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Comparison of total uncertainty in off-line dual inlet and online continuous flow IRMS measurement of ^{13}C in organic solids

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The stable isotope of carbon, ^{13}C, has a broad field of applications. Generally the measurement of this isotope is done in a differential way, which means comparison to a reference material for which a value was adopted. Expression of results in this technique, isotope ratio mass spectrometry, is illustrative of this way of working, since the delta-value in per mill is based on the relative difference of isotope ratios of the substance under study and a reference material. Within IRMS different techniques are possible: on the one hand dual inlet, in which the gas to measure and the reference gas are admitted through each side of a double inlet system and subsequently measured repeatedly; on the other hand continuous flow in which conversion of the sample to the gas suitable for IRMS measurement is done online with the IRMS and one actual measurement of the sample takes place. In the latter a carrier gas assures transport of the gases continuously from conversion module to mass spectrometer. Hence “continuous flow” (CF).

Switching to CF was a step forward in many respects. However, it should not mean the creation of a larger uncertainty in the total process of ^{13}C measurement. This study is aiming to assess the uncertainty involved in CF-IRMS for the measurement of ^{13}C in organic solids, as compared to uncertainty in the dual inlet technique, the latter including off-line sample preparation. Uncertainty statements play a major role in the establishment of transparency of the value assignment process. Knowing the major sources in an analytical procedure is of major importance, since this is the first step in optimisation of the value assignment process.

In the estimation of the total uncertainty of IRMS ^{13}C measurements for organic solids several components are present. Off-line preparation of different nature (CO_2 phosphoric acid liberation in case of carbonates, EA-combustion of organic solids) of reference materials and samples, the use of secondary reference materials and the mass spectrometric measurement yield different uncertainties in the different techniques, i.e. off-line EA-dual

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inlet IRMS and CF EA-IRMS. In this study uncertainties are shown for the off-line preparation of CO_2 from organic solids using elemental analysis equipment, followed by dual inlet IRMS and online measurement in EA-IRMS of organic solids.

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