MEASUREMENTS OF Li_2SrSiO_4, $K_3[Fe(CN)_6]$, Fe AND Mn_2CrO_4 APPLYING A SCINTILLATION COUNTER AND A SI PIN DIODE POINT DETECTOR

SETUP

Samples of Li_2SrSiO_4, $K_3[Fe(CN)_6]$, Fe and Mn_2CrO_4 were measured using transmission geometry on a STOE STADI MP diffractometer equipped with pure Cu $K\alpha_1$ radiation. Measurements were carried out with a scintillation counter (with and without secondary beam monochromator; 0.2mm slit size) and a Si PIN diode point detector (0.2mm slit size).

RESULTS

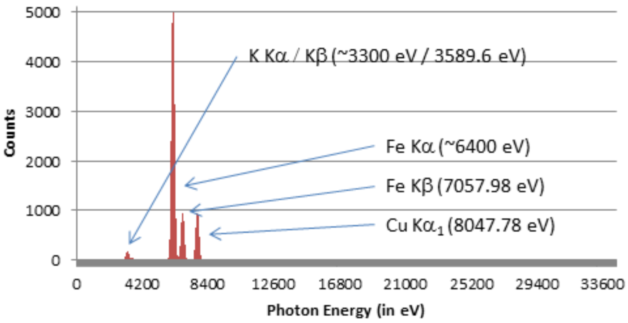

Figure 1: Photon energy spectrum of $K_3[Fe(CN)_6]$ after 15 h exposure time measured with a Si PIN diode point detector and 8.0 keV excitation energy (Cu $K\alpha_1$) ; clear separation of $K\alpha$ and $K\beta$ of Fe is visible.

A Si PIN diode enables the energy discrimination of the incident radiation, with a FWHM of the energy peak close to 200 eV. Therefore it is possible to clearly separate between $K\alpha$ and $K\beta$ radiation of Co, Cu, Mo and Ag and also cut off X-ray fluorescence appearing at absorption edges.

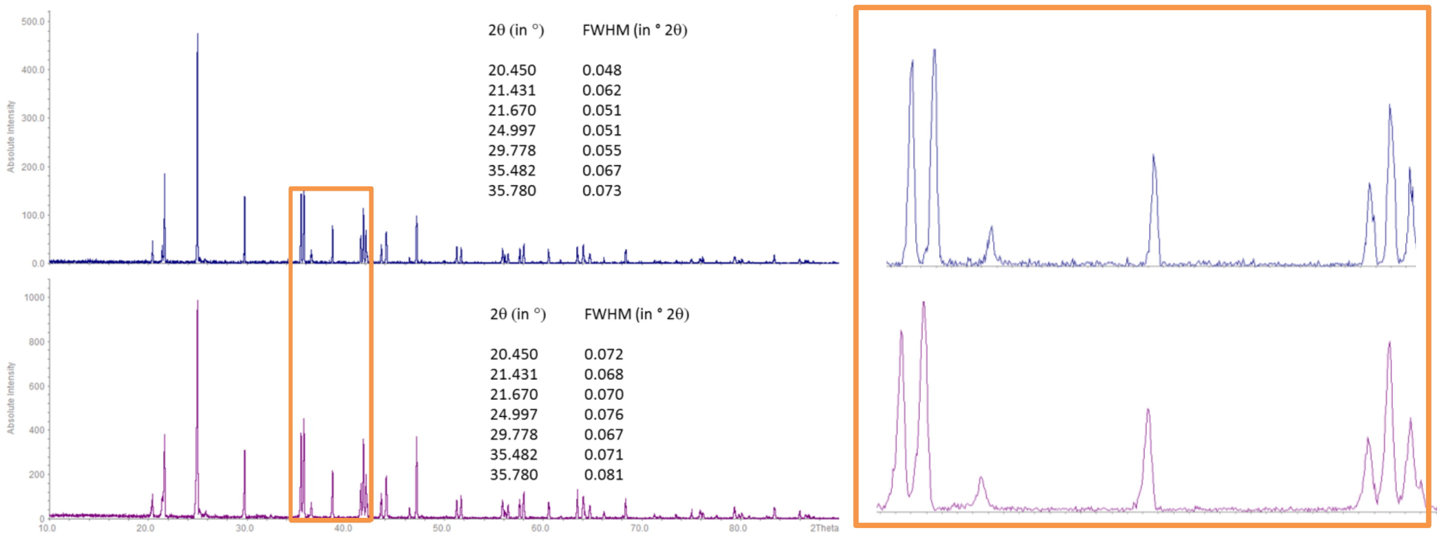

Figure 2: Measurement (17h measurement time) of Li_2SrSiO_4 with a Si PIN diode point detector (blue) and a scintillation counter without secondary beam monochromator (purple); FWHM were taken from a Le Bail[1] fit with asymmetry corrected according to Finger, Cox and Jephcoat[2]; The FWHM of the Si PIN diode point detector is up to 50% smaller compared to the scintillation counter (depending on the scattering angle). A close-up between 35 and 42.5° 2Θ shows complete separation of reflections for the data of the Si PIN diode.

[1] A. Le Bail, H. Duroy, J. L. Fourquet, *Mat. Res. Bull.* **1988**, *23*, 447.
[2] L. W.Finger, D. E. Cox, A. P. Jephcoat, *J. Appl. Cryst.* **1994**, *27*, 892.

MEASUREMENTS OF Li_2SrSiO_4, $K_3[Fe(CN)_6]$, Fe AND Mn_2CrO_4 APPLYING A SCINTILLATION COUNTER AND A SI PIN DIODE POINT DETECTOR

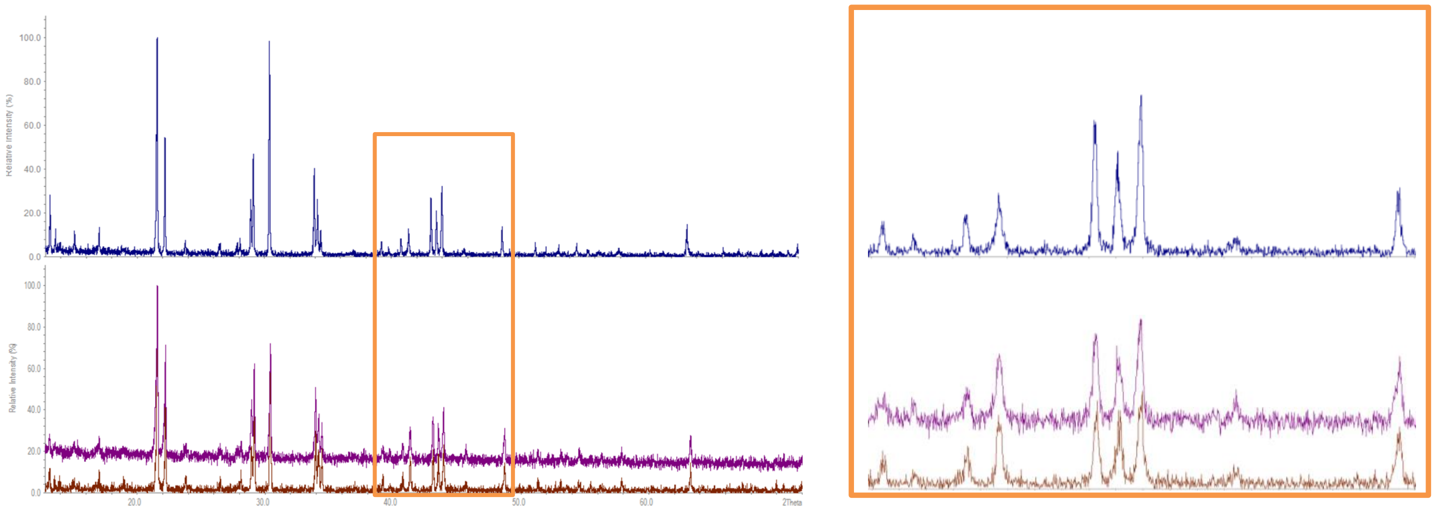

Figure 3: Measurements of $K_3[Fe(CN)_6]$ with Cu $K\alpha_1$ radiation; Si PIN diode point detector (blue) in comparison to scintillation counter with (brown) and without (purple) secondary beam monochromator after 17h measurement time. A close-up between 41 and 49° 2θ shows an improved signal-to-noise ratio for the Si PIN diode point detector compared to the scintillation counter.

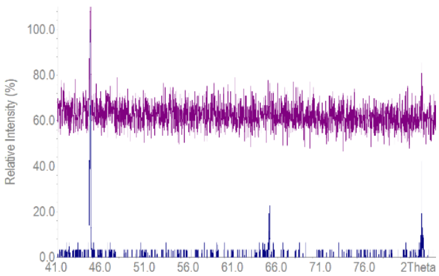

Figure 4: Fe measured with Si PIN diode point detector (blue) and scintillation counter without secondary beam mono-chromator (purple) with peaks scaled to 100%; increased background is due to fluorescence.

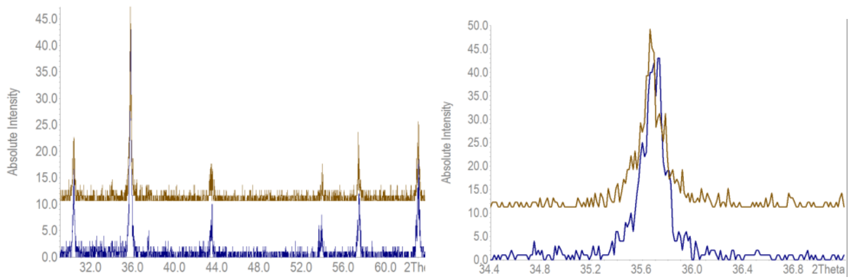

Figure 5: Mn_2CrO_4 (left) measured with Si PIN diode point detector (blue) and scintillation counter with secondary beam monochromator (brown); diffraction patterns are shifted vertically for better comparison. A close-up of the strongest reflection is shown (right).

CONCLUSION

Using a secondary beam monochromator for measurements of Fe (or Mn) containing samples with Cu $K\alpha_1$ radiation and a scintillation counter reduces the background and increases the signal-to-noise ratio but reduces the overall intensities to ¼.

The Si PIN diode point detector is able to completely cut off the fluorescence of Fe (and/or Mn) and therefore provides data with a signal-to-noise ratio up to two times higher than the scintillation counter with secondary beam monochromator. Due to the lower height divergence caused by the smaller detector size of the PIN diode compared to the scintillator crystal, the resolution is considerably better.

The slightly decreased intensity of the Si PIN diode due to the smaller detector size compared to the scintillation counter is overcompensated by the lower background and the higher quantum efficiency (~98 % vs. ~30 %).