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## Oxygen isotopes of biogenic silica: Recent technical developments and lacustrine temperature calibration study

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Relevance of the oxygen isotope composition of biogenic silica as a palaeotemperature indicator has increased ever since the relation between water temperature and oxygen isotope fractionation was shown for the opaline skeletons of diatoms in the 1970th. The broader application of this proxy parameter has, however, largely been hampered by technical difficulties of sample purification and oxygen extraction as well as by uncertainties with respect to the degree of the temperature dependence of the oxygen isotope composition of diatom valves as found in different calibration studies.

We here report about two new techniques which have recently been developed in our laboratory in order to overcome the technical problems of sample preparation. A new method for the quantitative separation of diatom frustules from soft sediments has been developed based on split-flow fractionation. The so-called SPLITT technique (Rings et al., 2004) allows to gain pure quantitative samples of diatom opal from a sediment sample in different sieve fractions by applying micro-sieving and particle separation in a flow channel based on the different hydrodynamical properties of diatom frustules relative to mineral grains. Since such opal samples normally contain high and variable amounts of adsorbed water molecules, hydroxyl-groups and weakly bound oxygen atoms, these contaminants have to be removed reliably to determine the "true" oxygen isotope composition of the biogenic silica. For this purpose a new and relatively easy technique was developed which enables dehydration and silica disintegration within a single reaction chamber and avoids fluorination. The new method is based on the reduction (*iHTR*), and uses currently ca 1.2 mg of opal (Lücke et the set of t

al., 2005). A fast, stepwise heating process eliminates oxygen containing contaminants prior to the reduction of the silica by which carbon monoxide is generated and analysed off-line. Analytical precision of oxygen isotope measurements by this method is better than  $\pm 0.15$  per mill.

These methods were applied to perform a novel calibration study directly within a small lake of the temperate zone (Moschen et al.). The study spans more than a seasonal cycle with water temperature changes of about  $15^{\circ}$ C and revealed identical linear relations between the oxygen isotope composition of diatom opal and water temperature for three different diatom size fractions.

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