

## **$^{15}\text{N}$ & $^{18}\text{O}$ ANALYSIS OF NITRATES ( $\text{NO}_3$ ) IN AQUEOUS SAMPLES**

### *Sample preparation:*

Nitrate is concentrated and collected in a 5 mL anion exchange column, after the resin has been cleaned and rinsed with acid and deionized water respectively. The volume of water required to collect sufficient nitrate for isotopic analysis is dependent on the concentration, and can range from 10 mL to several litres of water. Nitrate concentration data is required with sample submission. Prior to running samples through the column, sulfate is removed through additions of barium chloride to precipitate barium sulfate, which is then filtered out of the samples. Sulfate removal is required to prevent the column from preferentially binding sulfate over nitrates. Once samples have been collected from the column, chloride removal is required through additions of clean silver oxide. Silver chloride is then filtered from the samples prior to sample freezing. Samples are frozen overnight, freeze dried the following day, and ready for weighing for isotopic analysis.

### *Sample Analysis:*

Dried samples are weighed into tin cups for separate  $^{18}\text{O}$  and  $^{15}\text{N}$  analysis with a replicate every 3 samples. Approximately 0.1 mg of sample is used for  $^{18}\text{O}$  analysis.  $^{18}\text{O}$  samples are combusted at  $1430^\circ\text{C}$ , and purified by gas chromatography before continuous flow isotope ratio mass spectrometry. Analysis is carried out on a Finnigan Mat, DeltaPlus XL IRMS coupled with a Thermo Scientific TC/EA. Data is corrected and normalized using four international standards: USGS 32, NBS 127, IAEA SO5, and IAEA SO6, that bracket the samples. Standards are analyzed at the beginning and end of every run.

The analytical precision for analysis is  $\pm 0.5\%$ .

Analysis for  $^{15}\text{N}$  is carried out on a Finnigan Mat DeltaPlus IRMS with ConFlo III Interface coupled with a CE instruments EA 1110 CHN. Data is corrected and normalized using three international standards, IAEA-N1, IAEA-N2, IAEA-C6, and four calibrated internal standards, that bracket the samples. Standards are run at the beginning, middle and end of every run. The results are evaluated and corrected against standards run with the samples, and then reported against the international reference material.

The analytical precision for analysis is  $\pm 0.3\%$ .