A Review of the Performance of the “Imperial Batch EXtraction” (IBEX) Device for Clumped Isotope Analysis

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Because clumped isotopes are based on measuring rare isotopologues, a long-integration time to increase signal-to-noise ratio and thorough cleaning of the CO₂ gas to avoid contaminants are important prerequisites. As a consequence, relatively large samples are required, and the total integration time on the mass spectrometer is long (>6-9 hours/sample). Furthermore, the cleaning procedure used to prevent contaminated gas to enter the mass spectrometer is intricate and time consuming. The long preparation time coupled with the need for data consistency favors the use of automated lines: these allow for data acquisition over prolonged periods of time, and avoid variations in sample preparation that typically arise when researchers of varying experience use a manual vacuum system. The “Imperial Batch EXtraction” (IBEX), done in collaboration with Protium, is the first fully-automated preparation device using gas chromatographic separation designed for clumped isotope analysis. Other semi-automated devices coupled to GC systems exist at CalTech and elsewhere, but these generally do not include automatic liquid nitrogen refill. Alternatively, the Thermo Kiel device can be modified to include a poropak trap, but this lacks a full-GC capability with a carrier gas and thus may not be capable of cleaning difficult samples.

The IBEX design includes a common acid bath set at the operator’s temperature of choice, a multi positions (currently 40) auto-sampler, and a series of automated traps. The CO₂ resulting from carbonate acidification is first capture in a trap set at liquid nitrogen temperature, then released by heating the trap to -100°C. The gas is passed using a carrier gas (helium) through two sulfur traps containing silver wool and a 1-meter GC column containing poropak-Q. The gas is then captured in a second water-CO₂ trap, and finally transferred to a micro-volume before being inlet into the mass spectrometer for clumped isotope analysis. The traps are cooled using liquid nitrogen stored in a central dewer that is automatically refilled from a tank when needed. The IBEX has dedicated control software written in C#, a method editor allowing the operator to change the analysis parameter, and the system is currently fully integrated with the Thermo Isodat software. The IBEX also has the capacity to run on any IRMS currently available.

This presentation will focus on the results obtained on the IBEX, and the data will be shown using Easotope, a free software for clumped isotope corrections (John and Bowen, this meeting). We will discuss the reproducibility of the IBEX for both clumped isotopes and bulk isotopes, and show data from both standards and unknown samples. Of particular interest is the reliability of the IBEX, and how well samples can be cleaned using this system. Initial results suggest that sample cleaning is superior to the manual line.

Sample size is another important parameter to consider: current routine replicate size is 3 mg of carbonates, but initial testing suggests that the IBEX performs well with samples of 1.5 mg carbonate, and that this could potentially be further reduced. In conclusion, the IBEX has proven to produce reliable data from both samples and standards in a fully automated way, and the typical sample size needed for one replicate is the smallest currently performed in dual inlet mode using the bellows on a MAT 253.