National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2687

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), ferrite (C_4AF), and arcanite (K_2SO_4). A unit of SRM 2687 consists of three hermetically sealed vials, each containing approximately 10 g of crushed portland cement clinker. The materials selected for SRMs 2686a, 2687, and 2688 differ widely in phase abundance, crystal sizes, and distribution of crystals [1,2].

Certified Values: The certified values for SRM 2687, expressed as mass fractions, 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 [3]. The certified values listed are weighted averages, the results of analyses performed at NIST using quantitative X-ray powder diffraction (QXRD), point-counting of scanning electron microscope backscattered electron images, and data from an ASTM interlaboratory study that used the SRM clinkers [4]. The QXRD used Reitveld refinement of powder diffraction data [5,6].

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 is an expanded uncertainty, $U = ku_c$, with coverage factor $k = 2$, calculated by combining a between-method variance [7] with a pooled, within-method variance following the ISO Guide [8].

Information 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 [3]. Bulk oxide values determined by X-ray fluorescence and loss on ignition are provided in Table 2. Calculated compounds per ASTM C 150-07 are provided in Table 3.

Expiration of Certification: The certification of **SRM 2687** is valid, within the measurement uncertainty specified, until **01 December 2015**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for 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) will facilitate notification.

The overall direction and coordination of the analytical measurements leading to certification were performed by P.E. Stutzman and G. Lespinasse of the NIST Construction Materials Division.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Support aspects involved with the certification and issuance of this SRM were coordinated through the NIST Measurement Services Division.

Jonathan W. Martin, Chief
Materials and Construction Research Division

Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

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

INSTRUCTIONS FOR USE

Use and Handling: 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 epezite (calcium hydroxide), indicate change due to moisture exposure. Epezite exhibits a popcorn-like texture and high topographic relief. For XRD analysis, the presence of calcium hydroxide 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 Values for Phase Abundance (Mass Fraction in %) of SRM 2687 [2–8].

SRM 2687	Alite	Belite	Aluminate	Ferrite	Arcanite
Mean	71.24	12.57	11.82	2.81	0.92
U	1.27	1.22	1.03	0.68	0.15

Table 2. Information Values for Bulk Chemistry Mass Fractions by X-Ray Fluorescence [1] and Loss on Ignition (LOI).

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	TiO_2	P_2O_5	Mn_2O_3	SrO	LOI
21.43	5.53	1.98	67.20	1.48	0.83	0.14	0.72	0.27	0.29	0.04	0.11	0.17

Free Lime (Mass Fraction)[(a)]
2.2 %

[(a)] Based on optical microscopy and atomic absorption analysis of an ethyl acetoacetate extraction.

Table 3. Information Values for Calculated Compounds per ASTM C 150-07.

Phase	Mass Fraction (%)
alite	68.3
belite	9.9
aluminate	11.3
ferrite	6.0

REFERENCES

[1] Kanare, H.; *Production of Portland Cement Clinker Phase Abundance Standard Reference Materials (SRMs 2686, 2687, 2688)*; Final Report, Construction Technology Laboratories (1987).

[2] Stutzman, P.; Lespinasse, G.; Leigh, S.; *Compositional Analysis and Certification of NIST Reference Material Clinker 2686a*; NIST Technical Note 1602; U.S. Government Printing Office: Washington, DC (2008).

[3] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm (accessed Arp 2010).

[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; S. Leigh; *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).

[6] R.A. Young; *The Rietveld Method*; IUCr Monographs on Crystallography, Vol. 5, Oxford Science Publications, Oxford University (1995).

[7] Rukhin, A.L.; Vangel, M.G.; *Estimation of a Common Mean and Weighted Means Statistics*; J. Am. Stat. Assoc., Vol. 93, No. 441, pp. 303–308 (1998).

[8] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2010); 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 http://www.nist.gov/physlab/pubs/index.cfm (accessed Apr 2010).

Certificate Revision History: 14 December 2010 (Minor editorial changes); 15 April 2010 (Updated certified values and extended the certification period); 04 February 2002 (Original certificate date).

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) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.