Costech 4010 Elemental Analyzer Sample Size Determination v0.2

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Introduction

Determining the correct sample size for $\delta^{13}C$ and $\delta^{15}N$ analyses is important because the mass spectrometer signal intensity, sensitivity, and reproducibility is related to sample mass. These notes make three assumptions. 1. The the carbon and nitrogen content of a material is known prior to analysis. 2. Analysis is taking place in the University of California, Merced Stable Isotope Ecology laboratory using a *Costech 4010 Elemental Analyzer*. * Other laboratories have similar instrumentation setups, however instrument sensitivity will vary between configurations.

3. The *Delta V+ IRMS* CO_2 sample dilution is set to 89%. * This is the default configuration for routine analyses. Other configurations are possible but you should contact the Technical Director prior to sample preparation.

Background

The signal intensity of a sample gas measured by a *Delta V+ isotope ratio mass spectrometer* is directly related to the about of gas. In the case of the *Costech 4010 Elemental Analyzer* the signal intensity is linearly related to sample mass (Figure 1).

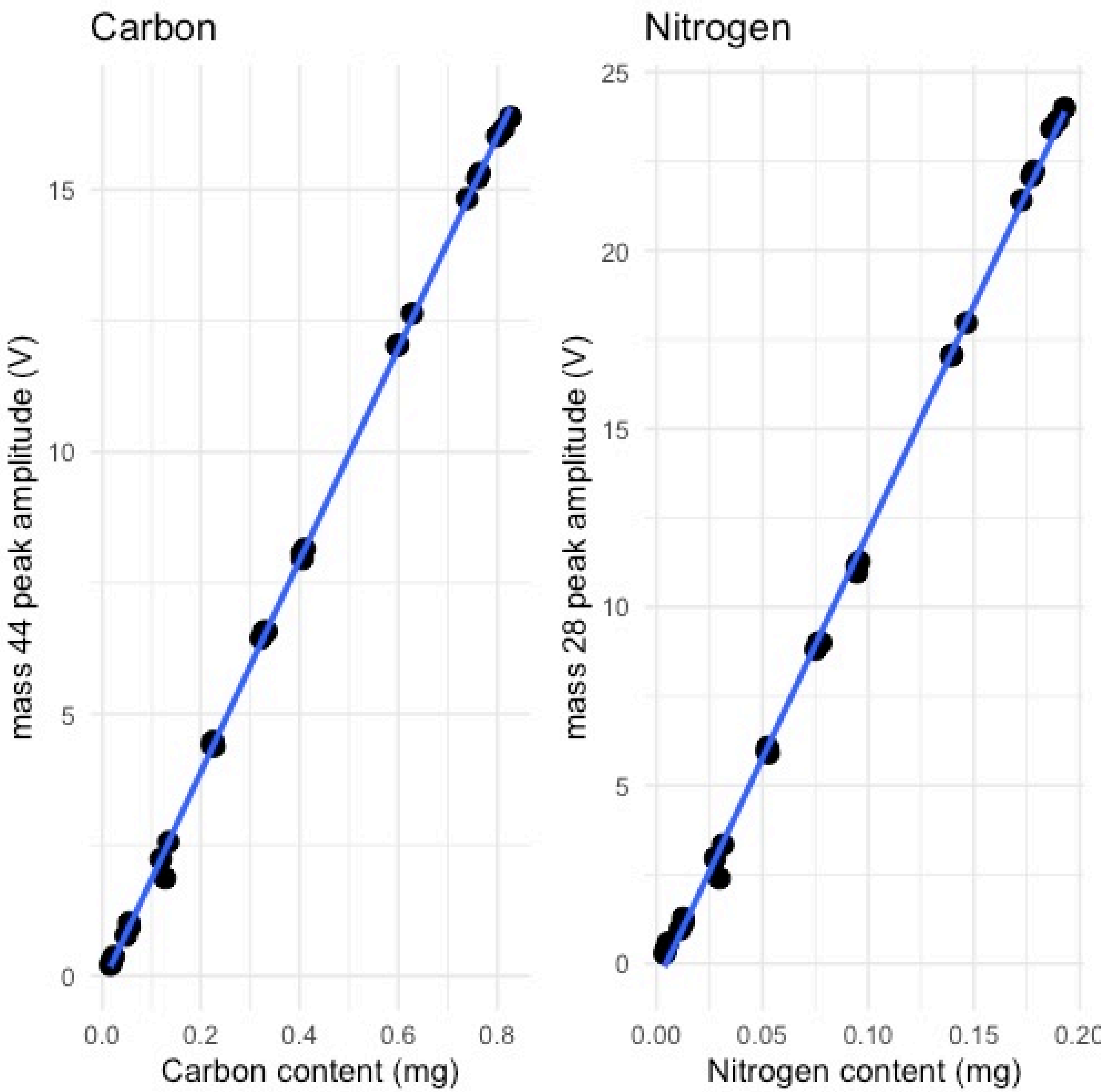

Figure 1: Sample carbon and nitrogen content versus signal intensity.

The *Delta V+ isotope ratio mass spectrometer* has a measurement range from 0 to 50 volts. However the sensitivity is best below about 30 volts. Furthermore, both $\delta^{13}C$ and $\delta^{15}N$ have have a non-linear relationship to signal intensity. This means that a sample with a small signal intensity will have a different measured δ value than a sample of the same material at a large signal intensity.

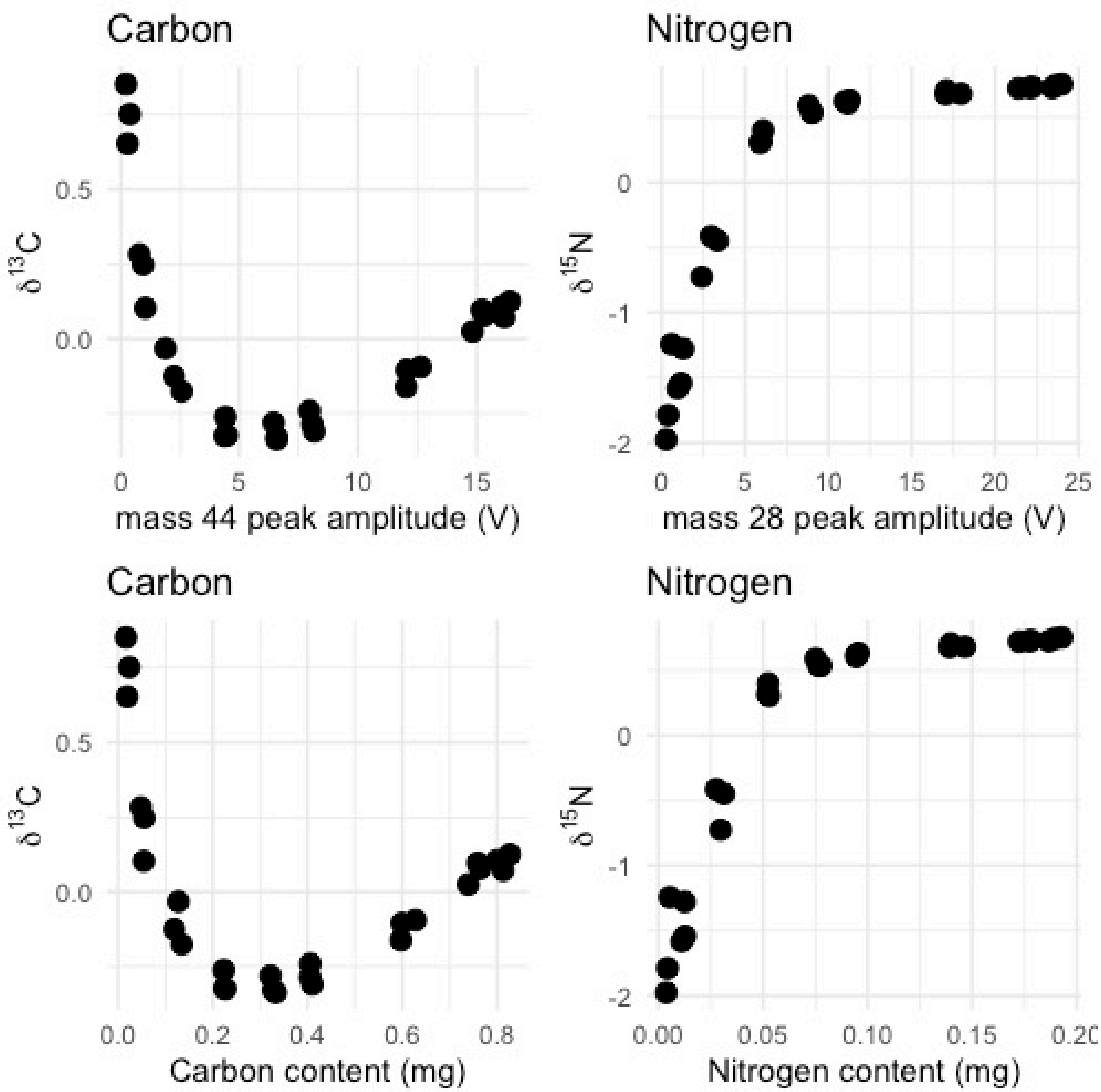

Figure 2: Mass dependence of $\delta^{13}C$ and $\delta^{15}N$ values.

The δ-value-mass-dependence is linear and lowest between about 5 to 15 volts for $\delta^{13}C$ and 5 to 25 for $\delta^{15}N$ (Figure 2). Since the relationship between signal intensity (volts) is linearly related to carbon and nitrogen content (Figure 1), this equates to the mass range shown in Table 1.

Table 1: minimum and maximum carbon and nitrogen contents for $\delta^{13}C$ and $\delta^{15}N$ analysis

isotope	minimum (mg)	maximum (mg)
$\delta^{13}C$	0.2	0.8
$\delta^{15}N$	0.05	0.2

Calculating Sample Size

If the carbon and nitrogen content of a material is known prior to analysis you can calculate the optimal sample size using the following equations to calculate the range of sample weights for carbon and nitrogen.

δ^{13}C Range

$$Sample\ size_{min} = 0.2 \div (sample\ carbon\% \div 100)$$

$$Sample\ size_{max} = 0.8 \div (sample\ carbon\% \div 100)$$

δ^{15}N Range

$$Sample\ size_{min} = 0.05 \div (sample\ nitrogen\% \div 100)$$

$$Sample\ size_{max} = 0.2 \div (sample\ nitrogen\% \div 100)$$

Measuring both $\delta^{13}C$ and $\delta^{15}N$

It is difficult to concurrently measure $\delta^{13}C$ and $\delta^{15}N$ in samples with high C:N ratios. Based on the sample ranges given in Table 1 the maximum C:N ratio is about 16 ($carbon\ maximum \div nitrogen\ minimum$ or $0.8 \div 0.05$) and the minimum C:N ratio is 1 ($carbon\ minimum \div nitrogen\ maximum$ or $0.2 \div 0.2$). For analyses of materials with C:N ratios outside this range please consult the Technical Director prior to sample preparation.

If your samples have a C:N ratio between about 1 to 16 then $\delta^{13}C$ and $\delta^{15}N$ can be measured concurrently. Calculate the range of acceptable sample sizes using the equations for carbon and nitrogen shown above and then choose a final sample size that overlaps both results. If your samples have a C:N ratio significantly outside this range then $\delta^{13}C$ and $\delta^{15}N$ may need to be measured separately. Please consult the Technical Director for guidance.

Commonly Analyzed Materials

Table 2: Carbon and nitrogen contents of commonly analyzed materials.

material	carbon %	nitrogen %
acetanilide	71.09	10.36
biochar	> 80	< 1.5
collagen	40 - 45	10 - 15
glutamic acid	40.3	9.52
keratin	40 - 45	10 - 15
plants	40 - 50	< 1 - 5

material	carbon %	nitrogen %
proteins	40 - 45	10 - 15
soil	highly variable	highly variable

Worked Example

Acetanilide is common reference material that is 71.09% carbon and 10.36% nitrogen (Table 2).

δ^{13}C

$$sample\ size_{min} = 0.28\,mg = 0.2 \div (71.09\% \div 100)$$
$$sample\ size_{max} = 1.13\,mg = 0.8 \div (71.09\% \div 100)$$

δ^{15}N

$$Sample\ size_{min} = 0.48\,mg = 0.05 \div (10.36\% \div 100)$$
$$Sample\ size_{max} = 1.93\,mg = 0.2 \div (10.36\% \div 100)$$

In this case the minimum sample size for δ^{13}C measurements is lower than the minimum for measuring δ^{15}N. the minimum sample size for δ^{15}N analysis is in the middle of the range for δ^{13}C so a sample size of **0.5 mg** would be appropriate.