

MATRIX-INDEPENDENT XRF METHODS FOR CERTIFICATION OF STANDARD REFERENCE MATERIALS

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The NIST Analytical Chemistry Division has as its primary mission the improvement of chemical metrology in support of the United States economy. One way we do this is to develop new analytical methods for application to industrial, environmental and healthcare problems. Another way is development of Standard Reference Materials[®], with which users can establish traceability to NIST calibration and measurement certificates. The broad scope of our mission causes us to analyze a wide variety of materials. To make an SRM[®] useful, all sources of analytical uncertainty must be understood and minimized. To create SRMs more efficiently and with lower uncertainties, we need matrix independent methods for our spectrochemical techniques.

The term 'matrix independent method' could be the subject of an interesting debate. Even if one restricts the debate to the technique of X-ray fluorescence spectrometry, the discussion might cover fundamental parameters, matrix correction algorithms, and a number of calibration techniques. This paper is based on one possible definition that is useful for reference materials development, viz.: 'A matrix independent XRF method is one through which the calibration of the spectrometer can be directly related to primary reference materials and through which a variety of materials of different matrix can be directly related to said calibration without the use of mathematical correction procedures and matrix-matched reference materials'. This approach sets aside matrix corrections in favor of destructive and somewhat labor-intensive sample preparation. Reference materials can be reserved for detection of bias in the analytical result instead of being used for calibration.

This discussion covers recent work on cements, zeolites and metals using the method of borate fusion to demonstrate this path toward matrix independence for XRF. Sources of random error are minimized by taking advantage of the excellent repeatability of borate fusion, analytical balances, and X-ray spectrometers. Sources of bias are minimized by preparing bracketing calibration standards from ultrahigh purity compounds and NIST Single Element Solution Standards. A typical error budget is itemized and explained. Values for expanded uncertainty are at the level of one percent relative or lower. The techniques are applicable to major, minor and sometimes trace constituents. Excellent agreement is obtained with classical methods and other instrumental techniques.