

A55 Choose Your Own (Accreditation) Adventure in the Defense POW/MIA Accounting Agency's (DPAA's) Isotope Testing Program: Part I—Sample Preparation

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Learning Overview: After attending this presentation, attendees will have learned about the work completed to accredit isotope sample preparation under International Organization for Standardization/International Electrotechnical Commission (ISO/IEC) 17025:2017 for Geographic Profiling (i.e., isotope testing) of human remains.

Impact on the Forensic Science Community: This presentation will impact the forensic community by illustrating the process used by the Defense POW/MIA Accounting Agency (DPAA) Laboratory to prepare for accreditation for isotope testing—specifically sample preparation—of unidentified individuals.

The DPAA Laboratory began investigating the utility of isotope testing for identification in 2012. The technique provides information about an individual's life history, as human tissues record the isotopic signatures of ingested water and food. These signatures can and do vary geographically. At the DPAA, the program is used to refine short lists for identification, to separate commingled remains, to eliminate non-United States remains from the DNA testing stream, and to add another line of evidence for an individual identification. Initially, isotope testing by the DPAA was completed through contracts with external providers for both sample preparation and analysis.

In 2017, the DPAA Laboratory in Hawaii began developing in-house capabilities for the preparation and analysis of osseous and dental remains for the “bio” elements—carbon, nitrogen, oxygen, and sulfur. One goal was to accredit isotope testing under ISO/IEC 17025:2017, appearing on the DPAA Laboratory's Scope of Work as “Geographic Profiling.” This presentation describes the work completed in 2019 to accredit isotope sample preparation with the American National Standards Institute National Accreditation Board (ANAB). Another presentation describes work completed in 2020 to accredit isotope sample analysis.

The initial step for accrediting isotope sample preparation was site validation, which focused on facilities, equipment, and training. Facilities were surveyed and deemed adequate, with sufficient security protocols, space, lighting, and climate control. Work areas were determined to be free of environmental influences that could adversely influence preparation processes (e.g., vibrations from the nearby airfield). Equipment was deemed appropriate and functional. A system of annual performance checks for equipment was developed. Full-time analytical personnel were hired in 2017 and 2018; their first priority was the development of a Standard Operating Procedure (SOP). The SOP codified the methods used in sample preparation. Once finalized, all personnel were trained and tested in specific SOP modules. Following initial competency certification, annual proficiency exams were implemented for the analysts.

Preparation methods used for osseous remains were adapted from ones developed and validated by the Stable Isotope Preparation Laboratory at California State University, Chico, an external provider for the laboratory since 2012. The methods were validated again at the laboratory to ensure comparability of measured isotope delta values, with 50 bone samples halved and then prepared separately at each location.¹ Replicability (i.e., samples having the same test results given multiple preparations over time) was successfully verified using six samples of human bone and six samples of animal bone that were subsampled, then prepared in triplicate over a four-month period.

Finally, monitoring activities were developed for quality management of sample preparation. Animal bones were identified that could be prepared alongside casework samples following the “Identical Treatment” (IT) principle.² Used as a means for monitoring consistency in isotope test results, the IT principle requires that samples and reference materials are similar in chemical nature, subject to the same preparation methods, and analyzed in the same manner. Test results for these animal bones can help analysts assess replicability; they are also useful for the detection of trends that could be indicative of detrimental changes in preparation processes. As an example, a change in acid strength that resulted in slower rates of bone demineralization was found to have no impact on measured isotopic signatures of the standards.

Most of the work was completed in-house by qualified personnel; however, a disinterested external subject matter expert was also utilized. The expert completed a review of the sample preparation modules in the SOP and an onsite “over-the-shoulder audit” (i.e., “witnessing”) prior to the final on-site assessment by ANAB. The results of the review and audit prepared the laboratory for the assessment and found no discrepancies between the validated methods and the expected outcome. After all work was completed, the DPAA applied for, and was granted, accreditation of isotope sample preparation in November 2019.

Reference(s):

1. Chesson, L.A., A.J. Edwards, E.J. Bartelink, T.H. Chau, G.E. Berg. 2020. Assessing Isotope Data Comparability: An Example from the Application of Isotope Testing to Unidentified Human Remains from Past Conflicts. *Proceedings of the American Academy of Forensic Sciences, 72nd Annual Scientific Meeting*, Anaheim, CA. 2020.
2. Carter J.F. and B. Fry. 2013. Ensuring the reliability of stable isotope ratio data—Beyond the principle of identical treatment. *Analytical and Bioanalytical Chemistry* 405:2799-2814.

Method Validation, Isotope Analysis, Quality Assurance (QA)