National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2688

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 2688 consists of three hermetically sealed vials, each containing approximately 10 g of crushed portland cement clinker [1,2].

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 [1]. A certified value is the present best estimate of the true value. Further detail regarding the preparation, analysis, and phases of the SRM are described in references 2 and 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. The QXRD used Rietveld refinement of powder diffraction data [4-6].

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 [3]. Bulk oxide values by X-ray fluorescence (XRF) and loss on ignition (LOI) are provided in Table 2. Calculated compounds per ASTM C 150-07 are provided in Table 3. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 2688** is valid, within the measurement uncertainty specified, until **08 March 2027**, 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) will facilitate notification.

Overall direction and coordination of the analytical measurements leading to certification were performed by P.E. Stutzman and G. Lespinasse of the NIST Materials and Structural Systems 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 Office of Reference Materials.

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Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND 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.

Certified Mass Fraction Values: The measurands are the mass fractions of the elements in cement listed in Table 1. Metrological traceability is to the SI derived unit for mass fraction (expressed as a percent). 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 with coverage factor $k = 2$, calculated by combining a between-method variance [7] with a pooled, within-method variance following the ISO Guide [8].

Table 1. Certified Mass Fraction Values for Phase Abundance of SRM 2688.

Phase	Mass Fraction (%)
Alite	64.95 ± 1.04
Belite	17.45 ± 0.96
Aluminate	4.99 ± 0.50
Ferrite	12.20 ± 0.84

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

Constituents[(a)]	Mass Fractions (%)
SiO_2	22.68
Al_2O_3	4.90
Fe_2O_3	4.07
CaO	66.50
MgO	0.98
SO_3	0.31
Na_2O	0.11
K_2O	0.35
TiO_2	0.24
P_2O_5	0.08
Mn_2O_3	0.03
SrO	0.13
LOI	0.21
Free Lime	0.2

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

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

Phase	Mass Fraction (%)
alite	58.7
belite	20.7
aluminate	6.1
ferrite	12.4

REFERENCES

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[2] Stutzman, P.; Lenker, S.; Kanare, H.; Tang, F.; Campbell, D.; and Struble, L.; *Standard Cement Clinkers for Phase Analysis*; in Proc. 11th Internat. Conf. on Cement Microscopy, International Cement Microscopy Association, New Oleans, LA, pp. 154-168 (1989).

[3] 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).

[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; 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).

[6] Young R.A; *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 Jul 2017); 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 Jul 2017).

Certificate Revision History: 23 August 2017 (Change of expiration date; editorial changes); 16 April 2010 (The certified values for alite, belite, aluminate and ferrite were updated and the arcanite value was decertified. The certification period was extended.); 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) 948-3730; e-mail mailto:srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.