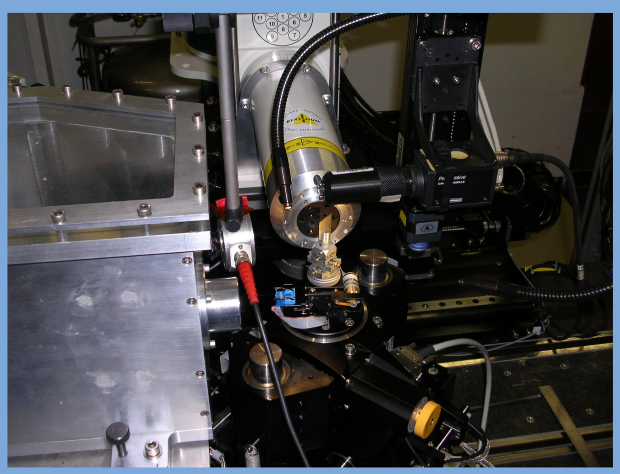
An energy dispersive X-ray detector for the ESRF hard X-ray microprobe beamline ID22

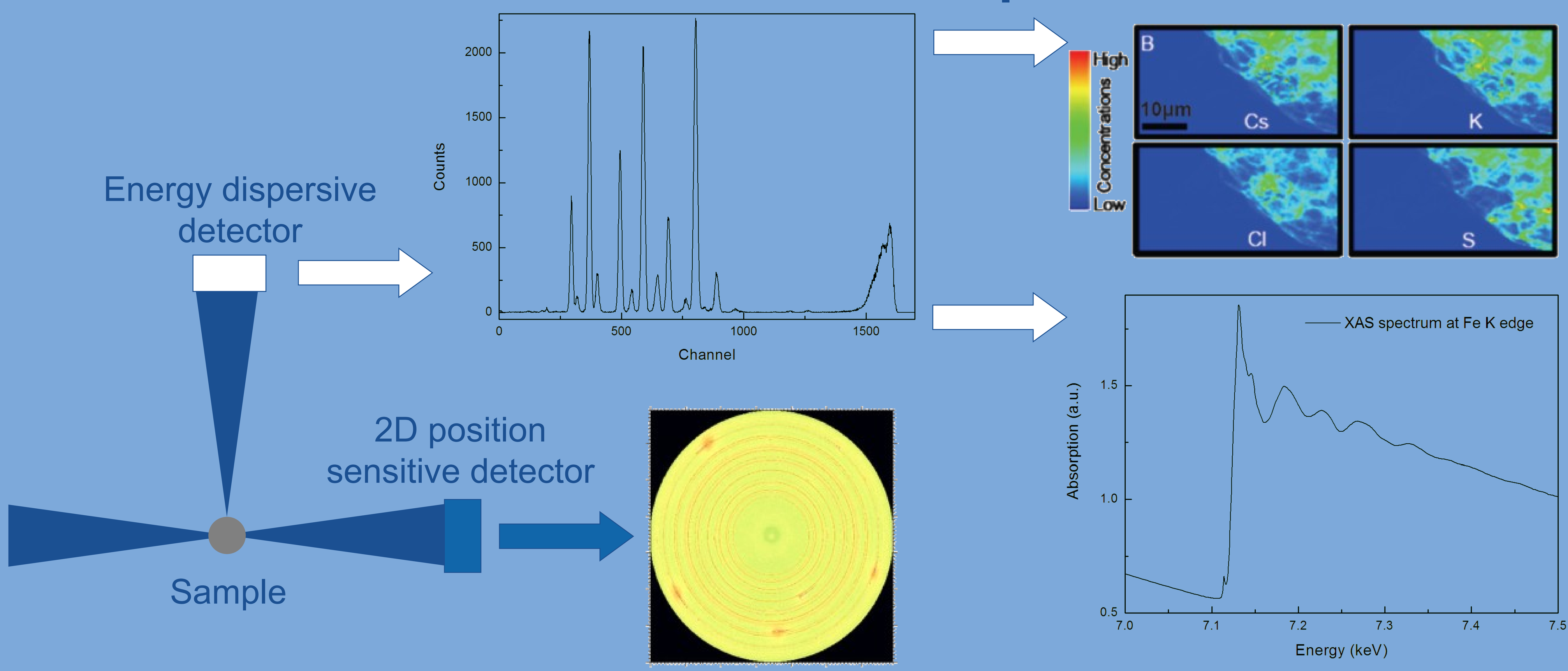
I. Kieffer (Letard), **P. Bleuet, S. Labouré, G. Martínez-Criado, J. Morse, E. Papillon, R. Tucoulou and J. Susini**
ESRF, Grenoble, France

A. Somogyi, Synchrotron Soleil, Gif-sur-Yvette, France
L. Vincze, University of Ghent, Belgium

ID22: combination of several techniques at the micron scale[1]



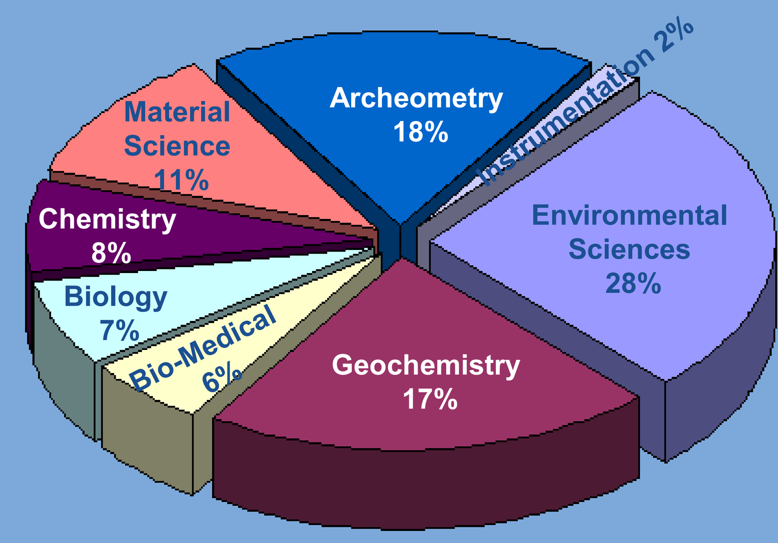
Energy range: 6-30keV
Spot size:
1st hutch: 1 x 4µm²
2nd hutch: 0.15x0.1 µm²
Photon flux:
1st hutch: 10¹²ph/s with ΔE/E=10⁻⁴ (Si(111) BW)
2nd hutch: 3x10¹¹ph/s with ΔE/E=10⁻² (pink beam + multi-layer Kirkpatrick-Baez focusing mirrors)



Micro-fluorescence → Quantification
2D mapping / 3D tomography → Co-localization

Micro-diffraction
→ Structural characterisation

Micro-XANES
→ Chemical speciation



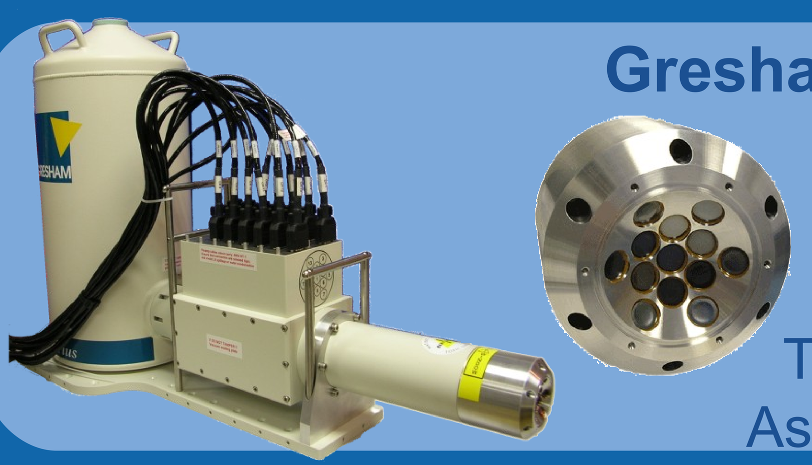
ESRF x-ray microprobe beamlines, 2002-2005: more than 210 experiments

13-element Si(Li) detector[2]

Introduction and detection requirements

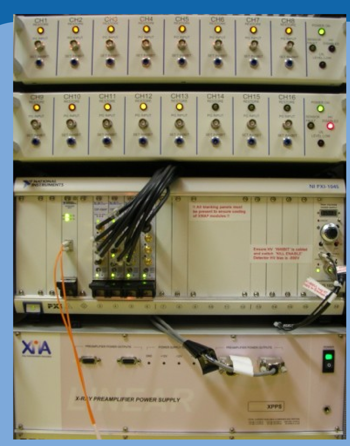
On ID22 beamline, energy dispersive X-ray detectors are used for both micro-fluorescence analysis (quantification and 2D/3D mapping) and micro-XANES. Those applications require high efficiency in the 2-30 keV energy range, high signal-to-noise ratio, good energy resolution and high throughput. To fulfill these requirements, a multi-element detector composed of Si(Li) crystals has been installed.

Gresham detector



13 x 50mm² Si(Li) crystals
Close packed focused array
No observable cross-talk
Thin DuraBeryllium windows (12µm)
Asynchronously restored preamplifiers

XIA electronics



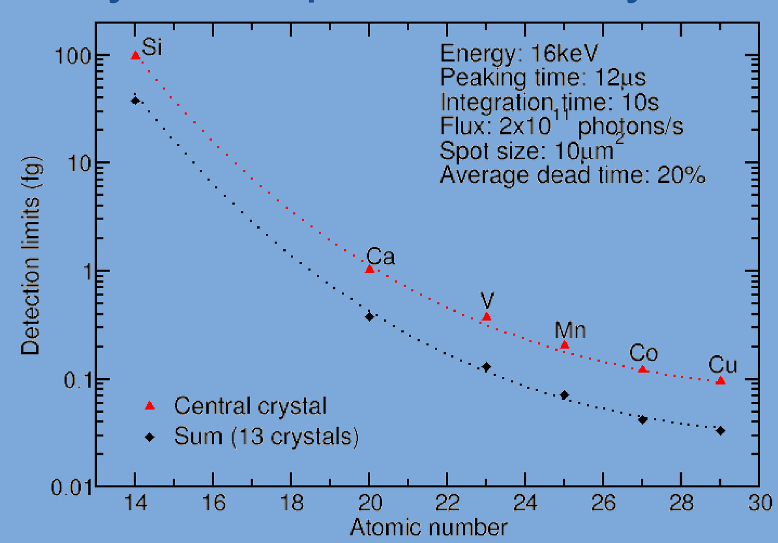
4 Digital X-ray Processor modules
Peaking time range: 0.1 to 100 µs
Maximum throughput: 1 000 000 cps/channel
External triggering – Spectrum read out: 15 ms
xManager: multi-element spectrum analysis software

Resolution

Peaking time	Count rate	FWHM (Mn K_α)
1µs	20kcps	**278eV**
12µs	1kcps	**148eV**

Detection limits

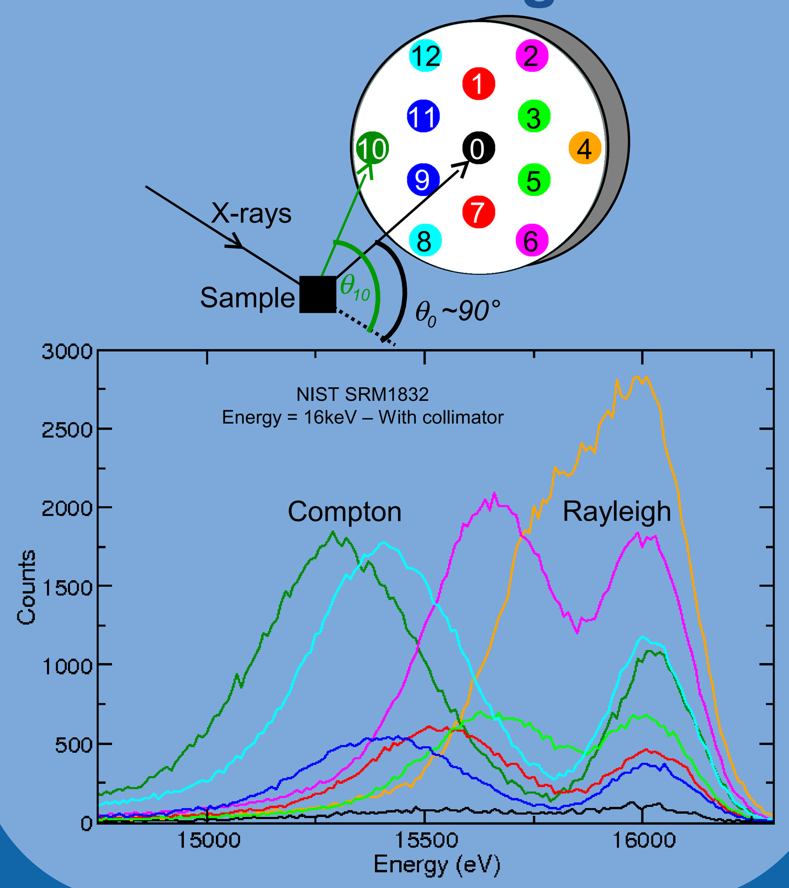
Detection limits are lowered by 3 compared to 1 crystal



SRM1832: Ca: 11.6% - V: 2.4% - Mn: 2.5% - Co: 0.61% - Cu: 1.6%
Spectra fitted with **PyMca**, A. Solé, ESRF

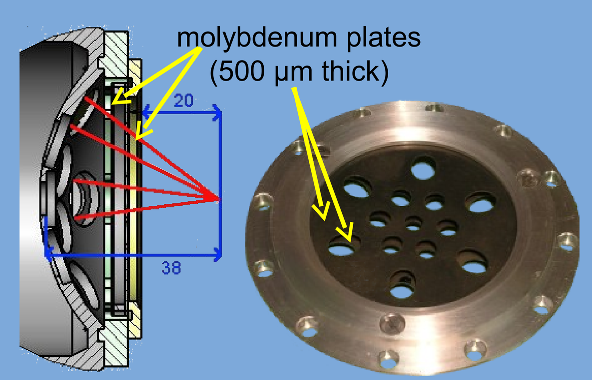
$$C_{Limit} = 3\frac{\sqrt{Bkgd}}{Signal}C$$

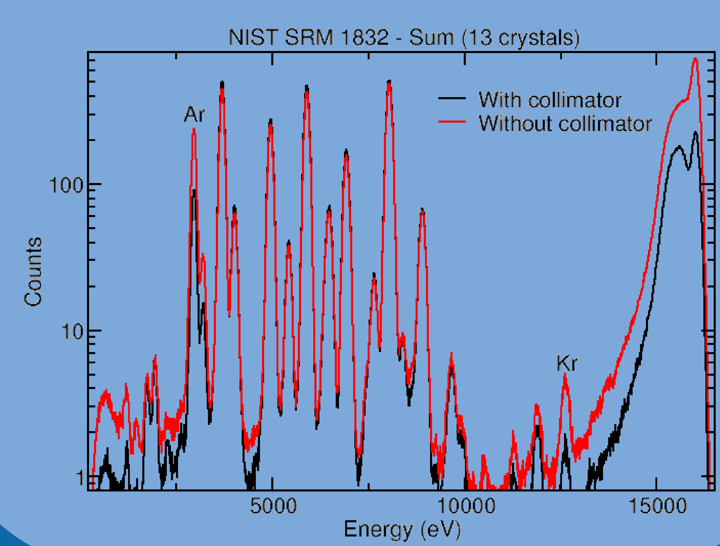
Scattering



Collimation to reduce scattering effects

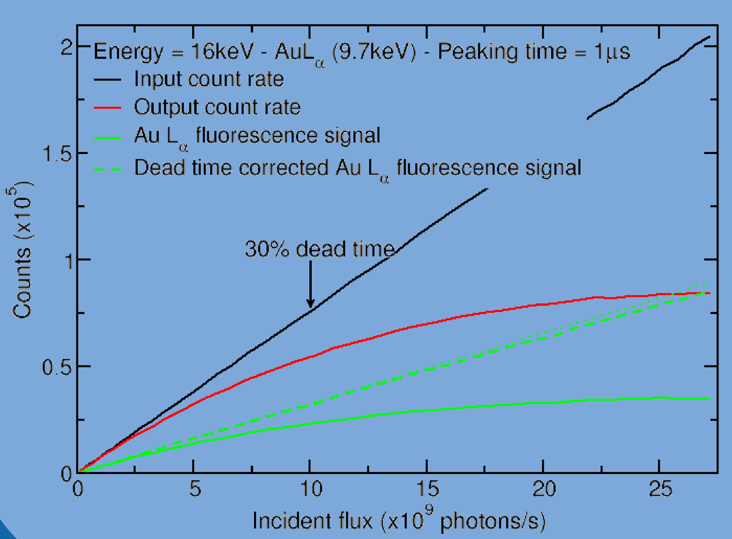
Air contribution is reduced
Scattering is reduced by 4
Dead time decreases from 25% down to 15%





Linearity

With dead time correction: up to 80 kcps/crystal



Integration times

For samples with low scattering, integration time is reduced by about 10. When scattering is high, it can be necessary to reduce the incident flux to maintain all the crystals in their linear range. In that case, the gain is less than 10.

Conclusion and perspectives

On a multipurpose beamline such as ID22, defining a detector optimized for all the developed applications is difficult. The optimization has to be a trade-off among linearity up to high count rates, resolution, and geometric considerations related to the scattering contributions. This does not lead necessarily to solid angle maximization. Silicon Drift Diodes (SDD) currently present resolution comparable to Si(Li) detectors, high count rate and easier mechanical integration. They constitute a promising possibility if several SDD can be located at different places according to each experiment requirements. Simulation codes, such as Monte Carlo based calculations,[3] let determine the scattering contributions of a given sample at a given energy and will help the optimization of detector technology and geometry for the NINA beamline (evolution of ID22 beamline in the frame of the ESRF upgrade programme).

[1] A. Somogyi et al., *ID22: a multitechnique hard X-ray microprobe beamline at the ESRF*, **Journal of Synchrotron Radiation**, vol. 12, p. 208 (2005)
[2] I. Letard et al., *Multielement Si(Li) detector for the hard x-ray microprobe at ID22 (ESRF)*, **Review of Scientific Instruments**, vol. 77, p. 063705 (2006)
[3] L. Vincze et al., *A general Monte Carlo simulation of ED-XRF spectrometers*, **Spectrochimca Acta B**, vol. 50, p. 127 (1995)