High precision analysis of three oxygen isotopes in silicates using an automated laser fluorination
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Introduction: The precise determination of the oxygen three isotope compositions in terrestrial/extraterrestrial materials is important to understand the geochemical/cosmochemical processes related to their origin and secondary effects. High precision oxygen isotope analysis can provide understanding for isotopic heterogeneity of early solar system and origin of undifferentiated and differentiated meteorites [1]. Furthermore, it also can reveal an isotopic variation of terrestrial silicates caused by mass dependent fractionation (e.g., rock-water interaction) [2, 3].

An infrared laser fluorination method coupled with dual-inlet isotope ratio mass spectrometer is widely used as a fundamental system for oxygen extraction from silicate samples and can perform most precise oxygen three isotope analysis. A CO2 laser-BrF5 fluorination system for oxygen three isotope measurement at the Korea Polar Research Institute (KOPRI) was refurbished a new vacuum line made of stainless steel [4]. O2 liberated from silicate sample (~2 mg) by laser fluorination is purified using cryogenic technique in vacuum line. The 17O/16O and 18O/16O ratios of the extracted O2 are measured with the mass spectrometer (MAT 253 Plus, Thermo Fisher Scientific). Recently, we have adopted an automated lasing method that provides a constant increase rate of laser power output with defocused laser beam at a fixed position. To confirm the reproducibility of the laser fluorination system, we have analyzed well-known international standard (UWG2 garnet, San Carlos olivine and NBS28 quartz) and in-house standard samples (mid-ocean ridge basalt glass and obsidian). The analytical precisions (1σ) for δ18O and Δ17O values of each standard samples are ±0.04‰ and ±11 ppm for UWG2 garnet (n = 24), ±0.07‰ and ±9 ppm for NBS28 quartz (n = 10), ±0.08‰ and ±11 ppm for San Carlos olivine (n = 9), ±0.09‰ and ±10 ppm for MORB glass (n = 45) and ±0.07‰ and ±11 ppm for obsidian (n = 42). The results showed that the overall external reproducibilities are better than 0.1‰ (1σ) for δ18O and 11 ppm (1σ) for Δ17O. Now we can provide the oxygen isotope data more precise than those obtained from previously used laser fluorination system [5].

Analysis of the VSMOW (Vienna Standard Mean Ocean Water) and SLAP (Standard Light Antarctic Precipitation) reference water is underway to achieve high accuracy. Precise calibration of working standard oxygen gas relative to the VSMOW is necessary for intercomparison of isotope data between different laboratories [6, 7, 8]. The two-point normalization using these reference waters is expected to provide us with an improved determination of oxygen isotope ratio in silicates. Analysis of meteoritic samples is also under way to validate that our system is available for extraterrestrial silicate samples.