

**Addressing the Amorphous Content Issue in Quantitative Phase Analysis:
The Certification of NIST SRM 676a**

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The measurement of phase abundances within mixtures is accomplished with relative ease via diffraction methods. Advances have led to improved accuracy; however, the issue of the amorphous, or non-diffracting, fraction within the unknown has relentlessly lurked in the background. The matter can be readily addressed with the use of the internal standard method if the diffraction experiment is accurate, and the standard is characterized with respect to phase purity. With this in mind, we certify the primary NIST quantitative analysis SRM 676a, alumina, with respect to amorphous content.

The experimental approach was based on the comparison of the phase abundance of mixtures of silicon and SRM 676a. The silicon used for this study was intrinsic, float-zone material used for SRM 640c. After comminution, the powder was fractionated into five lots with respect to particle size, and thus surface area, and admixed with SRM 676a. Neutron TOF and high-energy synchrotron, 25 keV, and on a different beamline, 67 keV data were collected. The mass fraction of silicon, determined via QRA, may then be plotted relative to the surface area, or amorphous content, of the silicon. An extrapolation to the point that silicon possesses “zero” amorphous content, with a comparison to the prepared mass fraction of the mixtures, yields the absolute phase purity of SRM 676a. The concurrence of results from the three diffraction methods lends credence to the result: a certified phase purity of SRM 676a of 99.02 % ± 1.11 %.