Quality Assurance in Trace Elemental Analysis-A Historical Perspective

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The first significant effort at evaluating the precision and accuracy of tract element determination was conducted under the auspices of the U.S. Geological Survey1 (USGS). The composition of two igneous rock standards, G-1 (granite) and W-1 (diabase), were evaluated in 35 laboratories around the world (with principally gravimetric, colorimetric, and spectrochemical methods), and the results of these intercomparisons were summarized by Chayes (Ref. 1, p. 67):

> Viewed in this light the scatter of chemical results…must profoundly disturb every petrologist. Normative variation is so extreme that in the absence of direct evidence to that effect there would by little reason to suppose all these analysises were of the same two rocks

These observations sparked the analytical community into beginning the arduous task of developing standards appropriate for validating methods for various sample types, particularly geochemical, biological, and environmental matrices. These included biological standards such as Bowen's Kale, environmental standards National Bureau of Standards (National Institute of Standards and Technology) Coal Fly Ash standard reference materials (SRM) 1633, Coal SRM 1632, additional geological standards (e.g., G-2, W-2, BCR-1, GSP-1, AGV-1 produced by the USGS), and many others. Instrumental and radiochemical neutron activation analysis (NAA) played and continues to play a significant role in the validation of these reference materials and SRMs that are critical to the development and evaluation of analytical methods. Instrumental NAA was of particular importance to the development of these standards because it allowed the analyst to directly analyze the samples with little chance of contamination caused by handling and dissolution. The data from the validation of these standards have been the subject of many publications. NAA also provided a very sensitive method for the determination of thorium and uranium, both of significant interest for nuclear materials. Even with the many advances in analytical methods today, NAA and radiochemical NAA still compete as the method of choice, particularly for difficult-to-dissolve matrices, because of their ability to avoid contamination caused by handling and dissolution.

From these early beginnings the modern programs of today have evolved with the same guiding principal, i.e., to ensure that the analytical data have acceptable precision and bias for the purposes for which the analyses were made. We provide a review of the historical development of quality assurance for trace element analysis with emphasis on nuclear techniques. In recent years, NAA and other radiochemical techniques have not been include in the Annual Fundamental Review of Analytical Chemistry, and many of today's chemists are untrained in these techniques. However, the historical significance of NAA in improving the reliability of trace element analysis standards has forged for it a role as one of the standard methods for validation of reference materials. However, only

with renewed support for research reactor facilities that conduct these experiments can NAA hope to continue to play a role in quality assurance.

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