The X-ray powder diffraction method has an exceptionally broad range of applications owing to its relative ease of use and its applicability to any crystalline material. Furthermore, the powder diffraction method can access a considerable breadth of sample characteristics: qualitative and quantitative phase analysis as well as crystal- and micro-structure determinations. Each application presents a specific challenge with regards to the development of a standard, both in the context of the character of the standard itself and the method by which it is certified. The success of an SRM is critically dependent on obtaining feedstock of the appropriate microstructure, either by selecting a material from commercial products or the development of a custom fabrication method. While the microstructure engineering may be individually tailored to each SRM, often the certification procedure falls into one of two methods. The majority of powder diffraction SRMs are certified with an SI traceable measurement of lattice parameter to yield a standard for calibration of line position. The second of the principal methods quantifies the crystalline phase purity, or conversely the amorphous content, and yields SRMs certified for quantitative analysis. The overall technical approach employed to certify NIST SRMs for X-ray metrology can be divided into three subtopics: 1) microstructure engineering or evaluation to obtain SRM feedstock suitable to address the measurement issue at hand, 2) the design and commissioning of the equipment that embodies the unique features required for SI traceable measurements on powders, thin films and single crystals, and 3) the design of experiments, measurement methods and data analysis strategies that allow for a valid assessment of systematic uncertainties of the measurement quantities.

For example, SRM 660b was prepared in a custom, dedicated processing run using the $^{11}$B isotope enriched to a nominal 99% concentration. The native $^{10}$B isotope is essentially opaque to neutrons and, as such, this isotopic enrichment renders this SRM relevant to the neutron diffraction community. The resulting powder was then annealed to "remove" microstructural defects and produce a uniform crystallite size of approximately 0.5 μm. The success of SRM 660b, reflected in the doubling of sales rates relative to SRM 660a, has been largely as a result of the microstructure engineering component of the NIST SRM effort. Owing to the specialized requirements of our program, all of our equipment is designed, built and commissioned in house and located in the temperature controlled NIST Advanced Measurement Laboratory. SI Traceable measurements of lattice parameters and layer spacings on powders and thin film structures can be performed with a uniquely featured, high-resolution parallel beam diffractometer. A second machine configured with a divergent beam is also used for SRM certification, homogeneity testing, microstructure and quantitative analysis. Additional equipment includes a double crystal diffractometer used to evaluate diffraction optics and a dedicated X-ray reflectometer. A NIST SRM must include valid estimates of uncertainty on certified measurement values. While the Type A uncertainties can be determined through statistical analysis, the Type B, systematic uncertainties can only be determined through a technical understanding of the measurement method(s). Experimental designs and data analysis methods are developed to impart to the experimentalist the information necessary for a meaningful assessment of Type B, systematic errors.