grasp the crucible with tongs, and slowly rotate the crucible so that the molten contents spread over the sides and solidify as a thin shell on the interior. Set the crucible and cover aside to cool. Rinse off the outside of the crucible and place the crucible on its side in a 300-mL casserole about one-third full of water. Warm the casserole and stir until the cake in the crucible disintegrates and can be removed easily. By means of a glass rod, lift the crucible out of the liquid, rinsing it thoroughly with water. Rinse the cover and crucible with HCl (1+3), and then add the rinse to the casserole. Very slowly and cautiously add 20 mL of HCl (sp gr 1.19) to the covered casserole. Remove the cover and rinse. If any gritty particles are present, the fusion is incomplete and the test must be repeated, using a new sample.

Warning—Subsequent steps of the test method must be followed exactly for accurate results.

8.3.2.2. Evaporate the solution to dryness on a steam bath (until it no longer appears gelatinous). Without heating the residue any further, treat it with 5 to 10 mL of HCl, wait at least 2 min, and then add an equal amount of water. Cover the dish and digest for 10 min on the steam bath or a hot plate. Dilute the solution with an equal volume of hot water, immediately filter through medium-textured paper and wash the separated SiO_2 thoroughly with hot HCl (1+99), then with hot water. Reserve the residue.

- 8.3.2.3. Again evaporate the filtrate to dryness, and bake the residue in an oven for 1 h at 105 to 110°C. Cool, add 10 to 15 mL of HCl (1+1), and digest on the steam bath or hot plate for 10 min. Dilute with an equal volume of water, filter immediately on a fresh filter paper, and wash the small SiO₂ residue thoroughly as described in Section 8.3.2.2. Stir the filtrate and washings, and reserve for the determination of the ammonium hydroxide group in accordance with Sections 9.1 through 9.3.
- 8.3.2.4. Continue the determination of silicon dioxide in accordance with Section 8.2.3.2.

9. AMMONIUM HYDROXIDE GROUP (REFERENCE TEST METHOD)

- 9.1. *Summary of Test Method*—In this test method, aluminum, iron, titanium, and phosphorus are precipitated from the filtrate, after SiO₂ removal, by means of ammonium hydroxide. With care, little if any manganese will be precipitated. The precipitate is ignited and weighed as the oxides.

- 9.2. *Procedure:*

- 9.2.1. To the filtrate reserved in accordance with Section 8.2.3.1 (see Note 20), which should have a volume of about 200 mL, add HCl if necessary to ensure a total of 10 to 15 mL of the acid. Add a few drops of methyl red indicator and heat to boiling. Then treat with NH₄OH (1+1) (see Note 21) dropwise until the color of the solution becomes distinctly yellow, and add one drop in excess (see Note 22). Heat the solution containing the precipitate to boiling and boil for 50 to 60 s. In the event difficulty from bumping is experienced while boiling the ammoniacal solution, a digestion period of 10 min on a steam bath, or on a hot plate having the approximate temperature of a steam bath, may be substituted for the 50- to 60-s boiling period. Allow the precipitate to settle (not more than 5 min) and filter using medium-textured paper (see Note 23). Wash, with hot ammonium nitrate (NH₄NO₃, 20 g/L) (see Note 24), twice for a small precipitate to about four times for a large one.

Note 20—If a platinum evaporating dish has been used for the dehydration of SiO₂, iron may have been partially reduced. At this stage, add about 3 mL of saturated bromine water to the filtrate and boil the filtrate to eliminate the excess bromine before adding the methyl red indicator. If difficulty from bumping is experienced during the boiling, the following alternate techniques may be helpful: (1) a piece of filter paper, approximately 1 cm² in area, positioned where the bottom and side of the beaker merge and held down by the end of a stirring rod may solve the difficulty, and (2) use of 400-mL beakers supported inside a cast aluminum cup has also been found effective.

Note 21—The NH₄OH used to precipitate the hydroxides must be free of contamination with carbon dioxide (CO₂).

Note 22—It usually takes 1 drop of NH₄OH (1+1) to change the color of the solution from red to orange and another drop to change the color from orange to yellow. If desired, the addition of the indicator may be delayed until ferric hydroxide (Fe(OH)₃) is precipitated without aluminum hydroxide (Al(OH)₃) being completely precipitated. In such a case, the color changes may be better observed. However, if the content of Fe₂O₃ is unusually great, it may be necessary to occasionally let the precipitate settle slightly so that the color of the supernatant liquid can be observed. If the color fades during the precipitation, add more of the indicator. Observation of the color where a drop of the indicator strikes the solution may be an aid in the control of the acidity. The boiling should not be prolonged as the color may reverse and the precipitate may be difficult to retain on the filter. The solution should be distinctly yellow when it is ready to filter. If it is not, restore the yellow color with more NH₄OH (1+1) or repeat the precipitation.

Note 23—To avoid drying of the precipitate with resultant slow filtration, channeling, or poor washing, the filter paper should be kept nearly full during the filtration and should be washed without delay.

- 9.2.2. Set aside the filtrate, and transfer the precipitate and filter paper to the same beaker in which the first precipitation was effected. Dissolve the precipitate with hot HCl (1+2). Stir to thoroughly