NIST NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

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Standard Reference Material® 2687a
Portland Cement Clinker
CERTIFICATE OF ANALYSIS

Purpose: 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), belite (C_2S), aluminate (C_3A), and ferrite (C_4AF).

Description: A unit of SRM 2687a consists of five hermetically sealed vials, each containing approximately 8 g of crushed portland cement clinker [1].

Certified Values: The certified mass fraction values are provided in the table below. 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 the table below. Metrological traceability is to the International System of Units (SI) derived unit for mass fraction (expressed as a percent).

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

Period of Validity: The certified values delivered by **SRM 2687a** are valid within the measurement uncertainty specified until **01 January 2029**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Aron Newman, Chief
Materials and Structural Systems Division

Steven J. Choquette, Director
Office of Reference Materials

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Storage: 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.

Use: 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.

Additional Information: Values of potential interest to users are provided in Appendix A.

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/system/files/documents/2019/05/14/sp260-195.pdf (accessed Nov 2024).

[2] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Nov 2024).

[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/en/committees/jc/jcgm/publications (accessed Nov 2024); 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 Nov 2024).

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Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Values of Potential Interest: Values of potential interest are presented in the tables below. Insufficient information is available to assess the uncertainty associated with values of potential interest [2]. Bulk oxide values by X-ray fluorescence (XRF) and loss on ignition (LOI) are provided in Table A1. Table A1 also includes information phase abundance values determined using only QXRD. Calculated compounds per ASTM C 150-18 are provided in Table A2. Values of potential interest cannot be used to establish metrological traceability.

Table A1. Mass Fraction Values of Potential Interest 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 A2. Mass Fraction Values of Potential Interest for Calculated Compounds per ASTM C 150-18

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

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