^{81}Br BROMINE ISOTOPE ANALYSIS BY CONTINUOUS-FLOW ISOTOPE RATIO MASS SPECTROMETRY

Sample Preparation:

Bromine stable isotope analysis is conducted on methyl bromide (CH_3Br) gas after converting bromide ions, in solution, to CH_3Br gas through a multi step procedure. Briefly, the separation is conducted in a special distillation apparatus. Potassium dichromate and sulfuric acid are used to oxidize the bromide in the sample. The produced bromine gas is trapped in a potassium hydroxide solution, where KBr and KBrO are formed. The solution is boiled after zinc powder addition in order to reduce all BrO^- ions in solution to Br^-. The solution is then filtered and precipitated as silver bromide through the addition of nitric acid, potassium nitrate, and silver nitrate. When complete precipitation has occurred, samples are rinsed with nitric acid (5%) and dried. Silver bromide is then reacted with methyl iodide to form CH_3Br gas. For the reaction to proceed to completion, vials are placed in an oven for 56h at 80°C. Typically, at least 2 mg of Br^- is required for analysis, however, samples with concentrations as low as 0.1 of Br^- can be analyzed using this method.

Sample Analysis:

The ratio of bromine stable isotopes ($^{81}Br/^{79}Br$) are determined by a continuous-flow isotope ratio mass spectrometry. An IsoPrime, Micromass is used to measure ^{81}Br, and an Agilent 6890 gas chromatograph (GC) equipped with a CTC Analytics CombiPAL autosampler is attached to the IRMS for CH_3Br separation. Sample and standard measurements consist of measuring the separated CH_3Br against a set of reference gas (CH_3Br) pulses. Typically, the reference gas is measured 6-8 times and the isotopic ratio of the sample or standard peak is determined against the average readings of the reference gas. Each sample is measured 2-4 times. All results are corrected and reported against the Standard Mean Ocean Bromide (SMOB). A calibrated internal standard is used during every run.

The analytical precision for analysis is ± 0.2‰.

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