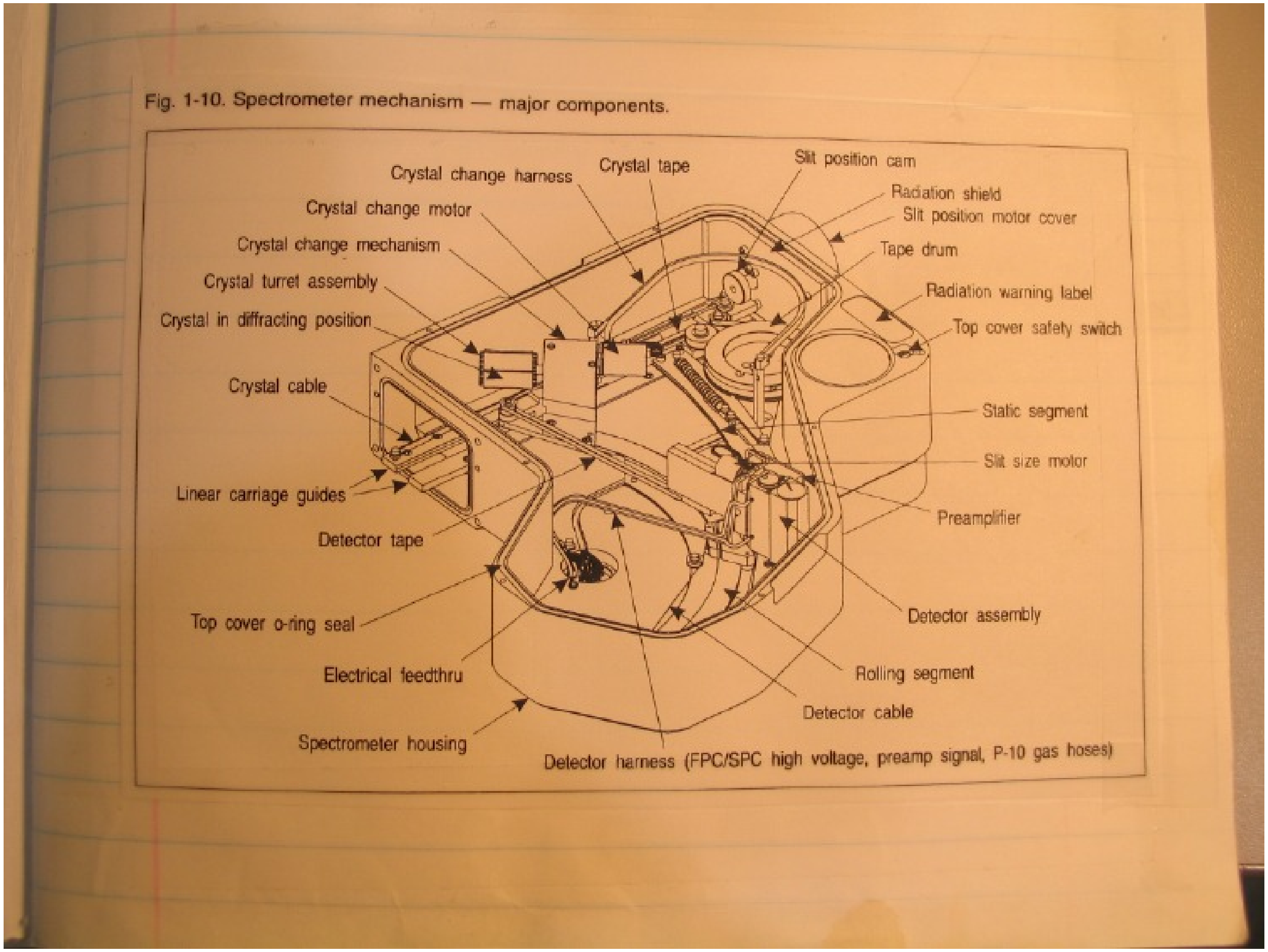
E-bay, A resource for scientists? Or: The wavelength dispersive spectrometer at Sector-20.

Julie Cross, PNC-CAT

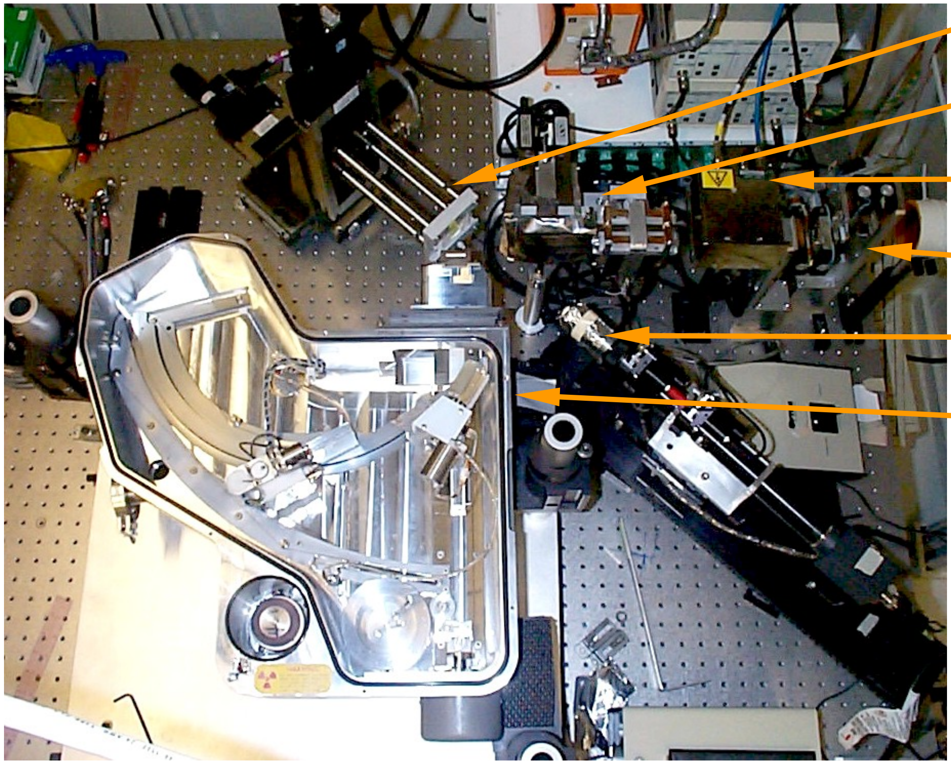
TWG meeting presentation, October 21, 2004

WDX 2A Schematic



The Wavelength Dispersive Spectrometer (Oxford WDX-600)

Borrowing technology developed for the electron-microscope community, the Wavelength Dispersive Spectrometer uses an analyzer crystal on a Rowland circle to select a fluorescence line. This has much better resolution (~30eV) than a solid state detector (~250eV), doesn't suffer from electronic effects like dead-time, and can have superior peak-to-background ratios. The solid-angle and count-rates are somewhat lower.



Sample and x-y-z stage

Kirkpatrick-Baez focusing mirrors

Ion chamber

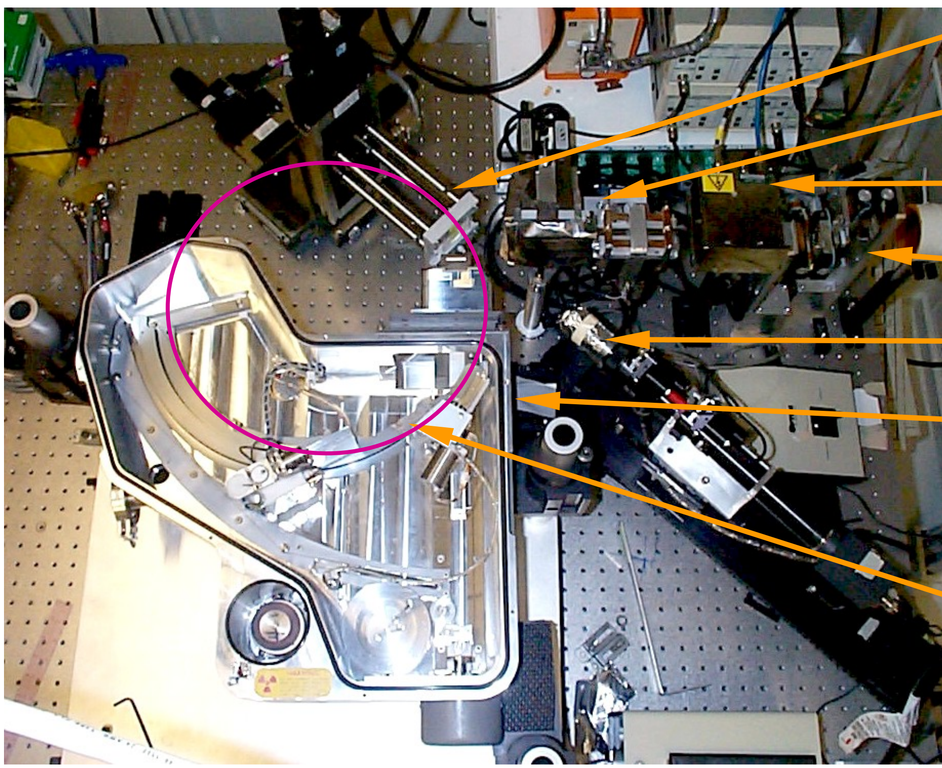
Table-top slits

Optical microscope

Wavelength Dispersive Spectrometer

The Wavelength Dispersive Spectrometer (Oxford WDX-600)

Borrowing technology developed for the electron-microscope community, the Wavelength Dispersive Spectrometer uses an analyzer crystal on a Rowland circle to select a fluorescence line. This has much better resolution (~30eV) than a solid state detector (~250eV), doesn't suffer from electronic effects like dead-time, and can have superior peak-to-background ratios. The solid-angle and count-rates are somewhat lower.

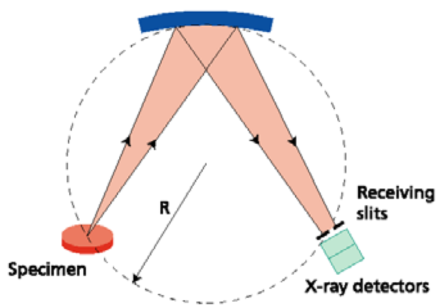


WDX-600: detailed view

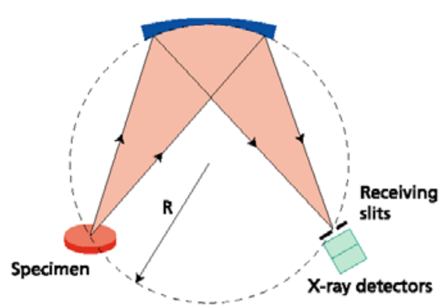
detectors = 2 proportional counters: (one flowing P-10 gas, and one sealed with 2 atm Xe) in tandem.

slits: define angular acceptance and energy resolution

crystals = LiF (200), LiF(220), LiF(420), and PET, on a six crystal turret. Crystal size ~45 x 15 mm



Johann geometry bent to radius 2R



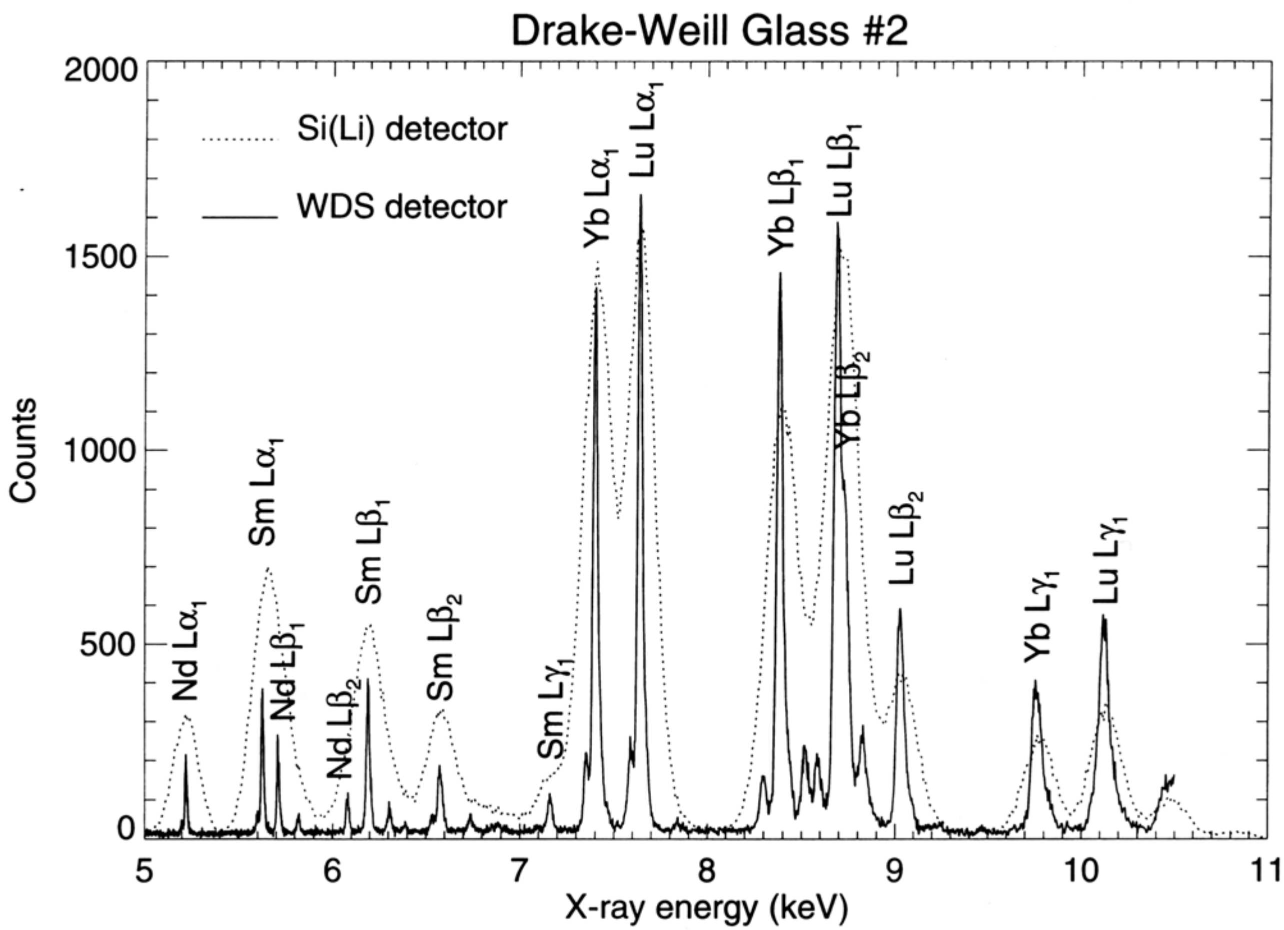
Johansson geometry bent to radius 2R and ground to radius R

By using a Johannson geometry Rowland circle, a point source focuses to a point at the detector slit. Aberrations are minimized, and the signal-to-noise ratio is improved.

Comparisons of the WDS and solid-state detectors

Steve Sutton and Mark Rivers, data collected at NSLS X-26A.

Detail of the XRF spectrum for a synthetic glass containing several rare-earth elements using both a Si(Li) detector and the WDS.

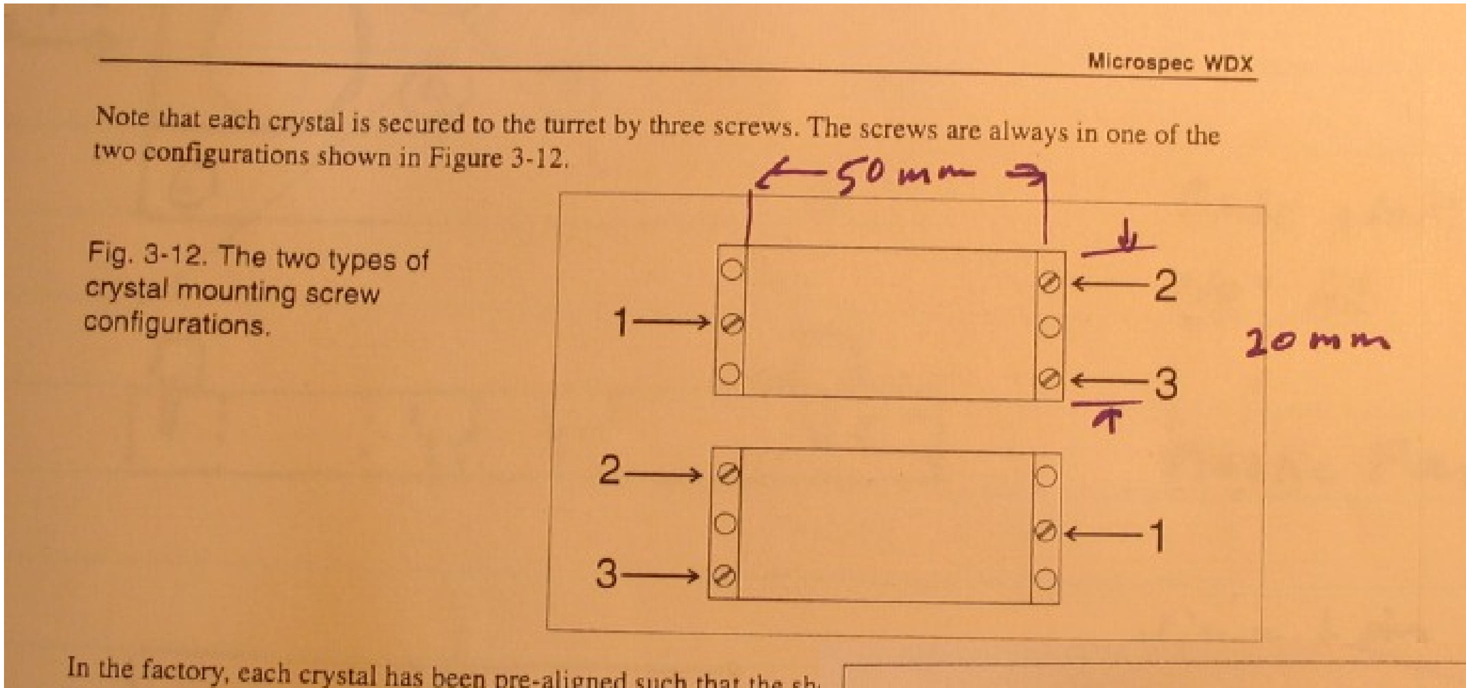


Comparisons of the WDS and solid-state detectors

Typical values for the WDS and a Ge solid-state detector

	WDS	Ge Solid-State
energy resolution:	~30eV	~100eV to ~300eV depending on shaping time
active area:	~500mm^2` (varies with angle)	100mm^2 (per detector, often 13X)
working distance:	~180mm	~100mm
max total count rate:	none	100KHz (per detector, often 13X)

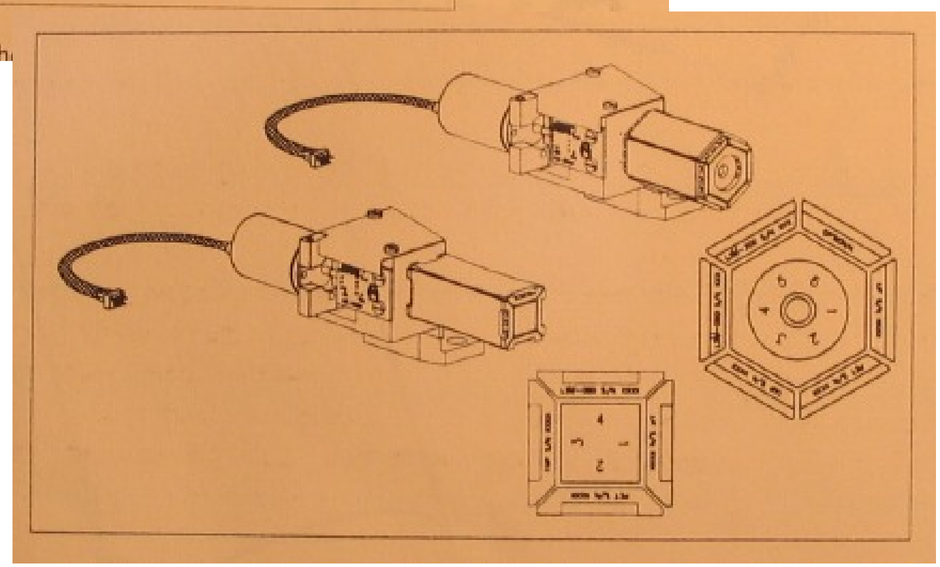
Aperture and Crystal Size


Microspec WDX

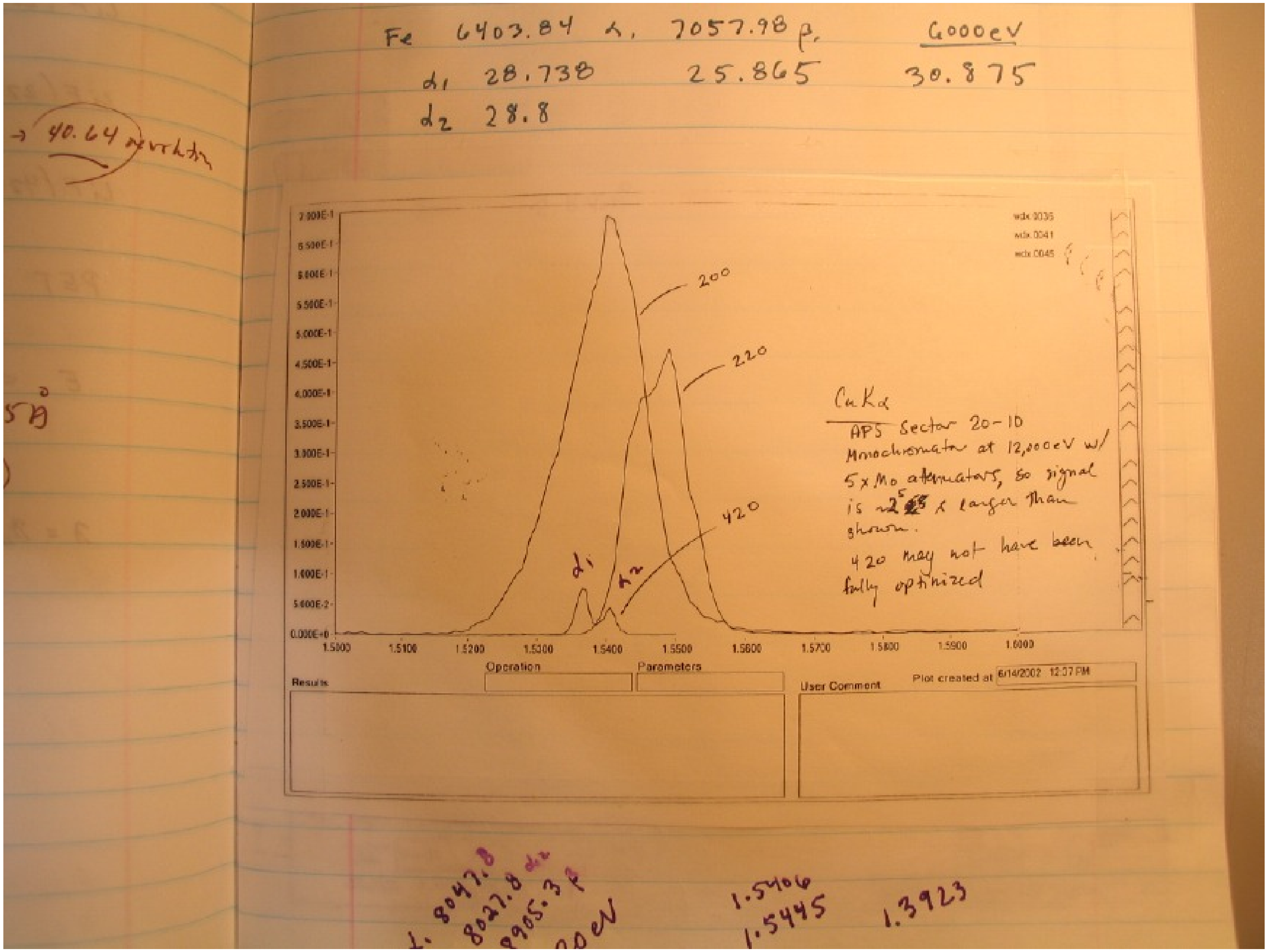
Note that each crystal is secured to the turret by three screws. The screws are always in one of the two configurations shown in Figure 3-12.

Fig. 3-12. The two types of crystal mounting screw configurations.

In the factory, each crystal has been pre-aligned such that the sh



Comparison of Analyzer Resolution and Efficiency at Cu Kα_1, α_2 LiF (400), (220) and (420)

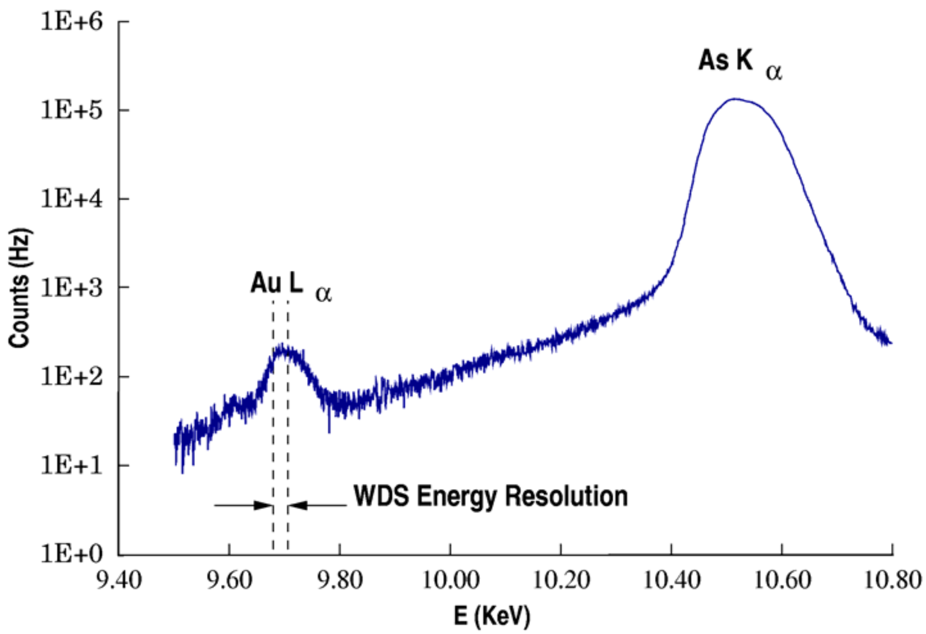


Using the WDS for XANES: 1000ppm Au in FeAsS (arsenopyrite)

Louis Cabri (NRC Canada), Robert Gordon, Daryl Crozier (Simon Fraser), PNC-CAT

1000ppm Au in FeAsS (arsenopyrite): The understanding of the chemical and physical state of Au in arsenopyrite ore deposits is complicated by the proximity of the Au L_{III} and As K edges and their fluorescence lines.

At the Au L_{III}-edge, As will also be excited, and fluoresce near the Au L_α line.



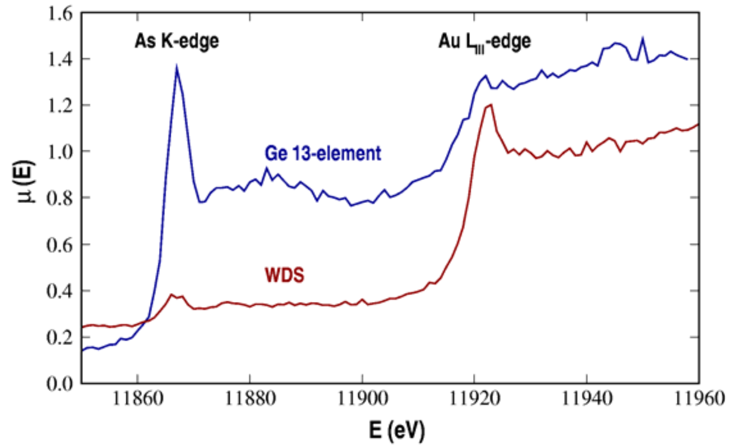
As K-edge	11.868 KeV
As K_α line	10.543 KeV
Au L_{III}-edge	11.918 KeV
Au L_α line	9.711 KeV

Even using the WDS, the tail of the As K_α line persists down to the Au L_α line, and is still comparable to it in intensity.

Using the WDS for XANES: 1000ppm Au in FeAsS (arsenopyrite)

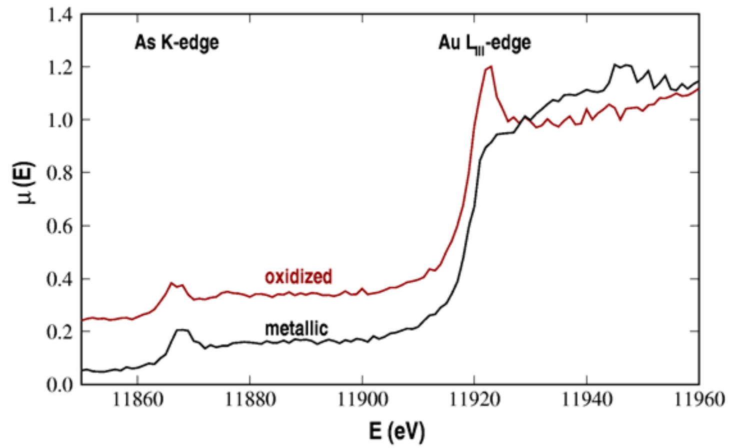
Louis Cabri (NRC Canada), Robert Gordon, Daryl Crozier (Simon Fraser), PNC-CAT

With a 13-element Ge detector (at PNC-CAT: ID-20), the tail of the As K_α line was still strong at the Au L_α energy, so the Au L_{III} edge-step was about the same size as the As *K* edge-step, and the Au XANES was mixed with the As EXAFS.



With the WDS, the As edge was visible, but much smaller, so the Au XANES was clearer.

Measuring two different natural samples of FeAsS, both with ~1000ppm of Au, we see evidence for both metallic and oxidized Au.



Issues using the WDS

Alignment: The WDS weighs ~30kg, and needs to be aligned fairly well:

~1 mm vertical
~1 mm in/out-board
~10μm up/down-stream

For our initial run, we adjusted the height by hand, and had a motorized in/out-board motion. For the up/down-stream position, we brought the sample to the spectrometer, which limits the focusing ability of the microprobe.

Tunability: The WDS selects one energy at a time, and looking at different energies requires a mechanical scan. So, unlike a solid-state detector, the WDS does not simultaneously measure multiple energies --- it does not have an MCA.

So XRF maps of multiple elements (like the Sr/Ca example) are not practical with the WDS.

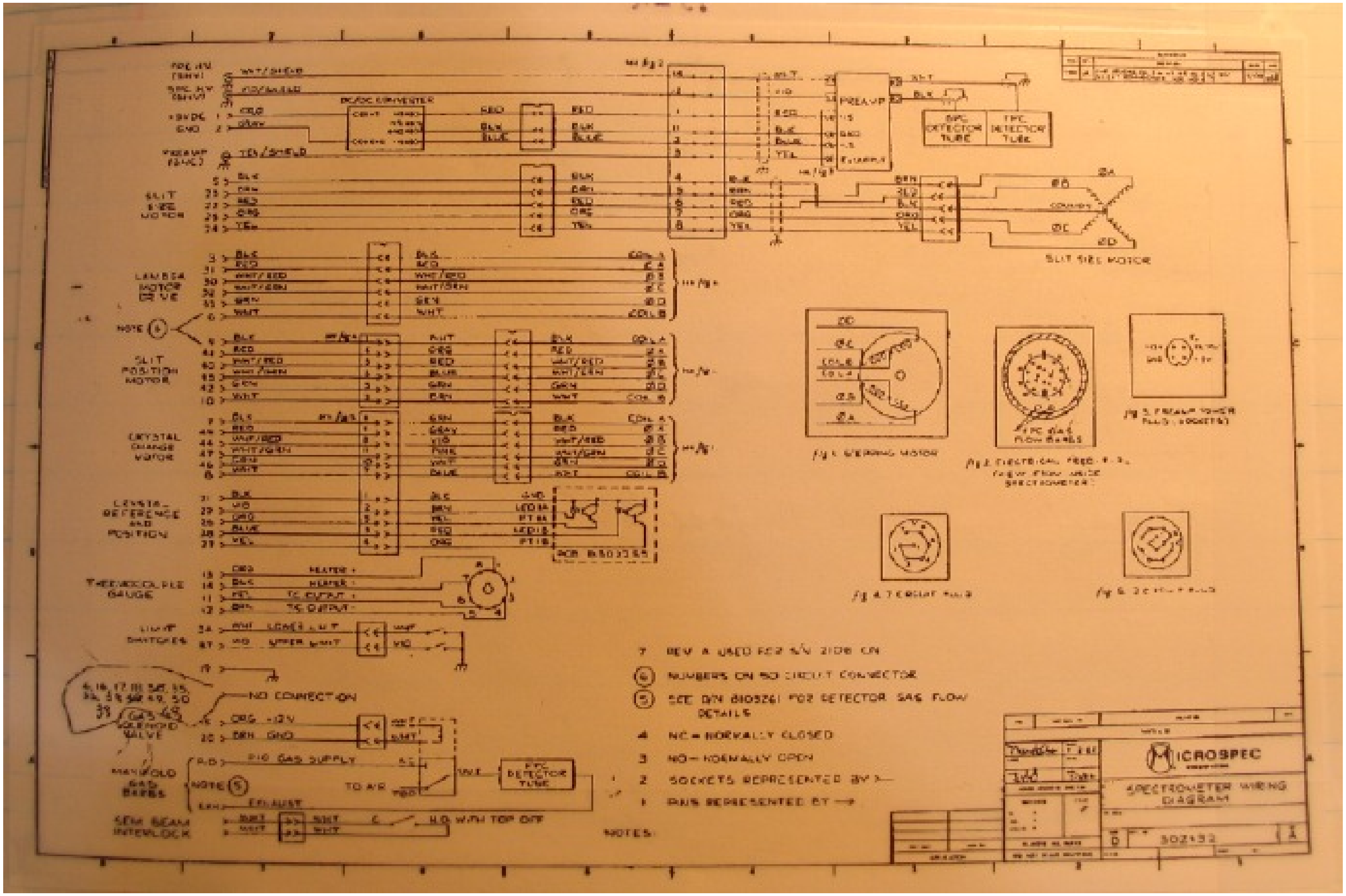
Breakdown of Components to Replace the Control Module

NIM crate and power supply	Ortec 4001A/4002D	$1800
Dual 0-2kV power supply	Ortec 660	1900
Spectroscopy amplifier	Ortec	2300
Single Channel Analyzer	Ortec 550A	650
Ratemeter	Ortec	900
Pre-amp power	Newark	80
Gas flow solenoid power	Newark	40
Break-out cable CEN-50 to Elko and BNC	Newark, misc. parts	50
Stepper motor control (4)	OMS, ACS	4400
TOTAL		$12,120

Breakdown of Additional Equipment Needed to Adapt the Oxford/Microspec WDX to the Synchrotron Microprobe

Stepper motor control (3)	ACS	$1800
P-10 gas bottle regulator	McMaster-Carr	250
Roughing vacuum pump	Varian	1200
Vacuum valve and lines	MDC	200
XYZ positioning stage	ADC	7200
Stepper motor control (3)	OMS, ACS	3300
Vacuum flight tube	Machine shop	180
Observation window	Machine shop	120
Mounting hardware, base	Machine shop	120
LiF(220) Johansson analyzer	SpexRay	2750
LiF(420) Johansson analyzer	SpexRay	2750
TOTAL		$19,870

Interface



SLIT SIZE MOTOR
LAMBDA MOTOR DRIVE
SLIT POSITION MOTOR
CRYSTAL CHANGE MOTOR
THERMOCOUPLE GAUGE
LIMIT SWITCHES
NO CONNECTION
PREAMP
DC/DC CONVERTER
SPC DETECTOR TUBE
FPC DETECTOR TUBE
7 REV A USED FOR S/N 2108 ON
6 NUMBERS ON 50 CIRCUIT CONNECTOR
5 SEE D/N 810326I FOR DETECTOR GAS FLOW DETAILS
4 NC = NORMALLY CLOSED
3 NO = NORMALLY OPEN
2 SOCKETS REPRESENTED BY
1 PINS REPRESENTED BY
NOTES:
MICROSPEC
SPECTROMETER WIRING DIAGRAM
D
302132

Cost Comparison Between Oxford and E-Bay WDX

Oxford/Microspec WDX-600 with Controller	$130,000
Modifications for Microprobe	$20,000
TOTAL	$150,000

E-Bay/Surplus WDX-400	$10,000
Equipment to replace Controller	$12,000
Modifications for Microprobe	$20,000
TOTAL	$44,000

Using the WDS for EXAFS: Re in $K_7[ReOP_2W_{17}O_{61}].nH_2O$

Mark Antonio (ANL)

The inorganic molecule α-$P_2W_{17}O_{61}$ is a candidate for stabilizing transition and rare-earth metal ions. It can lose a WO ligand and replace it with several valence states of Re (a nice, safe chemical analog of Tc).

The proximity of the Re and W L_{III}-edges, and their L_α lines, and the relative concentrations of Re and W (1::61) in this sample makes EXAFS measurements using a solid-state detector nearly impossible.

W L_{III}-edge	10.204 KeV
W L_α line	8.396 KeV
Re L_{III}-edge	10.534 KeV
Re L_α line	8.651 KeV

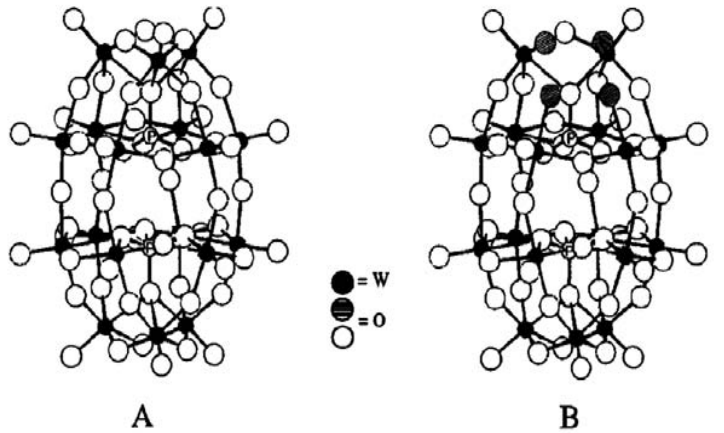
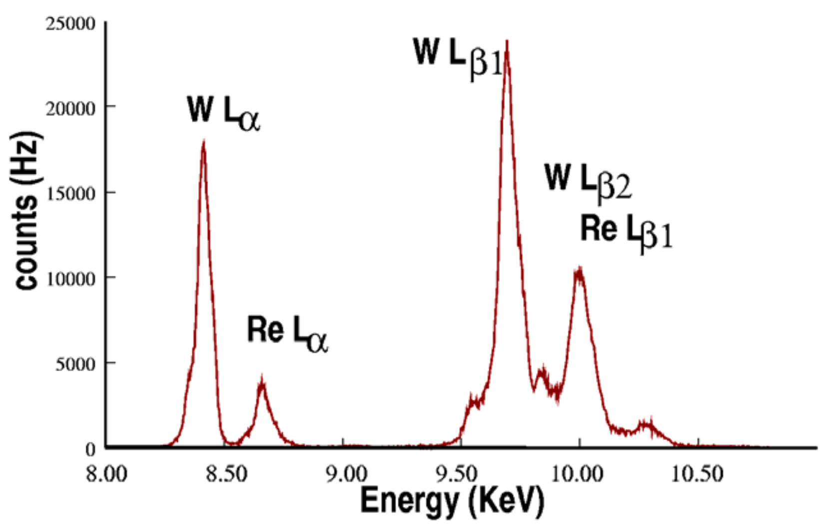
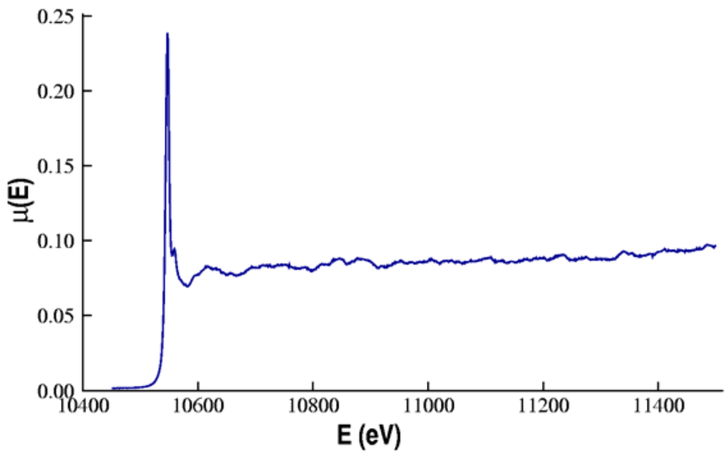


Fig. 1 Ball and stick model of A $[\alpha\text{-}P_2W_{18}O_{62}]^{6-}$ and B the lacunary $[\alpha_2\text{-}P_2W_{17}O_{61}]^{10-}$ ligand.

Venturelli, et al, J. Chem. Soc., Dalton Trans., p 301 (1999)

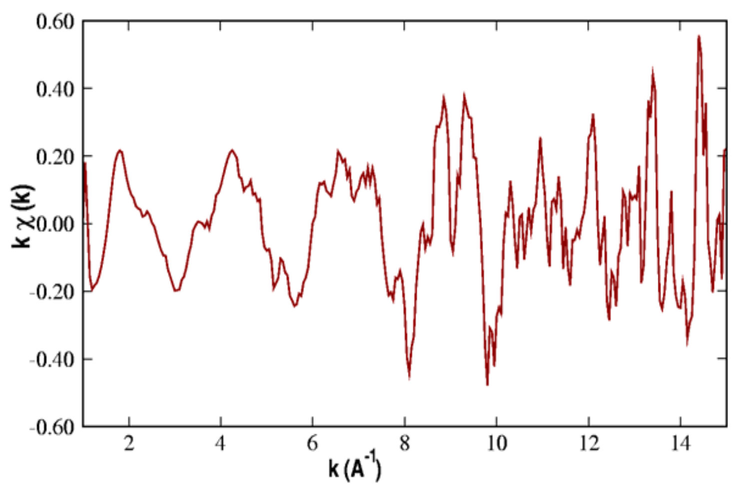


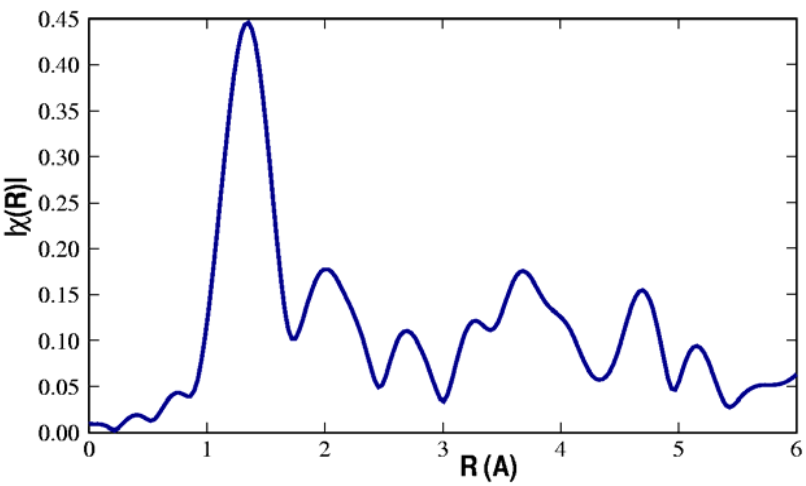
Using the WDS for EXAFS: Re in $K_7[ReOP_2W_{17}O_{61}].nH_2O$



Here are μ(E), the EXAFS kχ(k), and the Fourier transform of the EXAFS |χ(R)| for data collected with the WDS. The data is the average of 3 scans, each having an integration time of 5 seconds per point.

The data quality is acceptable up to ~12A^{-1}, and initial analysis supports a first shell with 4 oxygens at 1.8A.





Using a Wavelength Dispersive Spectrometer to measure XAFS

The Wavelength Dispersive Spectrometer can be used for XANES and EXAFS measurements. In some cases it is sometimes the only detector capable of such measurements.

In many cases, the WDS compares favorably with solid state detectors.

In some cases, the WDS is superior to solid-state detectors, and is the only detector capable of XRF, XANES, and EXAFS measurements.