EA-IRMS: No Intra-sample Memory for $\delta^{34}S$ Measurements of Inorganic Sulfur Materials Using the EA IsoLink IRMS System

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Key Words
$\delta^{34}S$, Carry-over, EA-IRMS, Intra-sample Memory

Goal
To demonstrate that the EA IsoLink IRMS System is free from intra-sample memory for $\delta^{34}S$ measurements by using inorganic international reference materials.

Introduction
Accurate and precise $\delta^{34}S$ measurements by EA-IRMS require that the system can process samples without intra-sample effects, generally referred to as “memory effect” or “carry-over”. Any intra-sample memory effect or carry-over will produce inaccurate $\delta^{34}S$ values and significantly reduce data reproducibility as well as reducing system productivity and consuming valuable laboratory resources.

This application brief demonstrates that the Thermo Scientific™ EA IsoLink™ IRMS System (Figure 1) does not show an intra-sample memory effect on $\delta^{34}S$ measurements and that accurate and precise $\delta^{34}S$ values are produced.

Figure 1. Thermo Scientific EA IsoLink IRMS System.
Intra-sample performance for $\delta^{34}S$ analysis

Figure 2 illustrates a series of 30 sequential $\delta^{34}S$ measurements of international standard reference materials IAEA S1, IAEA S2, IAEA S3, IAEA SO5 and IAEA SO6. Samples were weighed in amounts of 200–280 μg, which equates to 25–30 μg of sulfur. A 3-point calibration was produced, with an $r^2 = 0.999$. The standard deviation of the mean is 0.19‰ across a 56‰ range (Figure 3). The $\delta^{34}S$ precision was ≤0.18‰ for $n = 6$ showing excellent accuracy (Table 1).

Figure 2. Sequence of 30 sequential measurements of international reference materials.

Figure 3. $\delta^{34}S$ 3-point calibration curve.

Summary

The analysis of inorganic sulfur materials shows no intra-sample memory or carry-over effect on $\delta^{34}S$ measurements on the EA IsoLink IRMS System, therefore:

• There is no need to discard the first or second sample points from replicate measurements prior to analyzing the data.
• There are no trends in the replicate analysis of individual samples.
• There is no requirement to undertake memory correction.

The data presented in Table 1 are application data and are not warranted because they exceed product specifications. The warranted product specification for $\delta^{34}S$ is ±0.2‰ (1 sd) for 50 μg of sulfur measured on Sulfanilamide.

Table 1. Summary of measured $\delta^{34}S$ values and actual $\delta^{34}S$ values.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Weight (mg)</th>
<th>$n$</th>
<th>μg S</th>
<th>Measured $\delta^{34}S_{VCDT}$ (%) [Mean ± 1σ]</th>
<th>Actual $\delta^{34}S_{VCDT}$ (%) [Mean ± 1σ]</th>
</tr>
</thead>
<tbody>
<tr>
<td>IAEA S1 (AgS)</td>
<td>0.202–0.285</td>
<td>6</td>
<td>30.4</td>
<td>0.03 ± 0.08</td>
<td>-0.30 ± 0.20</td>
</tr>
<tr>
<td>IAEA S2 (AgS)</td>
<td>0.192–0.219</td>
<td>6</td>
<td>25.8</td>
<td>22.67 ± 0.10</td>
<td>22.70 ± 0.20</td>
</tr>
<tr>
<td>IAEA S3 (AgS)</td>
<td>0.191–0.234</td>
<td>6</td>
<td>28.0</td>
<td>-32.30 ± 0.15</td>
<td>-32.30 ± 0.20</td>
</tr>
<tr>
<td>IAEA SO5 (BaSO₄)</td>
<td>0.196–0.268</td>
<td>6</td>
<td>30.4</td>
<td>0.75 ± 0.10</td>
<td>0.50 ± 0.20</td>
</tr>
<tr>
<td>IAEA SO6 (BaSO₄)</td>
<td>0.188–0.272</td>
<td>6</td>
<td>29.2</td>
<td>-34.43 ± 0.18</td>
<td>-34.1 ± 0.20</td>
</tr>
</tbody>
</table>

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