FROM THE FIELD TO THE LAB –
FDA USE OF XRF TO MONITOR FOODS, DRUGS, AND OTHER CONSUMER PRODUCTS

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Although XRF is a mature technique, the first known published work by an FDA scientist on the use of XRF to analyze foods and/or drugs did not appear until 2003. Since that time, researchers at FDA's San Francisco District Laboratory, Center of Food Safety and Applied Nutrition, and Center for Drug Evaluation and Research have used it for a number of applications. In 2010, FDA invested significant funds to procure handheld, portable, and lab-based XRF equipment for two major purposes: (1) to rapidly screen for the presence of toxic elements in various products in the field, and (2) to enable accurate quantification of elements in samples for either regulatory purposes and/or to protect the public health.

The authors' experience with XRF spans only the past seven years. It should be noted that the workshops and scientific sessions at the Denver X-Ray Conference have played a significant role in their understanding of this technology. Initial FDA studies utilized a radioisotope-based portable XRF analyzer and later expanded to include more detailed evaluation of 10 different XRF instruments from various vendors. For field use, an integrated radiation shield was deemed necessary to ensure ALARA (keeping radiation exposure "as low as reasonably achievable"), and an integrated PC with a large display was preferred over the small handheld or palm-sized displays. For lab use, this same instrument is preferred by lab analysts to pre-screen samples prior to the low-level ICP-MS process stream, and occasionally to perform accurate and reliable quantification of various elements ranging from low ppm to percent levels. For accurate quantification of larger numbers of samples, lab analysts use a more conventional XRF instrument equipped with features such as sample rotation, an autosampler, full user control of excitation conditions and method parameters, and automatic report generation.

The authors developed two different 4-day workshops to train FDA staff to effectively utilize this equipment, each with a strong emphasis on hands-on training. FDA Consumer Safety Officers (CSOs) typically have only one year of college level chemistry, and hence are given appropriate training on XRF theory and spectral interpretation. Since the algorithms on portable XRF analyzers are surprisingly unsophisticated and still generate false positives and false negatives, the CSOs are instructed to *confirm* any positive findings of a toxic element by looking at the spectrum to verify that two peaks are present at the correct energies and proper intensity ratios. This training appears to be successful in that the CSOs have a 100% success rate in proficiency tests on "real world" samples, and have correctly identified a number of supplements containing toxic elements in several different field assignments. A second more in-depth training course was developed for lab analysts, who usually have a strong background in analytical chemistry and atomic spectrometry. Here, the analysts obtain experience in using various quantification models including Fundamental Parameters, Compton Normalization, empirical calibration with authentic standards, and the method of standard additions.

To date, FDA has used XRF in an ever growing number of applications. In field screening, XRF has been used to identify products which are grossly unsafe, such as a Sindoor powder and children's makeup that appeared to be pure lead tetroxide and lead oxide, respectively. FDA is also using portable XRF to screen for imported and domestic supplements that contain potentially toxic levels of chromium, lead, arsenic, mercury, and selenium. In the lab, implementation of XRF as a routine tool faces some resistance due to the predominance of ICP-MS and the perception of XRF as being "only semi-quantitative". Although quantification is rendered more difficult by the wide variability in sample matrices and the lack of suitable standards or reference materials, a number of studies have shown that XRF can give results that are statistically equivalent to more "proven" methods. More importantly, XRF sample preparation procedures typically involve homogenization, and hence are much faster and easier than ICP-MS procedures which require acid digestion and filtration.

Clearly, XRF is beginning to fill an important niche within the FDA and can be deemed to be complementary to ICP-MS for elemental analysis and toxic/heavy metal applications. This presentation will describe the evolution of XRF within the FDA, summarize the training programs for CSO's and laboratory analysts, and review some of the various consumer products that have been successfully analyzed via XRF.