Geophysical Research Abstracts
Vol. 15, EGU2013-5965-2, 2013
EGU General Assembly 2013

Application of a nanoEA-IRMS system for δ13C measurement of biomineral-bound organics in samples of diatom opal with nanomolar quantities of C

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We describe the isotopic measurement of δ13C in very small samples of diatom opal (nanomolar quantities of C) both from fossil sediments and cultures. We use a nano-EA system composed of a combustion elemental analyzer (EA3000 series, Eurovector), with standard 18 mm diameter quartz oxidation-reduction reactors and an ash removal device that aids in removal of uncombusted opal and ensures a long reactor lifetime. This is coupled to a custom designed trapping and cromatography system (Nano-CF, Nu Instruments Ltd.) which cryogenically removes CO_2 generated by sample combustion and introduces the gas into a low-flow helium carrier stream to the mass spectrometer (Nu Perspective IRMS instrument, Nu Instruments Ltd.).
This technique allows for an important reduction in the minimum sample requirements for analysis compared to a typical EA, however the need to reduce the contribution of the blank to the measured values becomes all the more critical. Blank from the capsules can be minimized through specific protocols including cleaning with solvents and reducing the size of the capsule by cutting it to a smaller size, attaining blanks as low as 13.75$\pm$2.15 nmol C. Under these conditions we can accurately measure both standards and diatom reference materials in the range of 100 to 330 nmol C, with a precision of 2σ < 1 ‰Ṫhe measured δ13C is independent of sample size in this range for standards or samples with δ13C < -11 ‰ which is the compositional range expected for natural diatom samples. Furthermore, no memory effect is observed in samples with an isotopic δ13C value differing by > 10 ‰ analysed in sequence.
Applied to measure biomineral-bound organics in cleaned diatom samples from sediments, the low sample size requirements of this technique allows us to analyse multiple size fractions within one sample, and explore isotopic fractionation patterns between them. We have analysed samples from sediments of both centric and pennate diatoms typically in the range of 200 to 1300 μg of cleaned opal. Analysis of culture samples of Thalassiosira pseudonana of 20 to 150 μg of cleaned opal, allows us to evaluate if there is covariation of the whole cell particulate organic carbon (POC) and diatom-bound δ13C values. Initial results suggest that diatom-bound δ13C is more depleted than POC.