

Biological XAFS on the BioCAT Undulator Beamline 18-ID

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Introduction

Experimental feasibility tests have been successfully carried out on a diverse set of biological and environmental systems, including site-directed mutants of bacterial dimeric hemoglobins, iron-sulfur centers from Photosystem I, iron bound to eumelanins, and the dilute mercury and selenium environments in cormorant liver tissues. Thermal and temporal stability of the beamline is now excellent for an undulator beamline, which can focus the full beam of $\sim 3 \times 10^{13}$ photons/sec into beam sizes as small as approximately 100 micrometer by 20 micrometer. Scans can be performed in tens of seconds, and analyzable data on sub-millimolar samples can be obtained in minutes. The Si(111) monochromator covers the range from 4 keV to 15 keV, while the Si(400) monochromator covers the ranges from 8 keV to 35 keV (70 keV in the second harmonic). A mirror is used to reject harmonics.

The 20-element multilayer array analyzer/detector has proven to be an effective instrument: it is essentially equivalent in performance to a conventional multielement solid-state detector, but importantly, because of the use of a fast, large-area current integrating detector following the analyzer, it effectively has no dead-time or pulse pile-up. This makes it especially suitable for the high fluxes delivered by the beamline, and it is suitable for fast time-resolved studies including pump-probe experiments synchronized to the time structure of the ring. It should be noted that Stern/Heald detectors with optimized filters and two-dimensionally focused slits are quite suitable detectors for many pur-

poses because of their immunity to deadtime issues and the large solid angle of collection.

The multilayer analyzer is by far superior in some cases, however. For example, this instrument provided in minutes analyzable EXAFS spectra on dilute (~ 10 ppm) Cu in a soil sample containing a high concentration ($\sim 30\%$) of Fe. This situation is problematic for filter/slit systems and solid-state detectors because the very intense Fe K-alpha fluorescence is at lower energy than the Cu K-alpha fluorescence and cannot be effectively rejected by a Z-1 filter, and the high Fe fluorescence intensity saturates solid state detectors. This detector is a useful addition to the range of available detectors for XAFS.

In summary, the BioCAT facility recently has been demonstrated to be a uniquely capable instrument for biological and environmental XAFS studies, particularly in those cases where a high-intensity focused beam is required. The primary remaining issues at this point for productive biological XAFS are sample related: how to minimize radiation damage and how to minimize artifacts due to ice crystallites in the samples.

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