

EA-IRMS: Simultaneous NCS Isotope Analysis

Christopher Brodie, Oliver Kracht,
Alexander Hartwig
Thermo Fisher Scientific, Bremen,
Germany

Keywords

EA-IRMS, EA IsoLink, $\delta^{13}\text{C}$, $\delta^{15}\text{N}$, $\delta^{34}\text{S}$, NCS analysis

Goal

Demonstrate simultaneous NCS analysis on the Thermo Scientific™ EA IsoLink™ IRMS System.

Introduction

Simultaneous $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and $\delta^{34}\text{S}$ measurements are increasingly performed on samples in food authenticity, forensic, geoscience and ecology and biology applications. Deriving accurate and precise $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and $\delta^{34}\text{S}$ values, from a single sample reduces cost per sample and increases system productivity.

This application brief reports data from simultaneous $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and $\delta^{34}\text{S}$ analysis on a range of sample matrices, spanning a range of N, C and S amounts, using the Thermo Scientific EA IsoLink IRMS system.

Analytical Set-up

For analysis, dried, homogenized samples were weighed into tin capsules and introduced to the combustion reactor from the Thermo Scientific™ MAS Plus Autosampler, where they were combusted in the presence of oxygen. Analysis time is less than 12 minutes, using 1.5 liters of helium per sample.

EA IsoLink Performance for Simultaneous $\delta^{13}\text{C}$, $\delta^{15}\text{N}$, $\delta^{34}\text{S}$ Analysis

Table 1 reports $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and $\delta^{34}\text{S}$ and wt% data as a mean value and one standard deviation from 5 replicate measurements. Mass traces from the TCD and IRMS were simultaneously acquired in the Thermo Scientific™ Isodat™ Software Suite with automated data evaluation and export. The precision for $\delta^{13}\text{C}$ is $\leq 0.08\text{‰}$, for $\delta^{15}\text{N}$ is $\leq 0.15\text{‰}$ and $\delta^{34}\text{S}$ is $\leq 0.2\text{‰}$ across the entire range of samples analyzed.

Samples containing 1-10 μg S show precisions for $\delta^{34}\text{S}$ $\leq 0.26\text{‰}$ and for 3-20 μg N show precisions of $\delta^{15}\text{N}$ $\leq 0.26\text{‰}$: this includes sample material analysed simultaneously for NCS with C/S ratio of 7900:1 (wood). The data presented in Table 1 are not warranted because they exceed product specifications. The warranted product specifications for $\delta^{13}\text{C}$ is $\pm 0.1\text{‰}$ (1 sd) for 50 μg of carbon, $\delta^{15}\text{N}$ is $\pm 0.15\text{‰}$ (1 sd) for 50 μg of nitrogen and $\delta^{34}\text{S}$ is $\pm 0.2\text{‰}$ (1 sd) for 50 μg of sulfur measured on sulfanilamide.

Summary

The EA IsoLink IRMS System achieves simultaneous $\delta^{13}\text{C}$, $\delta^{15}\text{N}$ and $\delta^{34}\text{S}$ and wt% analysis in less than 12 minutes, with complete analysis and data evaluation possible in as low as 9 minutes, on a single sample. By utilizing temperature ramped continuous flow gas chromatography, a unique feature in EA-IRMS, excellent peak shapes and baseline separation are achieved ensuring high quality data reproducibility. In addition, the Helium Management (He^M) Module provides at least 60% helium saving per NCS sample compared with previous Thermo Scientific™ EA-IRMS systems. Complete software automation, from sample drop to data export, provides a fast and reliable workflow making the system simple to use.

| Sample | $\delta^{13}\text{C}$ (‰) | $\delta^{15}\text{N}$ (‰) | $\delta^{34}\text{S}$ (‰) |
|---------------|---------------------------|---------------------------|---------------------------|
| Hair | -11.48 ± 0.04 | 6.95 ± 0.15 | 10.01 ± 0.10 |
| Serum | -14.63 ± 0.05 | 6.86 ± 0.11 | 8.11 ± 0.13 |
| Sulfanilamide | -18.64 ± 0.05 | -1.14 ± 0.07 | 19.23 ± 0.05 |
| Phenanthrene* | -14.97 ± 0.02 | 1.50 ± 0.29 | 1.65 ± 0.22 |
| BBOT | -17.40 ± 0.06 | -2.70 ± 0.15 | 15.67 ± 0.12 |

*sulfur was added to the sample material and is not part of chemical structure.

Forensics and Chemicals



| Sample | $\delta^{13}\text{C}$ (‰) | $\delta^{15}\text{N}$ (‰) | $\delta^{34}\text{S}$ (‰) |
|-------------|---------------------------|---------------------------|---------------------------|
| Wheat flour | -17.15 ± 0.08 | 2.45 ± 0.04 | 2.04 ± 0.22 |
| Protein | -17.81 ± 0.02 | 5.67 ± 0.03 | 9.12 ± 0.25 |
| Pasta | -15.02 ± 0.03 | 4.01 ± 0.07 | 4.47 ± 0.22 |
| Milk powder | -15.39 ± 0.02 | 4.64 ± 0.11 | 7.75 ± 0.26 |
| Vitamin B1 | -23.57 ± 0.04 | -1.24 ± 0.09 | 4.25 ± 0.17 |

Food Authenticity



Ecology and Biology



| Sample | $\delta^{13}\text{C}$ (‰) | $\delta^{15}\text{N}$ (‰) | $\delta^{34}\text{S}$ (‰) |
|----------------|---------------------------|---------------------------|---------------------------|
| Spruce wood | -24.07 ± 0.06 | 3.20 ± 0.23 | 5.92 ± 0.26 |
| Bird feathers | -6.77 ± 0.14 | 7.38 ± 0.04 | 7.53 ± 0.20 |
| Octopus tissue | -7.45 ± 0.02 | 10.24 ± 0.04 | 18.65 ± 0.20 |
| Bovine liver | -12.16 ± 0.05 | 7.32 ± 0.08 | 10.77 ± 0.06 |
| Tomato leaves | -17.24 ± 0.03 | 4.17 ± 0.08 | 7.39 ± 0.24 |
| Apple leaves | -17.04 ± 0.09 | 0.82 ± 0.09 | 7.88 ± 0.24 |
| Shark tooth | -9.09 ± 0.06 | 10.68 ± 0.08 | 17.59 ± 0.27 |

Geo- and Environmental Sciences



| Sample | $\delta^{13}\text{C}$ (‰) | $\delta^{15}\text{N}$ (‰) | $\delta^{34}\text{S}$ (‰) |
|----------------|---------------------------|---------------------------|---------------------------|
| Peat soil | -18.58 ± 0.06 | 3.42 ± 0.06 | -10.92 ± 0.24 |
| Pine needles | -15.45 ± 0.08 | -8.81 ± 0.02 | 3.31 ± 0.24 |
| Sediment | -16.87 ± 0.03 | 3.91 ± 0.08 | 7.53 ± 0.17 |
| River Sediment | -9.84 ± 0.18 | 4.94 ± 0.08 | -17.25 ± 0.20 |

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