A practical guide to the reliability of simultaneous $\delta^{13}C$ and $\delta^{15}N$ measurement at different C/N ratios

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Introduction

The simultaneous measurement of $\delta^{13}C$ and $\delta^{15}N$ values of solid materials in one single sample is handy to reduce analysis costs and often even necessary if sample amount is limited. However, the accuracy and reliability of the results especially for ^{15}N are uncertain. We therefore analysed samples from our daily routine covering a wide range of provenances, C/N ratios and sample amounts to give a guide under which circumstances simultaneous $\delta^{13}C$ and $\delta^{15}N$ measurements give reliable results.

Materials and Methods

Samples of a wide range of provenances analysed simultaneously for C and N with different amounts weighed in for combustion (Tab. 1). Routinely, 2 mg of sample was used to avoid GC column overload, which was reported by Brooks et al. (2003).

For comparison, samples were analysed separately for C with an average sample size of 600 µg C and N with an average sample size of 50 µg N.

All measurements were repeated ten times and were performed on a Thermo Finnigan Delta plus coupled with a Thermo Quest NC 2500 elemental analyser via a ConFlo III device.

Sample	C/N	$\delta^{15}N$ (‰) control	$\delta^{13}C$ (‰) control	sample weight (mg)	^{15}N (‰)	^{13}C (‰)
Bovine liver	5	7.65 (0.10)	-21.23 (0.08)	1	7.65 (0.04)	-21.35 (0.07)
				0.5	7.75 (0.05)	-20.80 (0.25)
				0.12	7.50 (0.21)	-20.99 (0.13)
Linden seeds (*Tilia cordata*)	17	0.35 (0.15)	-27.75 (0.27)	2	0.44 (0.09)	-27.55 (0,13)
Shrub leaves	23	2.65 (0.12)	-10.31 (0.12)	2	2.85 (0.12)	-10.29 (0.07)
Fine roots	29	0.07 (0.13)	-29,30 (0.14)	2	0.33 (0.06)	-29.37 (0.05)
Shrub leaves	29	-0.20 (0.08)	-29.46 (0.10)	2	-0.18 (0.12)	-29.52 (0.06)
Maize leaves	52	2.05 (0.10)	-12.62 (0.12)	2	2.08 (0.15)	-12.72 (0.08)
(*Zea mays*)				4	2.09 (0.12)	-12.23 (0.07)
Maize roots	83	2.48 (0.05)	-12.27 (0.11)	2	2.33 (0.34)	-12.39 (0.06)
(*Zea mays*)				6	2.50 (0.19)	-12.09 (0.05)

Tab. 1: Sample origin, sample weight and delta values for single and simultaneous C and N measurements (n=10).

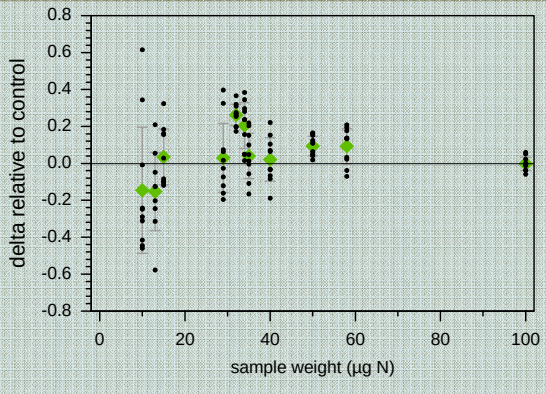

Fig. 1: N data of simultaneous C and N measurements expressed as deviation from control (n=10)

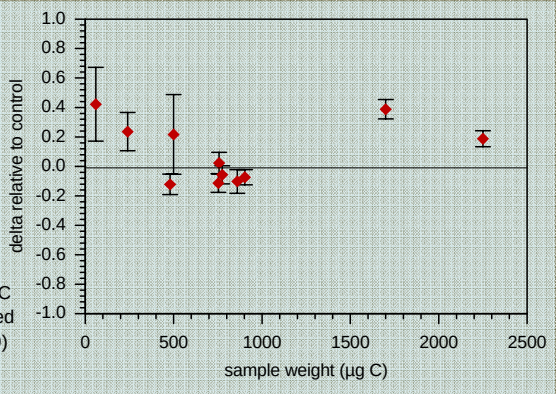

Fig. 2: C data of simultaneous C and N measurements expressed as deviation from control (n=10)

Conclusions

- **Although measurement precisions decreases strongly with decreasing sample size below 50 µg N, reasonable results could be obtained even for samples with C/N = 83.**
- **Sample weight above 1000µg C can help to improve the N results, and we did not observe column overload effects for a restricted number of samples (about 30).**
- **Care should be take to account for a sample size dependent drift of N data which must be accounted for using working standards (data not shown).**

Literature

P.D. Brooks, H. Geilmann, R. A. Werner, W. A. Brand, Rapid Communications in Mass Spectrometry 2003, **17**: 1924-1926