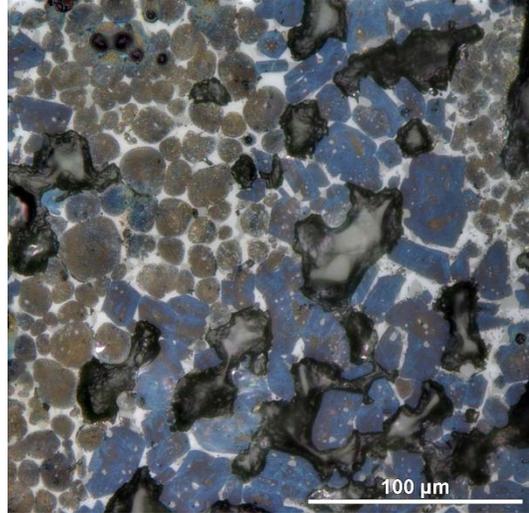


**Portland Cement Clinker for Phase Analysis:
Developing New Standard Reference Materials**
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A new Standard Reference Material® (SRM) for portland cement clinker has been produced for the Office of Standard Reference Materials at the National Institute of Standards and Technology (NIST). The SRM clinkers are intended for use in developing and testing quantitative methods of phase analysis for portland cement and cement clinker. The three available clinkers represent the range of textures and compositions of North American clinker production.

Clinker is composed of a set of crystalline phases that react with water to form the hydration products that bind the aggregates in portland cement concrete. Knowledge of the types and amounts of crystalline components of clinker is critical in monitoring quality control in clinker production and to produce cements conforming to specific cement types. Annual North American cement production of 94.4 million metric tons represents \$10.7 billion.



About 400 kg of clinker was subsampled by sieving to retain nodules ranging between +4 mm and -15 mm. Nodules were then stage crushed and sieved to capture the size fraction between 3 mm and 4 mm; re-crushing oversized material until the entire lot of clinker had been processed. The clinker was homogenized and packaged in glass jars each containing about 50 g of clinker. Jars were randomly selected for analysis with 25 containers for x-ray powder diffraction (XRD) and 12 for scanning electron microscopy (SEM).

SRM 2686b is a medium-grained, heterogeneous phase distribution clinker similar in phase composition and texture to the original SRM 2686. Certification of phase abundance was accomplished using quantitative XRD and SEM with image analyses. The XRD analyses incorporated data from selective extractions using salicylic acid to concentrate the interstitial phases. These methods provide mutually unique means to establish phase abundance, which subsequently are combined to establish certified values and uncertainties. The clinker selected for SRM 2686b contains medium-grained silicates with streaks and nests of belite along with a fine-grained interstitial matrix consisting of aluminates and ferrite. Periclase crystals as 1 μm to 10 μm equant inclusions scattered throughout the clinker and alkali sulfates occurring along grain boundaries. This clinker differs from the earlier SRMs by the presence of α -C₂S. Since SEM imaging does not distinguish between the α and β polymorphs of C₂S, a certified value representing the combined mass fraction is provided and informational values are provided for the individual polymorph fractions. For alkali sulfates and free lime, phase abundance is established using a single method (XRD) so only informational values are provided.