The mythen II detector: Practical aspects

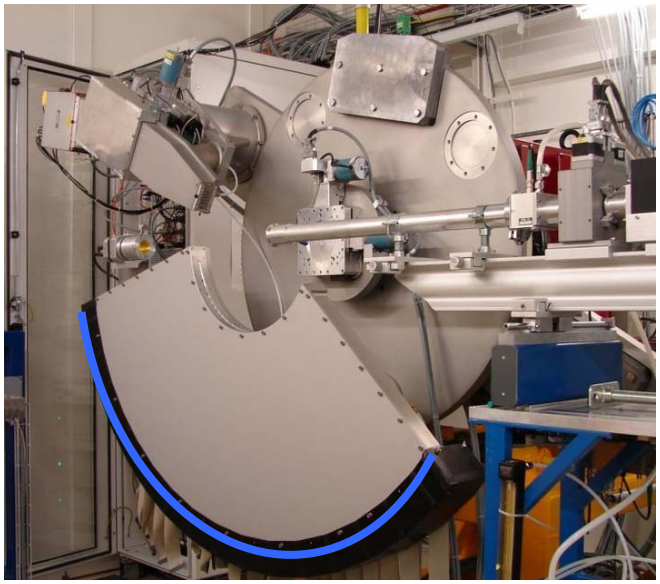
Inma Peral

PowderXRD2013

The Mythen II Detector: Microstrip System for Time resolved Experiments

Detector developed by the PSI Detector group

http://pilatus.web.psi.ch/mythen.htm



First installed in the Materials Science Beamline at the SLS (Synchrotron Light Source)

In the photo, 24 modules of mythenII
Covering 120 degrees of 2θ
Materials Science Beamline at SLS

Commercialized as today by Dectrics
https://www.dectris.com/

Why the mythenII detector?

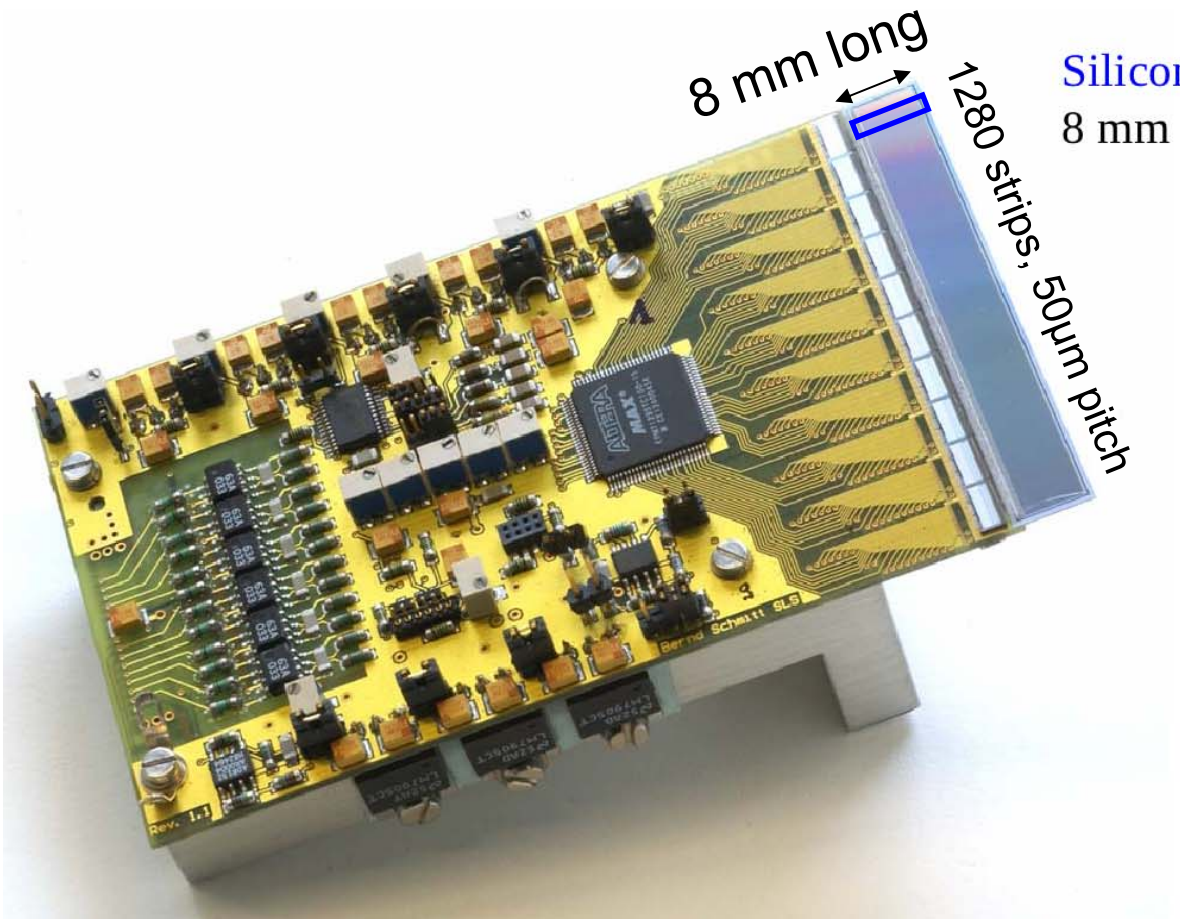
The quality of the synchrotron source: *monochromatic* and *collimated* allows for the high resolution detector data (... more later)

But ... high resolution detector trades photons for resolution

Fast Acquisition Detector specifications should be:

-High dynamic range (to detect strong and weak peaks)
-Time resolution ~0.1 s over 40 degrees of 2theta
-Intrinsic angular resolution better than 0.01 degrees

Mythen module



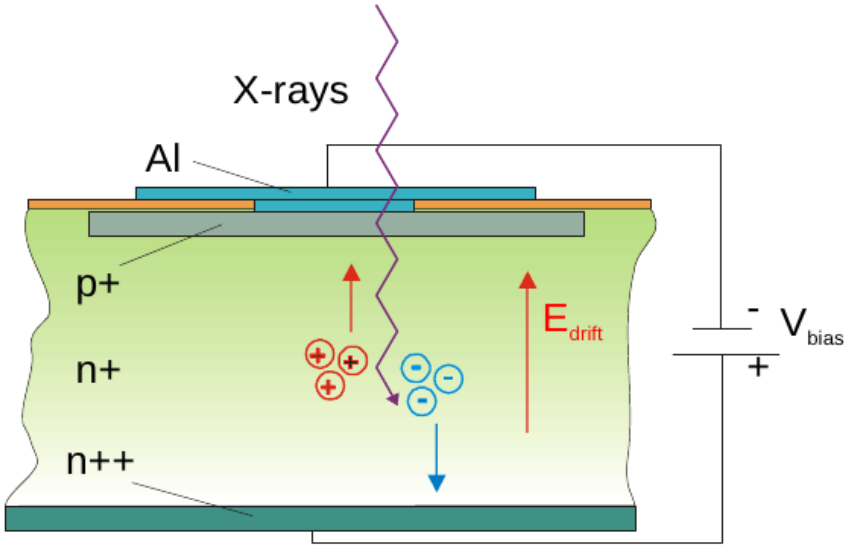
Silicon sensor with 1280 strips
8 mm long, 50 µm pitch, 300 µm thick

Read out chip:
128 channels
low noise preamp: 230 e-
24 bit counter
Readout Time: 250 µs
Count rate: 1 Mhz per channel

Detector principle

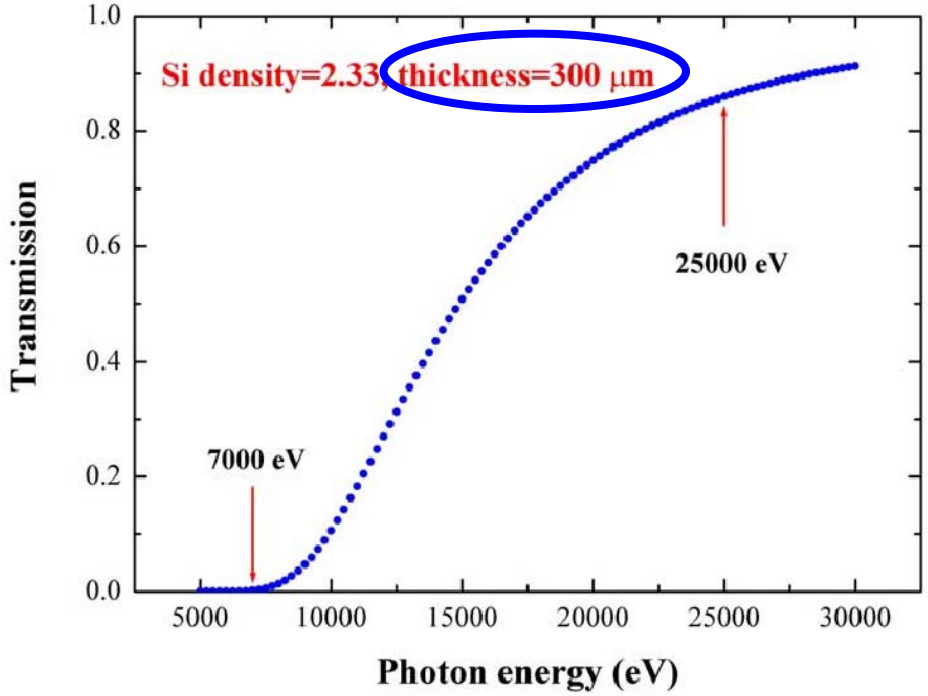
Silicon 300 µm thick wafer pn doped segmented on one side 1280 50 µm pitch 8 mm

X-ray is absorbed by the Silicon creating electron-holes that drift



small charge: 1400 -7000 e- for 5 keV – 25 keV X-rays
Enough charge for the front end electronics to count each photon directly

Silicon sensor efficiency



The detector efficiency is down to 15% already at 25 keV

Offered by Dectris as today	
Sensor thickness [μm]	320, 450, 1000

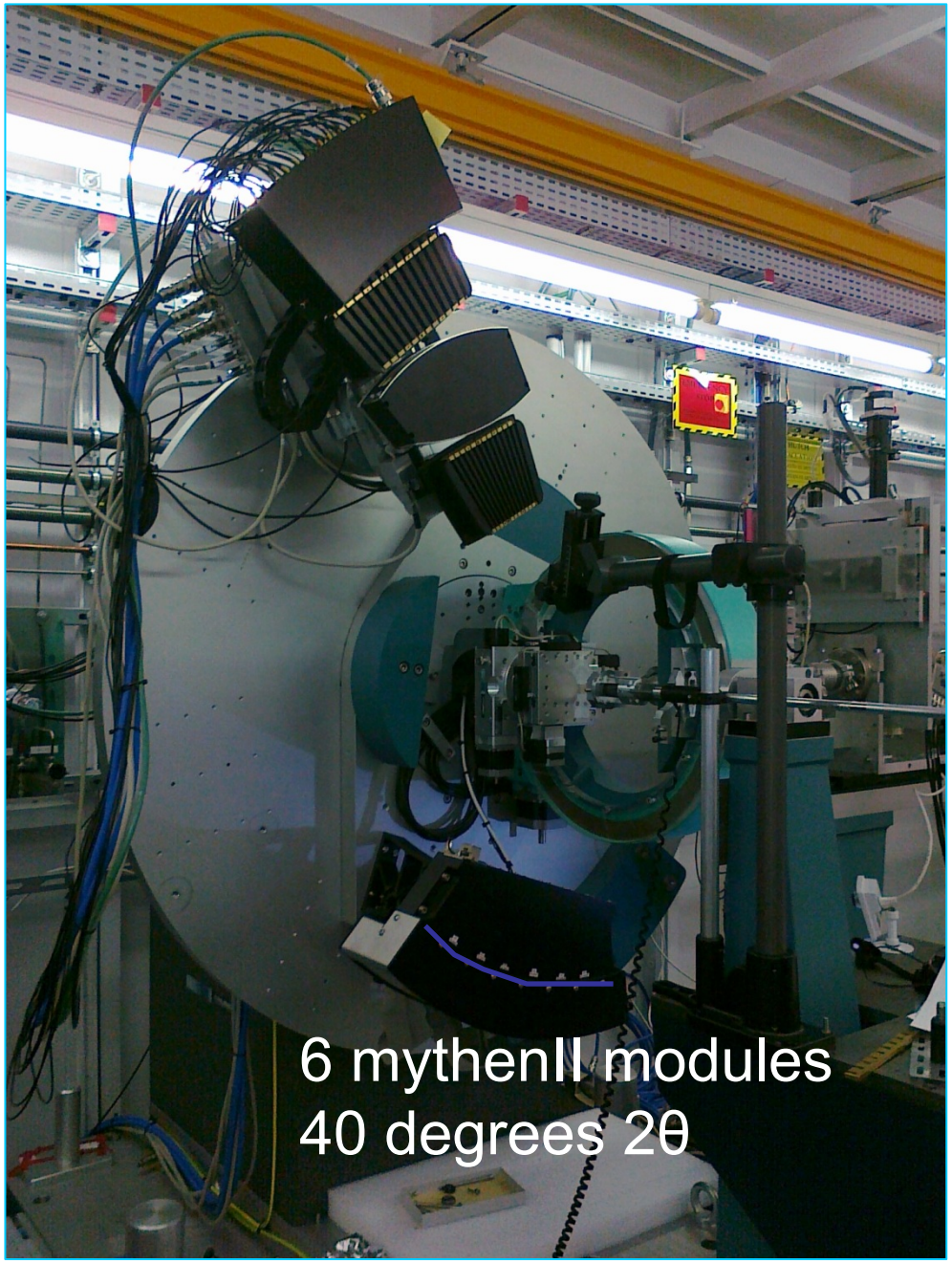
Our mythenII model is not efficient at E > 25 keV

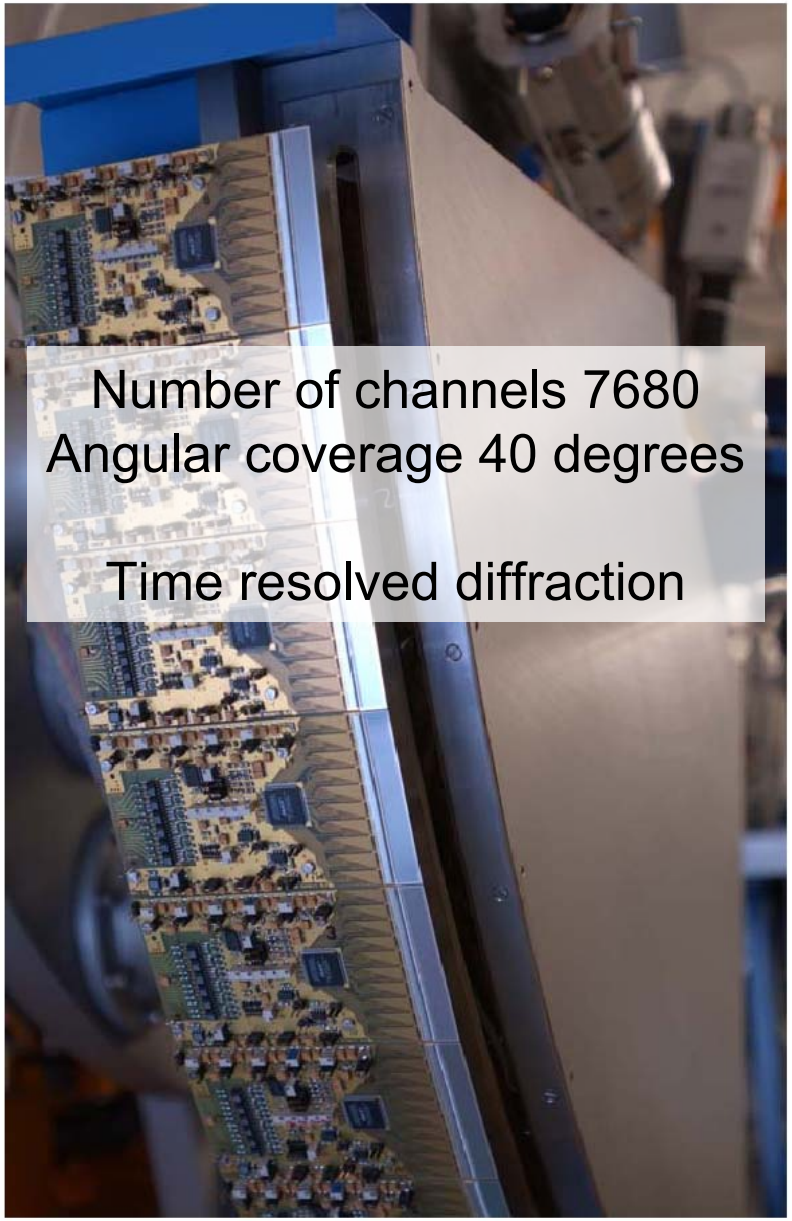
References

MythenII: A 128 channel single photon counting readout chip
Nuclear Instruments and Methods in Physics Research A 607 (2009) 250–252

The MYTHEN detector for X-ray powder diffraction experiments at the Swiss Light Source
Bergamaschi et al.
J. Synchrotron Rad. (2010). 17, 653–668

… and references therein





Mythen modules in detector casing
(without cover)

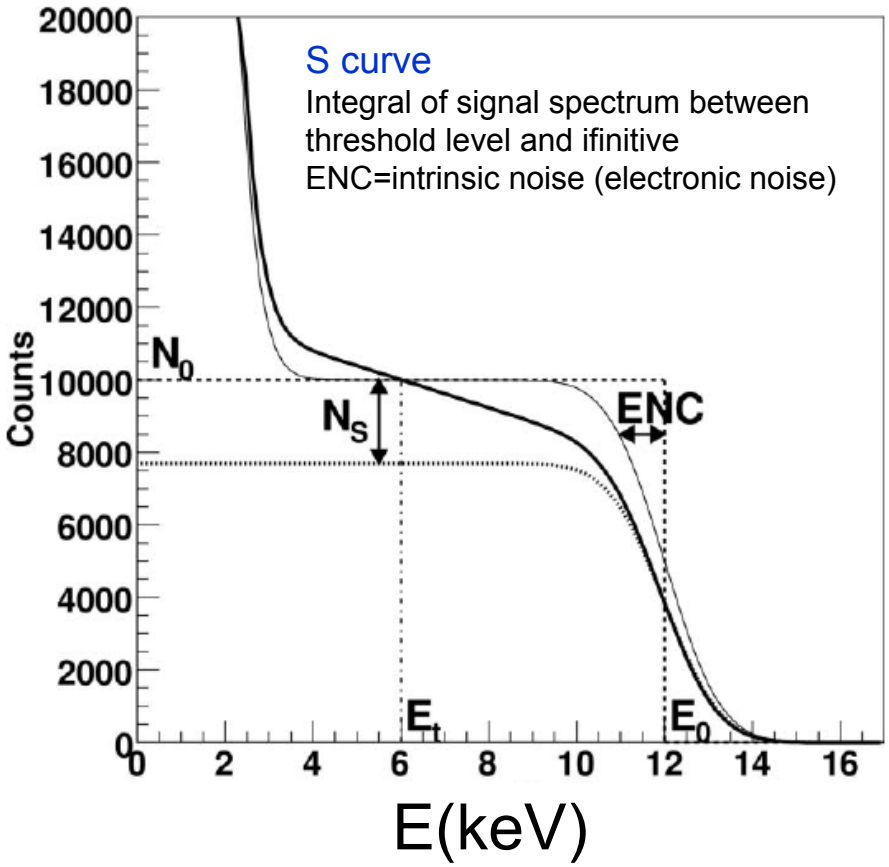
Detector calibration

- Energy calibration, relatively straight forward
- Angular calibration relatively easy: record diffraction peak (e.g. Si 111)every 0.5 degrees and fit channel number vs angle from encoder
- Intensity calibration: variations in gain and threshold cause count rate fluctuations use uniform illumination from coherent scatterer (quartz glass).

Detector calibration
(not for users!)

- Energy callibration, relatively straight forward
- Angular calibration relatively easy: record diffraction peak (e.g. Si 111)every 0.5 degrees and fit channel number vs angle from encoder
- Intensity calibration: variations in gain and threshold cause count rate fluctuations use uniform illumination from coherent scatterer (quartz glass).

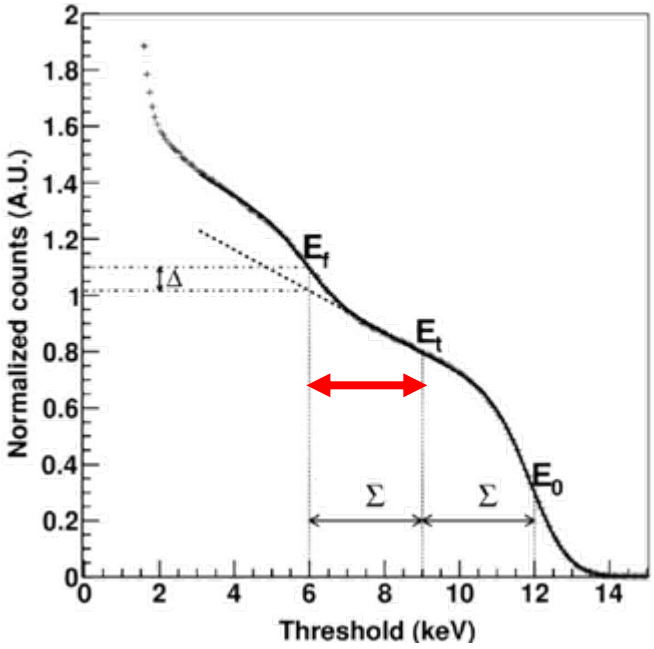
Energy calibration



Counts as a function of therhold by No photons of Eo energy

-Digitalised signal does not contain information on the photon energy.

-It is crucial to have the correct threshold in the comparator



Normally Et=Eo/2

To be revised when there are elements that emit fluorescence

Energy calibration

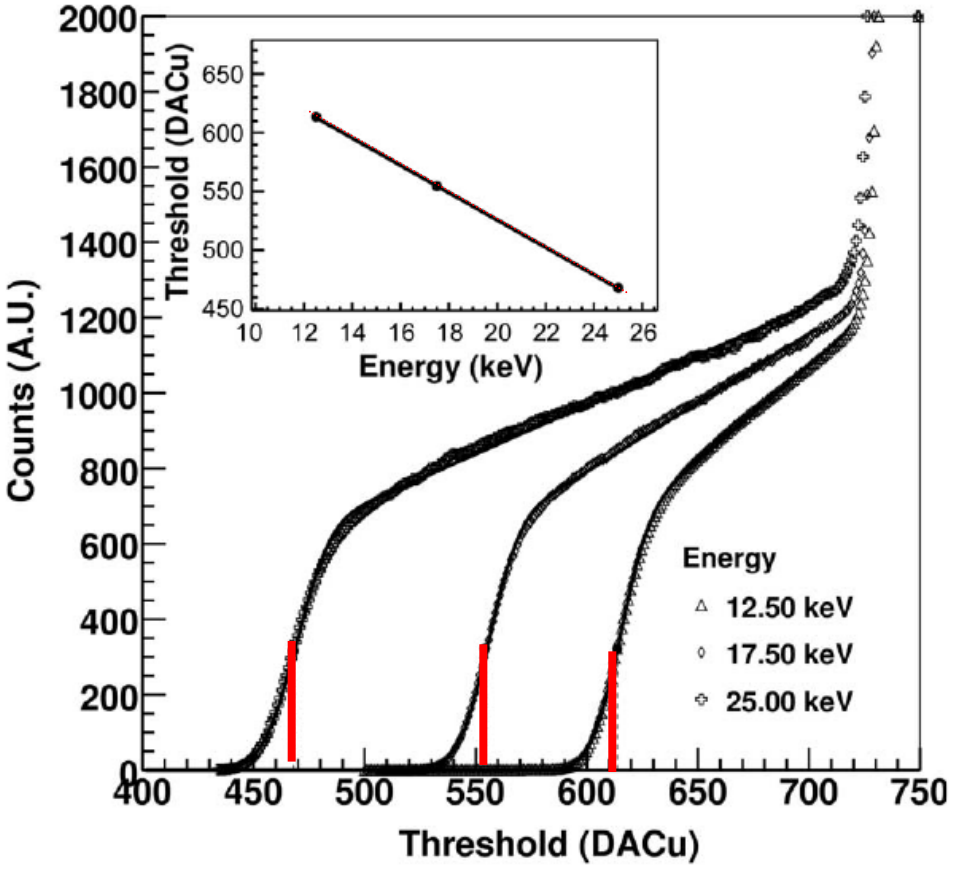


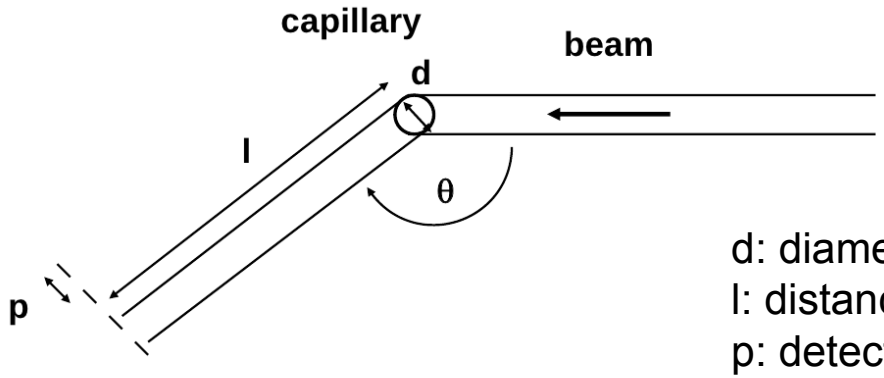
Figure 8
Median of the number of counts as a function of the threshold for X-rays of 12.5, 17.5 and 25 keV for one of the detector modules using *standard* settings. The solid line represents the fit of the experimental points with equation (1). In the inset the linear fit between the X-ray energy and the position of the inflection point of the curves is shown.

MythenII powder diffraction data

Angular resolution

intrinsic resolution 50 µm/550 mm =0.005 degrees

Angular resolution defined by a capillary



d: diameter of the capillary
l: distance detector sample=550 mm
p: detector “pitch= 50 µm

$\tan\theta=(p+d)/l$

d=300 µm => 0.036 degree

d=100 µm => 0.016 degree

MythenII powder diffraction data

Time resolution

Detector can be gateable, counting can be switched on or off (from 50ns to ∞)

Normal operation

Switch counting on, accumulate data, switch it off and read the detector out

Quite often, the same acquisition is repeated for several positions of the detector -> to be merged at the end

Pump and Probe operation

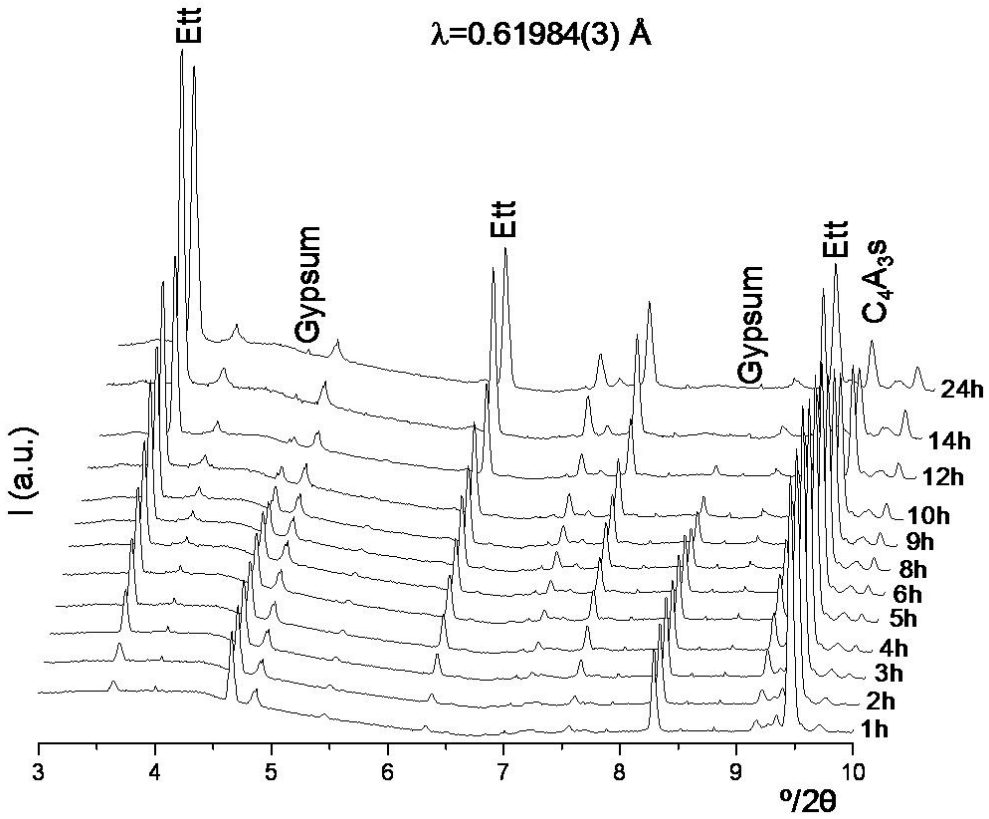
Pump sample (apply external excitation of sample) switch counting on for a time t1 after a delay t2, pump ... read data after N iterations

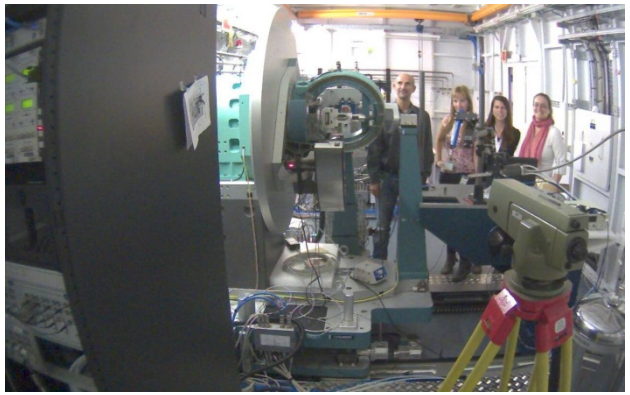
Some examples

Cement hydration

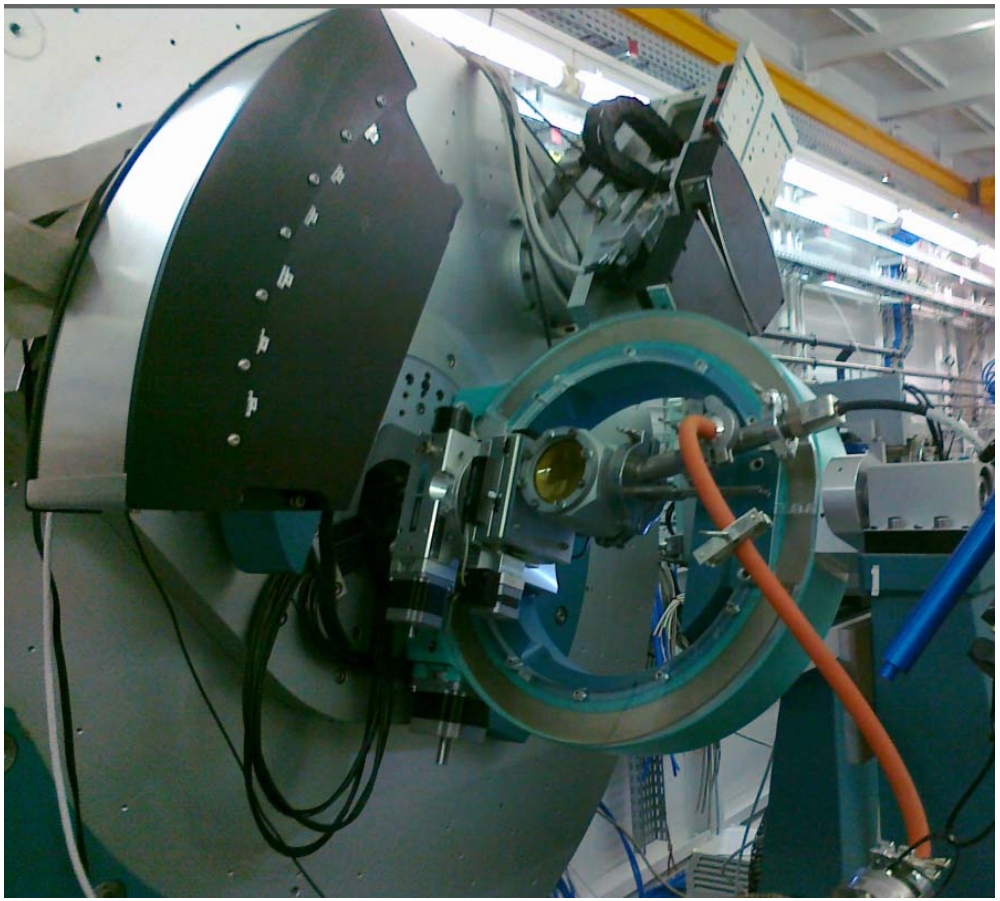
First mythen experiment

In-situ powder diffraction hydration study of eco-cements

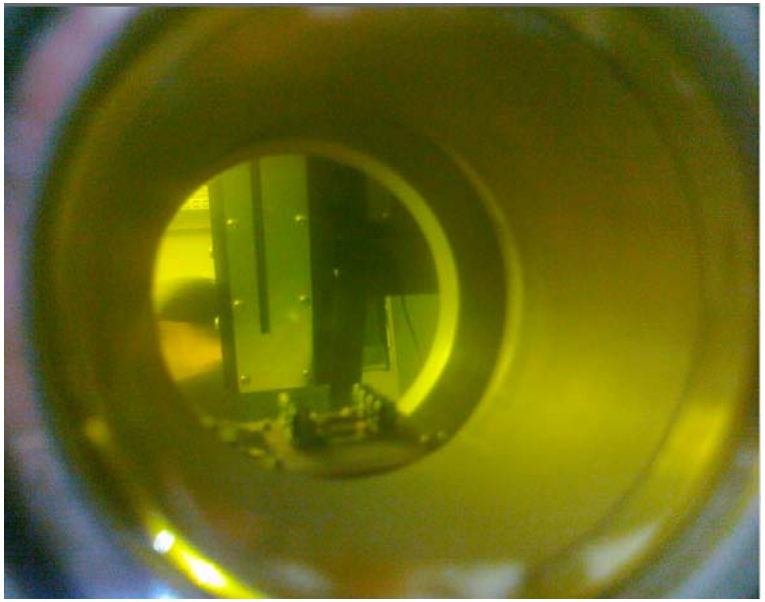




Combing techniques: nanocalorimetric data and x-ray diffraction



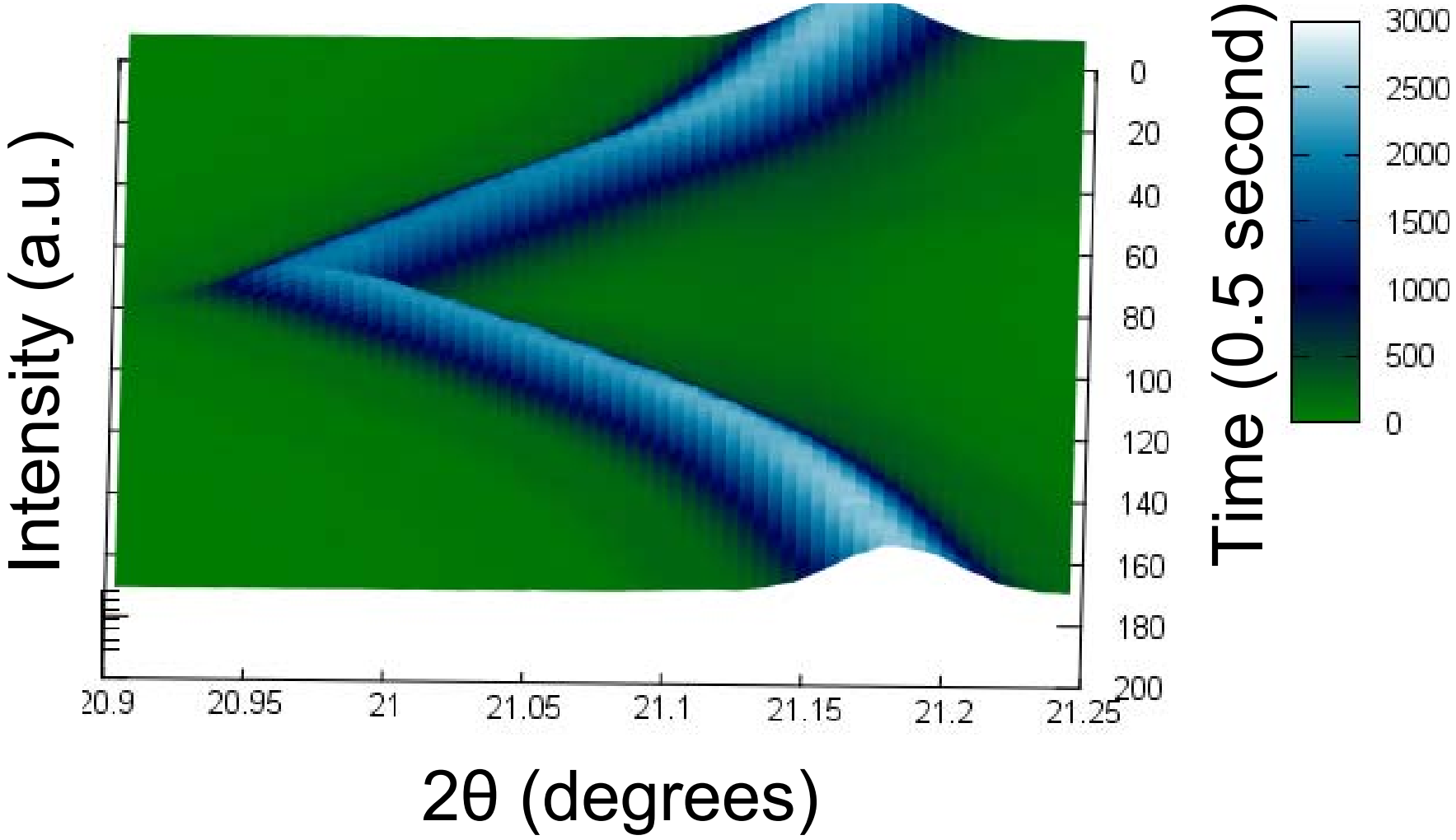
Nanocalorimeter cell in the powder diffraction endstation



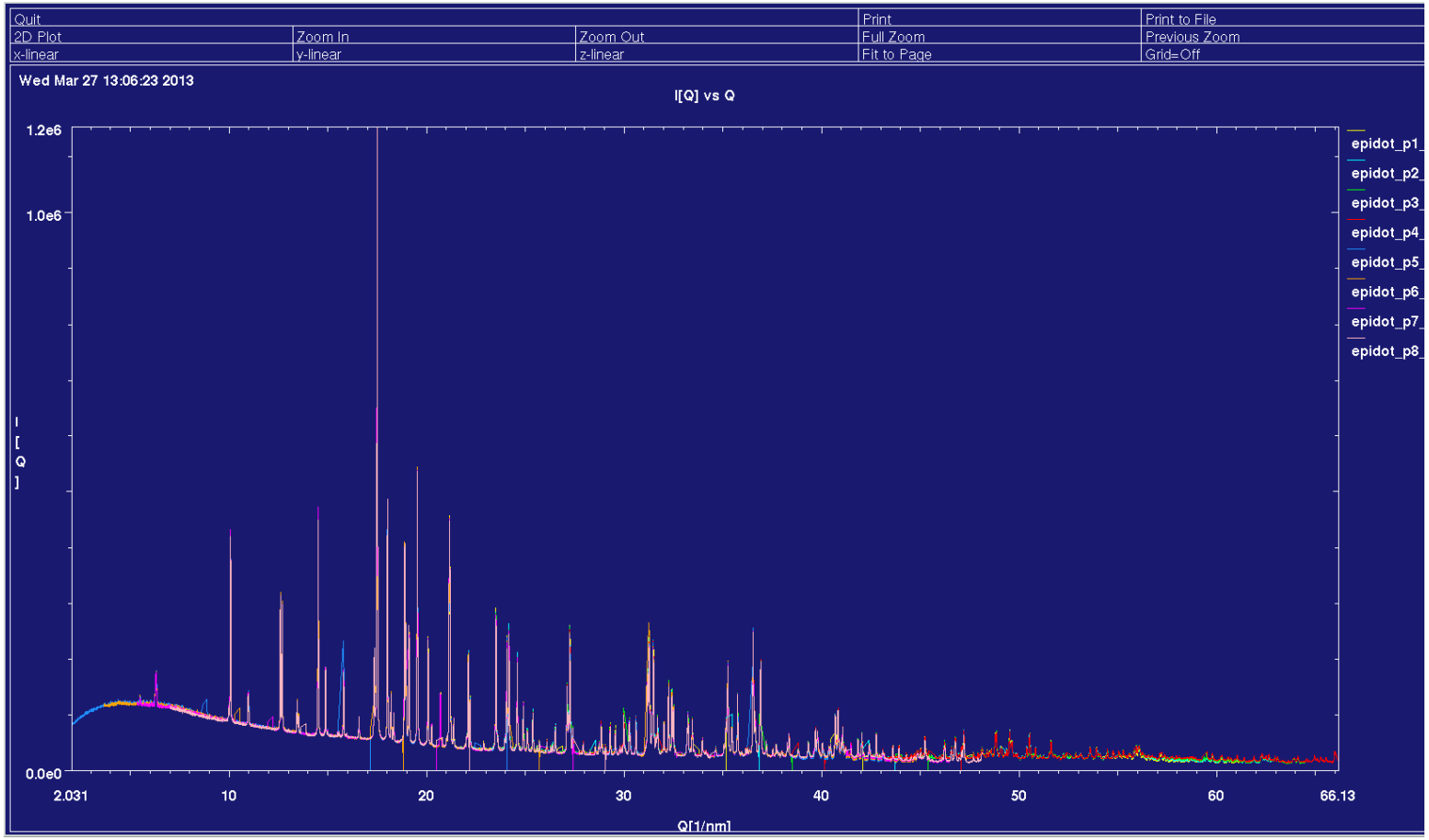
Nanocalorimeter seen through the kapton windows of the cell.
Mythen detector window can be seen.

http://gnam.uab.cat

Diffraction peak versus time during heating/cooling ramps at 10 K/s

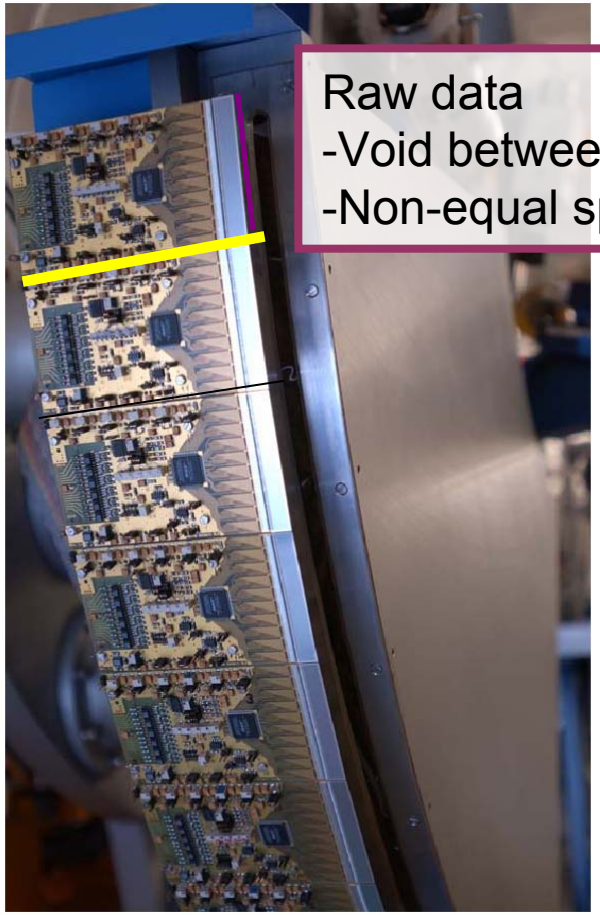


One example: epidote, mineral sample

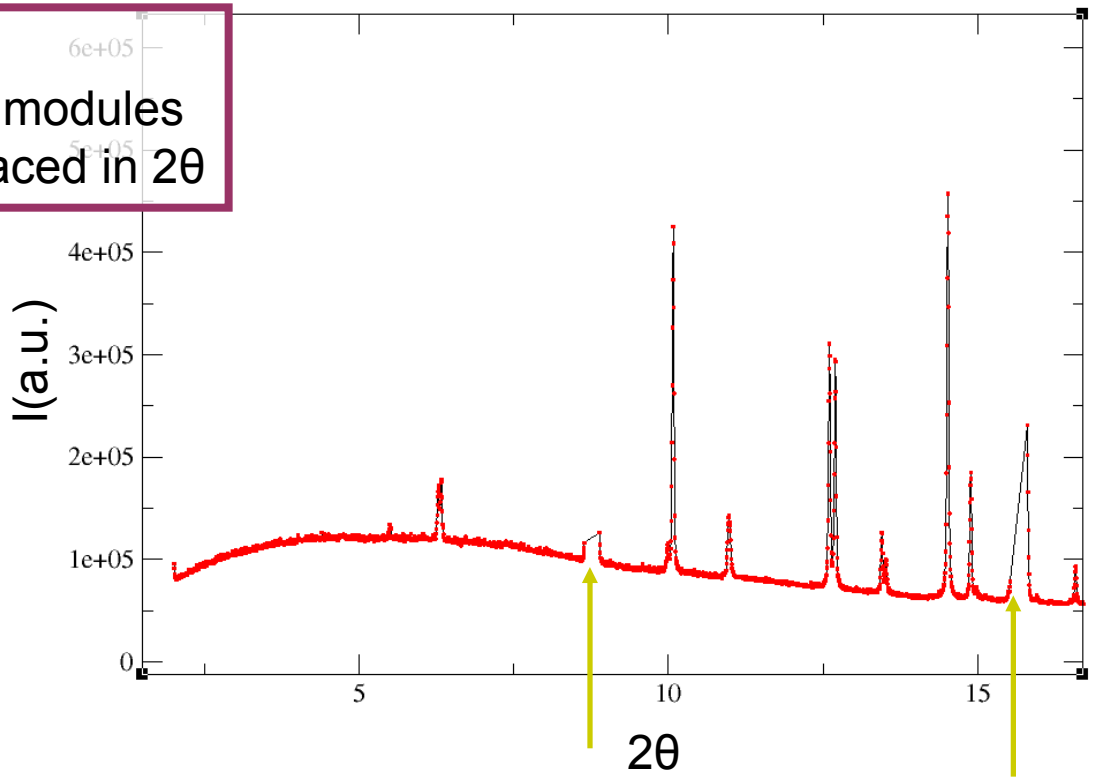


sample in a capillary at room temperature
typical experiment, different positions of the detector to be merged

The “typical”bl04 user experience
sample in a capillary at room temperature



Raw data
-Void between modules
-Non-equal spaced in 2θ

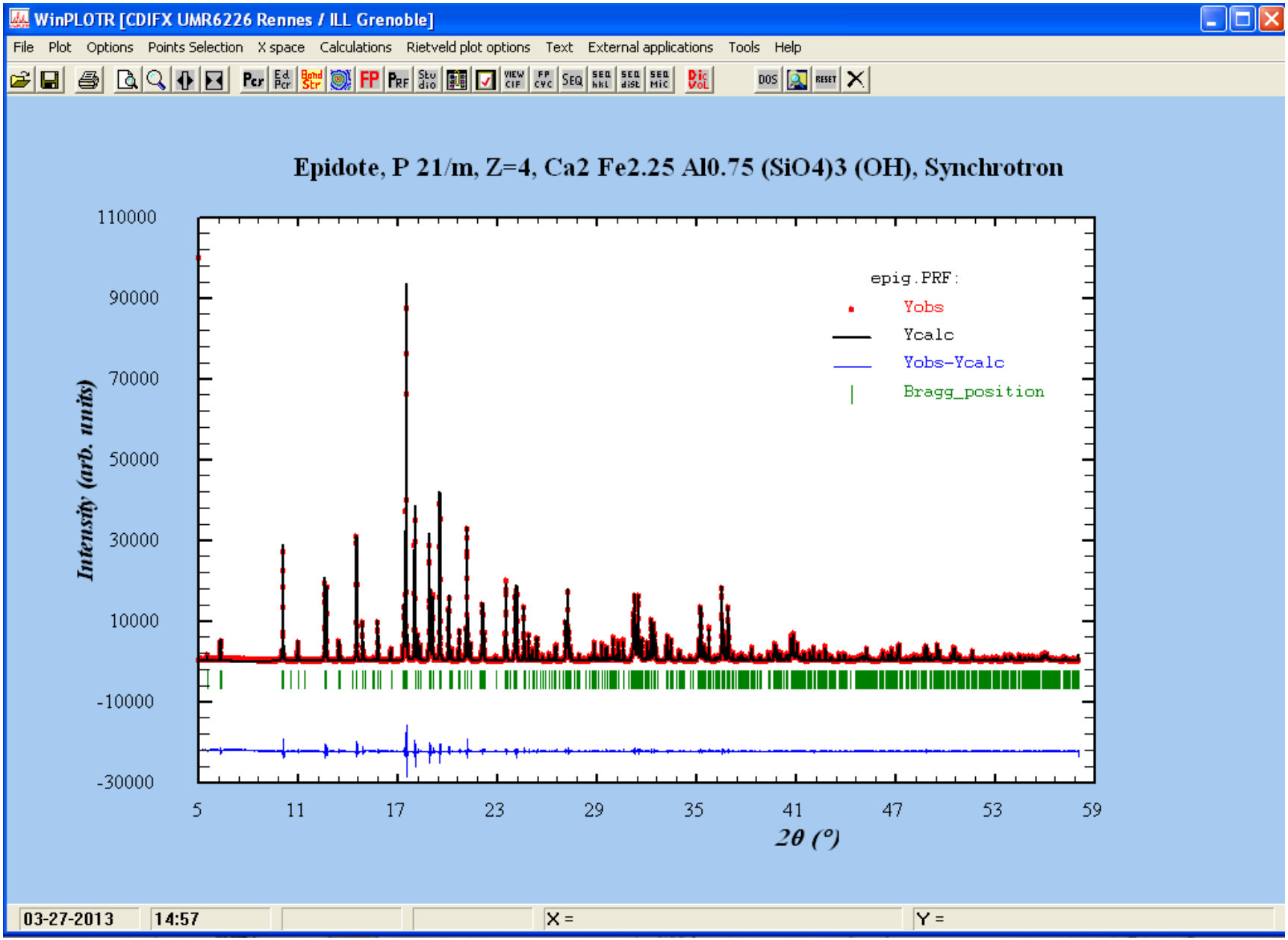


voids between modules

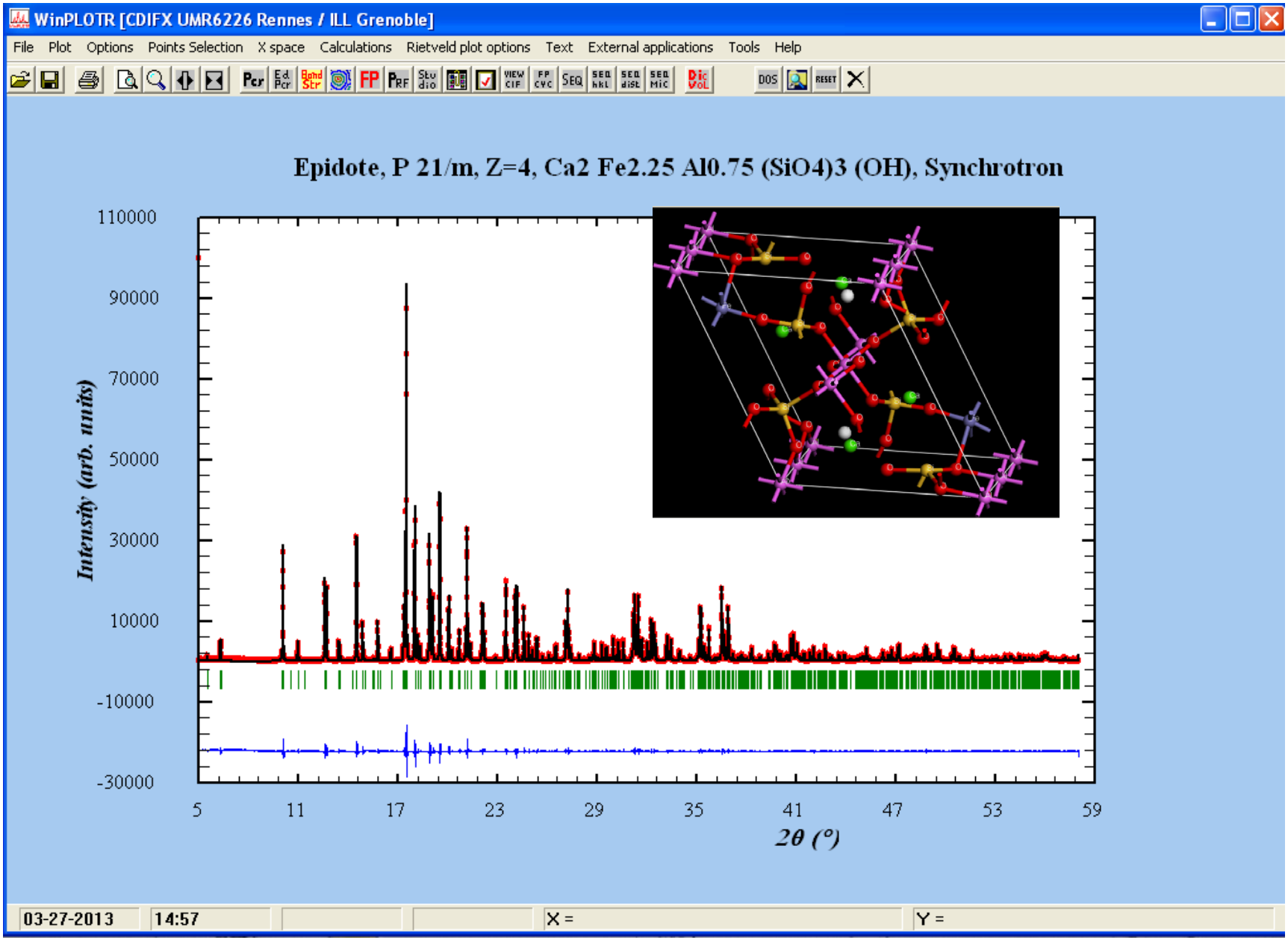
MythenII data reduction (done at Alba, during experiment)

- Standard measurement for wavelenght and offset
- Your measurement produces .raw and .dat files (ascii)
  - Counts vs channel raw files
  - Counts vs 2theta angle .dat files
- Several programs available to combine and transform (and also to monitor) the data into files for gsas, fullprof and dajust/sgaid (Francois Fauth)

Integrated intensities extracted by dajust



…and structure solved by direct methods (tomorrow's talk)



Organic samples, structure solution

- Important: Check the existence of radiation damage
- Wavelength=1Å, good compromise for resolution and 2θ range
- If possible, small diameter of the capillary. We are studying the possibility of flat container
- Complete data set can be achieved within minutes (depends on crystallinity, etc.)
- Verify peak shapes! It is not a multicrystal analyser detector, it is sensible to aberrations

Typical powder diffraction aplications

- temperature scans
- organic samples -> much reduced radiation damage
- screening of samples (measure 100's of samples in short time)
- fatigue measurements (piezo crystals for injection systems)
- time resolved measurements (chemical reactions, e.g. cement hardening)
- ... and what you come up to ;)

Thanks for your attention!