National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2686a

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), periclase (M), and alkali sulfates (aphthitalite and arcanite). A unit consists of four hermetically-sealed containers of approximately 7 g each of crushed portland cement clinker. The materials selected for SRMs 2686, 2686a, 2687 and 2688 differ widely in phase abundance, crystal sizes, and distribution of crystals [1,2].

Certified Value: The certified values for SRM 2686a, 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 unweighted averages, the results of analyses performed at NIST using quantitative X-ray powder diffraction (QXRD) and from image analysis of scanning electron microscope backscattered electron and X-ray images. The QXRD used Reitveld refinement of powder diffraction data [4-6].

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 ($2U_c$) 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 ISO Guide [8].

Reference Value: Reference values for SRM 2686a, expressed as mass fractions on an as-received basis, are provided in Table 2 [2]. Reference values are noncertified values that are the best available estimates of the true values; however, the values, which are based on determinations done by reliable methods, do not meet the NIST criteria for certification [3].

Information Value: 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 and atomic absorption and loss on ignition values are provided in Table 3.

Expiration of Certification: The certification of **SRM 2686a** is valid, within the measurement uncertainty specified, until **01 January 2018**, 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 Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of certification, 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 Building Materials Division.

Bulk chemistry analyses were performed by S. Lane, K. Stark, and G. Barger of the Ash Grove Cement Company, Overland Park, KS using seven specimens by wavelength-dispersive X-ray fluorescence on lithium borate fused beads and additionally for sodium and potassium, using atomic absorption on dissolved lithium borate fusions dissolved in dilute nitric acid.

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

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.

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) of SRM 2686a [2-8].

SRM 2686a	Alite	Belite	Aluminate	Ferrite	Periclase	Alkali Sulfates
Mean	63.53	18.80	2.46	10.80	3.40	0.86
$2U_c$	1.04	1.10	0.39	0.84	0.23	0.17

Table 2. Reference Values for Alkali Sulfate Phases by X-Ray Powder Diffraction (Mass Fraction)

	aphthitalite	arcanite
Mean	0.74	0.27
Standard Deviation	0.22	0.19
Number of samples (*n*)	98	98

Table 3. Information values for Bulk Chemistry by X-Ray Fluorescence (XRF), Atomic Absorption, and Loss on Ignition (LOI).[(a)]

	XRF											
	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
Mean	21.713	3.701	3.654	64.085	4.813	0.388	0.198	0.470	0.220	0.068	0.128	0.043
$\left(s/\sqrt{n}\right)$	0.034	0.014	0.015	0.083	0.008	0.010	0.011	0.005	0.000	0.002	0.002	0.002

	Atomic Absorption	
	Na_2O	K_2O
Mean	0.200	0.503
$\left(s/\sqrt{n}\right)$	0.003	0.032

	LOI
Mean	0.509
$\left(s/\sqrt{n}\right)$	0.024

[(a)] Expressed as the mean mass fraction and standard deviations of the means $\left(s/\sqrt{n}\right)$ from eight separate specimens.

REFERENCES

[1] SRM 2686, *Portland Cement Clinker*; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD 2002); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/archived_certificates/2686.pdf

[2] Stutzman, P.; Lespinnasse, 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, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136, U.S. Government Printing Office, Gaithersburg, MD (2000); available at http://www.cstl.nist.gov/nist839/NIST_special_publications.htm.

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

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

[6] 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).

[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, p. 571-579 (2000).

[8] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement;* (ISO GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology: BIPM, Sevres Cedex, France (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf; 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.physics.nist.gov/Pubs/contents.html.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.