

COMPARISON OF VARIOUS XRF QUANTITATIVE METHODS FOR DETERMINATION OF TOXIC ELEMENTS IN SUPPLEMENTS

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There has been increasing media and public scrutiny of the ongoing problem of toxic elements in dietary supplements. The CA Department of Public Health compiled a report describing Chinese herbal medicines known to contain one or more toxic elements (1). A study by Saper et al on 70 different Ayurvedic medicines sold in Boston area stores found detectable levels of Pb in 19% of the products with levels as high as 4%, and Hg in 9% of the products with median value of 2% and a maximum level of 10% (2). For the most part, these studies have relied on the use of techniques such as Atomic Absorption Spectrophotometry (AAS) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS) for quantitation. XRF is well suited for the analysis of products such as these which may contain toxicologically significant levels of elements in a more concentrated form.

The focus of this study is the XRF analysis of 28 different dietary supplement products via several different quantitative methods. A study by Dolan et al on these same products reported As, Hg, and Pb levels ranging as high as 4, 17, and 49 ppm via ICP-MS (3). These samples represent a considerable challenge for XRF given spectral overlaps of As and Pb, significant variability of sample matrices, and element concentrations close to the detection limits. Both portable and lab-based XRF instruments were employed in an attempt to mimic how such products might be screened in the field and/or analyzed using more rigorous lab-based quantitative methods. Samples were placed into sample cups and analyzed “as is” via a handheld XRF instrument using both factory and empirical calibrations, and the same sub-samples were homogenized and analyzed via lab-based XRF using fundamental parameter-based quantitation and empirical calibrations. This presentation will compare the results from these different quantitative methods and will hopefully demonstrate the suitability of XRF for this application.

1. Ko, R., Au, A. *Compendium of Asian Patent Medicines*, California Dept. of Health Services, 1997-1998.
2. Saper, R.B., Kales, S.N., Paquin, J., Burns, M.T., Eisenberg, D.M., Davis, R.B., Phillips, R.S., *JAMA* **2004**, *292*, 2868-2873
3. Dolan, S.P., Nortrup, D.A., Bolger, P.M., Capar, S.G. “Analysis of Dietary Supplements for Arsenic, Mercury, and Lead Using ICP-MS, *J. Agric. Food Chem.* **2003**, *51*, 1307-1312.