National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2687a

Portland Cement Clinker

This Standard Reference Material (SRM) is intended for use in evaluating methods of phase abundance analysis of major phases in cement clinkers: the percentages of alite (C_3S) [(1)], belite (C_2S), aluminate (C_3A), and ferrite (C_4AF). A unit of SRM 2687a consists of five hermetically sealed vials, each containing approximately 8 g of crushed portland cement clinker [1].

Certified Mass Fraction Values: The certified mass fraction values are provided in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or taken into account [2]. The certified values listed are weighted averages, the results of analyses performed at NIST using quantitative X-ray powder diffraction (QXRD) and image analysis of scanning electron microscope backscattered electron and X-ray images. The QXRD used Rietveld refinement of powder diffraction data [3-5].

Sampling for the X-ray study allowed assessment of within- and between-vial homogeneity and found the materials to be homogeneous. The uncertainty listed with each value ($2U_c$) is an expanded uncertainty, with coverage factor 2, calculated by combining a between-method variance [6,7] with a pooled, within-method variance following the JCGM/ISO Guide [8]. The measurands are the mass fractions of the phases in cement listed in Table 1. Metrological traceability is to the SI derived unit for mass fraction (expressed as a percent).

Information Mass Fraction Values: An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2]. Bulk oxide values by X-ray fluorescence (XRF) and loss on ignition (LOI) are provided in Table 2. Table 2 also includes information phase abundance values determined using only QXRD. Calculated compounds per ASTM C 150-18 are provided in Table 3. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 2687a** is valid, within the measurement uncertainty specified, until **01 January 2025**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before expiration, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of the analytical measurements leading to certification were performed by P.E. Stutzman and P. Dean of the NIST Materials and Structural Systems Division.

R. Eason of the NIST Materials and Structural Systems Division processed the bulk clinker in preparation for packaging.

Statistical consultation for this SRM was provided by N.A. Heckert of the NIST Statistical Engineering Division.

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Certificate Issue Date: 22 October 2019

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[(1)]Cement chemist's notation: C = CaO, S = SiO_2, A = Al_2O_3, F = Fe_2O_3.

Support aspects involved with the certification and issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Cement clinker is hygroscopic, so storage over desiccant is recommended to minimize the effects of exposure to humidity. Changes in the appearance of the etched surface of polished sections, particularly the appearance of free lime, which hydrates to portlandite [$Ca(OH)_2$], indicate change due to moisture exposure. Portlandite exhibits a popcorn-like texture and high topographic relief. For XRD analysis, the presence of portlandite or calcium carbonate may be taken as an indication that moisture has altered the free lime. For XRD powders, heat-treating to 450 °C converts calcium hydroxide back to free lime without other alteration.

Table 1. Certified Mass Fraction Values for Phase Abundance of SRM 2687a

Phase	Mass Fraction (%)	Mean $2U_c$ (%)
Alite	57.88	2.49
Belite	24.70	1.84
Aluminate	9.56	0.58
Ferrite	6.27	0.32

Table 2. Information Mass Fraction Values for Bulk Chemistry by XRF [1] and LOI

Constituents[(a)]	Mass Fractions (%)	Phase Abundance	Mean Mass Fractions (%)
SiO_2	22.07	Periclase	0.25
Al_2O_3	5.470	Arcanite	0.68
Fe_2O_3	2.403	Aphthitalite	0.16
CaO	66.172	Free Lime	0.51
MgO	1.035		
SO_3	0.695		
Na_2O	0.103		
K_2O	0.702		
TiO_2	0.246		
P_2O_5	0.524		
Mn_2O_3	0.031		
SrO	0.090		
ZnO	0.028		
LOI	0.49		

Table 3. Information Mass Fraction Values for Calculated Compounds per ASTM C 150-18

Phase	Mass Fraction (%)
Alite	59.47
Belite	18.41
Aluminate	10.43
Ferrite	7.31

REFERENCES

[1] Stutzman, P.; A. Heckert; *Certification of Standard Reference Material Clinker 2687a*; NIST Special Publication 260-195; U.S. Government Printing Office: Washington, DC (2019) available at https://www.nist.gov/sites/default/files/documents/2019/05/14/sp260-195.pdf (accessed Oct 2019).

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[3] Stutzman, P; Leigh S; *Phase Analysis of Hydraulic Cements by X-Ray Powder Diffraction: Precision, Bias and Qualification*; Journal of ASTM International, Vol. 4, No. 5, JAI Paper 101085 (2007).

[4] ASTM C 1356M, *Standard Test Method for Quantitative Determination of Phases in Portland Cement Clinker by Microscopical Point-Count Procedure*; Annul. Book of ASTM Stand., Vol. 4.01 (2006).

[5] Stutzman, P.; Feng, P.; Bullard, J.; *Phase Analysis of Portland Cement by Combined X-Ray Powder Diffraction and Scanning Electron Microscopy*; Journal of Research of the National Institute of Standards and Technology, Vol. 121, pp. 47–107 (2016).

[6] DeSimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; Control Clin. Trials, Vol. 7, pp. 177-188 (1986)

[7] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, No. 4, pp. 571-579 (2000).

[8] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Oct 2019); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at https://www.nist.gov/pml/nist-technical-note-1297 (accessed Oct 2019).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail mailto:srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.