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Standard Practice for Determination of Major and Minor Elements in Aqueous Pore Solutions of Cementitious Pastes by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)[1]

This standard is issued under the fixed designation C1875; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes a procedure for collection, sample preparation and analysis of aqueous pore solutions obtained from cementitious materials at different hydration times when analyzed by ICP-OES for the six most common readily soluble elements aluminum, calcium, potassium, silicon, sodium and sulfur.

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

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

1.4 *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:*

C25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime

C114 Test Methods for Chemical Analysis of Hydraulic Cement

C1738/C1738M Practice for High-Shear Mixing of Hydraulic Cement Pastes

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry

D7111 Test Method for Determination of Trace Elements in Middle Distillate Fuels by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

E1479 Practice for Describing and Specifying Inductively Coupled Plasma Atomic Emission Spectrometers

3. Terminology

3.1 *Definitions:*

3.1.1 Unless otherwise specified, for definitions of terms used in this practice refer to Terminology C1738/C1738M.

4. Summary of Practice

4.1 This document describes practice for determining the concentration of multiple dissolved components in the pore solution of cementitious pastes as a function of time using inductively coupled plasma optical emission spectroscopy (ICP-OES). A cementitious material is mixed with water at a desired water to solid ratio using a Practice D1129 certified constant mixer. Aliquots of the mix are divided into centrifuge tubes and stored in a water bath or environmental chamber at constant temperature. The samples centrifuged at different times. The extracted pore solution is filtered and diluted with a 5 % nitric acid solvent. The prepared test solutions flow into the plasma torch of the ICP instrument where it is vaporized and ionized, producing characteristic atomic wavelength emission spectra. The spectra are dispersed by a high resolution grating and the intensities of the individual wavelengths are measured. Elemental concentrations are determined by relating the emission intensities at specific wavelengths for an unknown sample to analytical curves made from reference standards of known composition. The concentration of the elements in the solutions can then be calculated. Internal standards compensate for variations in test sample flow rates in the instrument.

5. Significance and Use

5.1 The chemical composition of the liquid in cementitious pastes is an important indicator of the solid component reactivity at early times, being influenced by the content and rate of reaction of readily soluble alkali components, lime, and other soluble phases. Monitoring the solution composition with time can provide valuable diagnostic information about cement

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[1] This practice is under the jurisdiction of ASTM Committee C01 on Cement and is the direct responsibility of Subcommittee C01.23 on Compositional Analysis.

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quality and reactivity to supplement other sources of characterization data. This practice is intended to aid in the interpretation of the concentrations of readily soluble components in cement paste solutions, which may include portland cement, limestone, fly ash, ground granulated blast furnace slag, or other components. It provides guidance for measuring the time dependence of the concentrations of one or more components, on an elemental basis, including, but not limited to, aluminum, calcium, potassium, silicon, sodium, and sulfur.

6. Apparatus

6.1 *Spectrometer*—Spectrometer-An inductively coupled plasma emission spectrometer of the simultaneous or sequential type including RF generator, torch, nebulizer, connector tube, spray chamber, recommended peristaltic pump and host computer. Sample uptake is done by self-aspiration, or with a peristaltic pump.

6.2 *Mass Flow Controllers*—A mass-flow controller to regulate the nebulizer gas may be used as recommended by the instrument manufacturer.

6.3 *Balance*—Top loading or analytical, with automatic tare, with capacity of at least 500 g and a sensitivity of at least 0.1 g to weigh paste mix components.

6.4 *Centrifuge*—Use a centrifuge capable of accommodating two or more 50 mL centrifuge tubes at rotational speeds up to 5000 min^{-1}.

6.5 *Timer*—Calibrated timer for use of tracking blending and reaction times.

7. Reagents and Materials

7.1 Ordinary laboratory apparatus are not listed, but are assumed to be present.

7.2 *Purity of Reagents*—At a minimum, reagent grade or better chemicals shall be used in all tests.

7.3 *Purity of Water*—References to water shall be understood to mean deionized water. Tap water may be used only to prepare cement paste mixes if desired. If tap water is used for mixing the composition of the water itself must be separately measured as a test sample as described in Section 11.

7.4 *Argon*—High purity grade (99.99 %).

7.5 *Calibration Standards*—Single-element or multi-element calibration reference solutions that combine appropriate volumes of the stock solutions or weighed reagent-grade chemicals in acid-rinsed volumetric flasks. Dilute to the volume of the flask using a 5 % (v/v) nitric acid solution. Prior to preparing the multi-element solutions, analyze each stock solution separately to check for strong spectral interference and the presence of impurities. Take care when preparing the multi-element solutions to verify that the components are compatible and stable (that is, that they do not interact to cause precipitation) and that none of the elements present will contribute to mutual spectral interference. Calibration reference solutions must be verified initially using a quality control sample and monitored periodically for stability. Mixed calibration standards will vary, depending on the number of elements being determined.

7.6 *Calibration Blank Solution*—5 % (v/v) HNO_3.

7.7 *Nitric Acid*—5 % (v/v).

7.8 *Check Standards*—Quality control certified NIST traceable samples that are stable and representative of the samples of interest. These check standard samples represent ground truth samples and are used to check that the measurement process is in control.

7.9 *Internal Standards*—Internal standards are used to correct for instrument drift and physical interferences. Refer to instrument operating manual for suggested internal standards. Add internal standards to blanks, samples, and standards in a like manner.

7.10 *Membrane Filter*—Nylon 25 mm diameter, 0.2 µm.

7.11 *Syringe and Needle*—Capacity of at least 2 mL.

7.12 *Centrifuge Tubes*—Clean plastic centrifuge tubes the appropriate size for centrifuge.

7.13 *Glassware*—Volumetric flasks complying with the requirements of ISO 1042, made of borosilicate glass complying with the requirements of ISO 3585. Glassware should be cleaned before each use by soaking in nitric acid and then rinsing thoroughly with deionized water.

7.14 *Pipette*—Calibrated volumetric pipettes and pipette tips, for dispensing.

8. Hazards

8.1 The toxicity or carcinogenicity of each reagent used in this practice has not been precisely defined; however, each chemical should be treated as a potential health hazard. Adequate precautions should be taken to minimize exposure of personnel to chemicals used in this practice.

8.2 Gases under high pressure are used in this practice. Use only apparatus rated for handling the high gas pressures that occur in this practice.

9. Sample Handling

9.1 *Powder Homogenization*—A uniform distribution of powder components is essential to ensure representative sampling during the mixing process, especially for blended powder components such as portland cement and fly ash. Powders should therefore be homogenized for 30 min with a planetary mixer or similar device.

10. Sample Preparation and Collection

10.1 *High Shear Mixing:*

10.1.1 Blend the solid and liquid components to make a paste according the standard practice described in Practice D1129.

10.1.2 Set a timer to mark the end of mixing. The time on this timer will serve as the total reaction time for the remainder of the procedure.

10.1.3 Stir the paste with the spatula for no more than 15 s ensure homogeneity.

10.1.4 Transfer (25 ± 1) g of the paste into each of twelve centrifuge tubes with secured lids in the minimum amount of time possible, preferably less than 2 min.