Study on analytical technique of carbon isotopes for samples with minute and lean organic carbon content.

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Stable organic carbon isotope ratio ($\delta^{13}C_{org}$) of oceanic sediments is useful to discuss paleoceanographic change (e.g. increasing productivity on ocean surface, the global carbon cycle). However, very low total organic carbon content (TOC) makes carbon isotope analysis of organic carbon with regular equipment quite difficult. A shortage of stratigraphic record of $\delta^{13}C$ from pelagic sediments including chert is partly owe to it.

This study tried carbon isotope measurement of minuscule quantity of carbon and low TOC samples with techniques of elementary analyzer introduction interface (EA/IRMS) and dual inlet. EA/IRMS is online method that uses continuous-flow system and dual inlet is off-line method that is an analysis with IRMS of dual inlet type after cryogenic purification with a glass vacuum line. L-alanine were mainly used for experiments of EA/IRMS method as a standard material. For dual inlet method, standards (ANU-Sucrose, L-alanine, eicosane, triphenylamine) were mostly used. A few chert samples were used for each experiment as a natural sample. Experimental contents of EA/IRMS method were: 1) to know how minute the sample is enough to analyze TOC with elemental analyzer (EA), 2) to know how much weight of powdered sample can be introduced for the measurement of $\delta^{13}C_{org}$ and 3) to test actual organic carbon isotope analysis with chert sample. They demonstrated that EA/IRMS method can deal with carbon as much as 10 µg, one-fifth of suitable carbon weight for the conventional analysis in our lab. In spite of it, we judged that EA/IRMS method did not suit for measurement of chert as the volume of chert powder was too much to introduce to the equipment, as an analysis effected on the next analysis (memory effect), and as it damaged the analytical device. For dual inlet method, 1) estimation how minute amount of CO_2 gas is able for the analysis, 2) evaluation the stability of $\delta^{13}C_{org}$ for minuscule samples, 3) evaluation of calibration line by multiple standard samples, 4) preliminary organic carbon isotope analysis of actual chert sample, were conducted in this study. The results showed that the values obtained with 50 µgC standards were similar to that with 500 µgC standards (the amount for regular analysis). Moreover, dual inlet method demonstrated that $\delta^{13}C_{org}$ value from chert (~700 mg) were not stabilized (showed >1‰ difference) in replicated sample. On the other hand, the difference of $\delta^{13}C_{org}$ between 760 mg of diluted L-alanine (with baked quartz sand) controlled as low TOC (0.007%) and non-diluted L-alanine 0.1mg was about 0.5‰ showing more stable result than chert samples. Although analysis of standard samples with 50 µgC and low TOC condition with dual inlet method is nealy achieved, problems related to specific character of chert may be remained unresolved for organic carbon isotope analysis of chert.

Keywords: organic carbon isotope ratio, low total organic carbon content (TOC), dual inlet, Elemental Analyzer/ Isotope Ratio Mass Spectrometer (EA/IRMS)

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