Applications Note

QuantumLeap XAS

High Atomic Number (Z) Chemistry with Laboratory XAS

X-ray Absorption Spectroscopy (XAS) is a powerful technique used to study chemical states and the 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 H2000's outstanding performance for high energy XAS with a Zn sample at 17998 eV.

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

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High Atomic Number (Z) Chemistry with Laboratory XAS

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Background: X-ray Absorption Spectroscopy (XAS) is a powerful technique used to study chemical states and the coordination environment of elements of interest. In recent years, laboratory XAS has become commercially available but many systems struggle in performance for high energy XAS at >12 keV because of increasing sagittal error for Johann-based geometries at high energies. 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).

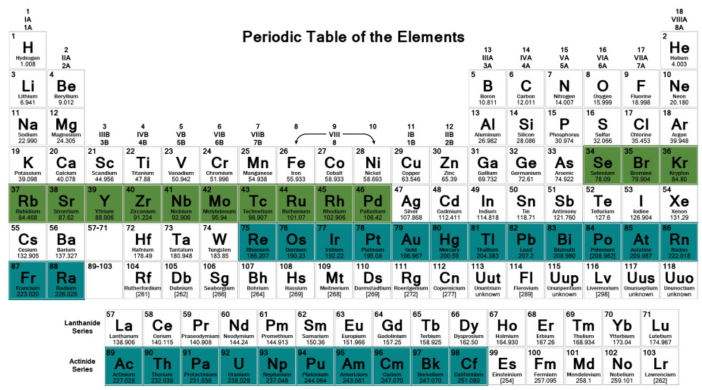

***Figure 1:** Periodic table of elements, with elements having L and K edges between 12 keV and 25 keV colored. L edges are colored in **green** and K edges are colored in **teal**.*

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, and
- system design.

The Sigray QuantumLeap H-series 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.

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). Spectrum with the energy from 17.8 - 18.7 keV (wavenumber k=~13 Å-1) was acquired in about 7 hours (see Figure 2). XAS data were processed and analyzed using 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 structure 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 the bond length and disordering factors between 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 through Sigray QuantumLeap™ can provide synchrotron-like performance for high energy XAS analysis. By using well-established software, measurements of a Zn foil were transformed into quantitative results (bond distance, coordination number, and local atomic disorder).

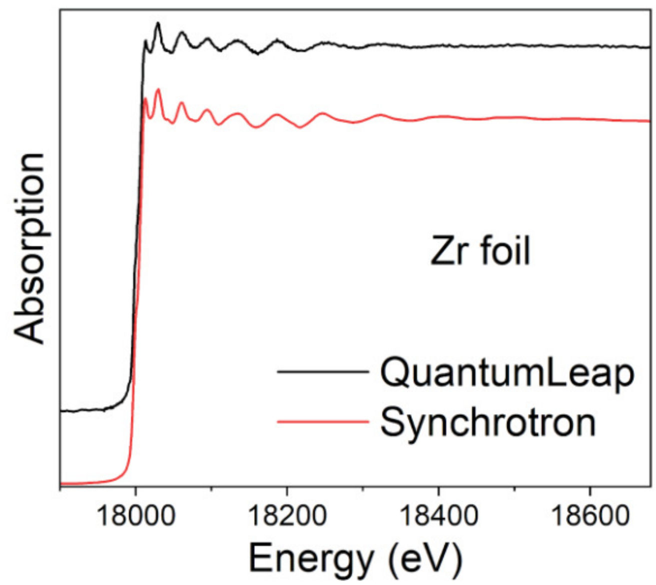

Figure 2: Zr K-edge (17998 eV) XANES and EXAFS of Zr foil acquired on the QuantumLeap (black), with comparative synchrotron data (red)

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 Athena. Results show excellent agreement with each other, demonstrating the high energy XAS capabilities of QuantumLeap.

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1. B. Ravel and M. Newville, ATHENA, ARTEMIS, HEPHAESTUS: data analysis for X-ray absorption spectroscopy using IFEFFIT, Journal of Synchrotron Radiation 12, 537–541 (2005).

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