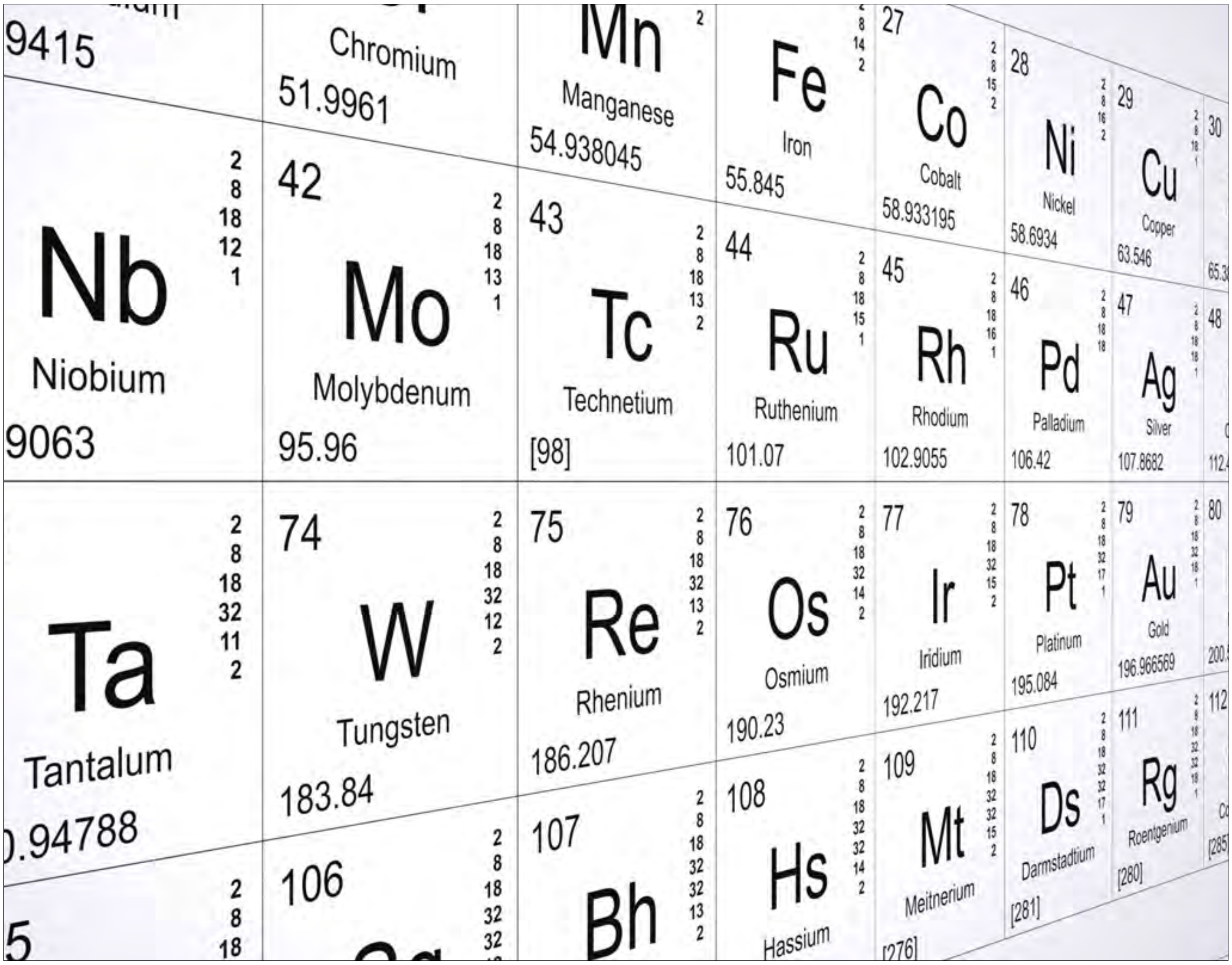

Barium Sulfate Analysis

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BRUKER TEST RESULTS

Objective

Barium Sulfate materials were analyzed to determine the concentration of impurities. Due to a lack of empirical reference standards, a theoretical approach was taken using fluorescence efficiencies.

Method

Data was collected at 40 keV with a current of 12 µA and a Ti/Al filter in dry air conditions. Validation is qualitative, as basic physical patterns of elemental fluorescence were used. One key assumption was made, that the number of Sulfur atoms will equal the sum of Barium and Strontium atoms due to the chemical formula of the known matrix (BaSO4 and SrSO4). This was used to develop a correction factor for the filter's effect on fluorescence (as the filter reduced the number of photons engaging with light elements). All data were normalized to Strontium, and concentrations calculated using the following equation:

$$C_i = [e_i * (F_{Sr}/F_i) * (D_{Sr}/D_i) * 28.5] * A_i$$

where C_i is the concentration of element i, e is the net photon count for element i as determined by bayesian deconvolution, Sr represents the net photon count for strontium as determined by bayesian deconvolution, f represents Fluorescence efficiency, D represents depth, and A represents the atomic weight of element i. Barium and strontium were calculated assuming they were present as sulfates (SO_4). The value 28.5 was determined using the correction needed to align sulfur net photon counts with barium and strontium net photon counts. This equation cannot correct for all uncertainties, and is used only for a rough estimate of elemental concentrations. It is strongly recommended that appropriate reference samples be used in future calculations for these elements. Results are present in Table 1 at the end of this document.

RESULTS

Bayesian Deconvolution

Bayesian Deconvolution was run using Bruker's Artax software, with 10 stripping cycles. This was used to generate net photon count rates that can be converted into quantitative results.

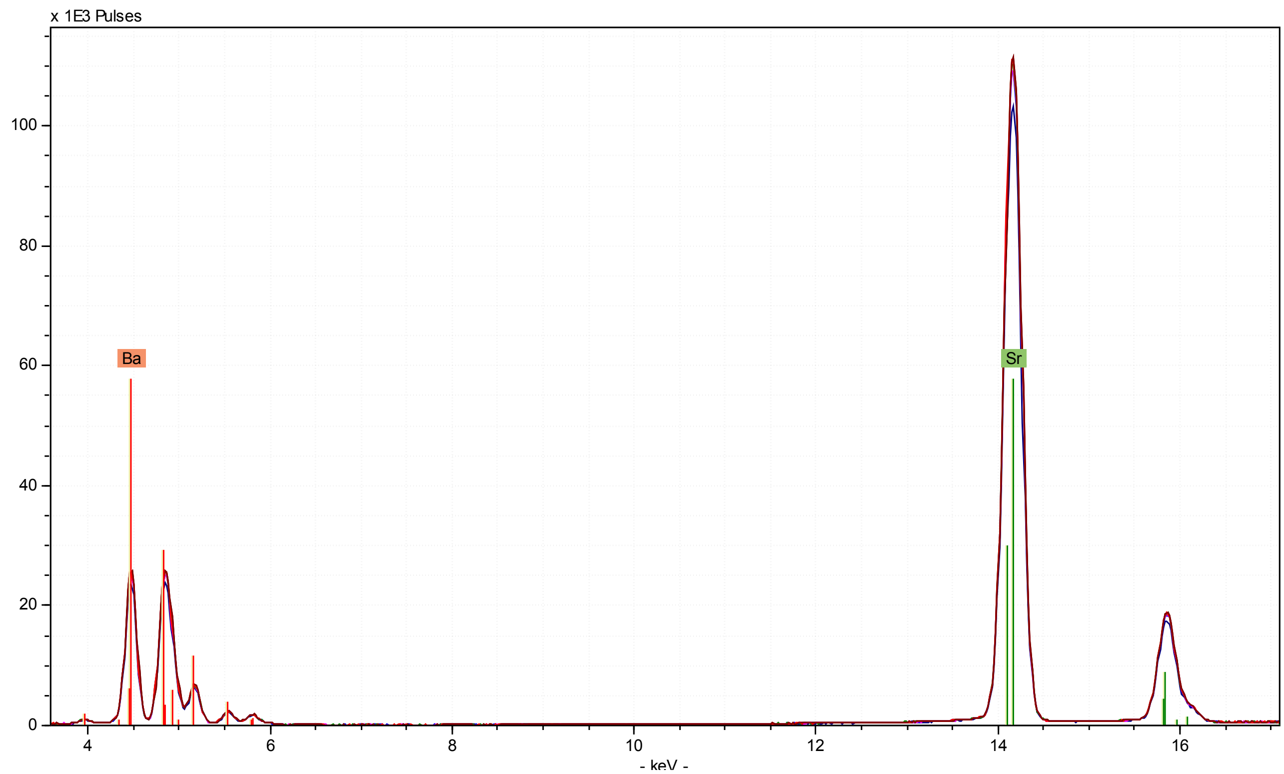

Figure 1: Spectral results of barium sulfate materials reveal very a very high strontium signal, this includes one material that had previously been reported as pure barium sulfate. Calculations that incorporate the fluorescence efficiencies of both strontium and barium indicate that this signal would be expected from a material that contained near 7 weight % strontium.

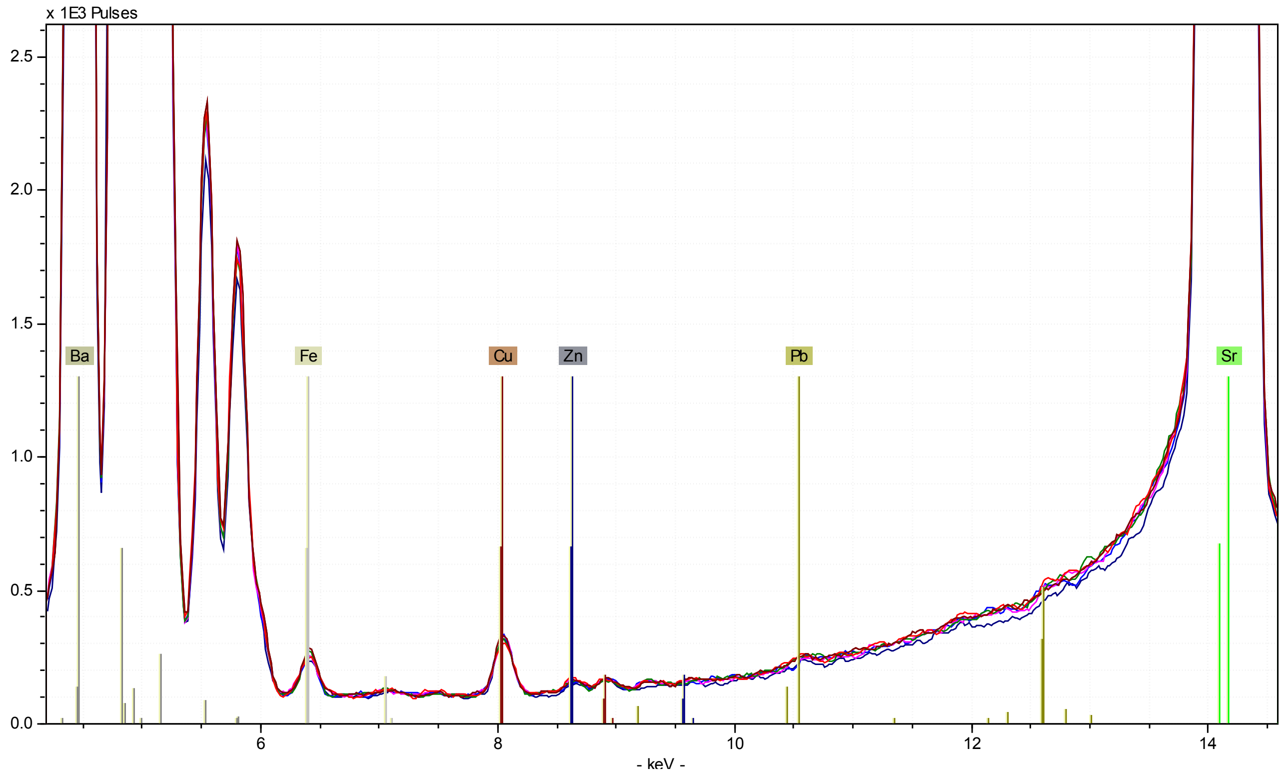

Figure 2: Spectral results for trace elemental composition. Small concentrations of iron, copper, zinc, and lead are visible in the spectra, these occur at the parts-per-million level.

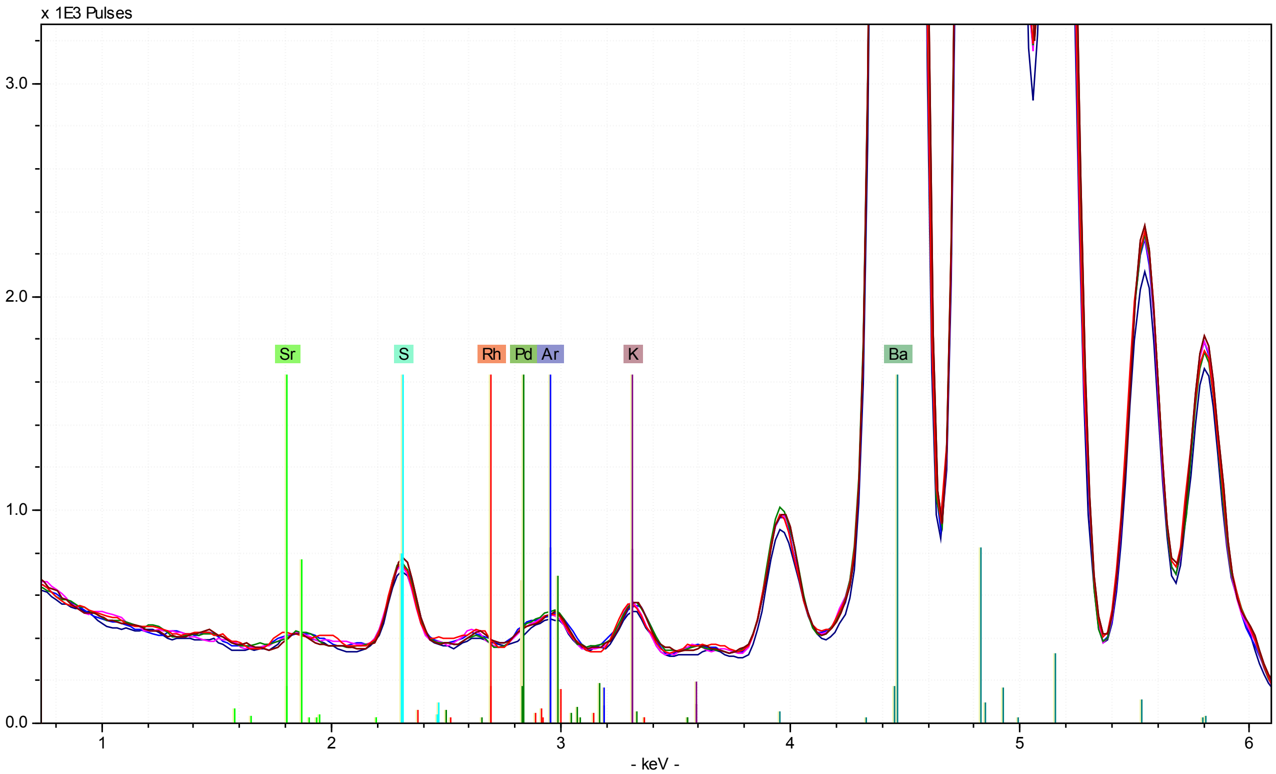

Figure 3: Spectral results for light elements. Potassium is clearly visible, while measurement parameters resulted in much lower sensitivity to sulfur. Rhodium is the x-ray excitation source, palladium is used as a collimator for the silicon-flash detector, and argon is present at ~1% of dry air composition.

	$BaSO_4$	$SrSO_4$	K	Fe	Cu	Zn	Pb
ba 1	93.27%	6.73%	368 ppm	98 ppm	65 ppm	12 ppm	117 ppm
ba 2	93.26%	6.74%	358 ppm	85 ppm	70 ppm	12 ppm	107 ppm
ba 3	93.25%	6.75%	365 ppm	85 ppm	72 ppm	11 ppm	99 ppm
ba 4	93.26%	6.74%	377 ppm	80 ppm	74 ppm	9 ppm	85 ppm
ba 5	93.22%	6.78%	344 ppm	80 ppm	80 ppm	9 ppm	97 ppm
“Pure Barium”	93.30%	6.70%	332 ppm	85 ppm	74 ppm	10 ppm	75 ppm

Table 1: Estimates of concentrations based on calculations originating from net photon counts.These should be regarded cautiously, as the calculations generate large uncertainty.

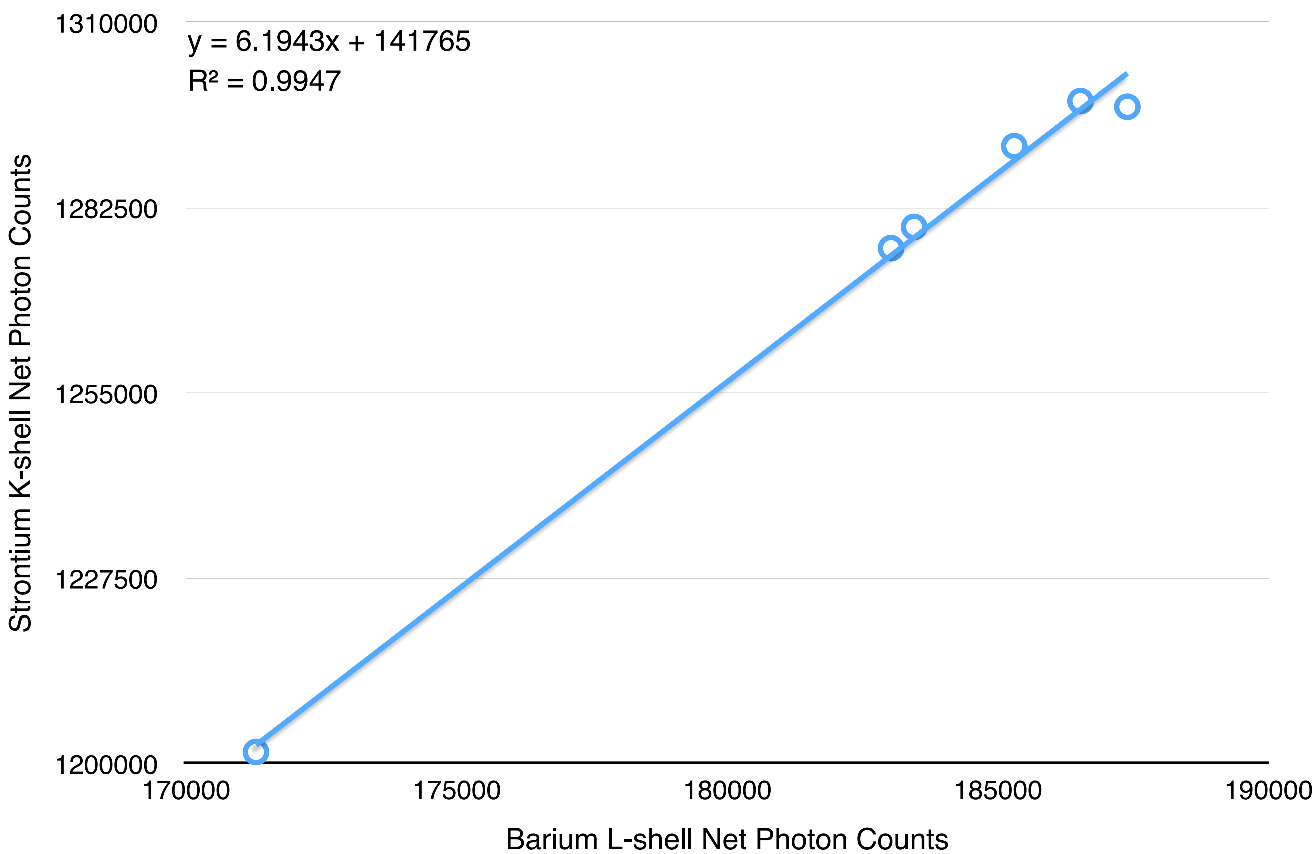

Figure 4: Calibration Validation. Barium net photons has a strong correlation with strontium net photons

Results

The data indicate that a substantial presence of strontium is in these materials - none can be correctly characterized as pure barium. The samples themselves are strikingly uniform even in trace elemental concentrations.

- Potassium, iron, copper, zinc, and lead are present as impurities in this material.
- The impurities seem strikingly uniform with regard to the matrix ($BaSO_4$).
- Barium and strontium concentrations are highly correlated.

These results should be treated as preliminary. The method for calibration, in absence of proper reference standards, generates large uncertainties. Concentrations in Table 1 should be treated as rough estimates, but are likely reflective of actual values.