Designation: C1356 – 07 (Reapproved 2020)

Standard Test Method for Quantitative Determination of Phases in Portland Cement Clinker by Microscopical Point-Count Procedure[1]

This standard is issued under the fixed designation C1356; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a systematic procedure for measuring the percentage volume of the phases in portland cement clinker by microscopy.

1.2 The values stated in SI units are to be regarded as the 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:*[2]

C150/C150M Specification for Portland Cement

C219 Terminology Relating to Hydraulic and Other Inorganic Cements

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

D75/D75M Practice for Sampling Aggregates

D3665 Practice for Random Sampling of Construction Materials

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of other terms relating to hydraulic cements, refer to Terminology C219.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *clinker phase, n*—a physically and chemically distinct optically identifiable portion of the clinker sample, including both principal phases (alite, belite, aluminate, and ferrite), minor phases (for example, free lime, periclase, and alkali sulfates), and voids.

3.2.1.1 *Discussion*—Voids, though not a phase in the sense of being a crystalline compound, are a distinct, identifiable portion of a clinker microstructure.

3.2.2 *voids, n*—isolated or interconnected open areas in the clinker, also called pores.

3.3 *Principal Clinker Phases:*[3]

3.3.1 *alite, n*—crystalline tricalcium silicate (C_3S), modified in composition and crystal structure by incorporation of foreign ions; the crystals are pseudo-hexagonal with well-defined faces, though less regular shapes commonly occur.

3.3.2 *aluminate, n*—tricalcium aluminate (C_3A) modified in composition and crystal structure by incorporation of a substantial proportion of foreign ions; aluminate forms cubic crystals when relatively pure, and forms identifiable elongated crystals commonly called "alkali aluminate" when in solid solution with significant amounts of potassium or sodium, or both.

3.3.3 *belite, n*—crystalline dicalcium silicate (C_2S), modified in composition and crystal structure by incorporation of foreign ions; belite usually occurs as rounded crystals marked by striations formed by cross sections of lamellae, and may occur as single crystals or in clusters.

3.3.4 *ferrite, n*—a solid solution of approximate composition tetracalcium aluminoferrite (C_4AF) modified in composition by variation in the Al/Fe ratio and by substantial incorporation of foreign ions; ferrite is characterized by high reflectivity in polished sections and is normally the only strongly colored compound among the principal clinker phases.

3.3.4.1 *Discussion*—Aluminate and ferrite form most of the interstitial material between the silicate crystals and, under

[1] This test method 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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[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.

[3] C = CaO, S = SiO_2, A = Al_2O_3, F = Fe_2O_3, $\bar{S}$ = SO_3, M = MgO, N = Na_2O, and K = K_2O in cement chemistry notation.

certain conditions of cooling, may not be easily identifiable or resolved by ordinary light microscopy.

3.4 *Minor Clinker Phases:*

3.4.1 *alkali sulfates, n*—sodium sulfate, potassium sulfate, and double sulfates such as calcium langbeinite (K_2SO_4–$2CaSO_4$).

3.4.2 *free lime, n*—calcium oxide (C) found mostly as round crystals.

3.4.3 *periclase, n*—crystalline form of free magnesium oxide (M), that has not been taken up in solid solution with other phases.

4. Summary of Test Method

4.1 The test method consists of the preparation and microscopical examination of a specimen produced by encapsulating clinker in a mounting medium and sectioning the specimen so as to expose the interior of particles for visual examination. Polishing the section surface and treating it with etchants to highlight specific phases complete the preparation. During microscopical examination phases are identified and their proportions determined by a point-count procedure. In this procedure, the specimen is moved in uniform increments on a microscope stage, and phases falling under the cross hairs of the eyepiece are identified and counted (**1-5**).[4]

5. Significance and Use

5.1 This test method provides a relatively simple and reliable microscopical means of measuring the phase abundance of portland cement clinker (Note 1). Microscopical point counting provides a direct measure of the clinker phase composition in contrast to the calculated Bogue phase composition (Note 2).

NOTE 1—This test method utilizes a reflected light microscope. Related methods such as transmitted light microscopy, scanning electron microscopy, and automated imaging techniques may also be used for clinker analysis but are not presently included in this test method.

NOTE 2—This test method allows direct determination of the proportion of each individual phase in portland cement clinker. This test method is intended to provide an alternative to the indirect estimation of phase proportion using the equations in Specification C150/C150M (footnote C in Table 1 and footnote B in Table 2).

5.2 This test method assumes the operator is qualified to operate a reflected light microscope and the required accessories, is able to correctly prepare polished sections and use necessary etchants, and is able to correctly identify the constituent phases.

5.3 This test method may be used as part of a quality control program in cement manufacturing as well as a troubleshooting tool. Microscopic characterization of clinker phases may also aid in correlating cement properties and cement performance in concrete, to the extent that properties and performance are a function of phase composition.

6. Apparatus

6.1 Reflected light microscope.

6.2 Mechanical stage with stepping increments ranging from 0.05 to 2.0 mm (to enable analysis of clinkers of different average crystal sizes) and vernier scales graduated in both X and Y directions.

6.3 Microscope objectives of magnification 5×, 10×, 20×, and 40× or other magnifications suitable for the task.

NOTE 3—The use of reflected light with oil immersion is optional. It is highly recommended for study of finely crystalline aluminate and ferrite which typically form the ground mass in which the silicates occur. Reflected light objective lenses with magnification up to 100× designed for use in oil-immersion are required.

6.4 Assorted eyepieces (5×, 10×, 20×) which when combined with the objectives described in 6.3 will provide magnifications up to 800×.

6.5 Eyepiece reticles (graticulae) with a linear grid pattern containing 9, 16, or 25 intersections.

6.6 Eyepiece micrometer for measuring dimensions of the object under investigation and calibrated for each magnification.

6.7 Stage micrometer for the calibration of the eyepiece micrometer.

6.8 Light source that provides uniform and consistent illumination of the field and light of constant intensity.

6.9 Counting (tallying) device capable of recording up to ten categories of data.

6.10 Crushing device capable of reducing sample particle size to between 1 and 4 mm.

6.11 Riffle sample splitter to reduce sample from initial volume to approximately 100 g.

6.12 Wire cloth sieves with openings suitable for sieving the entire clinker sample to broadly define the model size class, and sieves with 1- and 4-mm square openings to concentrate particles of recommended size for specimen preparation.

6.13 Vacuum impregnation device to force epoxy into clinker voids. (Vacuum bell jar or desiccator connected to a vacuum pump.)

6.14 Curing oven, hot plate, slide warmer, or ultraviolet light may be used to accelerate the epoxy hardening.

6.15 Thin, diamond-rimmed wafering saw for sectioning the encapsulated clinker.

6.16 Glass grinding (lapping) plates (300 × 300 × 5 mm) required only if the mechanical system is not equipped to handle the final grinding with alumina powder.

6.17 Ultrasonic cleaning device (optional) to clean the sample prior to, between, and after polishing steps.

7. Reagents and Materials

7.1 Consumable grinding (lapping) and polishing supplies. After the encapsulated specimen has been cut with the saw, all or most of the following grinding and polishing steps are required: 120-, 320-, and 600-grit silicon carbide grinding papers or equivalent and 5, 0.3, and 0.05 µm alumina polishing powders or their equivalent. Diamond grinding discs, silicon carbide paper, or polishing cloths and alumina polishing

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.