

Sulfur Analysis in Oil

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BRUKER TEST RESULTS

Objective

We have analyzed oil to determine the sulfur content. A sample was brought into Kennewick Washington, which was analyzed in addition to oil standards at 0 ppm, 50 ppm, 1,000 ppm, and 10,000 ppm. The goal was to identify the limits to which handheld XRF units can be used for this purpose.

Method

Data was collected at 15 keV with a current of 25 μ A in vacuum conditions within 16 torr on system T3S2614. Validation is qualitative, as known concentrations are being used. One reference standard was used, plasma standard solution, Specpure $(\text{NH}_4)_2\text{SO}_4$, with a concentration of 10,000 ppm. This solution was then diluted 1 in 10 in H_2O to create a second standard with 1,000 ppm. Water was used as a sample for 0 ppm, and

Background

The absorption edge of Sulfur is 2.47 keV with a fluorescence efficiency of 7% at 2.3 keV at its K-alpha emission. As the Rhodium L emission of Rhodium sits on 2.7 keV, it acts as a secondary target. As a consequence, with vacuum control Sulfur is easily identifiable to low concentration levels in organic materials.

Results

At both concentrations, Sulfur was clearly visible in its K-alpha emission of

- Limit of Detection of sulfur in oil with the parameters used in this report is 100 ppm, plus or minus 100 ppm. Limit of Determination of sulfur would 300 ppm.
- 50 ppm is detectable using both qualitative and quantitative approaches
- No sulfur was detected in the unknown sample provided to Bruker.

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.

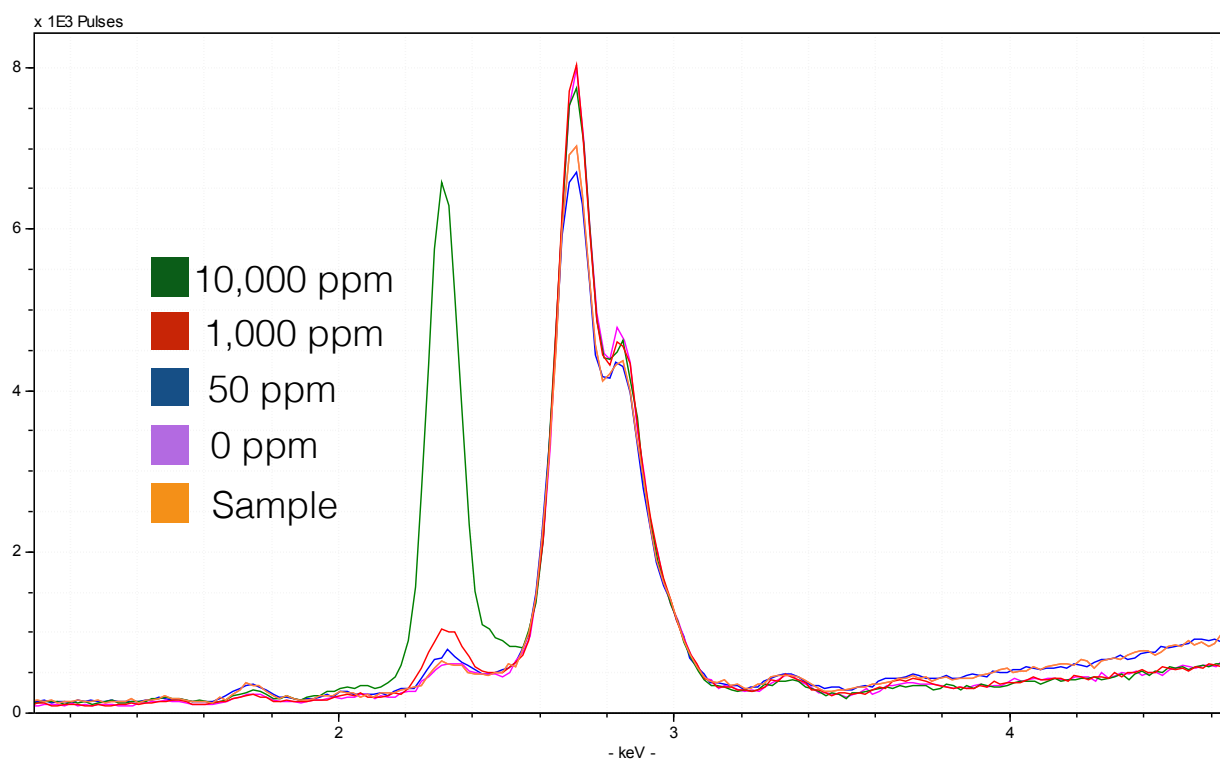


Figure 1: Spectral results of different oil standards. The unknown sample is not detectable, falling below a detection limit of around 100 ppm.

| Description | Net Normalized Sulfure | Quantified ppm for Unknown |
|----------------|------------------------|----------------------------|
| 0 ppm | 607 | |
| 50 ppm | 1229 | |
| 1,000 ppm | 3078 | |
| 10,000 ppm | 36221 | |
| Unknown Sample | 480 | 0 ppm |

Table 1: Quantitative and Bayesian Deconvolution Results. The net photon count of 480 falls within the photon count for 0 ppm, indicating no detectable sulfur is present using the current methods. 50 ppm was detectable, suggesting that the sample falls beneath even this limit.

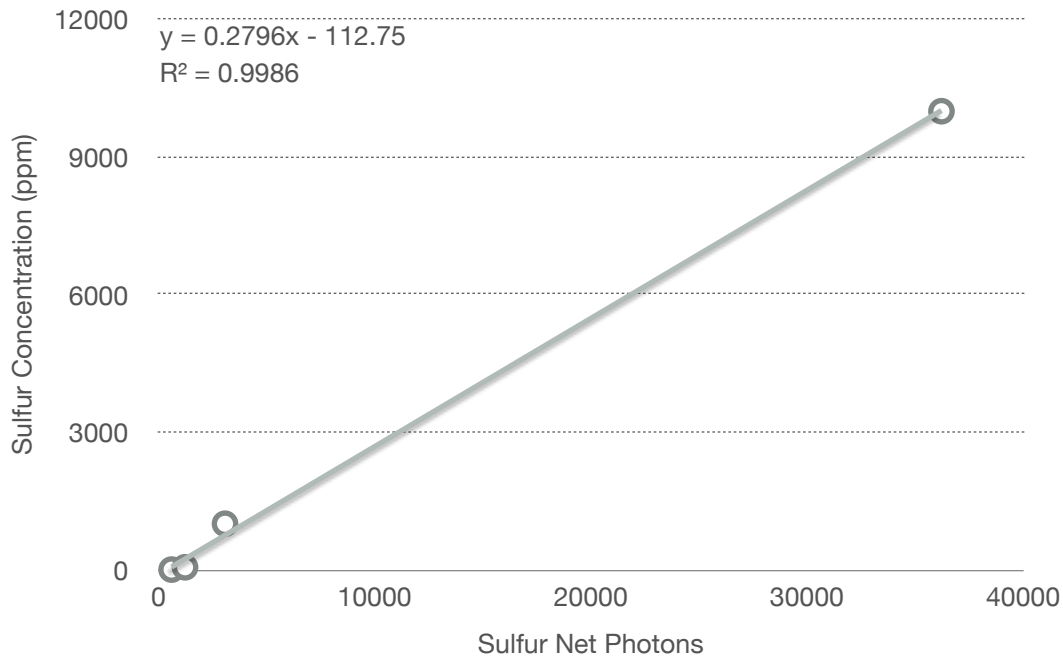


Figure 2: Calibration Validation. Sulfur Net Photons has a strong correlation with sulfur concentrations (ppm).