



Oxygen isotope analyses of fine powders using laser-extraction technique: application to sandstone and silcrete quartz cement analysis and comparison with oxygen isotope data obtained from ion microprobe analyses

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A simple protocol for oxygen isotope analyses of fine silicate grains using a laser fluorination technique is presented. A 30W CO₂ laser is used with a defocused beam, as advised by Spicuzza et al. (1998). Fine quartz standard powders lower than 500, 200, 100, 50, 20, 2 and 1 μm were processed successfully. Similar precision and analytical uncertainty of 0.15 per mille is obtained regardless of grain size. This is the first time an oxygen isotope analysis using laser fluorination of silicate particles lower than 20, 2 and 1 μm is presented.

This protocole is used for investigating paleoenvironmental conditions leading to formation of five silicified layers of a Cretaceous sedimentary outcrop (Vaucluse, France). On the basis of field and petrographic observations, diagenetic sandstone, groundwater silcretes and pedogenic silcretes were identified from the bottom to the top of the sequence (Basile-Doelsh et al., in press). $\delta^{18}\text{O}$ compositions of the cements are analysed using both the laser-fluorination technique after quartz separation, and the in-situ ion microprobe technique (SIMS analytical uncertainty: 0.3 per mille). Comparison of the results shows that when cements are composed of micro- and macro-crystalline quartz only, the two methods give similar values. In such a case, analysis of

fine particles after laser extraction is very interesting. When the cements are made of overgrowths, quartz and chalcedony, the laser technique gives an average $\delta^{18}\text{O}$ value lower than the $\delta^{18}\text{O}$ value of micro- and macro-crystalline quartz. The quartz cements of both groundwater and pedogenic silcrete give a similar $\delta^{18}\text{O}$ value of 26 per mille vs V-SMOW, indicating that they precipitate from similar low temperature conditions. $\delta^{18}\text{O}$ value of the cement of the sandstone reaches 31 per mille vs V-SMOW. Assuming a temperature of 40°C obtained from fluid inclusion analysis (Sabouraud and parron, 1977), the cement may have formed from a sea water with a $\delta^{18}\text{O}$ value of -1 per mille vs V-SMOW, in agreement with pre-glacial ocean $\delta^{18}\text{O}$ value. Assuming a terrestrial temperature ranging from 15 to 30°C, silcretes formed from water with $\delta^{18}\text{O}$ value ranging from -11 to -7 per mille vs V-SMOW, lower than the mean value of about -7 per mille vs V-SMOW assigned to modern precipitation in the area. A plausible paleo-environmental scenario is discussed.

Oxygen isotope analysis of fine silicate grains lower than 20 μm , using the laser fluorination technique, is a promising tool for investigating $\delta^{18}\text{O}$ compositions of chert formations as well as fossil biogenic siliceous particles such as diatoms and phytoliths, and their related paleo-environments.