APPLICATIONS OF XRF IN CRM DEVELOPMENT AT THE NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY

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At NIST, XRF is applied to many different materials of interest to industrial laboratories, product testing laboratories, and others for the purpose of value assignment of certified reference materials (CRMs). The XRF specialists in the Analytical Chemistry Division are located in the Inorganic Chemical Metrology Group, but they work with both inorganic and organic materials in both solid and liquid forms. When it comes to materials, nothing can be considered routine. Rather, there are a few functions that are almost routine, including development of Standard Reference Material (SRM) candidate materials, homogeneity testing, borate fusion sample preparation, and quantitative calibrations. Therefore, the presentation will illustrate these functions by highlighting applications to different materials.

The most recent example of development of an SRM candidate material is the design, prototype evaluation, and production of paint films for validation of test methods for Pb in children's products. In this project, the analysts have applied WDXRF, microXRF, high-definition XRF, and portable XRF instrumentation to characterize paints on toys, study the paint film drawdown process, and evaluate prototype films from a contractor.

Homogeneity testing procedures are divided into bulk materials and microheterogeneity. Evaluation of bulk powder, alloy, and liquid materials is done using WDXRF with large spot sizes to acquire high-precision data for ANOVA characterization of within and between unit variances. Microheterogeneity is evaluated using microXRF to measure centimeter scale areas with a 50 μm beam. Principle components analysis is used to characterize heterogeneity arising from dispersion of elements as a function of the quantity of material viewed.

For quantitative determinations, borate fusion is the preferred sample preparation tool at NIST. Nearly all inorganic materials are prepared by borate fusion because it allows creation of customized, synthetic calibrants. As a consequence, the XRF analysts are continuously creating new fusion procedures, which involves anything from optimizing an existing procedure for a related material, to adaptation of a published procedure, to creation of a new procedure. The most recent example is fusion of nanoparticulate TiO_2 to determine total Cl in 0.25 g specimens.

Quantitative determinations frequently involve upwards of 20 elements and one of a number of calibration options: semi-empirical calibrations with influence coefficients, reconstitution using fused beads, and high-precision comparisons to existing SRMs of nearly identical composition, for example low alloy steel SRMs. One of the more interesting recent examples is the analysis of lead-free solder manufactured by a semi-chill casting process. Calibrations for SRM 1728 used a variety of old NBS Standard Samples and newer commercial reference materials for the elements Al, Si, P, S, Cr, Fe, Ni, Cu, Zn, As, Ag, Cd, Hg, Pb, Bi, and Sn.