



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material® 2686b

#### Portland Cement Clinker

This Standard Reference Material (SRM) is intended primarily for use in evaluating methods of phase abundance analysis of major phases in cement clinkers: the percentages of alite ( $C_3S$ )<sup>(1)</sup>, belite ( $C_2S$ ), aluminite ( $C_3A$ ), ferrite ( $C_4AF$ ), and periclase (M). A unit consists of one glass bottle containing approximately 50 g of crushed portland cement clinker.

**Certified Values:** The certified values for SRM 2686b, expressed as mass fractions, are provided in Table 1 [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 [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 ISO/JCGM Guide [8].

**Information Values:** An information value is considered to be a value developed using reliable methods that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2]. Information values for arcanite and free lime by X-ray powder diffraction are provided in Table 2. Bulk oxide values by X-ray fluorescence are provided in Table 3. Estimates of the clinker phase composition following ASTM C150 [9] are provided in Table 4. Information values cannot be used to establish metrological traceability.

**Expiration of Certification:** The certification of **SRM 2686b** is valid, within the measurement uncertainty specified, until **01 January 2030**, provided the SRM is handled and stored 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 the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of the analytical measurements leading to certification were performed by P.E. Stutzman of the NIST Materials and Structural Systems Division, and L. Mundy, formerly of NIST.

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<sup>(1)</sup> Cement chemist’s notation: C = CaO, S = SiO<sub>2</sub>, A = Al<sub>2</sub>O<sub>3</sub>, F = Fe<sub>2</sub>O<sub>3</sub>, M = MgO.

Bulk clinker processing in preparation for packaging was performed by R. Eason of the NIST Materials and Structural Systems Division.

Statistical consultation for this SRM was provided by A. Heckert of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

## INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

The cement clinker is hygroscopic. Store bottle in original, unopened pouch until ready to use. After opening, store in the original bottle, tightly capped, in a desiccator over desiccant to minimize exposure to moisture. Changes in the appearance of the etched surface of polished sections, particularly the appearance of free lime, which hydrates to portlandite ( $\text{Ca}(\text{OH})_2$ ), indicates change due to moisture exposure. Portlandite exhibits a popcorn-like texture and high topographic relief. 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.

Table 1. Certified Values for Phase Abundance (Mass Fraction) of SRM 2686b

| Phase     | Mass Fraction<br>(%) |
|-----------|----------------------|
| Alite     | 64.82 ± 2.57         |
| Belite    | 16.68 ± 3.35         |
| Aluminate | 3.76 ± 1.00          |
| Ferrite   | 10.42 ± 1.88         |
| Periclase | 3.31 ± 0.70          |

Table 2. Information Values for Phase Abundance (Mass Fraction) of SRM 2686b

| Phase     | Mass Fraction<br>(%) |
|-----------|----------------------|
| Arcanite  | 0.20                 |
| Free Lime | 0.53                 |

Table 3. Information values for bulk chemistry by X-Ray Fluorescence

| Constituents            | Mass Fraction<br>(%) |
|-------------------------|----------------------|
| $\text{SiO}_2$          | 21.33                |
| $\text{Al}_2\text{O}_3$ | 4.18                 |
| $\text{Fe}_2\text{O}_3$ | 3.72                 |
| $\text{CaO}$            | 63.97                |
| $\text{MgO}$            | 4.37                 |
| $\text{SO}_3$           | 0.04                 |
| $\text{Na}_2\text{O}$   | 0.24                 |
| $\text{K}_2\text{O}$    | 0.45                 |
| $\text{TiO}_2$          | 0.23                 |
| Cl                      | 0.0019               |

Table 4. Information Mass Fraction Values for Calculated Compounds per ASTM C 150-20<sup>(a)</sup>

| Phase     | Mass Fraction (%) |
|-----------|-------------------|
| alite     | 64.80             |
| belite    | 12.27             |
| aluminate | 4.78              |
| ferrite   | 11.32             |

<sup>(a)</sup>ASTM C 150-20 provided in reference 9.

## REFERENCES

- [1] Stutzman, P.; Mundy, L.; Heckert, A.; *Certification of Standard Reference Material Clinker 2686b*; NIST Special Publication 260-204 (2021); U.S. Government Printing Office: Washington, DC (2021); available at <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-204.pdf> (accessed Jul 2021).
- [2] 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: Washington, DC (2020); available at <https://www.nist.gov/system/files/documents/srm/SP260-136.PDF> (accessed Jul 2021).
- [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(5), JAI101085 (2007).
- [4] ASTM C1365-18; Standard Test Method for Determination of the Proportion of Phases in Portland Cement and Portland-Cement Clinker Using X-Ray Powder Diffraction Analysis; Annual Book of ASTM Stand., Vol. 04.0 (2018).
- [5] Stutzman, P.E.; Feng, P.; Bullard, J.W.; *Phase Analysis of Portland Cement by Combined Quantitative X-Ray Powder Diffraction and Scanning Electron Microscopy*; J. Res. Natl. Inst. Stand. Technol., Vol. 121, pp. 47–107 (2016).
- [6] DerSimonian, 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(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/publications/guides> (accessed Jul 2021); 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 2021).
- [9] ASTM C 150/C150M-20; *Standard Specification for Portland Cement*; Annual Book of ASTM Stand., Vol. 04.01 (2020).

*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 at: telephone (301) 975-2200; email [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <https://www.nist.gov/srm>.*