APPLICATION BRIEF

AB30356

EA-IRMS: Simultaneous $\delta^{13}C$, $\delta^{15}N$, and $\delta^{34}S$ Analysis of Wood with the EA IsoLink IRMS System

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Key Words

C/S Ratio, C/N Ratio, EA IsoLink, Gas Chromatography, NCS Analysis, $\delta^{13}C$, $\delta^{15}N$, $\delta^{34}S$, Wood

Goal

To demonstrate simultaneous NCS analysis on wood samples, with a C/S ratio > 7000:1, using the EA IsoLink IRMS System.

Introduction

A prominent challenge in analyzing sulfur in sample matrices with very high C/S ratios (e.g. > 5000:1), such as wood, is the very low amount of sulfur in the sample alongside the extremely high carbon amounts. In addition, simultaneous NCS weight% (wt%) and isotopic analysis of very high C/S ratio samples has been a significant analytical challenge due to difficulties in complete baseline separation of N_2, CO_2 and SO_2, the analysis of high amounts of carbon relative to small amounts of nitrogen and sulfur, and EA-IRMS system sensitivity.

Figure 1. Thermo Scientific EA IsoLink IRMS System.

Analytical Configuration

For analysis, 5 replicates of approximately 12–16 mg of dried, homogenized wood were weighed into tin capsules, along with 2 mg of V_2O_5 and introduced to the reactor from the Thermo Scientific™ MAS Plus Autosampler, where it was combusted with 41 ml of oxygen. Analysis time is less than 10 minutes, using less than 1.5 liters of helium per sample, and it is based on standard sensitivity mode of the Thermo Scientific™ Delta V™ Series Isotope Ratio Mass Spectrometer.

Simultaneous NCS analysis of wood

Using the Thermo Scientific™ EA IsoLink™ IRMS System (Figure 1), simultaneous NCS analysis is demonstrated on wood samples (C/S ratio ≥ 7000:1). Table 1 shows excellent precision on simultaneously measured wt% N, wt% C, wt% S, $\delta^{13}C$, $\delta^{15}N$ and $\delta^{34}S$ values. The analysis shows that for a sample containing very small nitrogen and sulfur amounts, alongside extremely high carbon amounts, precision of 0.26‰ for n = 5 is achieved on 1 µg S, which equates to less than 0.007% or 70 ppm of sulfur.

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Table 1. Data from simultaneous NCS analysis of wood samples.

Sample	Weight (mg)	wt% C	wt% N	wt% S	$\delta^{13}C$ (‰)	$\delta^{15}N$ (‰)	$\delta^{34}S$ (‰)	µg C	µg N	µg S	ppm S	C/S Ratio
Spruce	12.69 (11.72–14.24)	54.954 ± 1.086	0.088 ± 0.003	0.007 ± 0.0002	-24.10 ± 0.06	3.20 ± 0.23	5.92 ± 0.26	7036.48	11.14	0.89	70	7905
Iroko	15.63 (15.00–16.07)	32.018 ± 0.814	0.123 ± 0.01	0.005 ± 0.0005	-26.43 ± 0.04	10.13 ± 0.32	6.36 ± 0.27	5004.75	19.20	0.71	70	7048

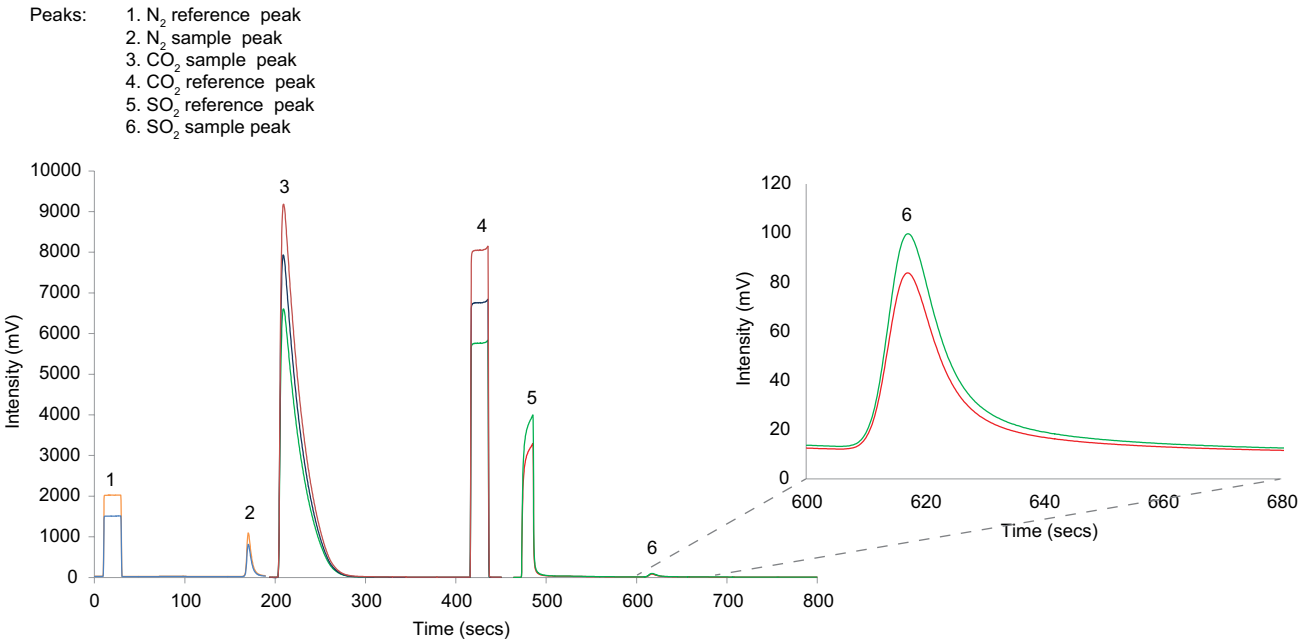

Figure 2. Isotope chromatogram for simultaneous NCS measurements on a spruce wood sample.

From a single sample, complete baseline separation of N_2, CO_2, and SO_2 peaks is achieved and sharp peak shapes with no peak tailing is ensured by temperature ramped continuous flow gas chromatography (Figure 2). The excellent dilution capabilities for high CO_2 amounts in the presence of very low N_2 and SO_2 amounts, using the Thermo Scientific™ ConFlo IV™ Universal Interface, makes simultaneous NCS analysis routine on very high C/S, C/N, and C/N/S ratio sample matrices.

However, the sample weight required for simultaneous NCS analysis of wood samples will vary. This depends on the sulfur content of sample. The data presented in Table 1 are application data and are not warranted because they exceed product specifications. The warranted product specification for $\delta^{13}C$ is ±0.1‰ (1 sd) for 50 µg of carbon, $\delta^{15}N$ is ±0.15‰ (1 sd) for 50 µg of nitrogen and $\delta^{34}S$ is ±0.3‰ (1 sd) for 10 µg of sulfur measured on Sulfanilamide.

Summary

From a single sample with a high C/S, C/N and C/N/S ratio, outstanding precision, low cost per sample, and short analysis times are ensured for wt% and isotopic measurements in simultaneous NCS mode by the EA IsoLink IRMS System. The method presented here demonstrates simultaneous NCS analysis on wood samples with a C/S ratio > 7000:1 and a nitrogen and sulfur content of <20 µg and 1 µg, respectively.

Cost per analysis is reduced by the Helium Management (He^M) Module, using less than 1.5 liters per sample.

Find out more at **thermofisher.com/EAIsoLink**

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