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Mixture Stability of Stable Isotopic Gases

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The dependence of stable isotopic analyses in geochemistry and environmental measurements are steadily increasing. However, the ability to compare measurements between laboratories can be quite challenging owing to differences in measurement and calibration. The key to obtaining reliable data is by designing experiments that utilize sampling methodologies that represent the environment intended for the study and meet the data-quality objectives. These samples in turn must then be calibrated against suitable reference materials containing low levels of uncertainty. The precision and accuracy of the analytical result is directly related to the precision and accuracy of the standard used to calibrate the analytical instrument.

Isotopic primary reference materials, which utilize various laboratory extraction techniques (depending on the isotope of interest), are available in extremely limited quantities from the International Atomic Energy Agency (IAEA) and NIST. However, quantities of these materials are typically limited to one reference material per laboratory every 3 years. Laboratories are encouraged to develop their own working standards that can be used to calibrate samples. Preparation of these standards often increases the total uncertainty of the measurements. It is good analytical practice to matrix match the standard and sample as well as bracket the standards around the concentration of the samples. Ideally, 3 standards are the minimum required to assess linearity and accuracy. How can this be accomplished with the limited range of standards available?

This presentation will examine the isotopic mixture preparation process, for both molecular and isotopic concentrations, for a range of components and delta values illustrating stable shelf life. The role of precisely characterized source material will be presented. Analysis of individual cylinders within multiple batches will be presented to demonstrate the ability to dynamically fill multiple cylinders containing identical compositions without isotopic fractionation. Additional emphasis will focus on the ability to adjust isotope ratios to more closely bracket sample types without the reliance on combusting naturally occurring materials, thereby improving analytical accuracy.