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Analysis of dissolved organic carbon concentration and 13C isotopic signature by TOC-IRMS – assessment of analytical performance

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Stable carbon isotopes provide a powerful tool to assess carbon pools and their dynamics. Dissolved organic carbon (DOC) has been recognized to play an important role in ecosystem functioning and carbon cycling and has therefore gained increased research interest. However, direct measurement of 13C isotopic signature of carbon in the dissolved phase is technically challenging particularly using high temperature combustion. Until recently, mainly custom-made systems existed which were modified for coupling of TOC instruments with IRMS for simultaneous assessment of C content and isotopic signature. The variety of coupled systems showed differences in their analytical performances. For analysis of DOC high temperature combustion is recognized as best performing method, owing to its high efficiency of conversion to CO_2 also for highly refractory components (e.g. humic, fulvic acids) present in DOC and soil extracts. Therefore, we tested high temperature combustion TOC coupled to IRMS (developed by Elementar Group) for bulk measurements of DOC concentration and 13C signature. The instruments are coupled via an Interface to exchange the carrier gas from O_2 to He and to concentrate the derived CO_2 for the isotope measurement.

Analytical performance of the system was assessed for a variety of organic compounds characterized by different stability and complexity, including humic acid and DOM. We tested injection volumes between 0.2-3 ml, thereby enabling measurement of broad concentration ranges. With an injection volume of 0.5 ml (n=3, preceded by 1 discarded injection), DOC and 13C signatures for concentrations between 5-150 mg C/L were analyzed with high precision (standard deviation (SD) predominantly <0.1‰, good accuracy and linearity (overall SD <0.9‰. For the same settings, slightly higher variation in precision was observed among the lower concentration range and depending upon specific system conditions. Differences in 13C signatures of about 50‰ among samples did not affect the precision of the analysis of natural abundance and labeled samples. Natural DOM, derived from different soils and assessed at various concentrations, was measured with similar good analytical performance, and also tested for the effect of freezing and re-dissolving.

We found good performance of TOC-IRMS in comparison with other systems capable of determining C concentration and isotopic signatures. We recognize the advantages of this system providing:

- High sample throughput, short measurement time (15 minutes), flexible sample volume
- Easy maintenance, handling, rapid sample preparation (no pretreatment)

This preliminary assessment highlights wide-ranging opportunities for further research on concentrations and isotopic signatures by TOC-IRMS to elucidate the role of dissolved carbon in terrestrial and aquatic systems.