REE Identification and Characterization of Coal and Coal By-products Containing High Rare Earth Element Concentrations

Contract No. DE-FE-0026527
Robert Uhrin, Ph. D.
XLight Corporation

Application of Field Portable X-ray Fluorescence to the Identification of Rare-earth Elements
The issue is to find a suitable technique that can be used in the field as well as the laboratory for identifying Rare-earth Elements (REEs):

• XRF is a fast analysis technique not applied with accuracy to identifying REEs
• To circumvent this obstacle standard sample compositions will be synthesized
• Many REE concentrations in random coal-associated samples are < 1ppm (w)
• Standard sample REE concentrations of 1 ppm, 10 ppm, 100 ppm and 1000 ppm (where appropriate) will be prepared to serve as standards
• This will be accomplished typically by dissolving RE oxides in hydrochloric acid (HCl) and precipitating the rare-earth orthophosphates using phosphoric acid (H3PO4)
• Field or laboratory data will be compared to the standard sample values obtained by XRF to determine actual REE concentrations in field samples
• Correlation can then be made with ICP-MS values
Figure 1. Typical XRF Spectrum of Coal Spoil (WPFACO08)
Figure 2. XRF Spectrum of 1% Yb:YPO4
Current Properties of Field-portable XRF

- A Niton XL3t 500 field portable instrument was used.
- Only Sc and Y are currently contained in the libraries used for peak identification.
- However, the emission energies of all elements are in the database, so all peaks can be identified manually using a cursor.
- Figure 1 shows that Y (Kα1, at least) can be identified in most coal-related spectra (broad peak at 14.96 keV) with interference from Rb (Kβ1).
- Figure 2 shows that a high-concentration of Y can be readily identified along with 1% levels of REEs or REE contaminants, i.e., Yb(Lα1, Lα2, Lβ1, Lβ2, Ly 1) misidentified in the insert.
- Table I shows a brief XRF analysis of the sample in Figure 1.
- Figure 3 demonstrates a dependence of Light REE (LREE = La-Nd) on thorium *Th) using 45 Penn State University samples of bituminous coal.
- Figure 4 demonstrates the spectrum for a coal-related sample containing 44 ppm Th. The spectrum reveals a definite, but unquantified, Y content.
<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration (ppm)</th>
<th>2σ</th>
<th>Element</th>
<th>Concentration (ppm)</th>
<th>2σ</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mn</td>
<td>27</td>
<td>3</td>
<td>As</td>
<td>9</td>
<td>3</td>
</tr>
<tr>
<td>Zr</td>
<td>121</td>
<td>3</td>
<td>Zn</td>
<td>27</td>
<td>4</td>
</tr>
<tr>
<td>Sr</td>
<td>126</td>
<td>3</td>
<td>Cu</td>
<td>48</td>
<td>13</td>
</tr>
<tr>
<td>Rb</td>
<td>79</td>
<td>3</td>
<td>Nb</td>
<td>31</td>
<td>2</td>
</tr>
<tr>
<td>Th</td>
<td>11</td>
<td>2</td>
<td>Bi</td>
<td>21</td>
<td>4</td>
</tr>
<tr>
<td>Pb</td>
<td>34</td>
<td>4</td>
<td>V</td>
<td>397</td>
<td>12</td>
</tr>
<tr>
<td>Fe</td>
<td>2.78 %</td>
<td>0.05 %</td>
<td>Ti</td>
<td>4222</td>
<td>2</td>
</tr>
</tbody>
</table>
Figure 3. Comparison of LREE concentration with Th content

\[ y = 14.13x \]

\[ R^2 = 0.7882 \]
Figure 4. Emission Energy Spectrum for Sample SF14-004-02