Analysis of Sulfur Isotopes Using EA-IRMS

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Sulfur powder
Project Goal:

To develop a capability in our lab to help support future analog and mission studies involving sulfur.

Curiosity Rover Self Portrait
The EA-IRMS Process:

Elemental Analyzer

Isotope Ratio MS

Auto-sampler carousel

Internal Reactor, Columns, and Oven

GC Column behind electronic interface of EA

Delta V Plus Isotope Ratio MS Instrument Suite
The EA-IRMS Process:

1. Solid sample loaded in autosampler drops
2. Solid is combusted and gas carried through the reactor column by helium
3. Gas is sorted in GC column after exiting reactor column
4. IRMS determines isotope ratios of gases ($\delta^{34}S$)
5. Sorted gases exit GC column and carried to IRMS by helium
Current Work

- Calibration of Standards:
  - Barium Sulfate (BaSO₄)
  - Silver Sulfide (Ag₂S)
  - Sulfanilamide (C₆H₈N₂O₂S)

- Samples:
  - SAM Melanterite (unsieved)
IRMS Chromatograms:

SO$_2$ Reference Gas

SO$_2$ from BaSO$_4$

SO$_2$ Reference Gas
Results of Preliminary Melanterite Runs

<table>
<thead>
<tr>
<th>Sample</th>
<th>Average $\delta^{34}S$</th>
<th>Standard Dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Barium Sulfate</td>
<td>14.4496</td>
<td>0.16432</td>
</tr>
<tr>
<td>Silver Sulfide</td>
<td>-36.0206</td>
<td>0.36995</td>
</tr>
<tr>
<td>Sulfanilamide</td>
<td>11.2688</td>
<td>0.80115</td>
</tr>
<tr>
<td>SAM Melanterite</td>
<td>-8.5735</td>
<td>0.77248</td>
</tr>
<tr>
<td>SAM Melanterite + Vanadium</td>
<td>-8.646</td>
<td>0.99327</td>
</tr>
</tbody>
</table>

- All sulfur isotopes are calibrated to the Vienna-Canyon Diablo Troilite (VCDT) isotope value.
- A negative $\delta^{34}S$ value indicates that the value is less than that of VCDT, and vice versa for a positive $\delta^{34}S$ value.
Linear trend is expected. Deviations indicate the need for further study.

Concentration of S (Standards)

\[ y = 366.9x - 17.958 \]

Concentration of S (SAM Melanterite)

\[ y = 369.38x - 12.991 \]

Area 64 vs. \( d \, ^{34}\text{S} \) (Standards)

Area 64 vs. \( d \, ^{34}\text{S} \) (SAM Melanterite)
Future Studies/Next Step…

- Closer examination of SAM Melanterite samples to determine more effective methods of combustion
  - Modify the mode of combustion
  - Different oxidative agents
- Example of a benefit to the above:
  - Proper conservation/consumption of precious consumables on Curiosity Mars Rover