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innovative coating technologies

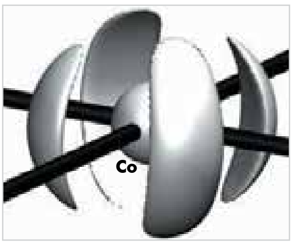
IμS for Ag-Kα Radiation

The Microfocus X-ray Source for Short Wavelength Radiation

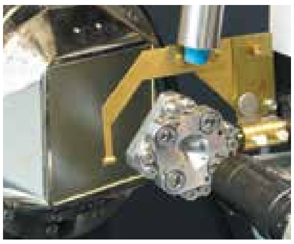
The Incoatec Microfocus Source IμS and IμS High Brilliance

Huber system equipped with a Ag-IμS, Univ. of Durham

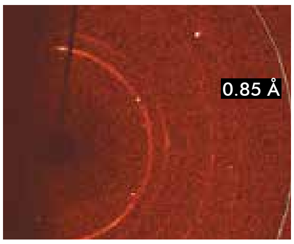
Combined XRF/XRD setup with Ag-IμS, Univ. of Antwerp



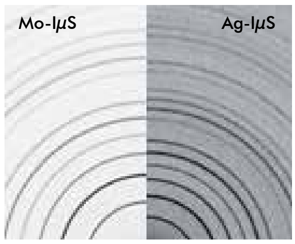
Laplacian of the charge density in $Sc_3Co(C_2)_2$



Diamond anvil cell on a SMART APEX II goniometer



Diffraction pattern of an organic compound in a DAC



Comparison of powder diffraction patterns of LaB_6

Higher resolution - Less absorption

- Air-cooled low-power microfocus X-ray source
- 2D focused beam (FWHM = 0.09 mm; 5 mrad divergence)
- Diffracted intensity up to 4 times higher than that of a 1.5 kW Ag sealed tube system with graphite monochromator
- Ideal for high-resolution crystallography, especially for strongly absorbing materials, and high-pressure experiments on single crystals and powders

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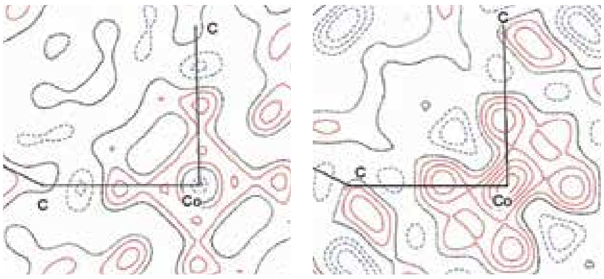
High-Resolution Crystallography

The IμS for Ag radiation delivers a focused beam with a FWHM of 0.09 mm. The advantages of Ag radiation are the reduced absorption and extinction, and the „compression" of the reciprocal space. Thus, data are less biased by extinction and absorption, and access to a larger range of *d*-spacings at a fixed 2θ setting is gained. This is beneficial for high-resolution diffraction experiments, such as charge density measurements or studies on inorganic solid state compounds. Table 1 and 2 show comparisons of two single crystal diffraction experiments on inorganic samples measured with Ag and Mo radiation.

Ag-IμS Mo-ST



Source	Ag-IμS	Mo-ST
R1; *w*R2	0.0091; 0.0217	0.0122; 0.0160
	$\rho(r)_{BCP}$[eÅ^{-3}] / $L(r)_{BCP}$[eÅ^{-5}]	
Co-C	0.534 / 5.2	0.581 / 5.4
C-C	1.845 / -8.7	1.813 / -12.0

Table 1: Comparison of the residual density of $Sc_3Co(C_2)_2$ after multipole refinement: Ag-IμS (left, APEX II detector), Mo-ST (right, scintillation counter)

Source	Ag-IμS	Mo-IμS
Sample	$Cu_6PbO_{8-x}(Cl,Br)_{2x}$	
Size [mm^3]	0.12 x 0.09 x 0.06	
μ [mm^1]	20.6	38.4
Exposure time [s/0.3°]	10	10
max- resolution [Å]	0.61	0.77
Unique data	127 (43)*	69 (17)*
<I/σ>	158.9 (126.9)*	137.9 (121.2)*
<I/σ> (to 0.80 Å)	181.4 (168.2)*	140.1 (126.9)*
R1 (all); *w*R2 (all)	0.0196; 0.0525	0.0197; 0.0536

Table 2: Details for comparative measurements on $Cu_6PbO_{8-x}(Cl,Br)_{2x}$ (values for highest resolution shell)*

High-Pressure Crystallography

The larger reciprocal space that is accessible with the Ag-IμS and the small beam cross-section make the Ag-IμS an interesting alternative to sealed tube sources for high-pressure diffraction studies on single crystals and powder samples. The sharp beam profile from the focusing optics reduces the background that is due to the scattering from the gasket of the diamond anvil cell (DAC), as shown in Figure 1. The improved completeness and resolution of the data facilitate structure solution and refinement of high-pressure phases.

Ag-IμS Mo-ST

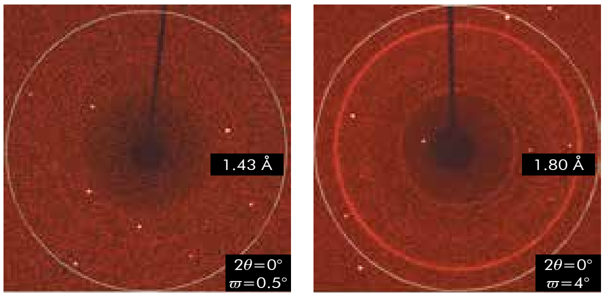


Figure 1: Diffraction patterns of a single crystal (organic sample; Ø ≈ 0.25 mm) grown in-situ in a Be-free DAC (0.8 GPa, recorded with a Bruker AXS APEX II diffractometer, DX=71 mm): Ag-IμS (left), Mo sealed tube (graphite monochromator, 0.5 mm collimator, right).

Source	Ag-IμS	Mo-ST
Power	0.03	2.0
Exposure time [s/0.3°]	20	20
<I>	368.8 (64.9)*	378.0 (61.0)*
<I/σ>	19.6 (3.2)*	18.3 (4.7)*
Unique data	866 (170)*	721 (135)*
<Redundancy>	1.5 (0.9)*	1.1 (0.7)*
<Completeness>	40.6 (28.9)*	33.7 (22.6)*
R(int)	0.0306 (0.1636)*	0.0342 (0.1489)*
R(σ)	0.0415 (0.2877)*	0.0361 (0.2073)*
R1 ($I<2\sigma(I)$)#	0.0487 (630)#	0.0532 (523)#
*w*R2 (all)&	0.1025 (860)&	0.1232 (705)&

Table 3: Data statistics for the comparative measurement (resolution 0.90 Å (1.00 - 0.90); #, & number of reflections used).*

Acknowledgement

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