



High Atomic Number (High-Z) Chemistry with Laboratory XAS

X-ray Absorption Spectroscopy (XAS) is a powerful technique used to study the chemical states and coordination environment of elements of interest. In recent years, laboratory XAS has become commercially available, but many systems struggle with high-energy XAS at >12 keV. In this application note, we demonstrate Sigray QuantumLeap's outstanding performance for high-energy XAS with a Zirconium sample at 17,998 eV.

This white paper will review high energy performance of Sigray QuantumLeap XAS.



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Background: X-ray Absorption Spectroscopy (XAS) is a powerful technique used to study the chemical states and coordination environments of elements of interest. In recent years, laboratory XAS has become commercially available, but many systems struggle with high-energy XAS performance at >12 keV due to increasing sagittal errors for Johann-based geometries. This is problematic because many important catalysts (Pt and Zr) and actinides (U, Pu, Th, etc.) have L and K edges in the range between 12 and 25 keV (see Fig. 1).

Novel Approach: Sigray QuantumLeap XAS

Sigray's QuantumLeap™ x-ray absorption spectroscopy (XAS) product line represents the first laboratory XAS instruments with synchrotron-like capabilities. The QuantumLeap product line features multiple patented technologies, including its:

- ultrahigh brightness x-ray source technology
- acquisition approach
- system design

The Sigray QuantumLeap is optimally designed for a wide operating energy range between 4.5 and 25 keV. In particular, its high-energy capabilities between 12 and 25 keV are critical for a wide range of catalysis and nuclear applications.

The figure shows a standard periodic table of elements. Elements with L and K absorption edges between 12 keV and 25 keV are highlighted. K edges are colored in green, and L edges are colored in teal. The highlighted elements include: Zr, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Cd, In, Sn, Sb, Te, I, Xe, Ba, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Ac, Th, Pa, U, Np, Pu, Am, Cm, Bk, Cf, Es, Fm, Md, No, and Lr.

Figure 1: Periodic table of elements, with elements having L and K edges between 12 keV and 25 keV colored. K edges are colored in green and L edges are colored in teal.

Experiments and Results

In this report, we applied the Sigray QuantumLeap™ to a Zr foil of 7.5 μm to demonstrate its capabilities for high-energy XANES (X-ray Absorption Near Edge Spectroscopy) and EXAFS (Extended X-ray Absorption Fine Structure).

Method

Using QuantumLeap's intuitive software interface, the Zr K-edge was selected from a periodic table of elements. The system automatically selected the appropriate crystal: a cylindrically curved Johansson crystal Ge (800). A spectrum with energy ranging from 17.8 - 18.7 keV (wavenumber $k \approx 13 \text{ \AA}^{-1}$) was acquired in approximately 7 hours (see Figure 2). XAS data were processed and analyzed using the Athena and Artemis programs of the IFEFFIT package [1].

Results and Discussion

Quantitative analysis of Zr K-edge EXAFS was performed by fitting theoretical EXAFS spectra to the experimental data in R-space to obtain the structural parameters (see **Table 1**), including the coordination number N , the bond distance R , and the disorder factor σ^2 . The scattering contributions from the shortest Zr-Zr bond of 3.21 Å are included.

The correlation between bond length and disorder factors obtained from synchrotron and QuantumLeap data is excellent. The error bars of all parameters are small, demonstrating the reliability of the results.

Summary

We have demonstrated that laboratory XAS using the Sigray QuantumLeap™ can provide synchrotron-like performance for high-energy XAS analysis. By using well-established software, measurements of a Zr foil were converted into quantitative results, including bond distance, coordination number, and local atomic disorder.

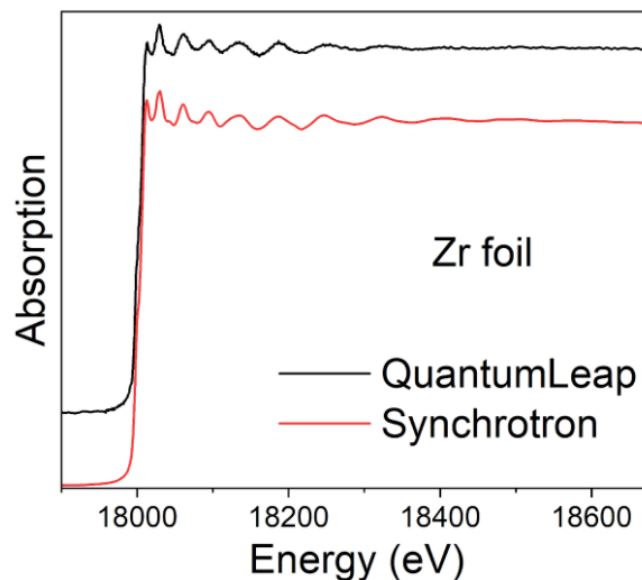


Figure 2: Zr K-edge (17998 eV) XANES and EXAFS of Zr foil acquired on the QuantumLeap (black), with comparative synchrotron data (red)

Structure Parameter	Synchrotron	Sigray QuantumLeap
N	12	12
R (Å)	3.23 ± 0.01	3.21 ± 0.01
σ^2 (Å ²)	0.009 ± 0.001	0.012 ± 0.002

Table 1: Synchrotron and Sigray QuantumLeap Zr (17.8 to 18.7 keV) spectra were analyzed with Artemis. Results show excellent agreement with each other, demonstrating the high energy XAS capabilities of QuantumLeap.

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