



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] SRM 2686

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, belite² (β - C_2S), aluminate (C_3A), ferrite (C_4AF), and periclase (M). Each unit consists of three hermetically-sealed containers of approximately 10 g each of crushed portland cement clinker. The materials selected for SRMs 2686, 2687, and 2688 differ widely in phase abundance, crystal sizes, and distribution of crystals [1,2].

Certified Values: The certified values for SRM 2686, 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 accounted for by NIST. The certified values listed are based on the results of analyses performed at NIST using quantitative X-ray powder diffraction (QXRD) and from optical microscopy point count analyses conducted by both Construction Technology Laboratories, Inc. (CTL)³ and ASTM C 1.23 Microscopy Task Group (MPC) [3,4]. The MPC analyses followed ASTM C 1356M [5] and the QXRD used Reitveld refinement of powder diffraction data [6,7].

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, with coverage factor 2, calculated by combining a between-method variance [8,9] with a pooled, within-method variance following the ISO/NIST Guide [10].

Informational Values: Information values for SRM 2686, expressed as mass fractions on an as-received basis, are provided in Table 2. Information values are noncertified values with no uncertainty assessed that are provided for information purposes only.

Expiration of Certification: The certification of this SRM is valid, within the measurement uncertainties specified, until **01 December 2007**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Use and Handling). However, the certification will be nullified if the SRM is damaged, contaminated, or otherwise modified.

Coordination of technical measurements for certification was accomplished under the direction of P.E. Stutzman of the NIST Building Materials Division. Analytical measurements for certification of this SRM were performed by P.E. Stutzman of the NIST Building Materials Division, D. Campbell of CTL, Skokie, IL, and ASTM C 1.23.01 Task Group on Microscopy Methods.

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

Support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

Geoffrey Frohnsdorff, Chief
Building Materials Division

Gaithersburg, MD 20899
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John Rumble, Jr., Acting Chief
Standard Reference Materials Program

¹ Cement chemist's notation: C = CaO, S = SiO₂, A = Al₂O₃, F = Fe₂O₃, M = MgO.

² Belite is the β crystalline form of dicalcium silicate, C₂S.

³ Certain commercial organizations, services, equipment, or materials are identified in this certificate to specify adequately 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 organizations, services, materials, or equipment identified are necessarily the best available for the purpose.

Stability: This material is considered to be stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser as mentioned at the end of this document.

Source and Processing: A portion of the bulk material used for SRM 2686 had been used to make approximately 2500 vials of RM 8486 Portland Cement Clinker released in 1989. Details of the original processing of this bulk material are provided in the Report of Investigation for RM 8486 [1]. In 2000, the remaining bulk material was divided by the spinning riffler technique into vials containing approximately 10 g each and capped. Each vial was sealed in a foil bag and subsequently issued in sets of three vials as SRM 2686.

Use and Handling: Cement clinker is hygroscopic and 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, if present, which hydrates to epepite (calcium hydroxide) indicate change due to moisture exposure. Epepite 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 SRM 2686

Major phases in cement clinker, expressed as mass fractions (in %), with mean and expanded uncertainty ($U = 2 u_c$) at an approximate confidence level at 95 % [9,10].

Phase	Mass Fraction (%)
Alite	58.6 ± 4.0
Belite	23.3 ± 2.8
Ferrite	14.1 ± 1.4
Aluminate	2.3 ± 2.1
Periclase	3.3 ± 1.9

Table 2. Informational Values for SRM 2686

Elemental constituents determined by X-ray fluorescence spectrometry [1] and expressed as the chemical forms and in the order given in ASTM C 114-00, Section 3, Table 1 [12].

Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)	Constituent	Mass Fraction (%)
SiO ₂	22.48	MgO	4.73	TiO ₂	0.25
Al ₂ O ₃	4.70	SO ₃	0.27	P ₂ O ₅	0.06
Fe ₂ O ₃	3.60	Na ₂ O	0.10	Mn ₂ O ₃	0.10
CaO	63.36	K ₂ O	0.42	SrO	0.05
				L.O.I.	0.16

Free Lime (mass fraction): 0.3 %*

*Based on optical microscopy and atomic absorption analysis of an ethyl acetoacetate extraction

Calculated Compounds (per ASTM C 150-98)	Mass Fraction (%)
C ₃ S	50
C ₂ S	27
C ₃ A	6
C ₄ AF	11

REFERENCES

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- [2] Stutzman, P. and Leigh, S., "Phase Composition Analysis of the NIST Reference Clinkers by Optical Microscopy and X-ray Powder Diffraction," NIST Technical Note 1441.
- [3] Struble, L., "A Review of Clinker Analysis by QXRD," in Characterization and Performance Prediction of Cement and Concrete, J.F. Young, Ed., New York, Engineering Foundation, pp. 31-37, (1982).
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- [6] Young, R.A., Ed., The Rietveld Method, International Union of Crystallography Monographs on Crystallography 5, Oxford University Press.
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- [8] Rukhin, A.L. and Vangel, M.G., "Estimation of a Common Mean and Weighted Means Statistics," Journal of the American Statistical Association, **Vol. 93**, No. 441, pp. 303-308, (March 1998).
- [9] Levenson, M.S., Banks, D.L., Eberhardt, K.R., Gill, L.M., Guthrie, W.F., Liu, H.K., Vangel, M.G., Yen, J.H., and 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, (2000).
- [10] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and 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://physics.nist.gov/Pubs/>.
- [11] ASTM C 150-98, "Standard Specification for Portland Cement," ASTM Standards, **Vol. 04.01**, American Society for Testing and Materials, West Conshohocken, PA, (1999).
- [12] ASTM C 114-00, "Test Methods for Chemical Analysis of Hydraulic Cement," **Vol. 04.01**, American Society for Testing and Materials, West Conshohocken, PA, (1999).

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) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet <http://www.nist.gov/srm>.