

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

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Application of Field Portable X-ray Fluorescence to the Identification of
Rare-earth Elements



**U.S. DEPARTMENT OF
ENERGY**



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 (WPFAC008)

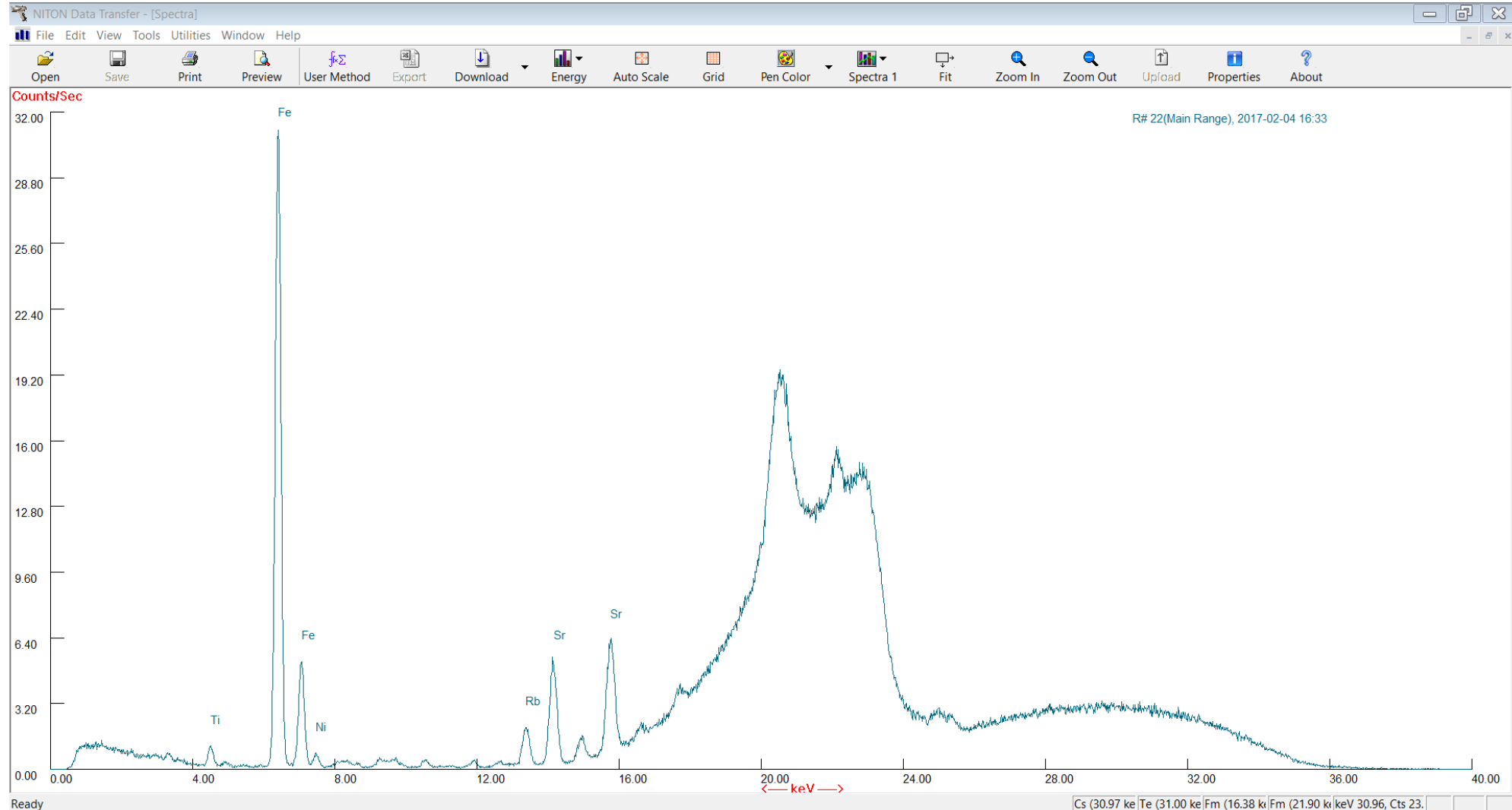
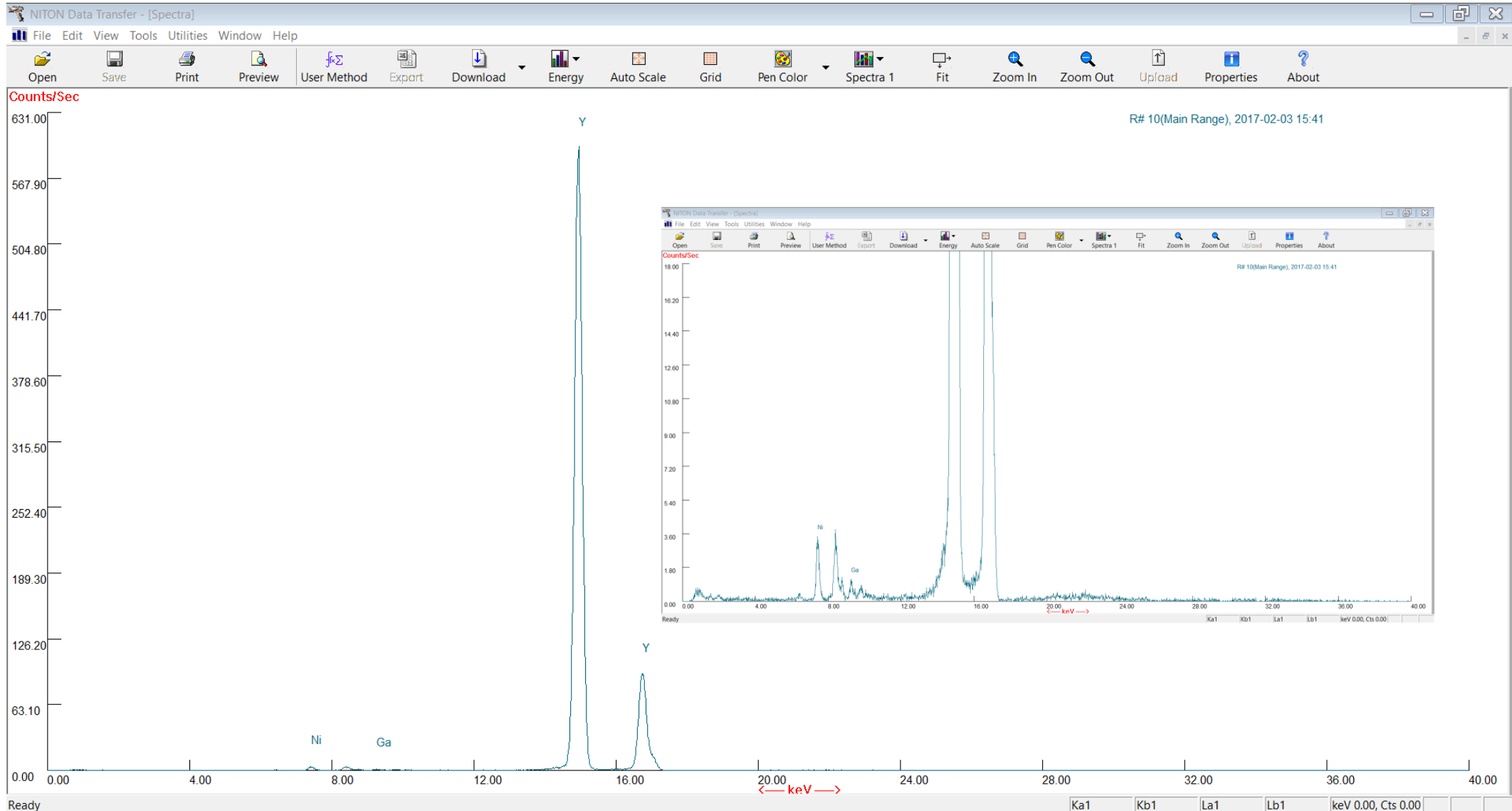


Figure 2. XRF Spectrum of 1% Yb:YPO₄



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 ($K\alpha_1$, at least) can be identified in most coal-related spectra (broad peak at 14.96 keV) with interference from Rb ($K\beta_1$).
- 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($L\alpha_1$, $L\alpha_2$, $L\beta_1$, $L\beta_2$, $L\gamma_1$) 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 I. XRF Analysis of Sample in Figure 1

Element	Concentration (ppm)	2 σ	Element	Concentration (ppm)	2 σ
Mn	27	3	As	9	3
Zr	121	3	Zn	27	4
Sr	126	3	Cu	48	13
Rb	79	3	Nb	31	2
Th	11	2	Bi	21	4
Pb	34	4	V	397	12
Fe	2.78 %	0.05 %	Ti	4222	2

Figure 3. Comparison of LREE concentration with Th content

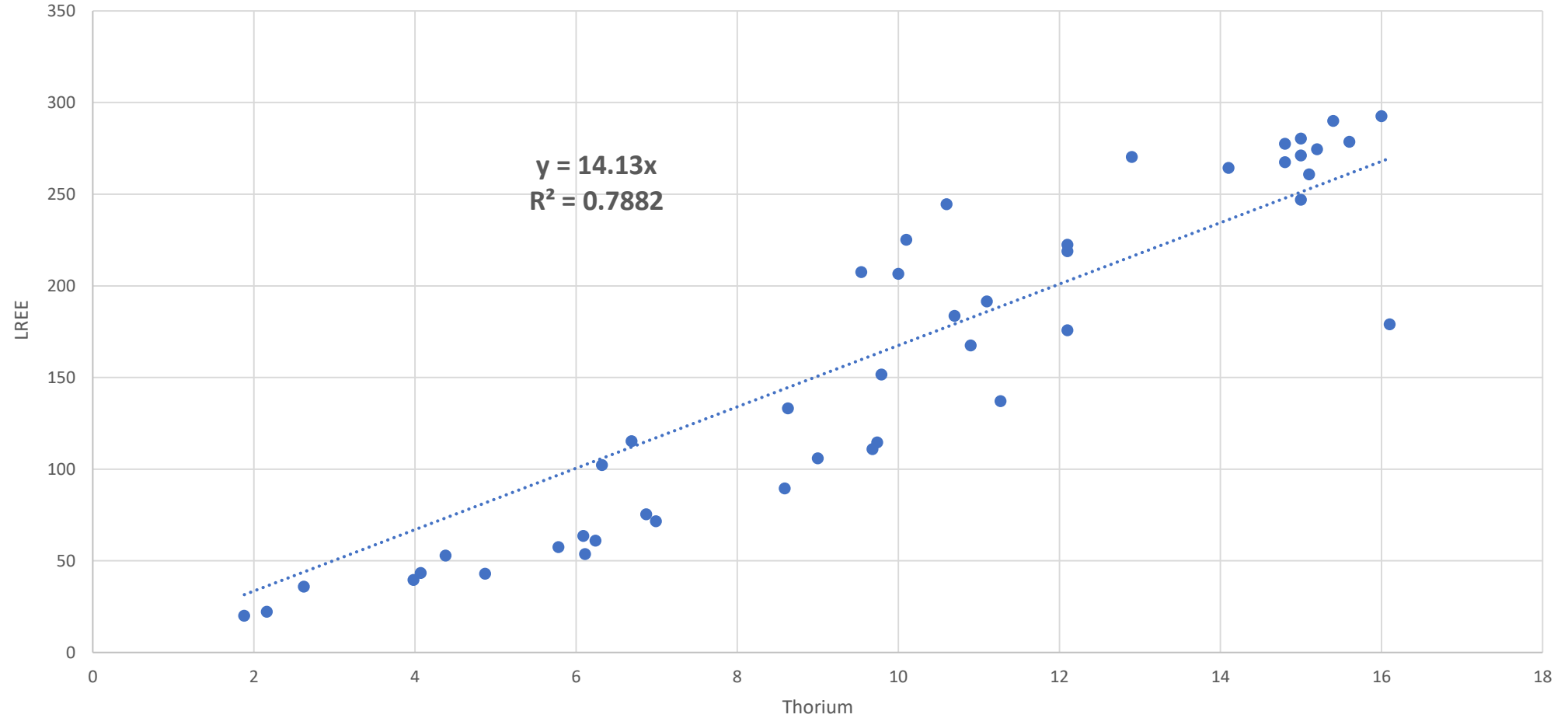


Figure 4. Emission Energy Spectrum for Sample SF14-004-02

