



Oxygen isotopes of diatom silica from El'gygytgyn Crater Lake, NE Russia - Analyses using a remotely-operated laser-fluorination based mass spectrometry unit

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The analysis of oxygen isotopes from diatom silica in sediment cores has reached importance for reconstructing the palaeoclimate and is especially valuable in non-carbonate lakes of cold regions, where no other bioindicators such as ostracods are available. A new approach for samples in sub-mg range has been developed to provide a better chronological resolution and to expand the method to periods where little biogenic silica is available.

After extracting the diatoms from sediment cores with various preparation steps including wet chemistry, sieving and heavy liquid separation, a minimum of $\sim 700 \mu\text{g}$ fine material from 5 g of wet sample is obtained. The pure sample is melted to a bead to eliminate the enclosed water and reduce the surface. The used periphery of the mass spectrometer (PDZ Europa 20-20) consists of a laser fluorination system operated under vacuum. The silica bead is reacted with a CO_2 laser in a BrF_5 atmosphere. The non-oxygen gas components are trapped in a -150°C cold trap, whereas oxygen passes on to a molecular sieve cooled with liquid nitrogen. It is then transferred to the PDZ Europa 20-20, analysed and compared with a reference standard of known isotopic

composition.

Specially designed software and a video camera are used to survey and record the process in the reaction chamber, allowing an automatised, remote operation. This guarantees maximum safety as the instrument is installed in two different rooms with the reagent (BrF_5), the reaction chamber, and the laser unit arranged under a hood. The fluorination periphery is directly coupled to the mass spectrometer, making an on-line analysis possible. In addition, a direct comparison of oxygen is enabled due to omitting the intermediate step of reacting oxygen to CO or CO_2 .

The reference oxygen directed through different circles of the system shows a standard deviation of <0.2 permil. Tests on standard material (NBS 28, Campolungo) show a standard deviation <0.2 permil as well. The best laser settings for the preparation of the beads were determined in various test series. Using these settings no isotopical fractionation could be detected, neither in the preparation of the bead nor when reacting the bead.

Sample material from Lake El'gygytgyn, NE Russia (core Lz1024) is analysed and a $\delta^{18}\text{O}$ curve of the last 280.000 years complements a strong climate proxy to the existing data. The lake lies inside a meteorite impact crater formed approximately 3.6 million years ago and hence offers a unique option to fill the spatial gap of locations in the Arctic where palaeoclimate reconstructions are sparse. Former drilling operations show that the lake is likely to contain the longest, most continuous terrestrial record of past climate change in the entire Arctic back to the time of impact.

The pilot results from core Lz1024 are the base for studying the climate history using stable isotopes in lacustrine diatoms of the whole 400 m sedimentary sequence on sediment cores at Lake Elgygytgyn to be drilled within the frame of the ICDP deep drilling in 2009.