Description and practical details of mad26 detector

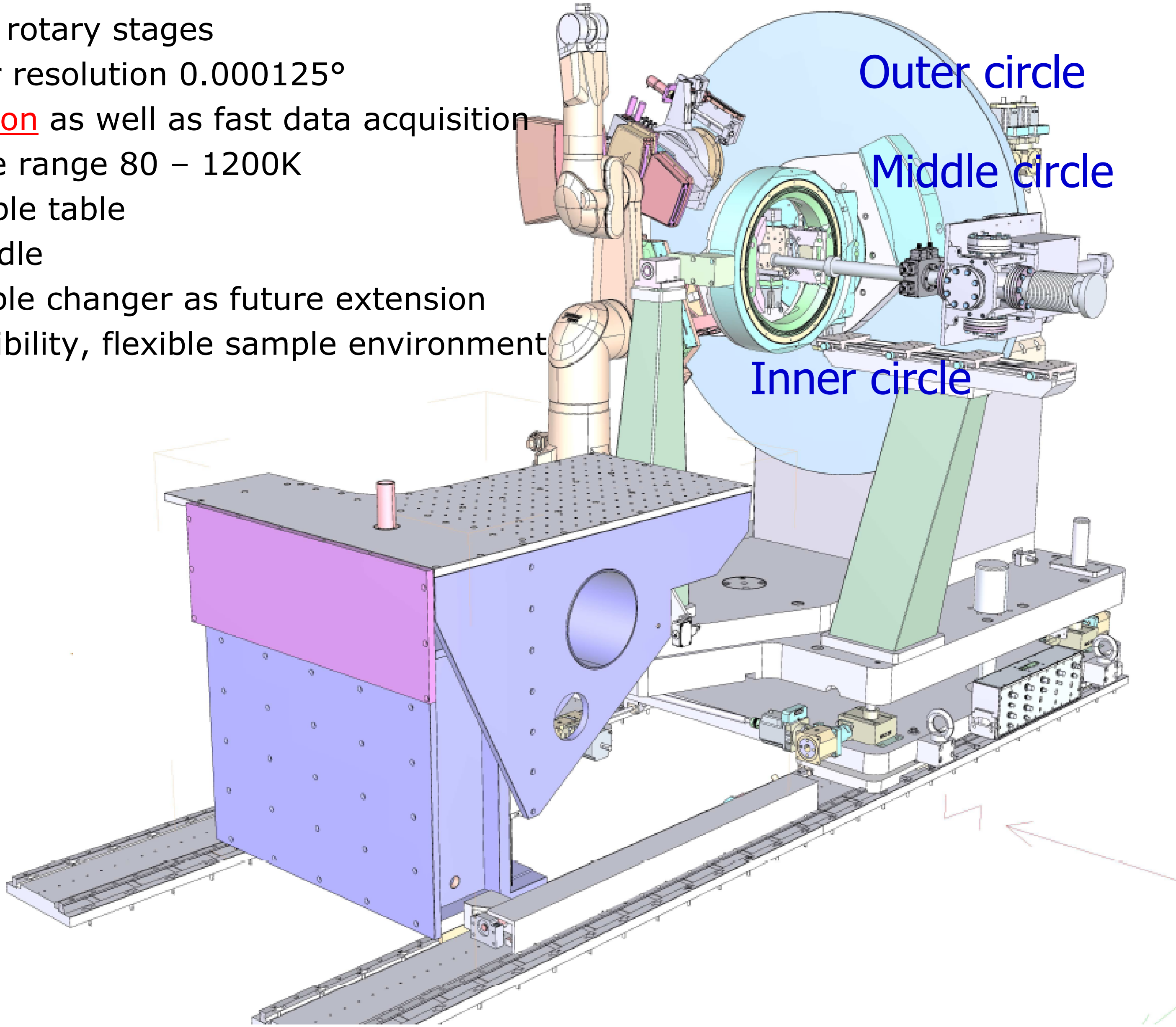
(high resolution detector)

powderxrd2013

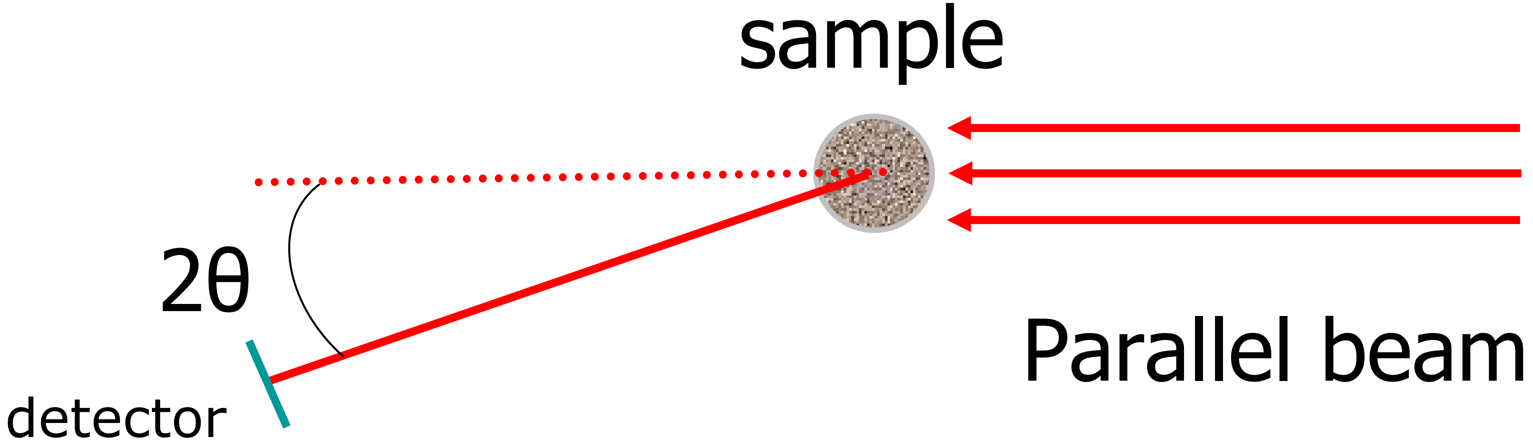
www.powderxrd2013.cells.es

MSPD, Powder Diffraction station

- 3 concentric rotary stages
- high angular resolution 0.000125°
- high resolution as well as fast data acquisition
- Temperature range 80 – 1200K
- second sample table
- Eulerian Cradle
- robotic sample changer as future extension
- good accessibility, flexible sample environment



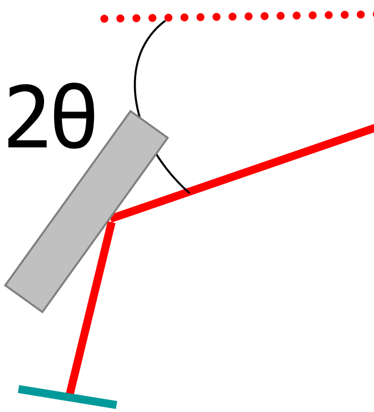
Power Diffraction

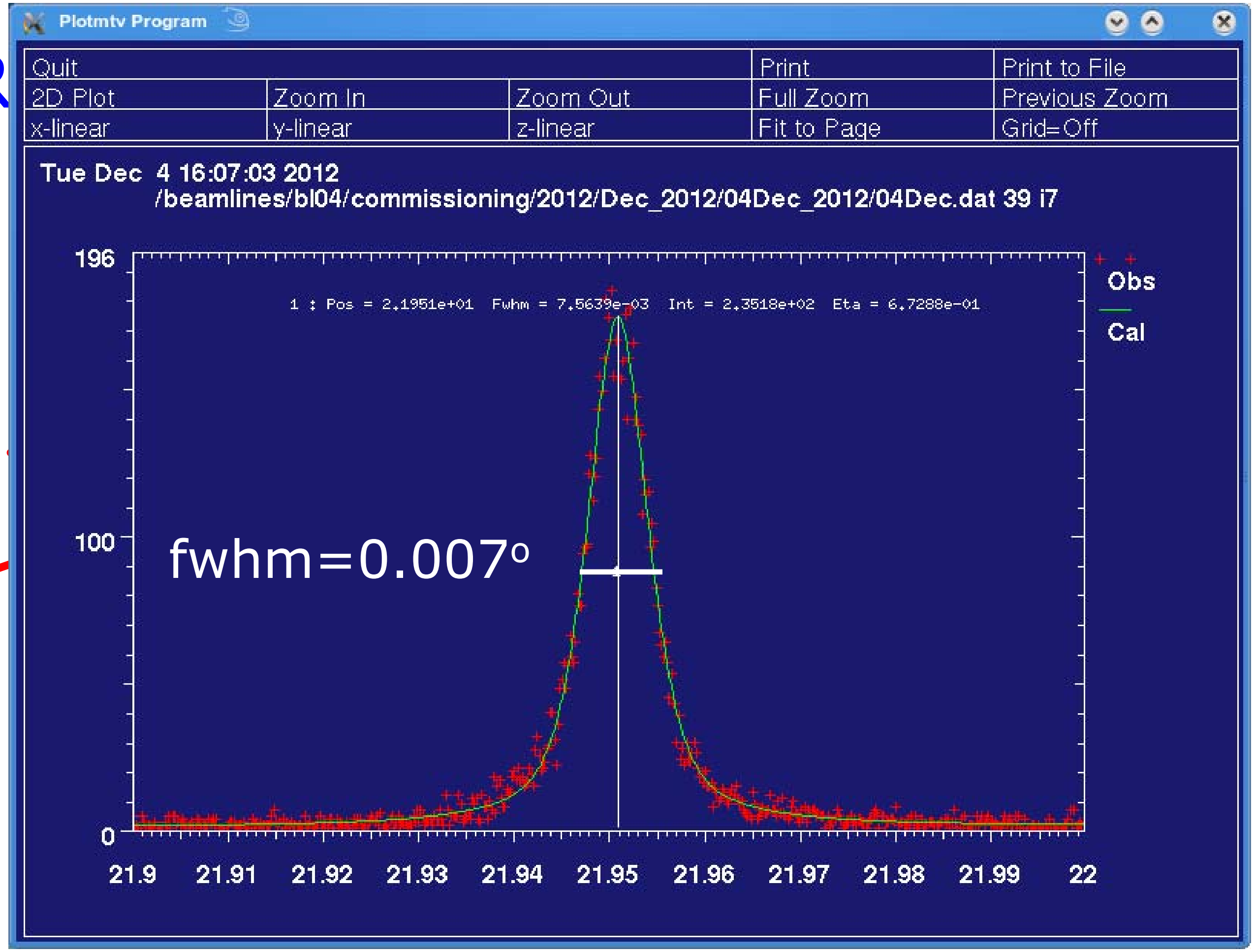


A detector system at a given 2θ will detect

- The beam defined by the slits (if any)
- And other effects: fluorescence, for example, depending on the energy window of the detector

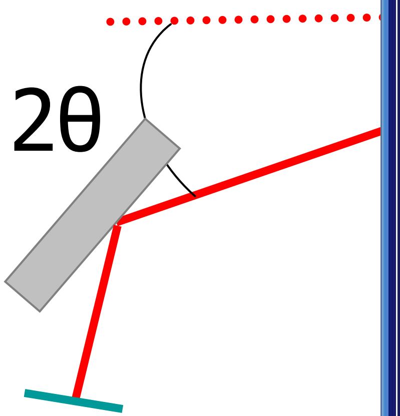
High R

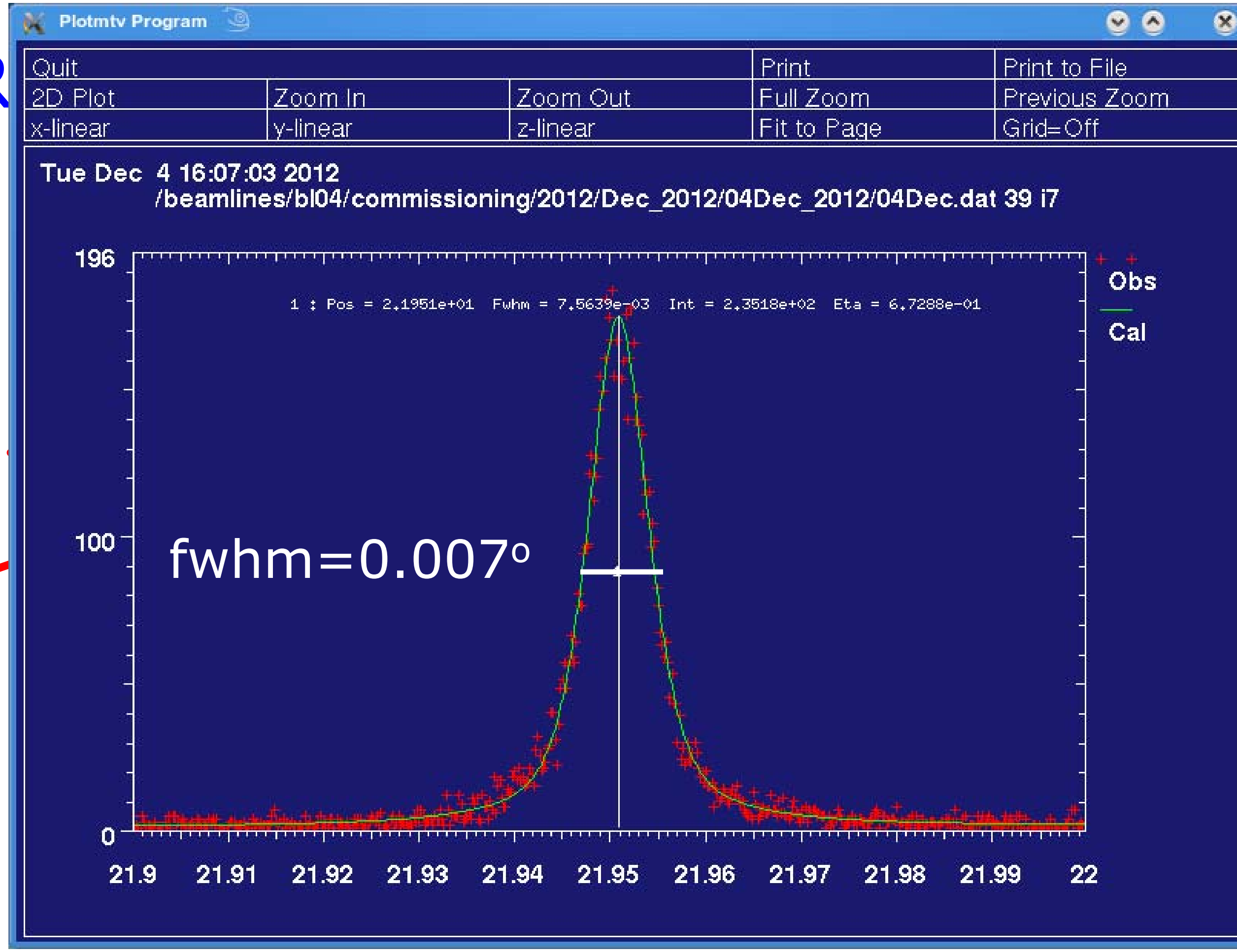




- Angular resolution system given by the angular acceptance of the crystal analyzer ~0.005 degrees for Si111.
- Note that the crystal analyzer is not sensitive to misaligments sample to center of difffractometer and other aberrations.

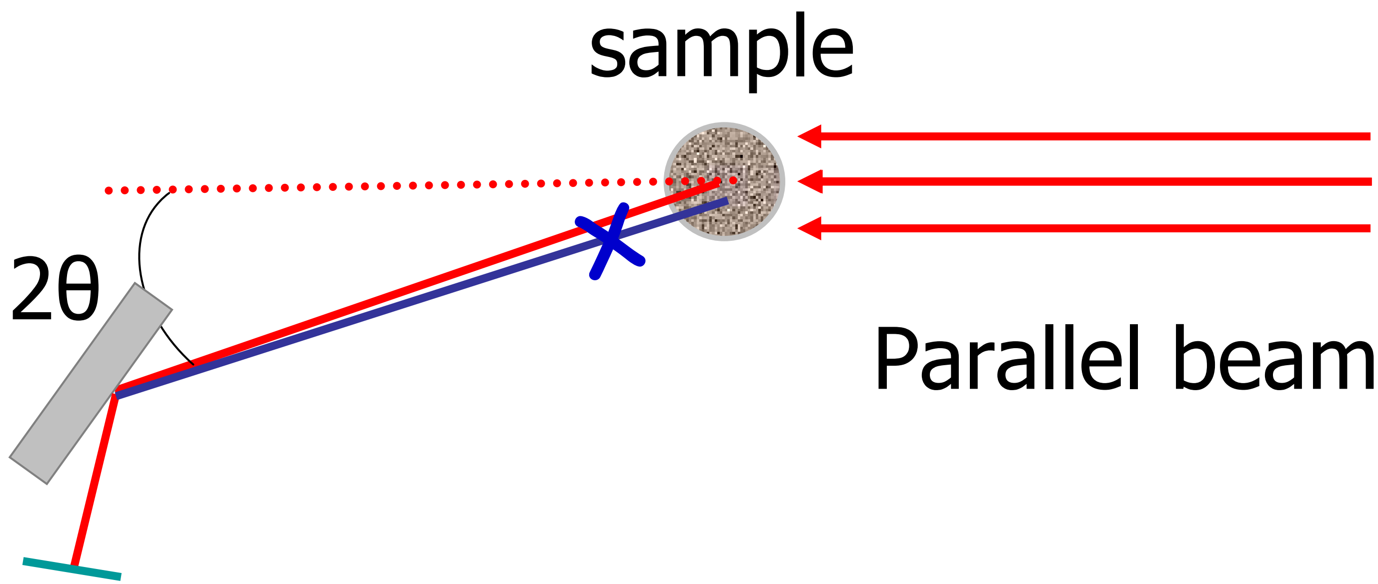
High R





- Angular resolution system given by the angular acceptance of the crystal analyzer ~0.005 degrees for Si111.
- Note that the crystal analyzer is not sensitive to misaligments sample to center of difffractometer and other aberrations.

High resolution power diffraction

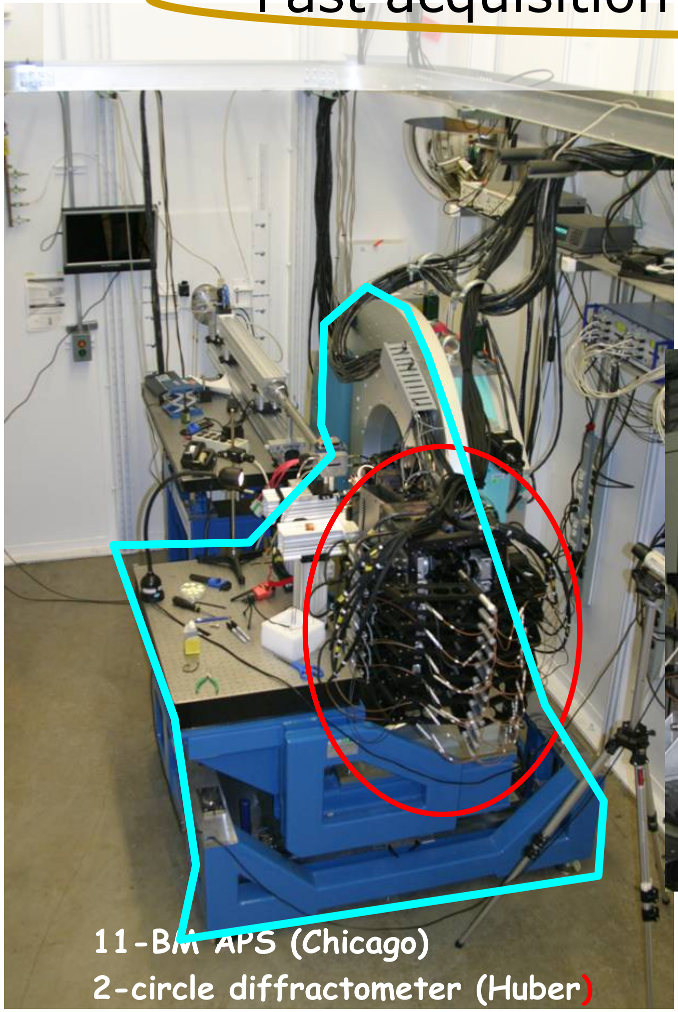


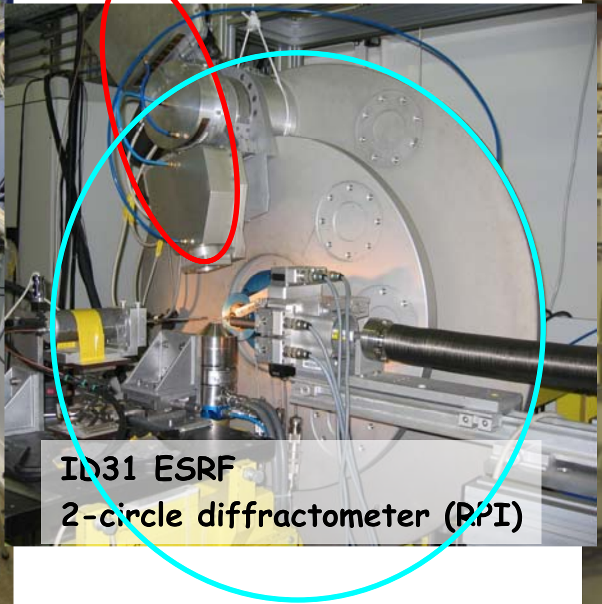
- angular resolution system given by the angular acceptance of the crystal analyzer ~0.005 degrees for Si111.
- Note that the crystal analyser is not sensitive to misaligment sample to center of difffractometer and other aberrations.

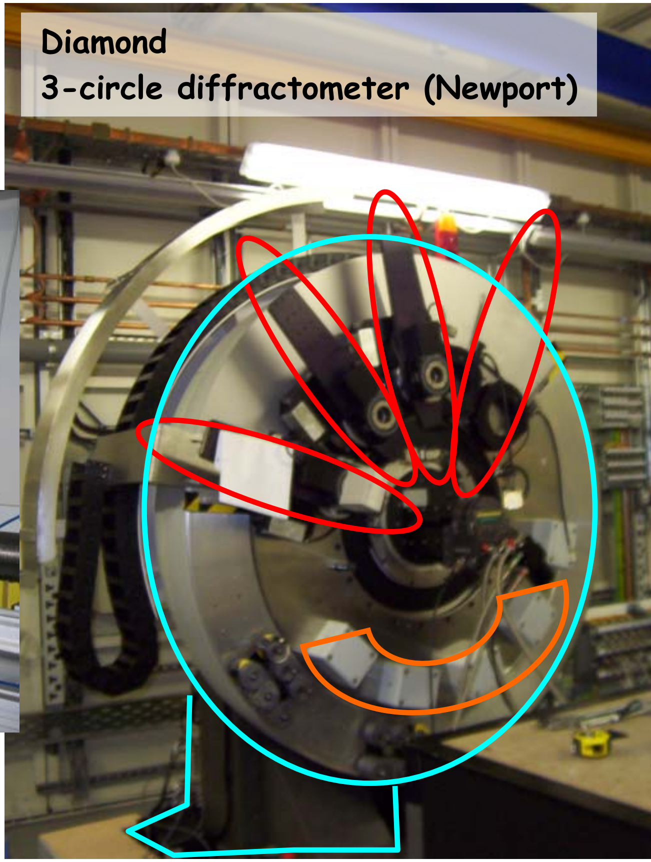
This system provides high resolution but not enough statistics just one crystal analyser -> multicrystal analyzer systems

Powder diffraction endstations

-Diffractometer
-Detectors
High resolution detector -> Multicrystal Analyzer
Fast acquisition detector (low resolution data)

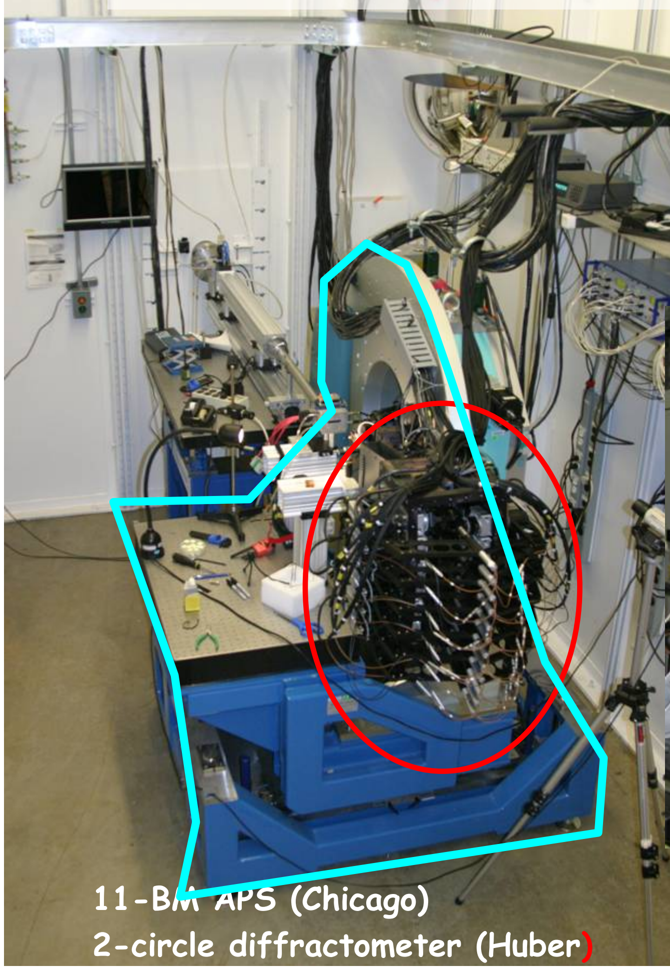


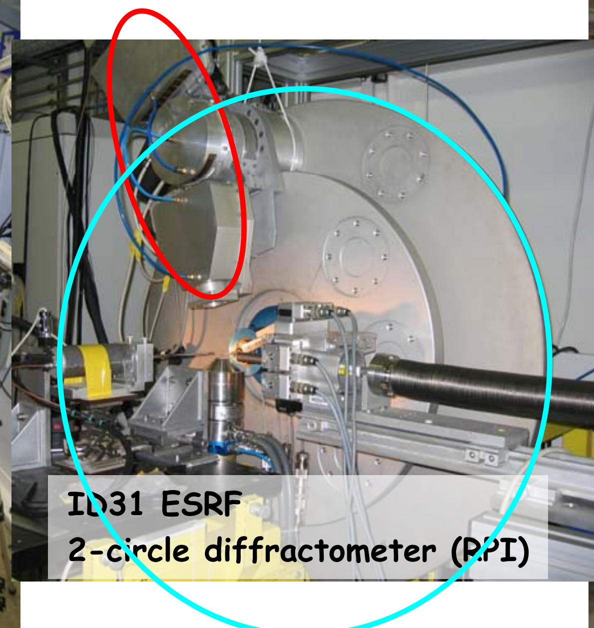


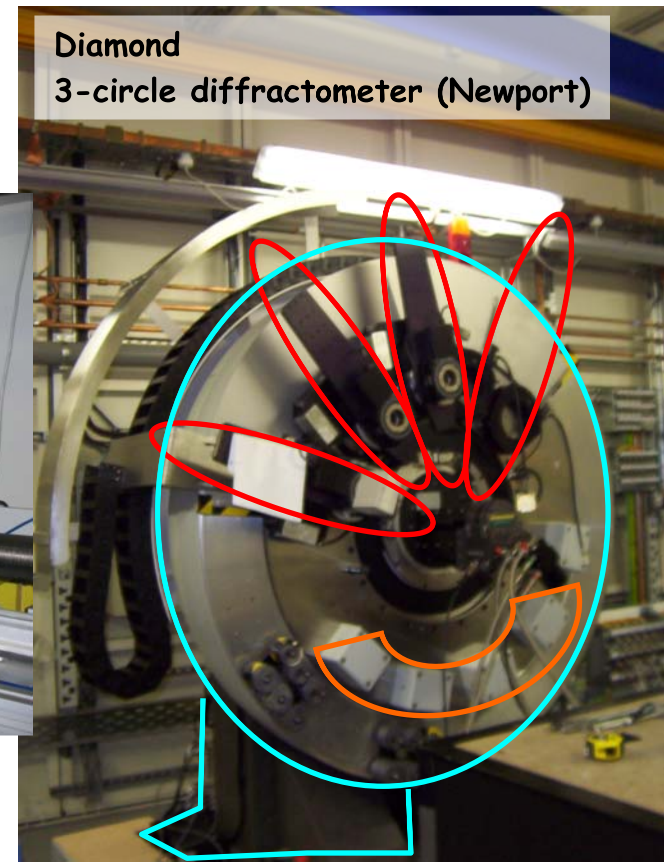


Multicrystal Analyzer designs

- In house designs adapted to the energy range of the beamline
- They should be integrated into the endstation
- Different number of crystals: 12/5/9/45 … etc.
- Diferent mechanical realitations -> implications on the operation







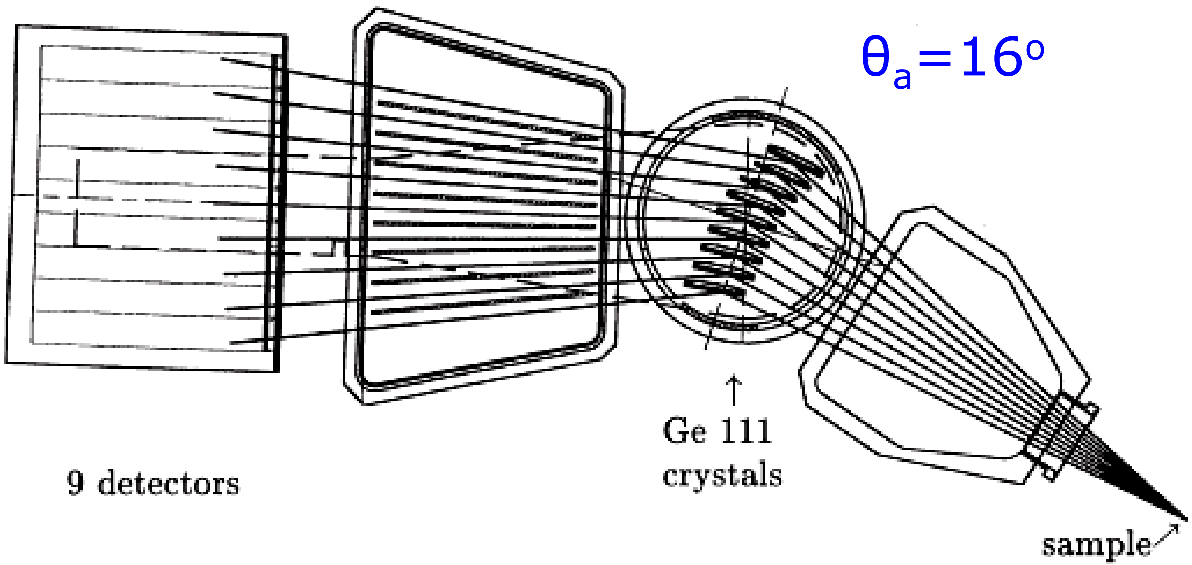
Multicrystal Analyser Detector

MAD is the main detector for high resolution powder diffraction,

The crystal analyzer set-up is the best choice

E= 6 keV

θ_a=16^o



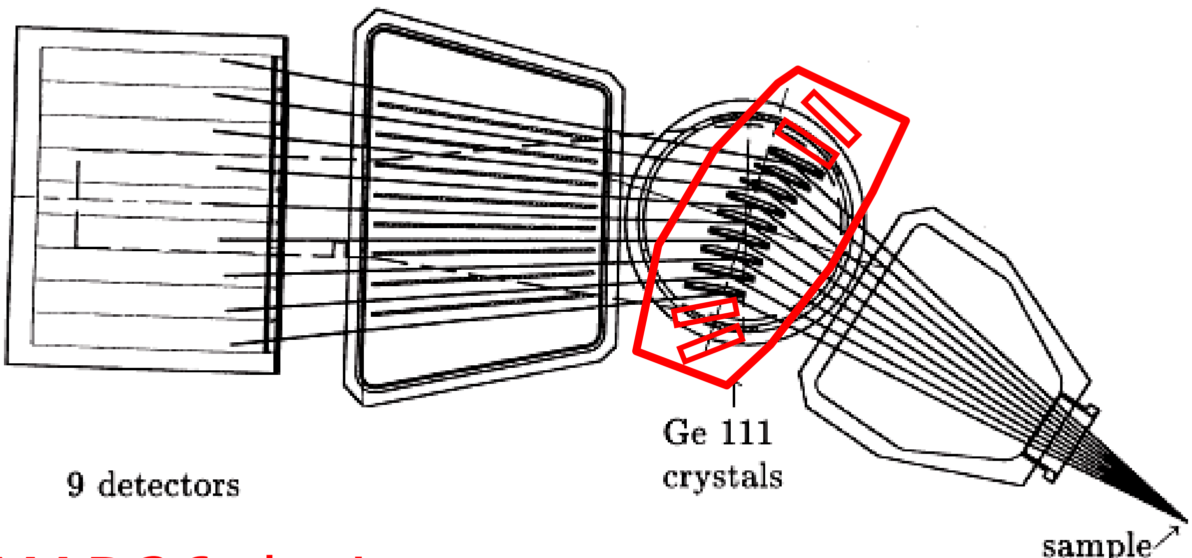
Original design by Hodeau et al. Proc. SPIE (1998)

First installed in BM16, ESRF

Multicrystal Analyser Detector MAD26

- MAD is main detector
- Basic layout:

13 channels
Si 111 or Si220 single bounce crystals
Angular separation 1.5deg
Radius (Sample – Crystal): 550mm



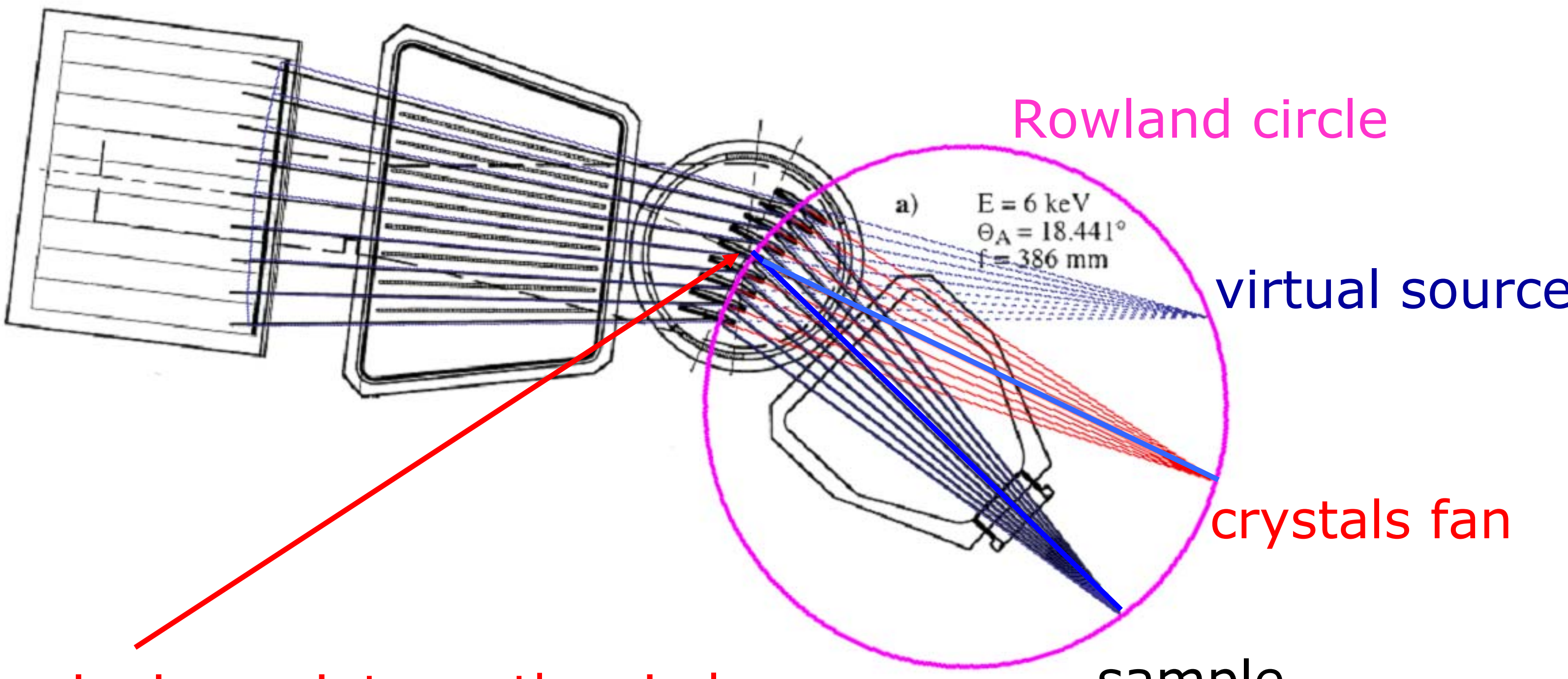
MAD26 design:

-Optimization of the position of the crystals

-Two sets of analyser crystals Si111 & Si220

Multicrystal Analyser Detector MAD26

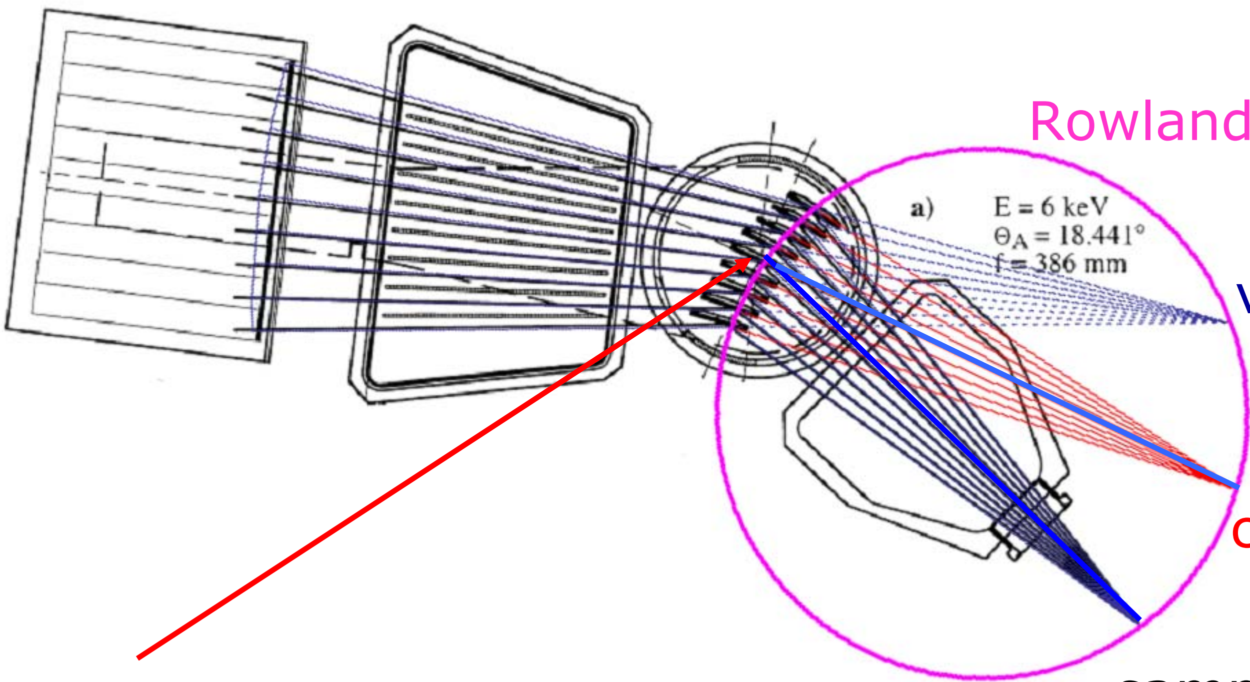
We have developed a method that describes the MADs in terms of the Rowland circles and also defines the independent parameters.



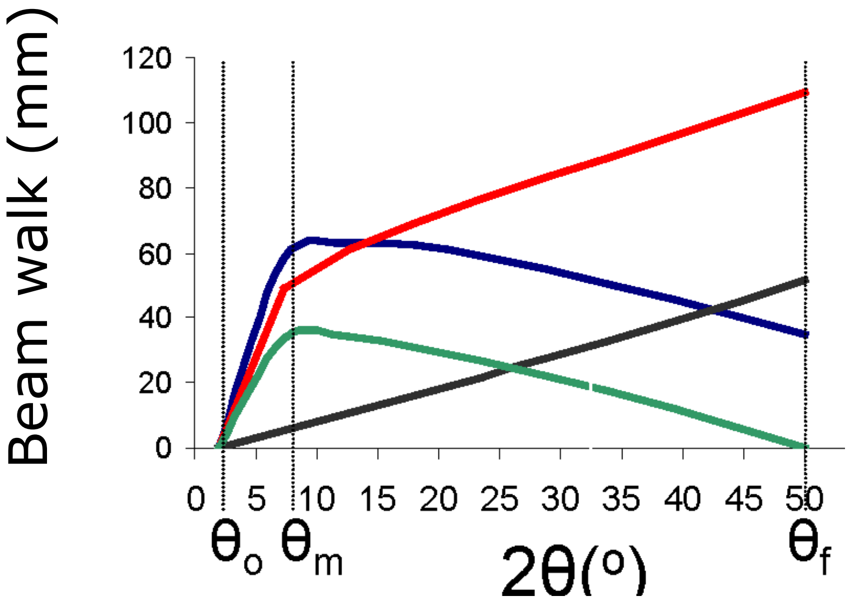
Multicrystal Analyser Detector MAD26

We have developed a method that describes the MADs in terms of the Rowland circles and also defines the independent parameters.

Angular offset between crystals, SO and SA



Mad26 design



MAD26 design:

Optimization of the crystals position for a given energy range (or θ analyser crystal)

Minimization of beam walk by imposing that the impinging points at Emin and Emax is the same.

OA_{opt}

OA=SO

OA > SO

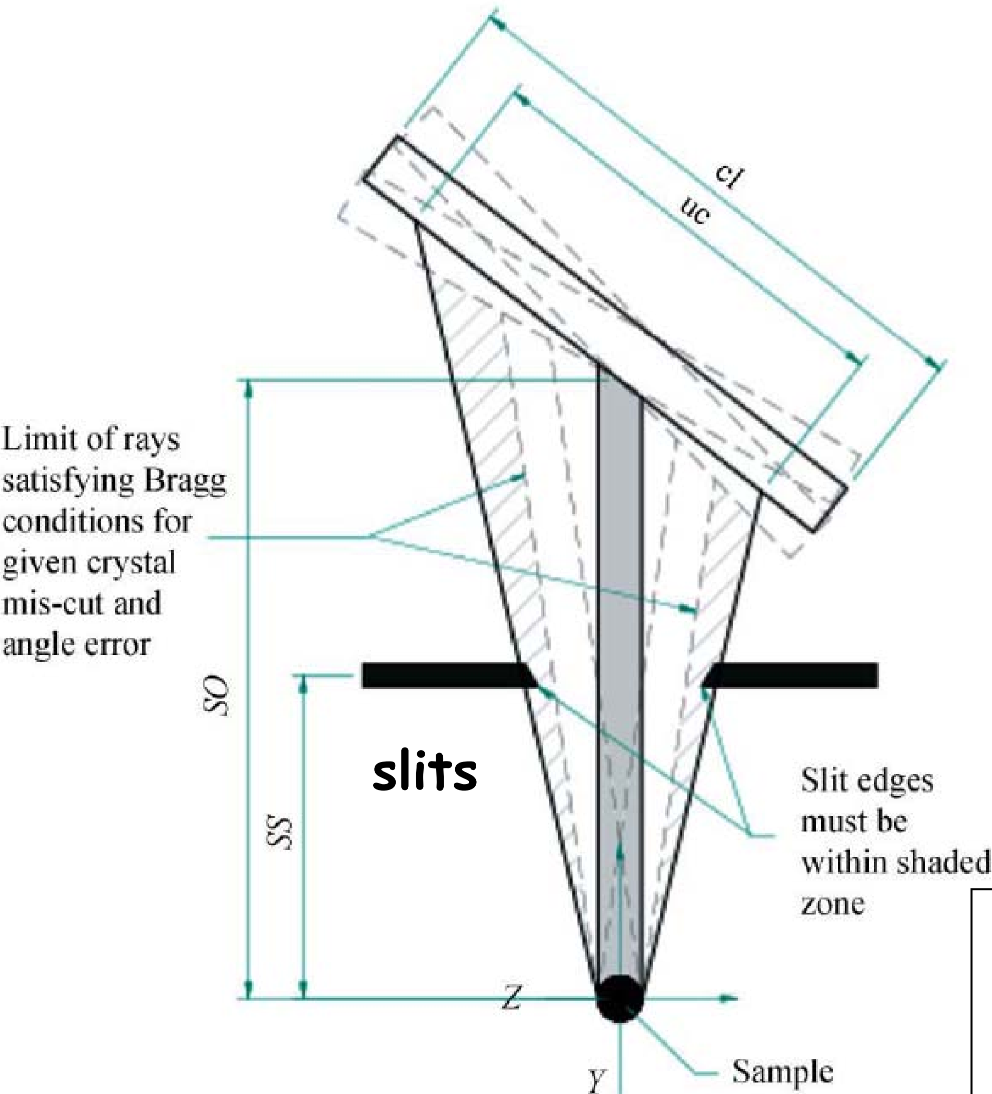
OA < SO

$$OA_{opt} = SO \frac{\cos\left(\frac{1}{2}\left(\theta_o + \theta_f\right)\right)}{\cos\left(\frac{1}{2}\left(\theta_o - \theta_f\right)\right)}$$

OA: distance crystal fan focus to center of the crystal

SO: sample to crystal stage center

MAD26 design realization



Error budget approach

(normally used in design of accelerators)

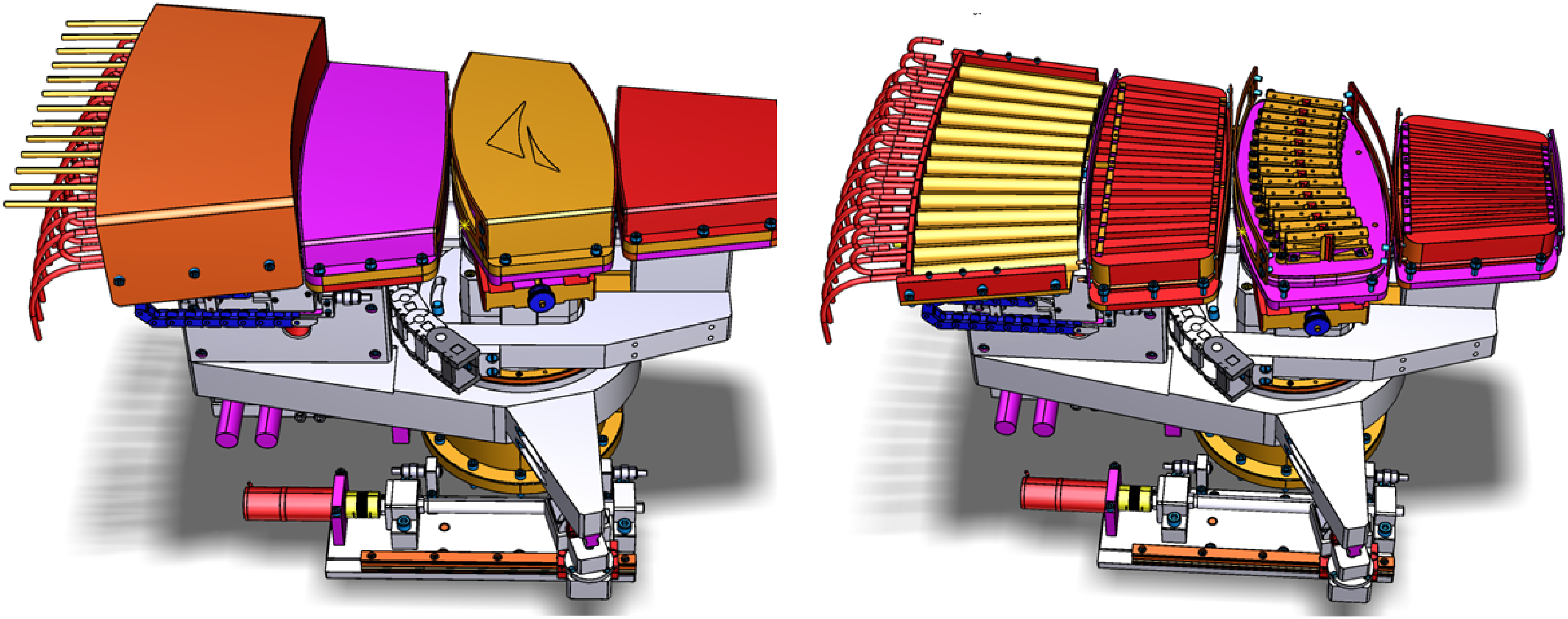
Breaking down the different sources and effects of errors to determine the manufacturing and assembly requirements of the system.

- slits should not touch the beam
- slits+crystal must block the beam

**Shaded zone is the "budget" for positioning the slits*

**Portion of crystal length is the "error budget" for crystal positioning*

3d model of mad26, the multicrystal analyzer

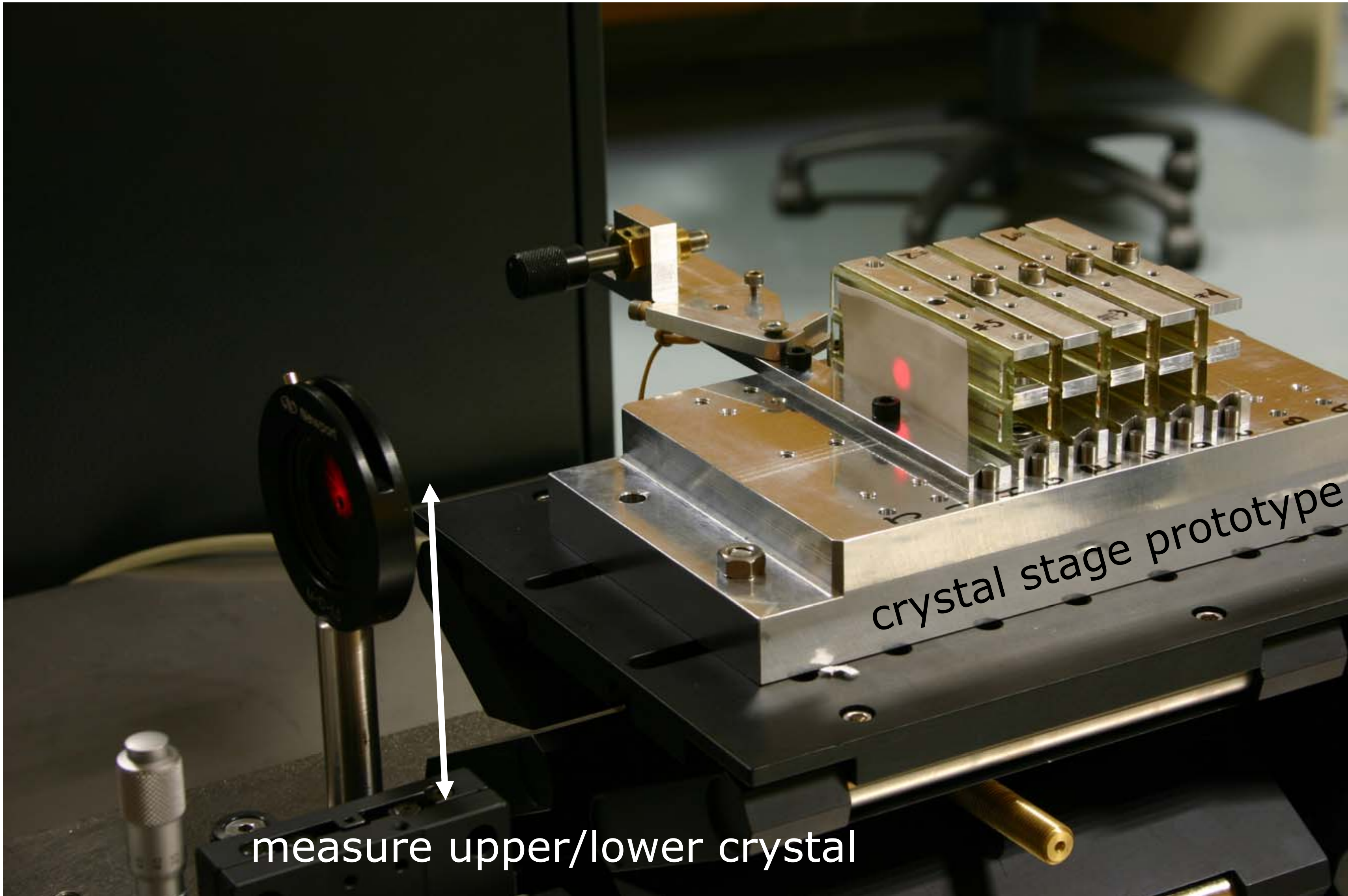


I. Peral, J. McKinlay, M. Knapp and S. Ferrer
J. Synchrotron radiation (2011) 18 , 1-9

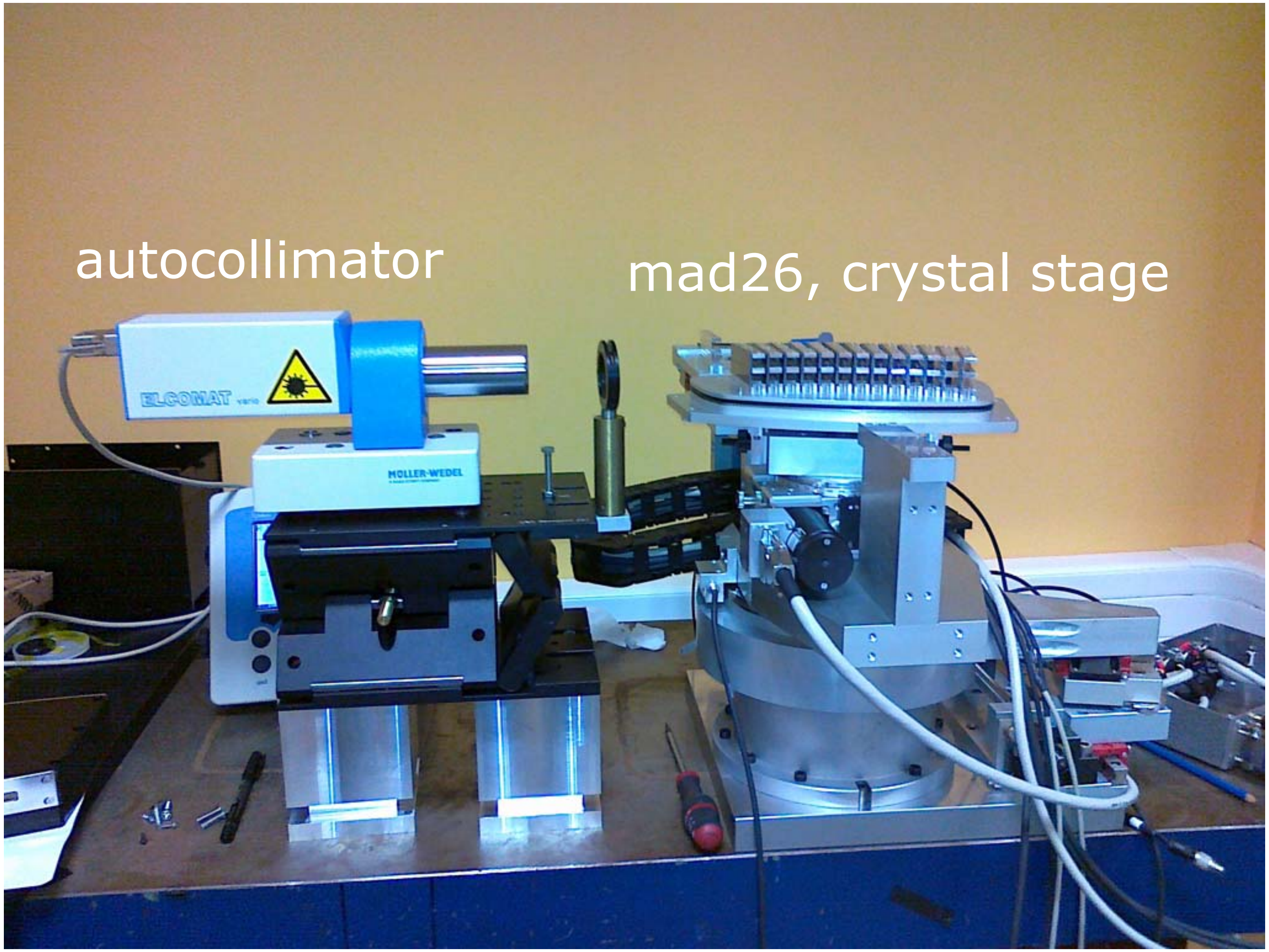
Alignment of the 26 crystals

Alignment with a prototype, crystals (pieces of glass)

Crystal-Crystal (same type) alignment the crystals, +/-0.023 degrees



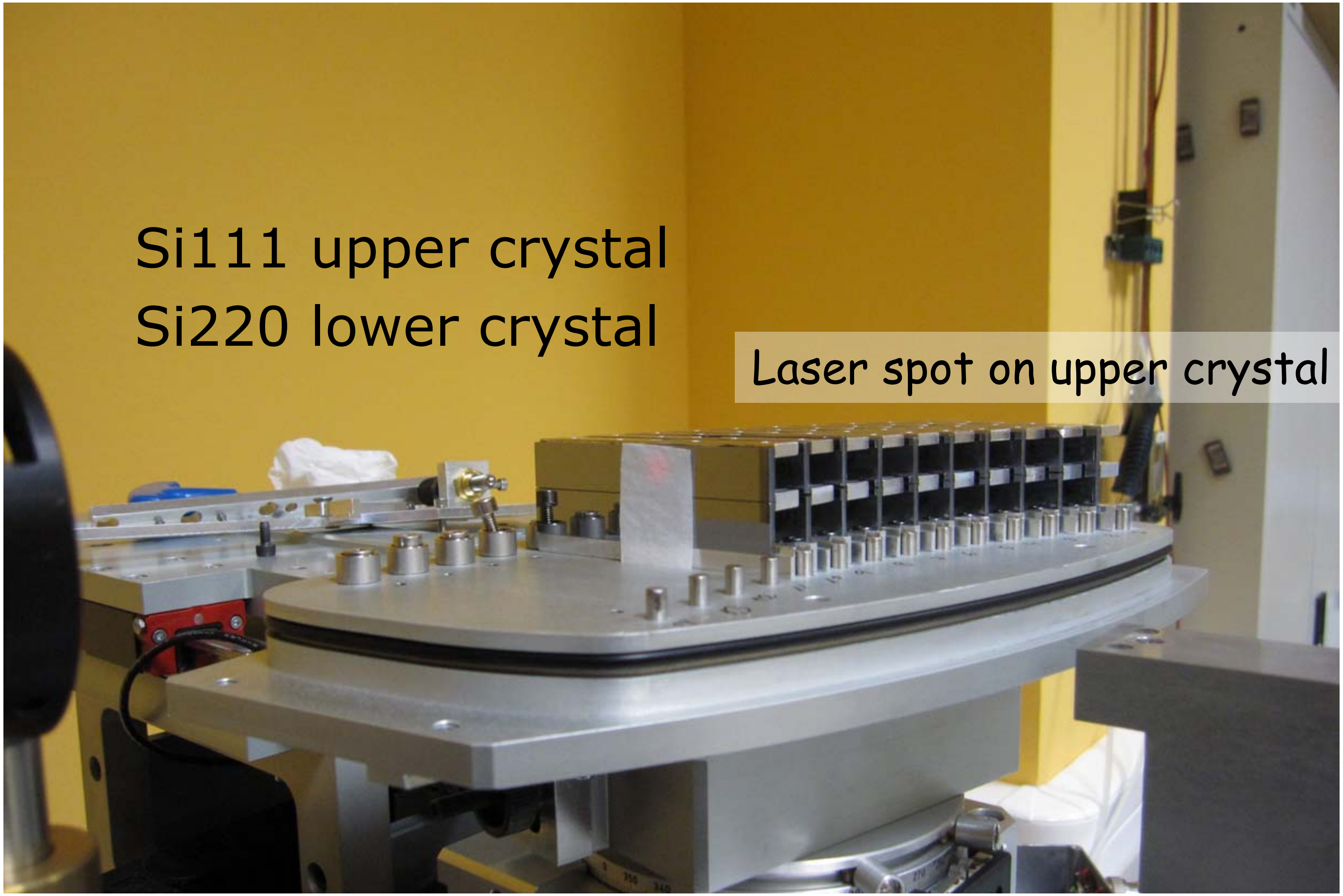
Alignment of the 26 crystals



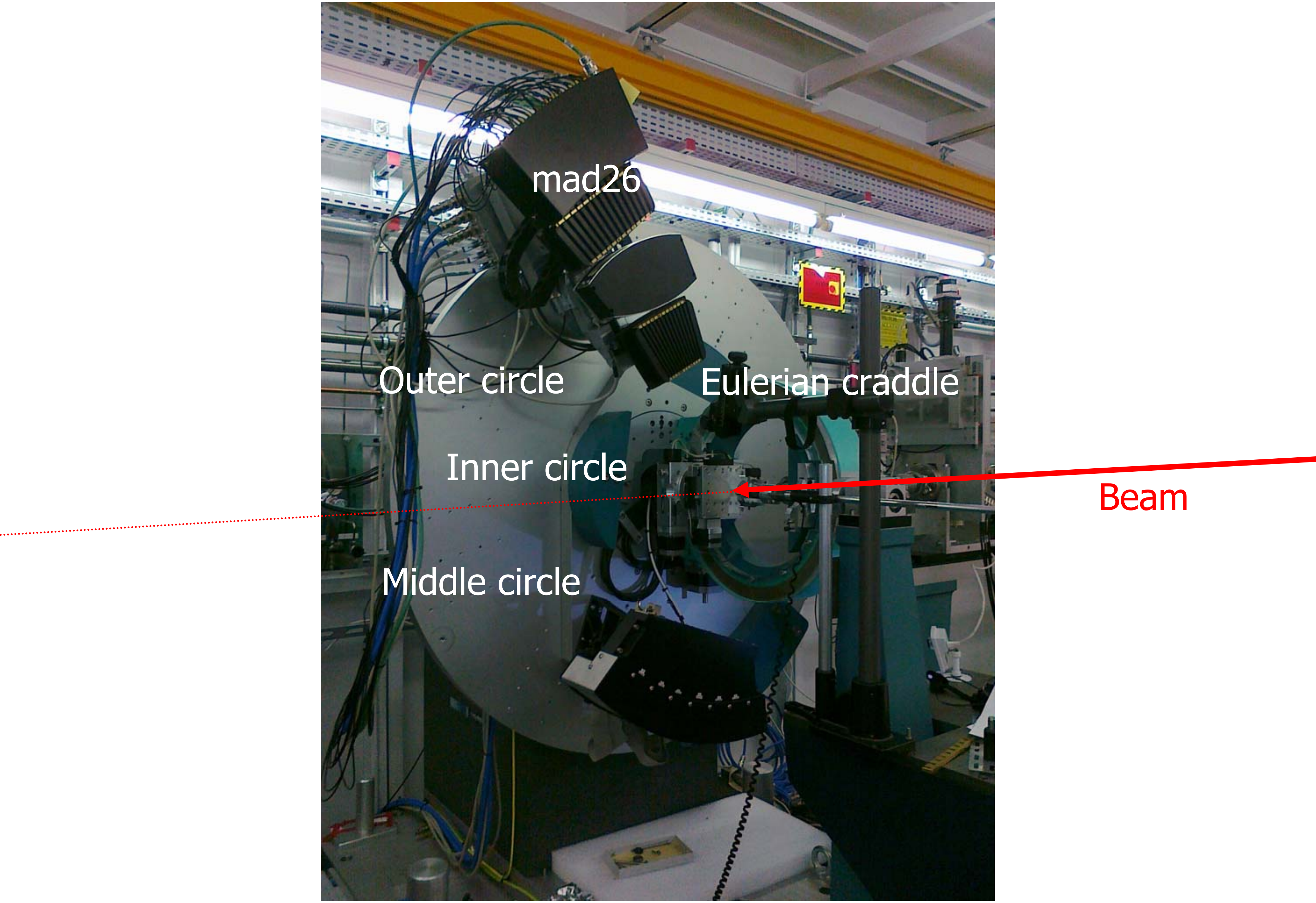
All set-up in the same table

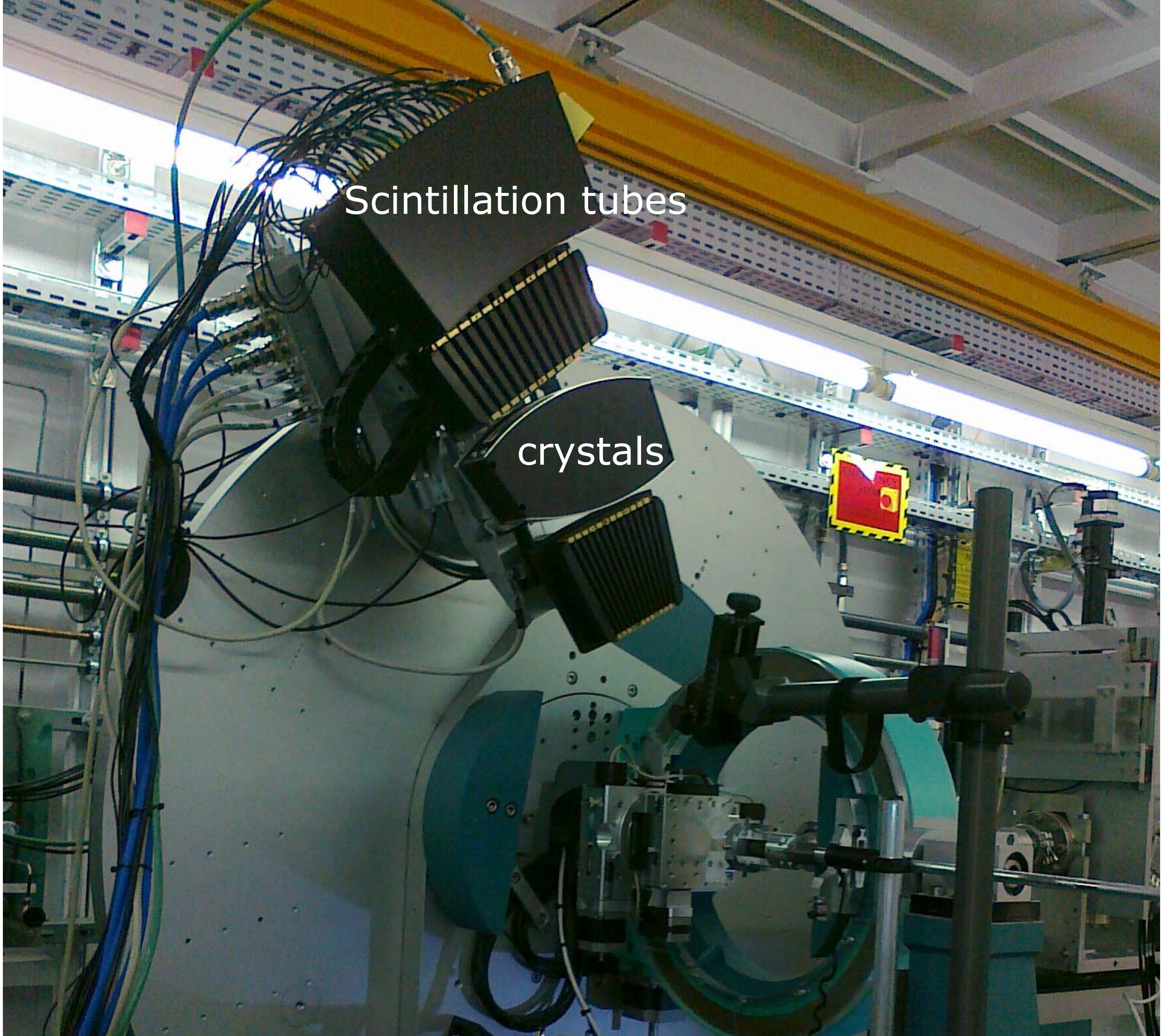
Stability better than 0.0005degrees

Alignment of the 26 crystals

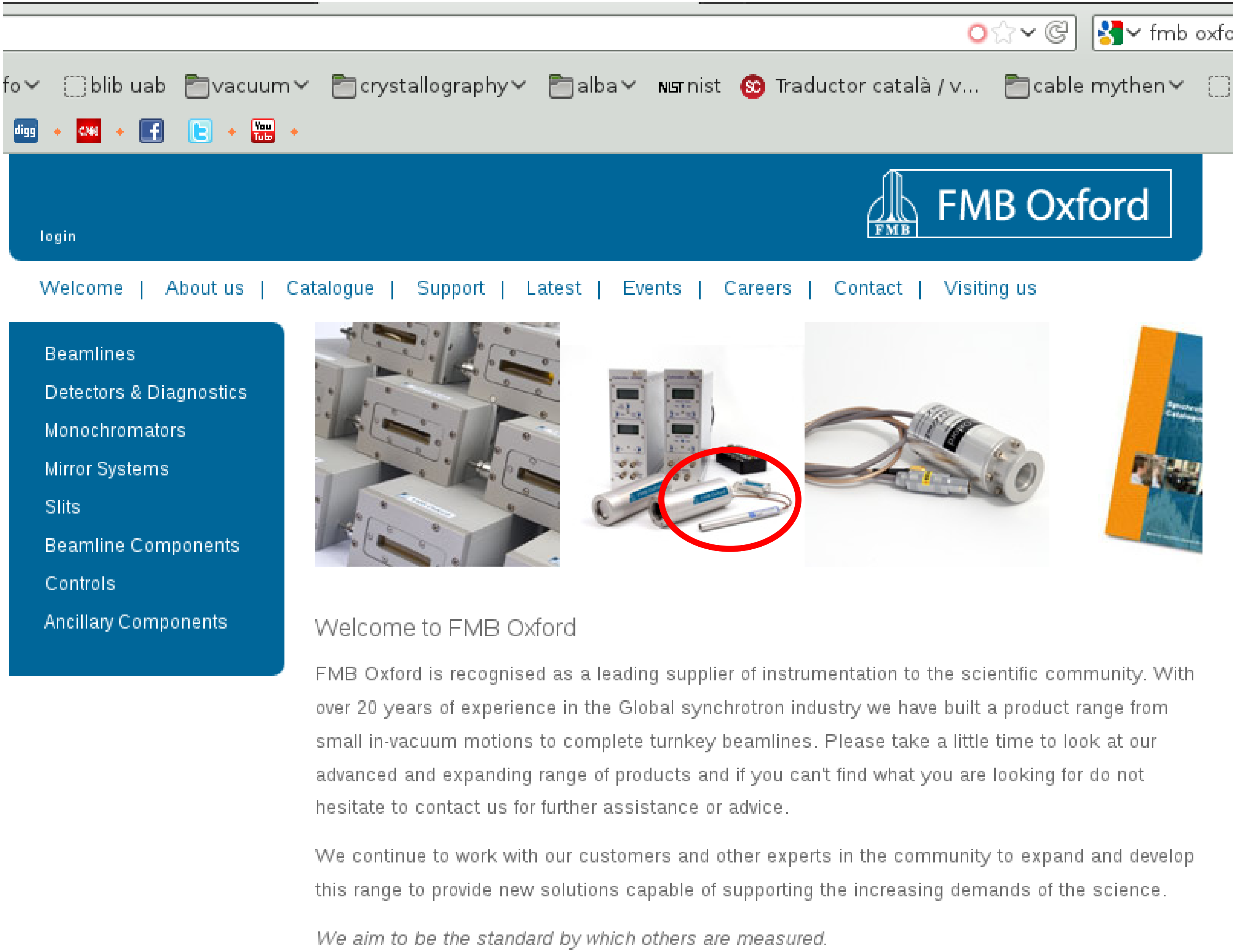


... but the beam gave us the final alignment!


mad26
Outer circle
Eulerian craddle
Inner circle
Middle circle
Beam


Scintillation tubes
crystals

Scintillation detectors and electronics


fo blib uab vacuum crystallography alba nist Traductor català / v... cable mythen

fmb oxfo

login

FMB Oxford

Welcome | About us | Catalogue | Support | Latest | Events | Careers | Contact | Visiting us

Beamlines
Detectors & Diagnostics
Monochromators
Mirror Systems
Slits
Beamline Components
Controls
Ancillary Components

Welcome to FMB Oxford

FMB Oxford is recognised as a leading supplier of instrumentation to the scientific community. With over 20 years of experience in the Global synchrotron industry we have built a product range from small in-vacuum motions to complete turnkey beamlines. Please take a little time to look at our advanced and expanding range of products and if you can't find what you are looking for do not hesitate to contact us for further assistance or advice.

We continue to work with our customers and other experts in the community to expand and develop this range to provide new solutions capable of supporting the increasing demands of the science.

We aim to be the standard by which others are measured.

Scintillation detectors and electronics

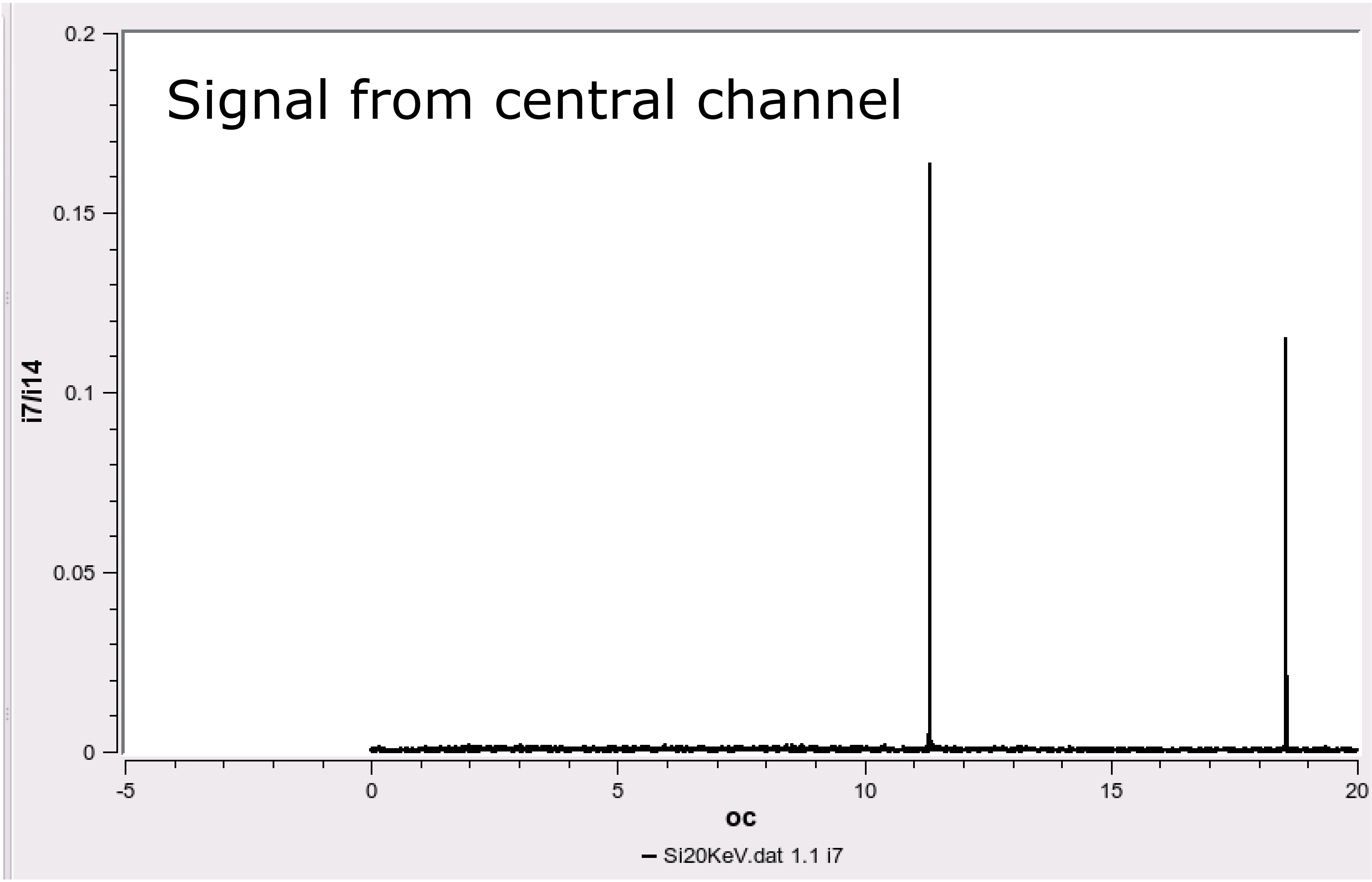
In parallel the design of the type of detectors and electronics to get the signal from the 13 channels.

Specifications: Mcps and energy range=8-50 KeV

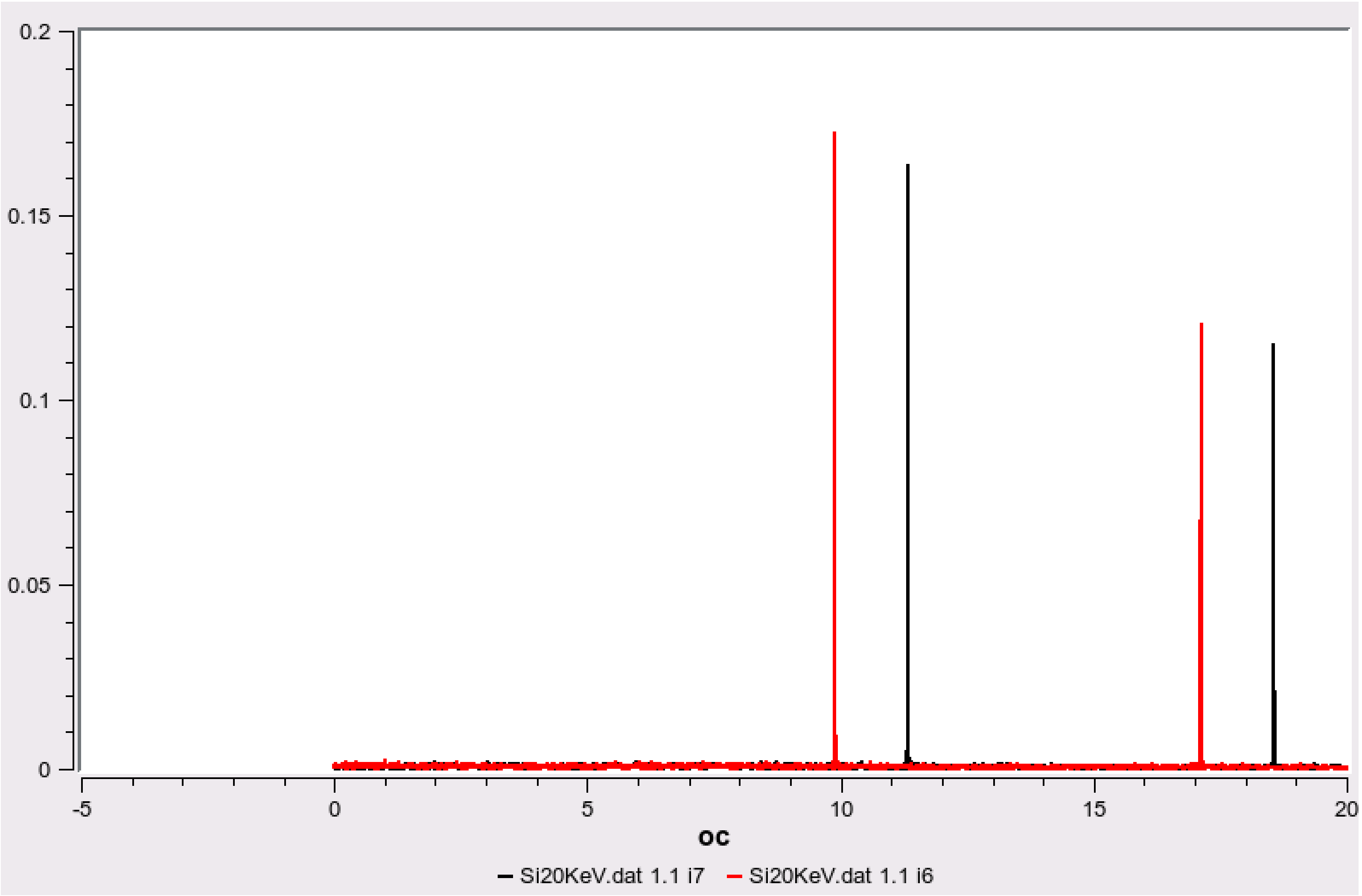
Scintillators by scionix (same as in SOLEIL and in Diamond).

- Pre-amplifier designed and built in house (electronics group)
- We tested the design with radiation sources to prove the design
- The final test: The esrf borrowed us a detector so we could compare our electronics with the already working electronics at the esrf

How to combine the signals from the 13 channels?

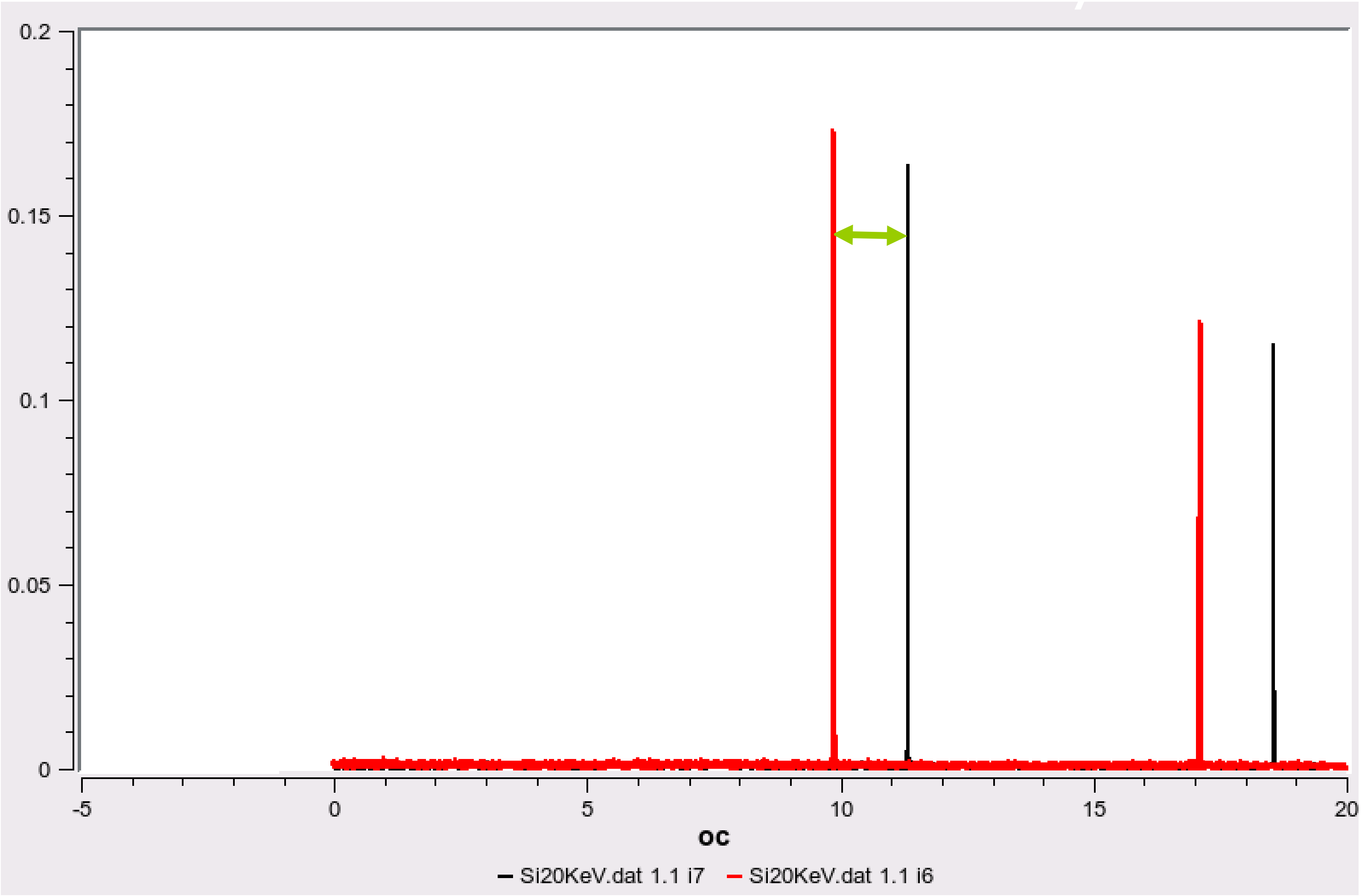


Combine signals from neighbouring channels:

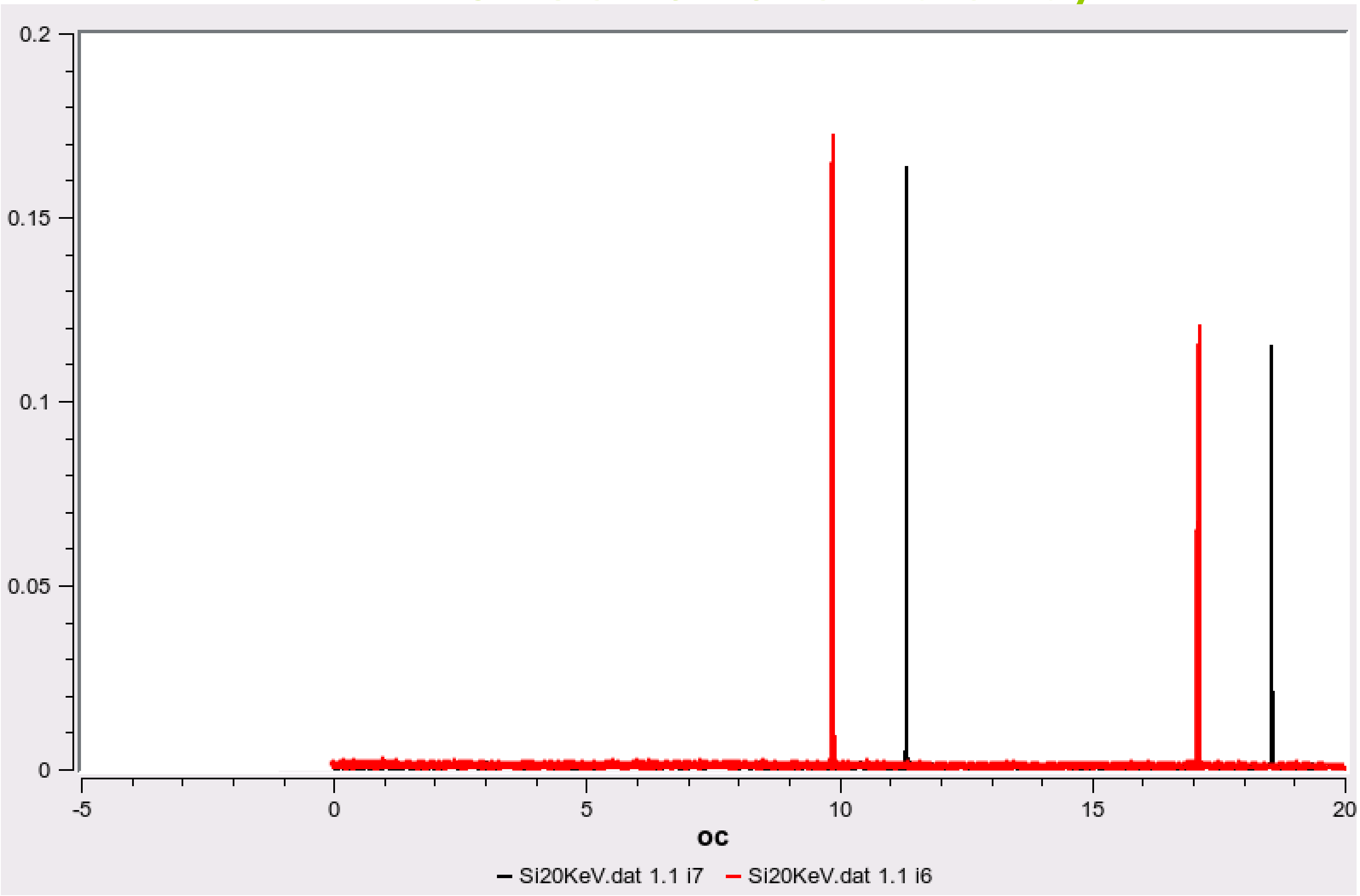


Combine signals from neighbouring channels:

offset



Combine signals from neighbouring channels: offset and efficiency



Data processing at Alba

The same is done with the 13 channels

One sample, typically Silicon standard is measured with good statistics to obtain the proper values of offset and efficiency for each channel

The merging is only possible if the channels see approximately the same signal.

Data processing at Alba

Merging data from a multi-detector continuous scanning powder diffraction system. *J. P. Wright, G. B. M. Vaughan, and A. N. Fitch.*

IUCr Computing Commission Newsletter, 1, 92 (2003).

It has been adapted to mad26 detector by Francois Fauth

albasum/albaoffsets

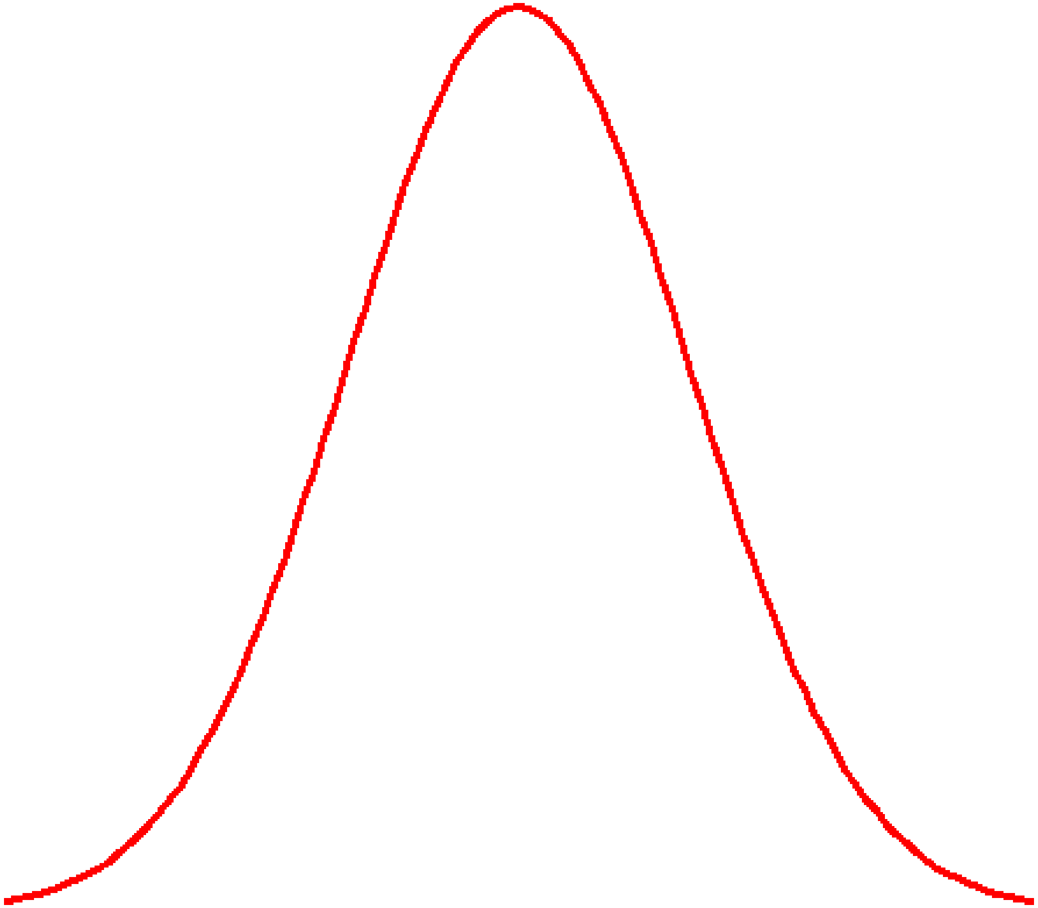
Scanning mad26 at mspd

Typically ~1 degree/minute

High resolution data from mad detectors

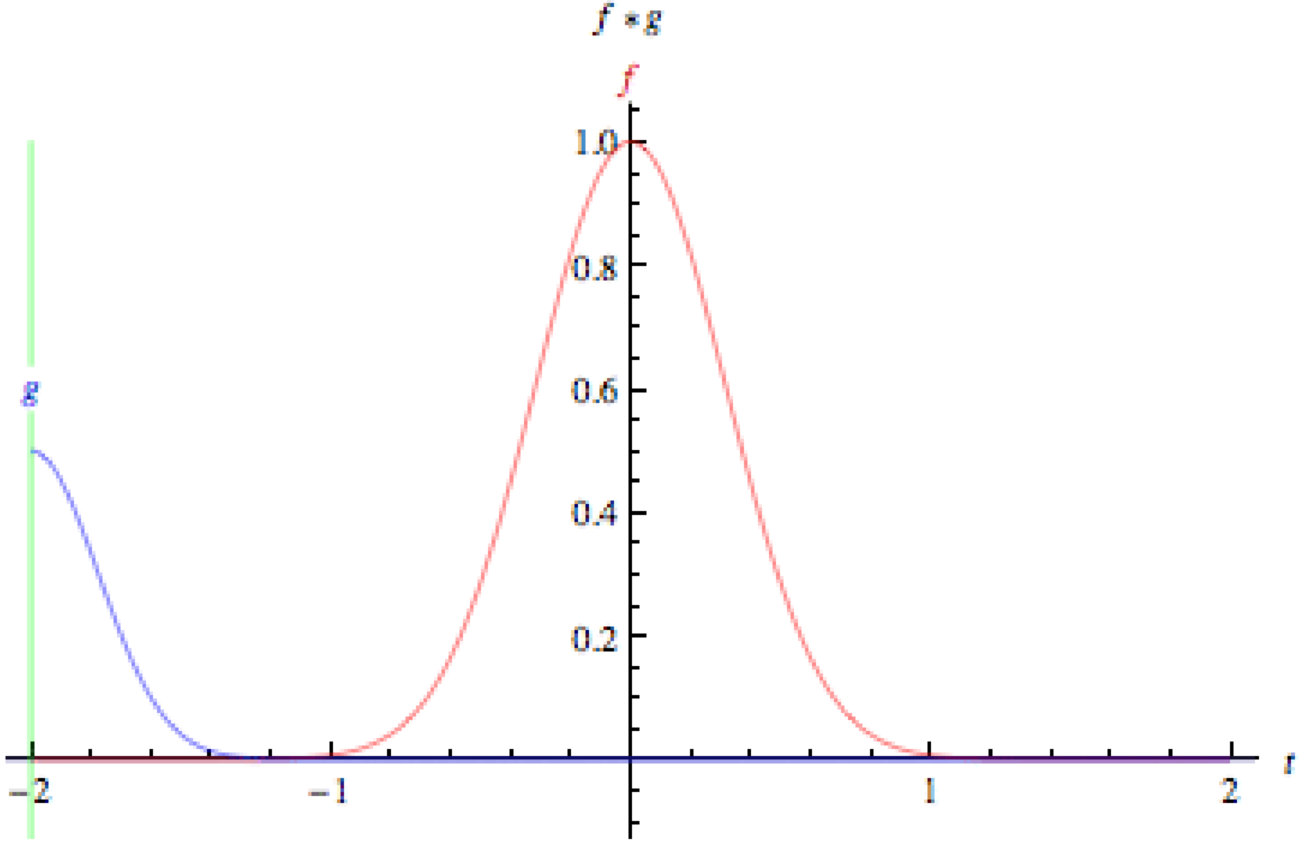
- High resolution data → instrumental resolution given mainly by angular acceptance of the crystals
- Excellent signal to noise ratio!

REAL SAMPLES



... also have curves

The effect of the real sample



When is it needed?

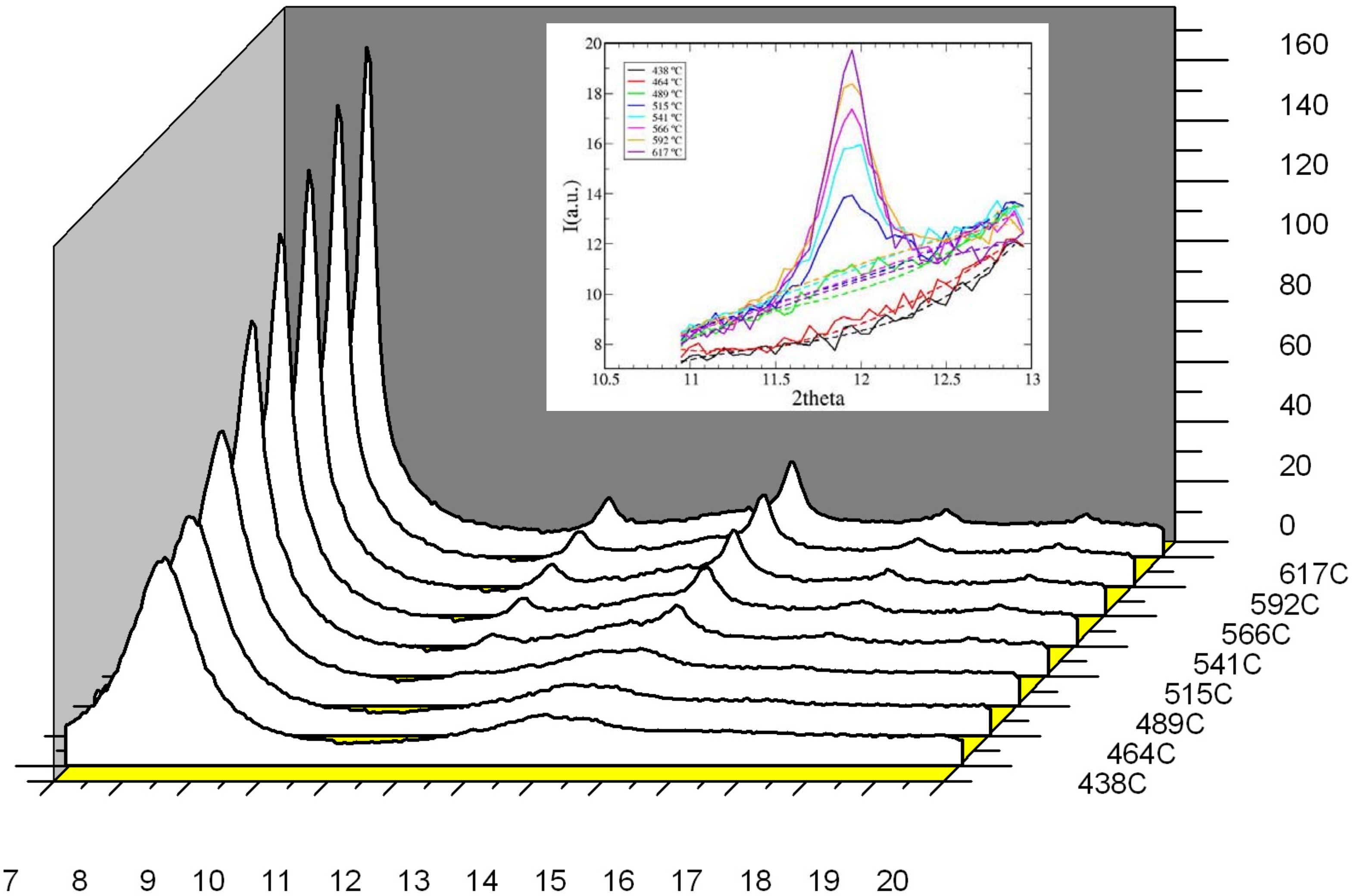
Real samples have curves

Nanocrystalline sample measured at ID31

Microstructure evolution and grain size distribution in nanocrystalline FeNbBCu from Synchrotron XRD and TEM analysis

J. Torrens-Serra, I. Peral, J. Rodriguez-Viejo and M. T. Clavaguera-Mora

J. Non Crystalline Solids 358 107–113 (2012)

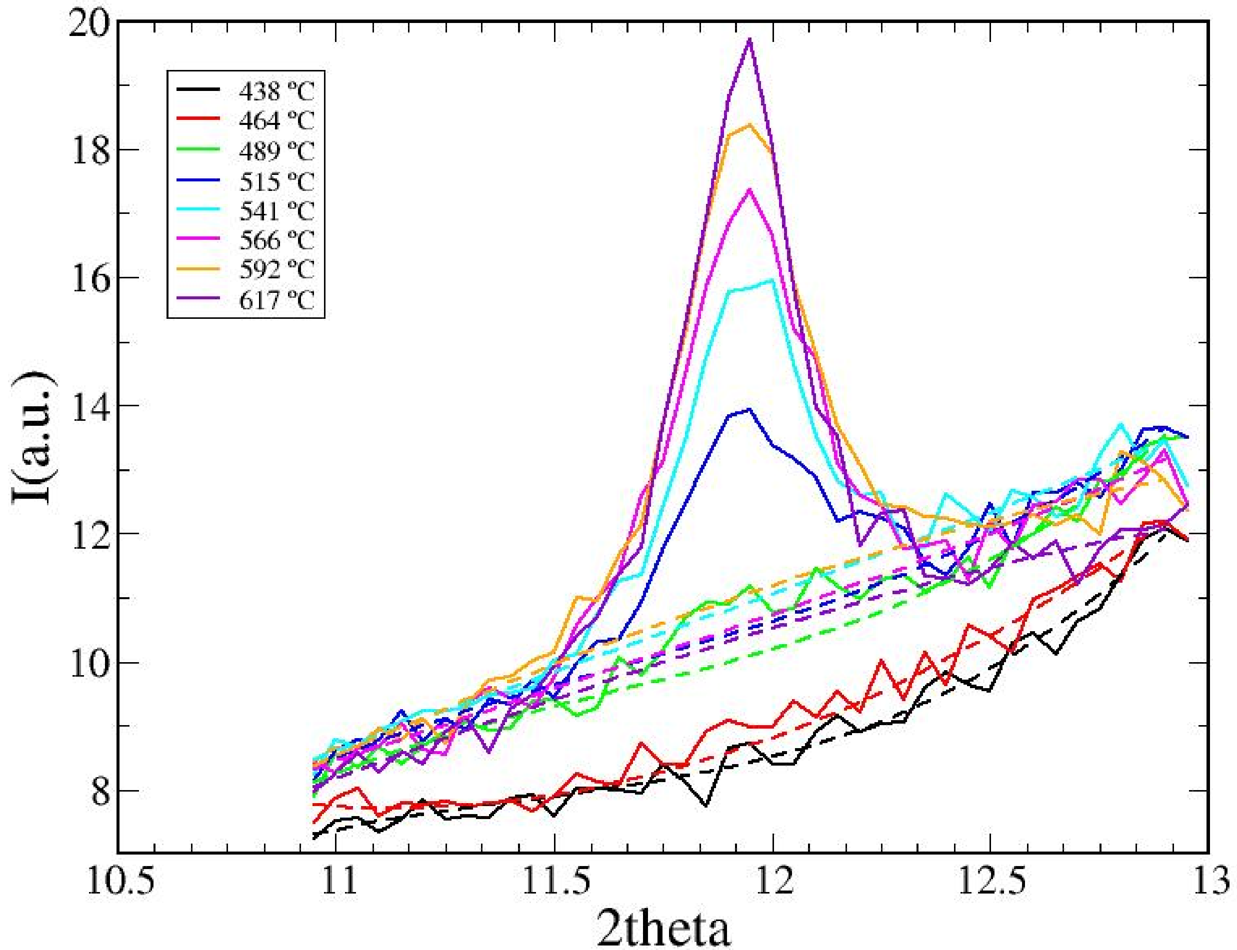


Nanocrystalline sample measured at ID31

Microstructure evolution and grain size distribution in nanocrystalline FeNbBCu from Synchrotron XRD and TEM analysis

J. Torrens-Serra, I. Peral, J. Rodriguez-Viejo and M. T. Clavaguera-Mora

J. Non Crystalline Solids 358 107–113 (2012)

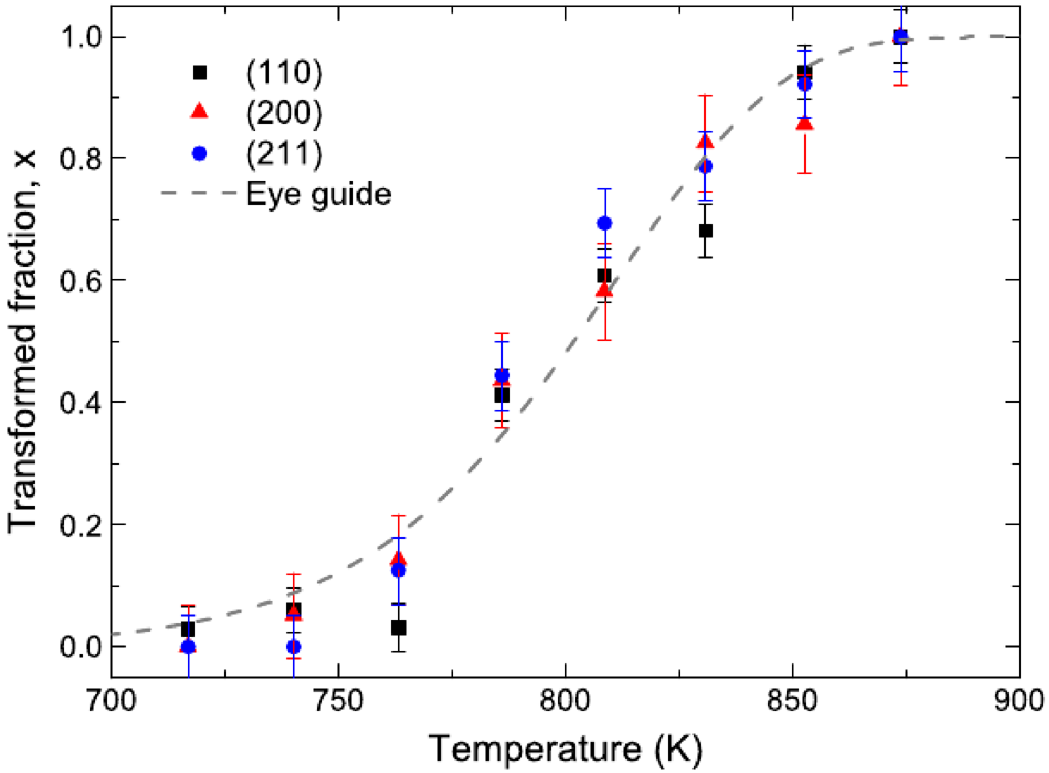


Nanocrystalline sample measured at ID31

Microstructure evolution and grain size distribution in nanocrystalline FeNbBCu from Synchrotron XRD and TEM analysis

J. Torrens-Serra, I. Peral, J. Rodriguez-Viejo and M. T. Clavaguera-Mora

J. Non Crystalline Solids 358 107–113 (2012)



Nanocrystalline sample measured at ID31

Microstructure evolution and grain size distribution in nanocrystalline FeNbBCu from Synchrotron XRD and TEM analysis

J. Torrens-Serra, I. Peral, J. Rodriguez-Viejo and M. T. Clavaguera-Mora

J. Non Crystalline Solids 358 107–113 (2012)

Table 2

Calculated values of the surface- ($<D>_A$) and volume-averaged ($<D>_V$) domain sizes, average radius (R) and dispersion (σ) from the Warren–Averbach and the Langford methods; from the TEM experimental histogram and from the fitting analysis with the Debye equation.

	TEM	Profile analysis		Modelling
		W-A	Lang.	
$<D>_A$ (nm)	4.6	3.9	3.3	4.6
$<D>_V$ (nm)	5.9	5.2	5.2	5.6
$\bar{R}$ (nm)	2.6	2.1	1.3	3.0
σ (nm)	1.0	0.9	0.8	0.8

Examples of application

(examples from the Alba newsletter)

Structural studies

Structure solution (beware of the radiation damage! with organic samples)

In-situ studies, trackig the structure vs. temperature, electric field, etc.

Catalysis (tracking of a range in 2theta in minutes!)

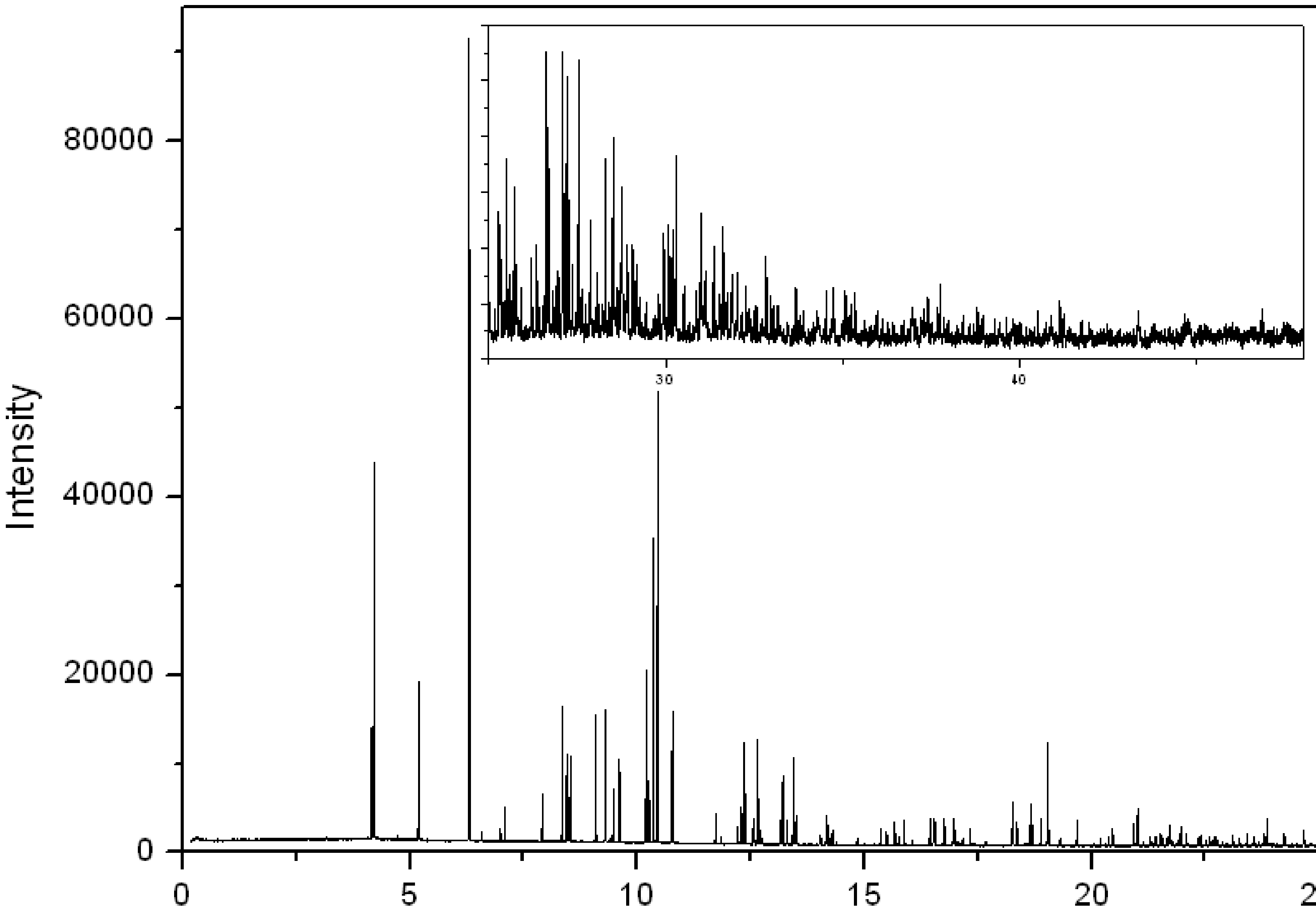
Profile analysis

Phase transitions

...

Advantages:

- Whole energy range of the beamline
- Well resolved peaks
- Low background



The diffraction pattern of an as-made zeolite containing organic cations and fluoride anions. Miguel Camblor group (Alba newsletter)
It was measured in 187 minutes, with 0.6198 Å up to 48 º (0.76 Å).

Thank you for your attention!