Photon counting microstrip detector for time resolved powder diffraction experiments

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Outline

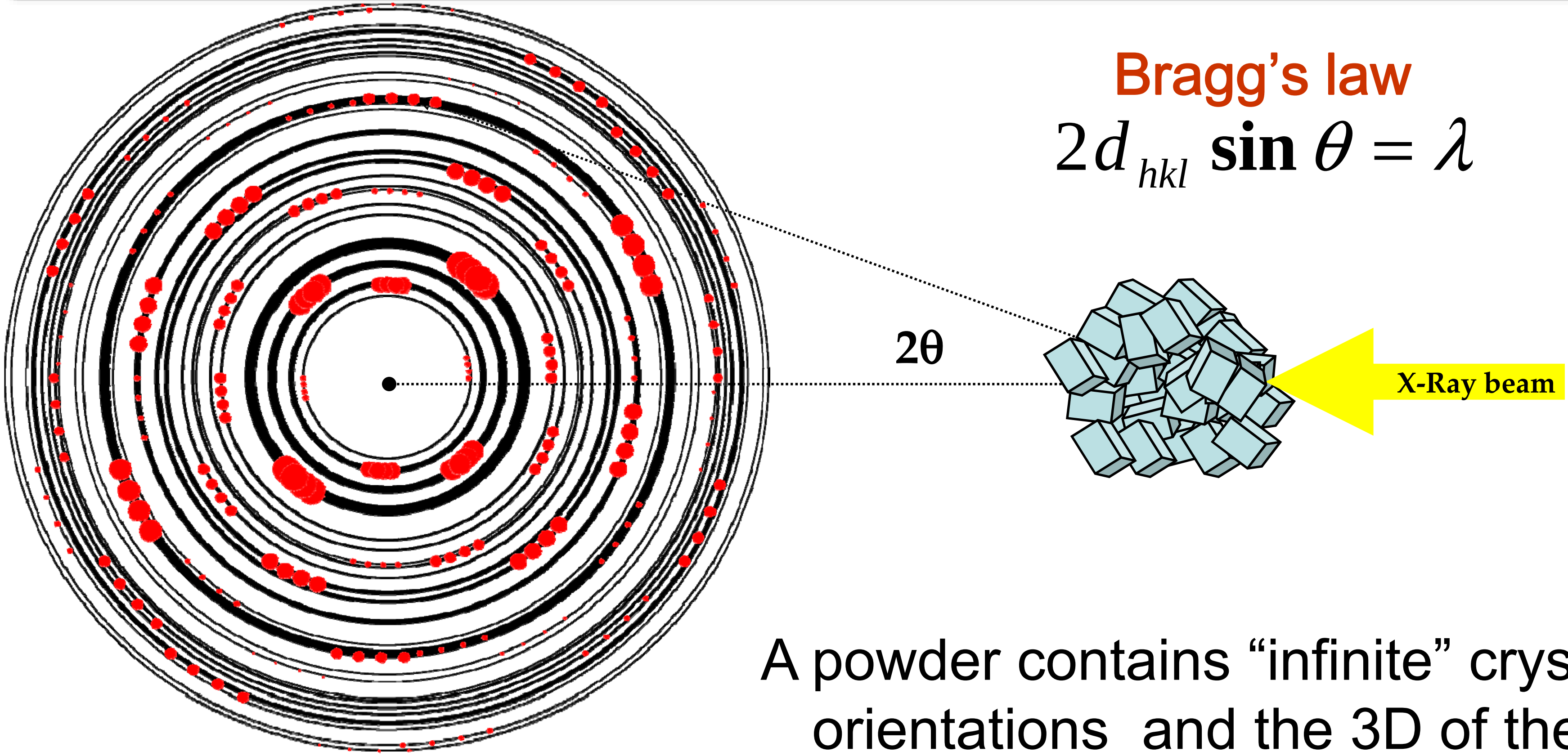
➔Powder diffraction detector requirements

➔Description of the MYTHEN detector

➔Detector performances

➔Example applications

Powder diffraction

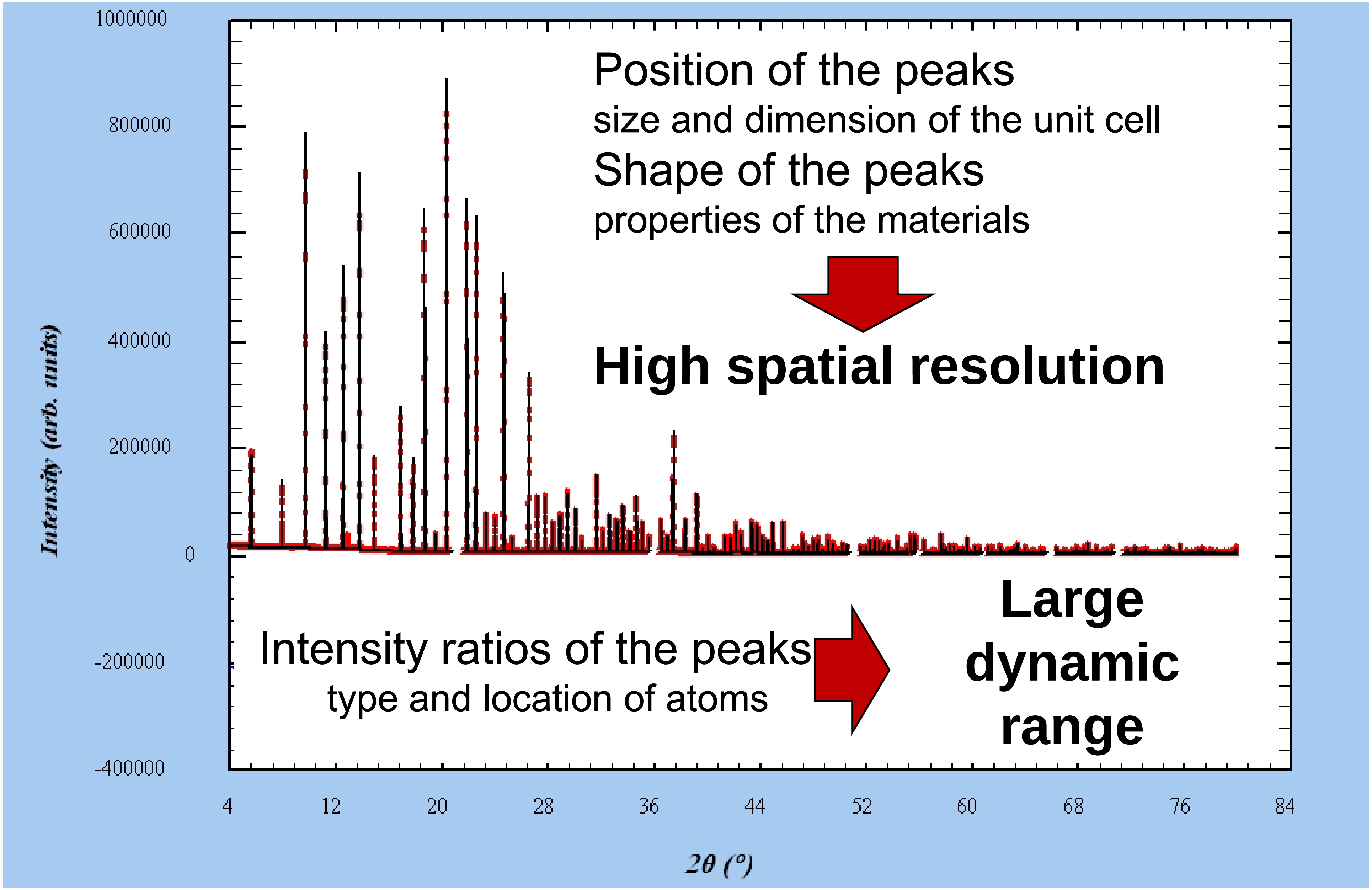


Bragg's law

$$2d_{hkl}\ \mathbf{sin}\ \theta = \lambda$$

A powder contains "infinite" crystal orientations and the 3D of the reciprocal space collapse onto 1D

Detector requirements



Time resolved experiments

➔ Samples in a variable environment

- Phase changes in the sample
- Many cycles to check the reversibility of forms

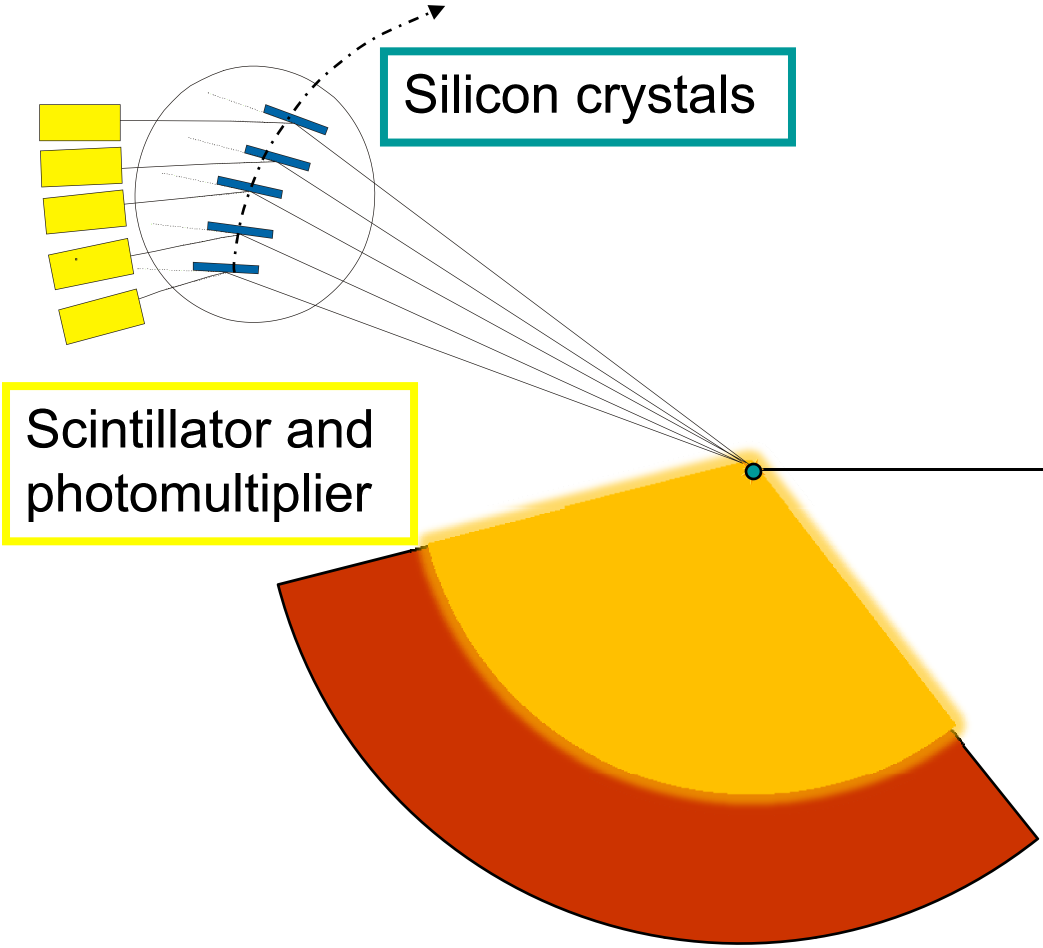
➔ Radiation sensitive samples

- Organic samples degrade already after a few minutes of exposure

➔ Pump and probe experiments

- Gating and triggering to synchronize with stimulus

Powder diffraction detectors



Crystal analyzer detector

- ➔ Better than 0.003° resolution
- ➔ Angular selectivity
- ➔ Long measurements
  - 15 min-several hours

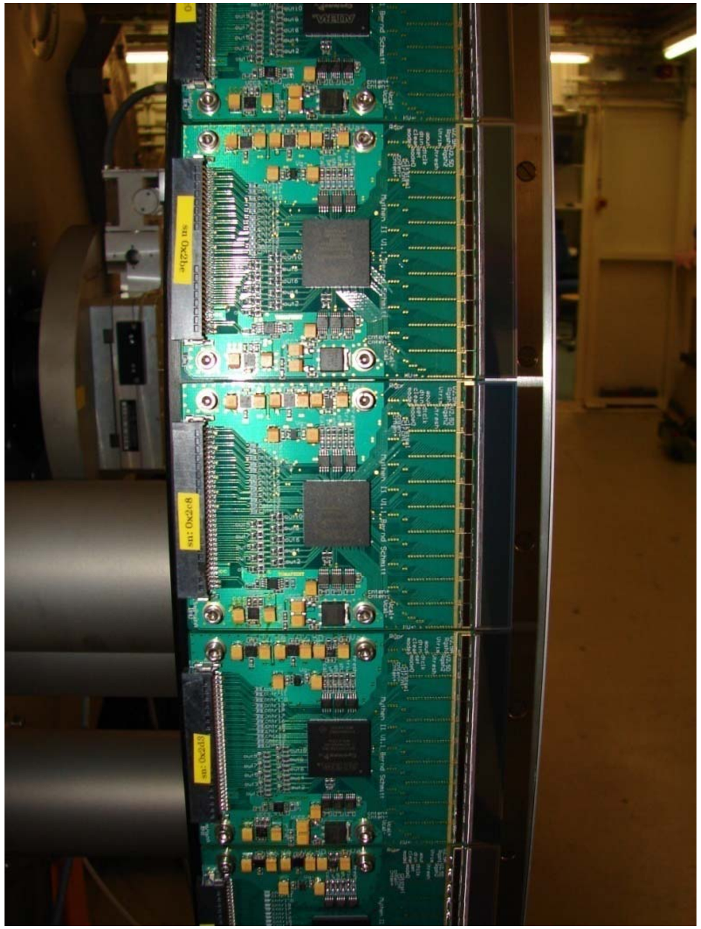
Silicon microstrip detector

- ➔ 120° acquired in parallel in fractions of a second

Microstrip sYstem for Time rEsolved experimeNts

➔ Silicon microstrip sensor
  - Position sensitive
    - 50 μm pitch
  - 1280 independent channels

➔ Single photon counting readout
  - Large dynamic range
    - 24 bits
  - Poisson-like statistics



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The wide angle diffractometer

➔ Massive Parallel detection

- 120° angular coverage
- 30k independent channels
  - 0.03% bad
- 0.004° angular resolution
  - Usually limited by the sample size

➔ Time resolved powder diffraction is possible

- Average acquisition time 1s
- The acquisition can run 30000 faster than using the single channel crystal analyzer

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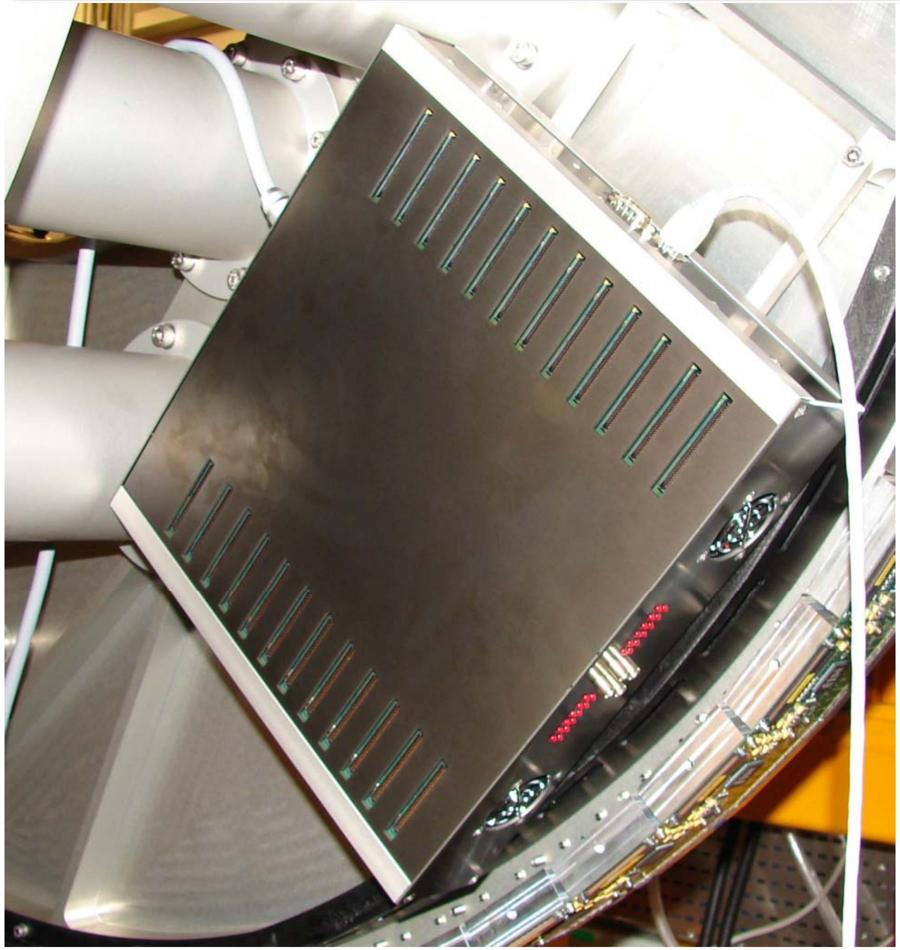
Mythen Control System



- ➔ Embedded Linux system
- ➔ Client-Server TCP/IP communication
- ➔ Real time data taking
  - ➔ Memory on board
- ➔ External gating and triggering
  - ➔ Interfacing to external hardware
  - ➔ Pump and probe experiments

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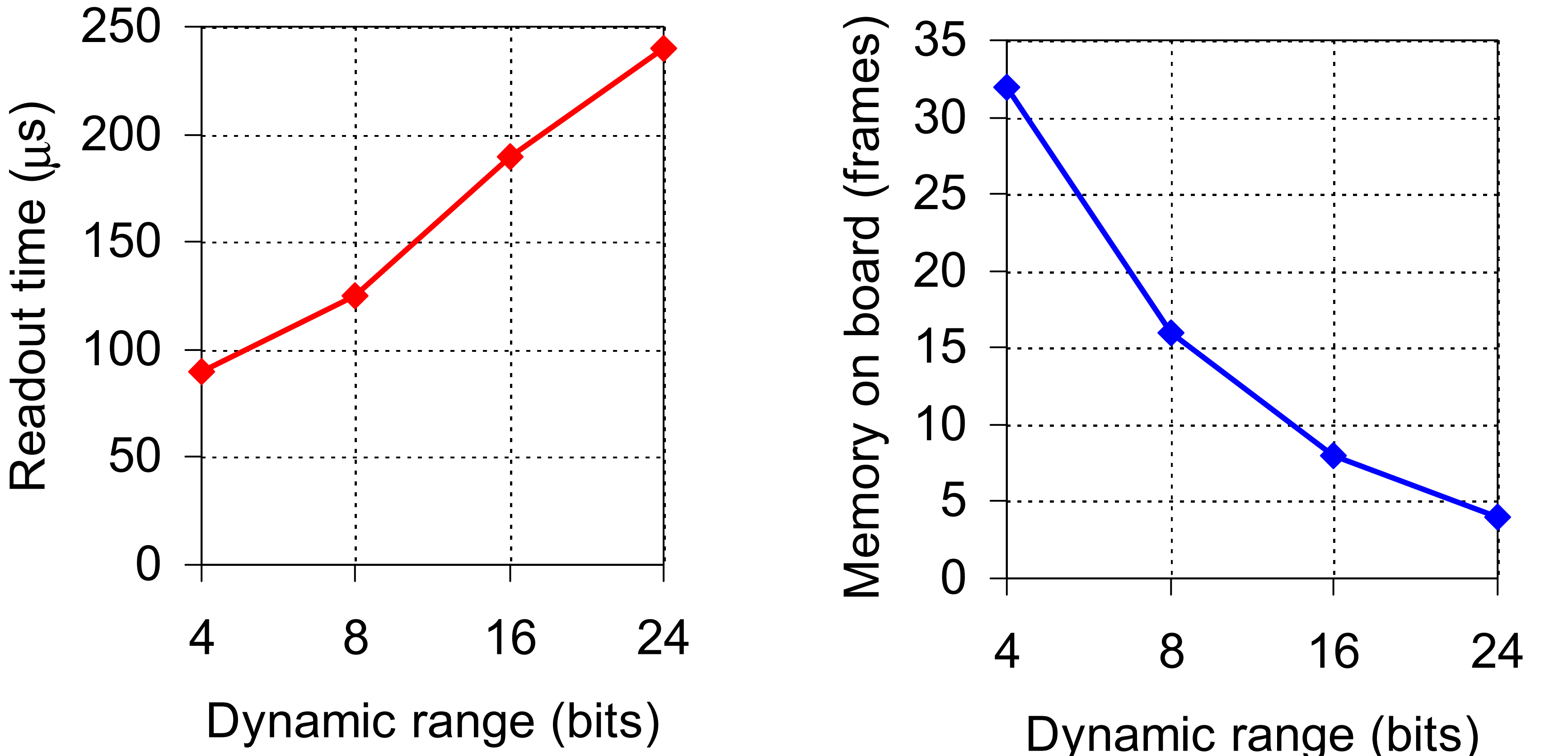
Readout time

➔Selectable counter dynamic range 4-24 bits



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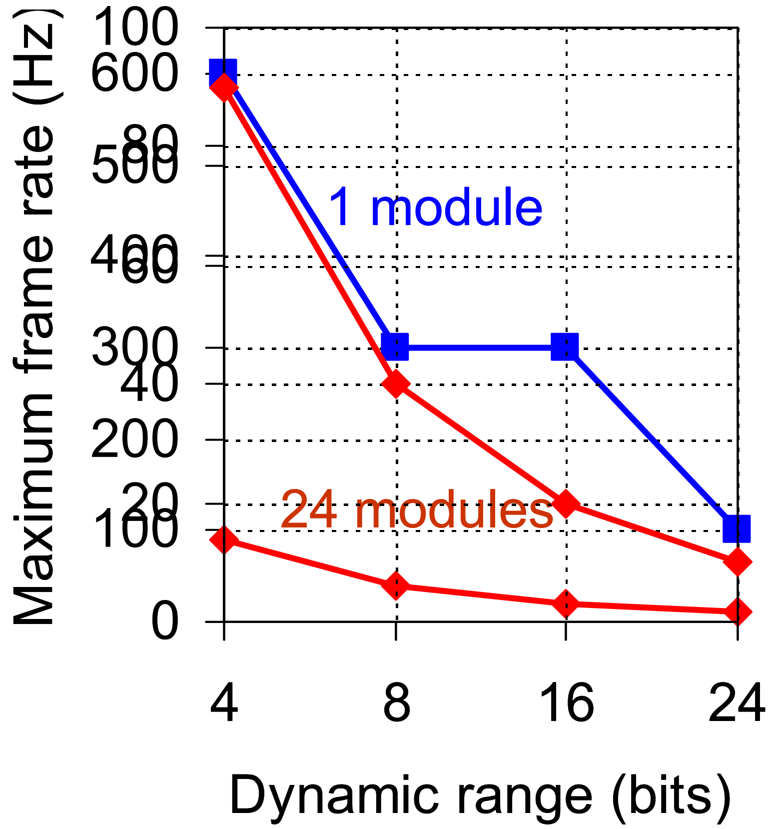
Frame rate

➔ The maximum frame rate is limited by the data transfer rate from the MCS to the PC

- Configurable number of modules to increase the frame rate



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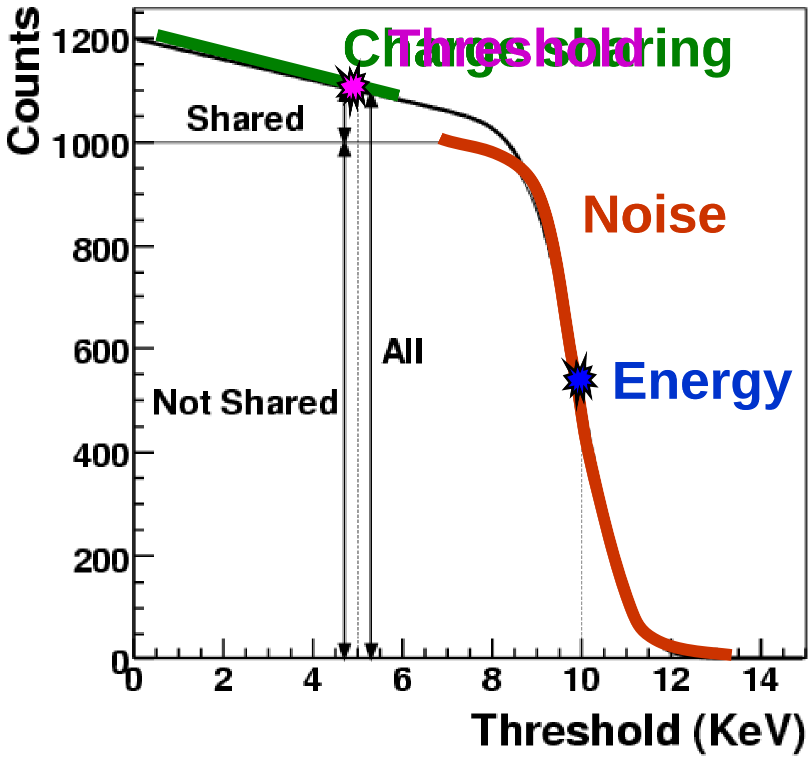
Energy calibration



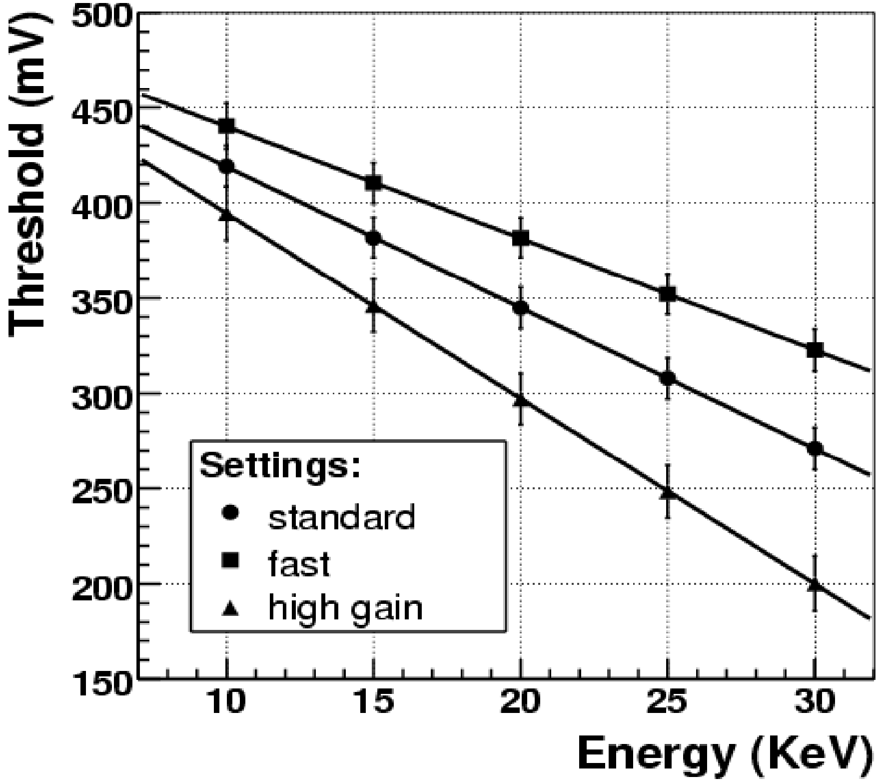
➔ A correspondence between threshold value and X-ray energy should be found

- The threshold is normally set at half of the energy value

➔ The comparator threshold should correspond to the same energy for all channels

- Energy resolution
- Count uniformity

Comparator threshold linearity

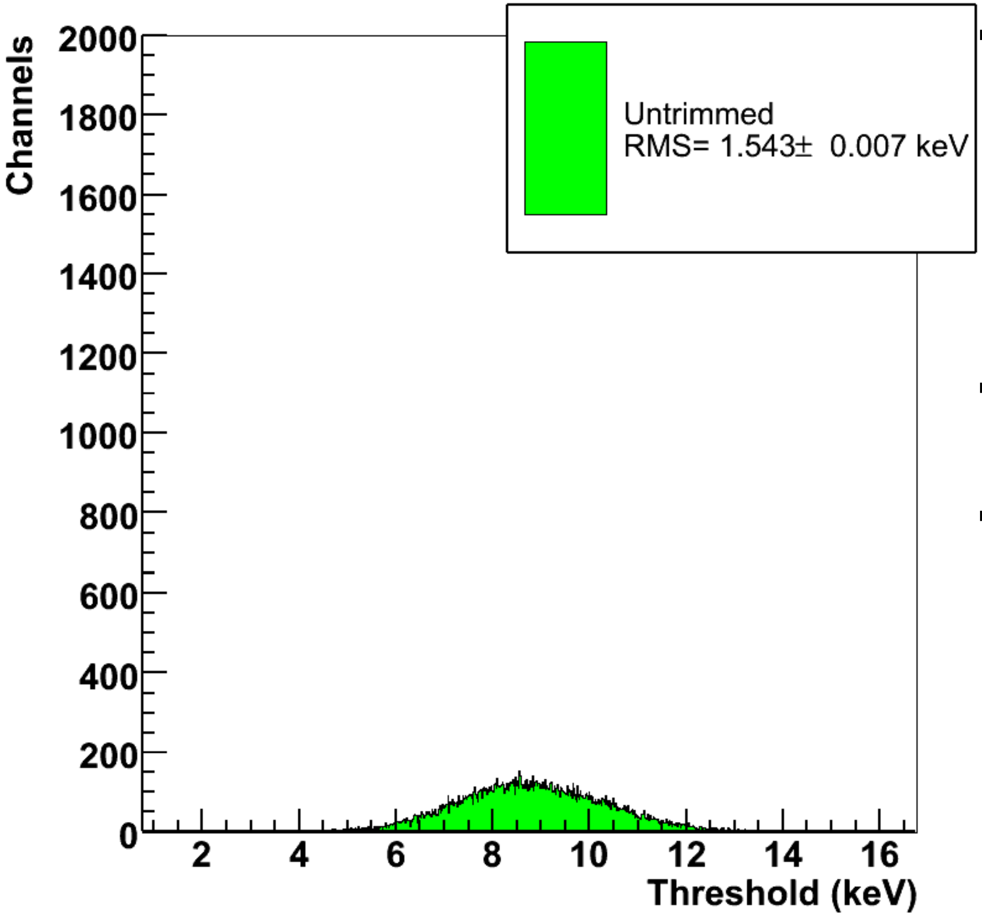


➔ Shaper and amplifier settings depending on the application

- Standard
  - E_{min} = 8 keV
  - $\Phi_{\varepsilon=90\%}$ = 1 MHz
- Fast
  - E_{min} = 10 keV
  - $\Phi_{\varepsilon=90\%}$ = 3 MHz
- High gain
  - E_{min} = 5 keV
  - $\Phi_{\varepsilon=90\%}$ = 300 kHz

➔ The comparator threshold is adjustable on a module base

Threshold dispersion

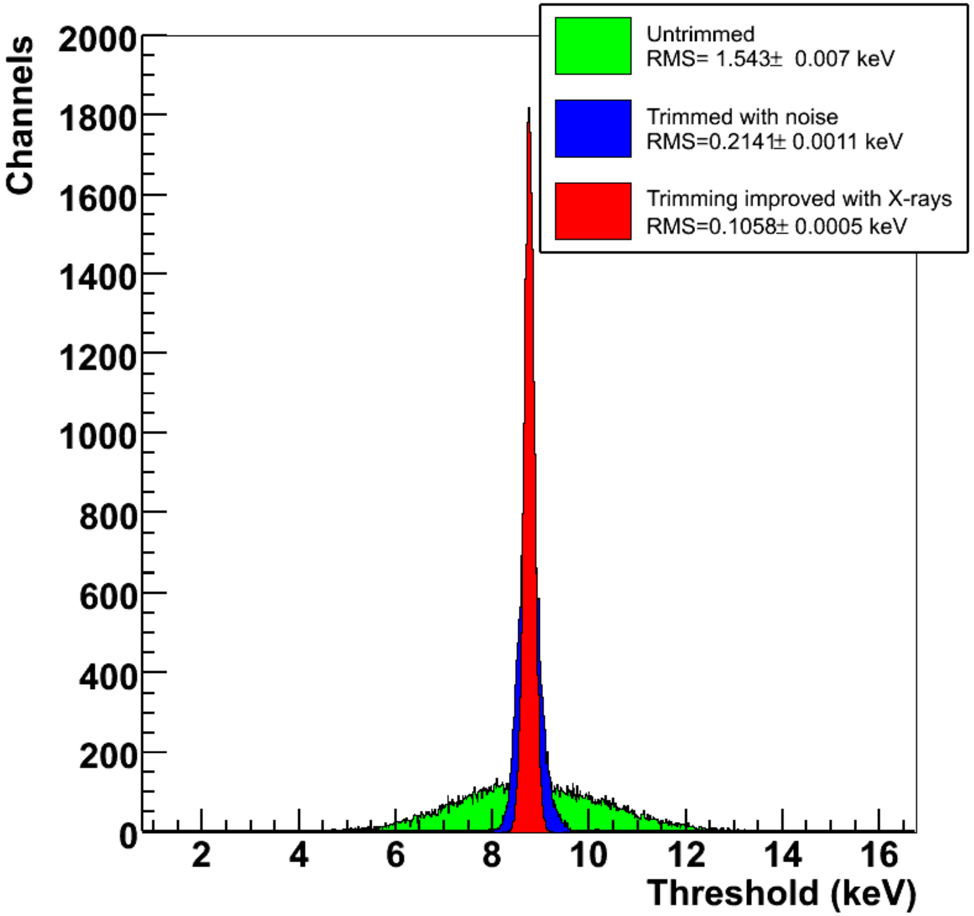


- ➔ The same voltage threshold does not correspond exactly to the same energy for all channels
  - Trimming the threshold for each channel internally with the internal 6 bits DAC
- ➔ Count differences due to the charge sharing slope
- ➔ A threshold dispersion larger than the electronic noise ENC places a limit on the minimal detectable energy

  $\sigma_{tot}^2 = ENC^2 + \sigma_{threshold}^2$

  $E_{min} = 10\ \sigma_{tot} = 15$ keV

Trimming methods



➔ Trimming with noise
- 1 minute
- $\sigma_{threshold}$ lower than ENC

➔ Trimming with X-rays
- Detector illumination by scanning in front of the beam
- 45 minutes
- $\sigma_{threshold}$ further improved

Count uniformity

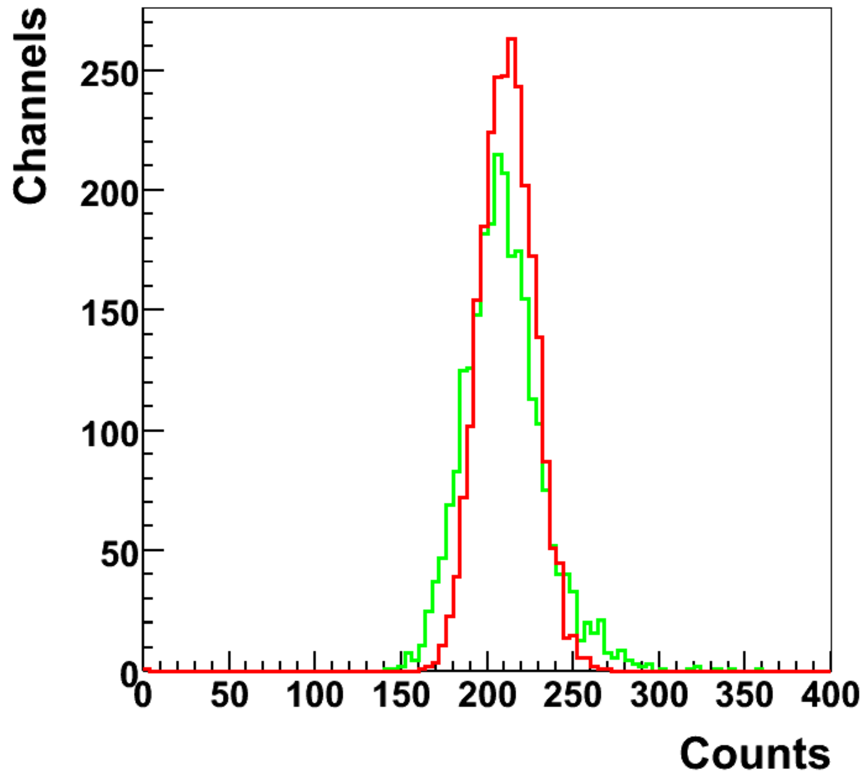
➔ A flat field correction of the data is still necessary to obtain a Poisson-like statistic

$\Sigma = \sigma_{counts}^2 / \langle counts \rangle = 1$

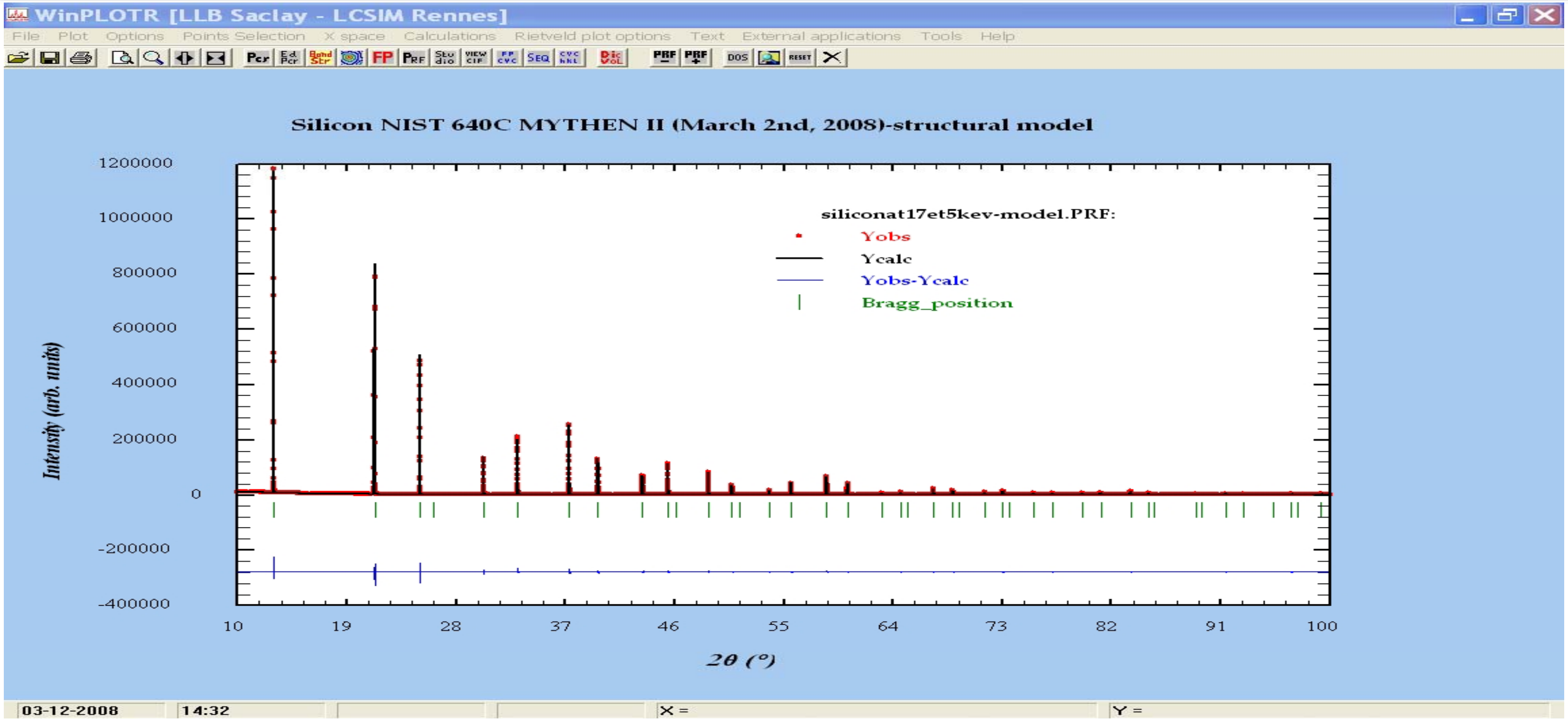
- $\Sigma_{untrimmed}$ = 2.6
- $\Sigma_{improved}$ = 1.2

➔ A further improvement of the trimming is possible by optimizing the DAC dynamic range

Energy=17.5keV
Threshold=8.75keV



Ritveld refinement



➔Structures can be determined from microstrip data

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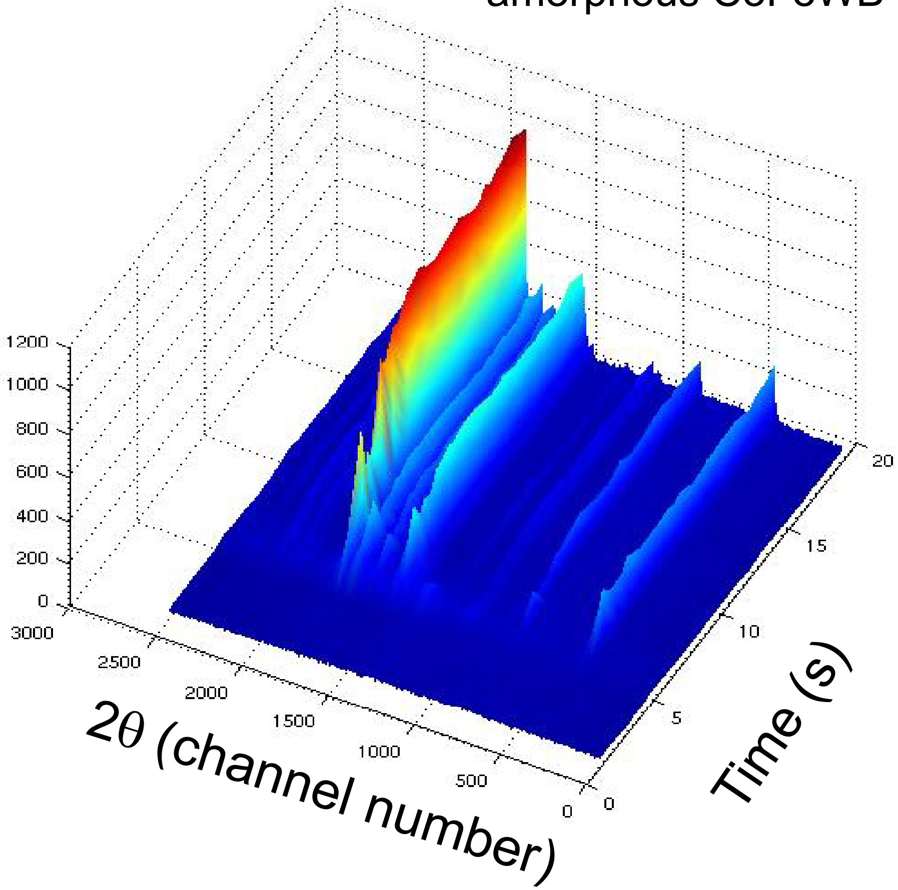
Crystallization of Co-rich alloys under microwave field

amorphous CoFeWB ⇨ Co(Fe) + $Co_{21}B_6W_2$



- ➔ Co-rich amorphous alloys for stable high temperature use as soft-magnetic nanomaterials
- ➔ Single-pulse microwave field application
- ➔ One-step nanocrystallization.

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EMPA Materials Science & Technology

Courtesy of S. Vaucher

Conclusions

➔ Calibration is essential for a proper operation of a large angular range detector
  - Energy calibration and trimming
  - Flat field correction
  - Angular calibration

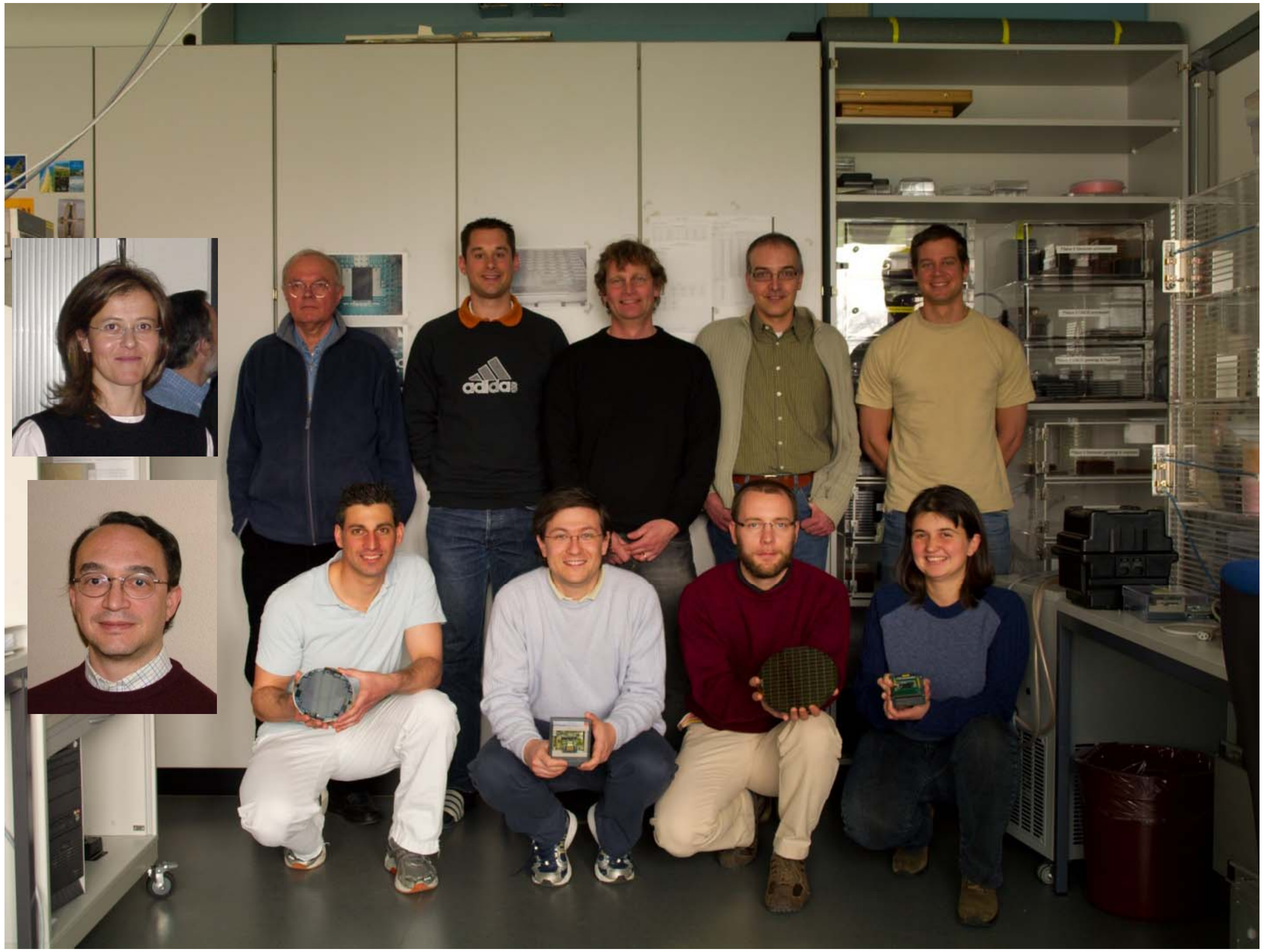
➔ The quality of the data acquired with the new Mythen detector is comparable to that obtained with the analyzer detector
  - Fast and time resolved measurements are also possible!
  - Not only Powder diffraction: Time resolved, pump and probe, FEMTO, Imaging, SAXS

➔ Mythen is a unique detector for 1-D X-ray applications
  - Large diffraction systems for synchrotrons
  - Smaller systems available also for lab diffractometers (Dectris AG)

Perspectives

- ➔ Faster data taking
  - 10 kHz for the single module
  - 1 kHz for the 24 modules
- ➔ Higher intensities
  - Higher count rate (time over threshold mode)
  - Integrating readout under test
- ➔ Higher spatial resolution
  - 25 μm pitch sensors wire-bonded on both sides
- ➔ Higher efficiency
  - Thick sensors
  - High Z-materials
- ➔ Mythen3 ASIC…

Thanks



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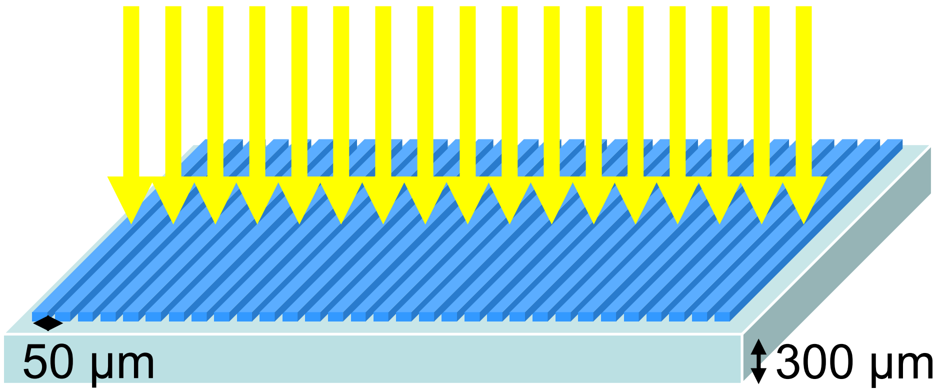
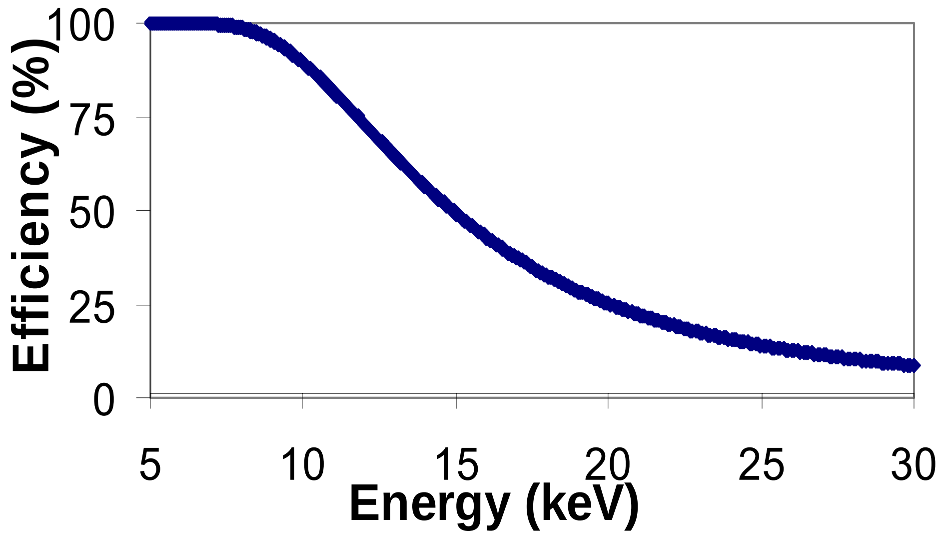
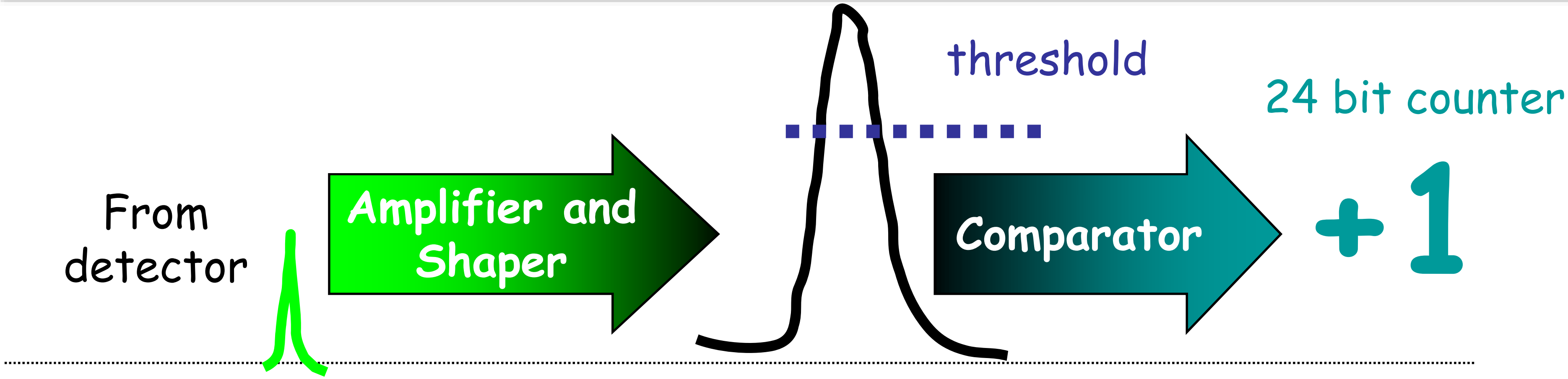
The microstrip sensor

- ➔Direct conversion of X-rays into electric charge
- ➔The spatial resolution is defined by the 50 µm strip pitch





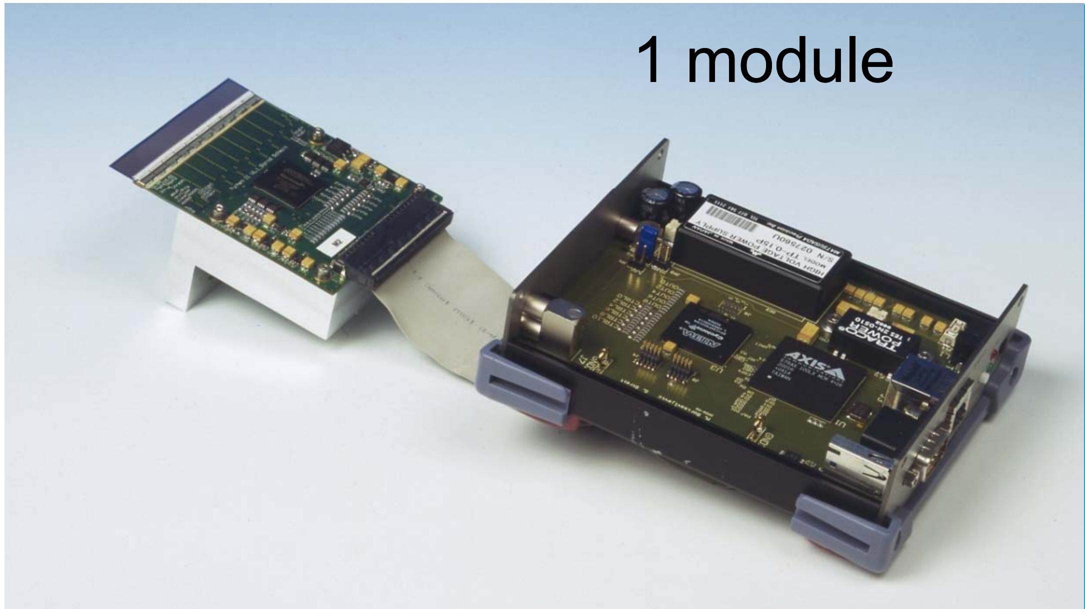
Single Photon counting

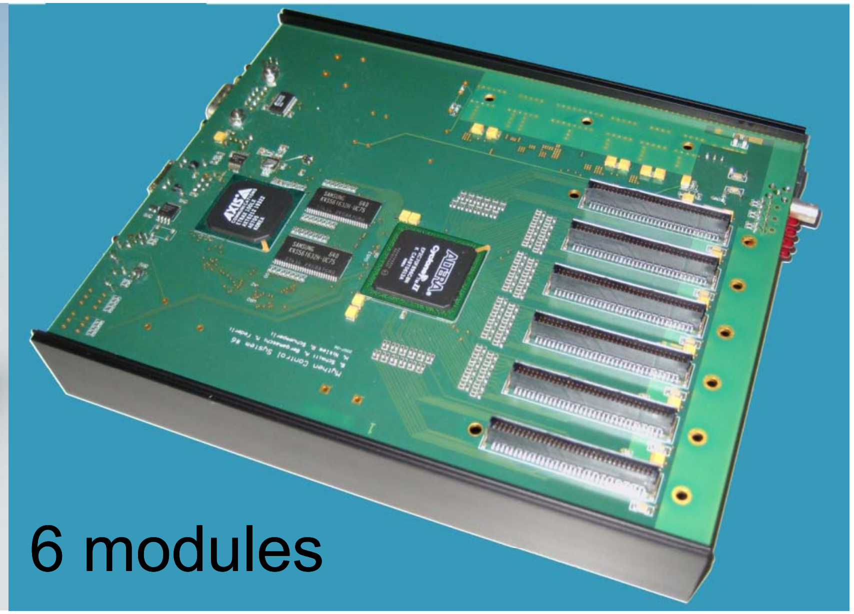


➔High dynamic range

- Essentially noiseless ➔Low signal applications
- No saturation ➔High dynamic range

Smaller systems

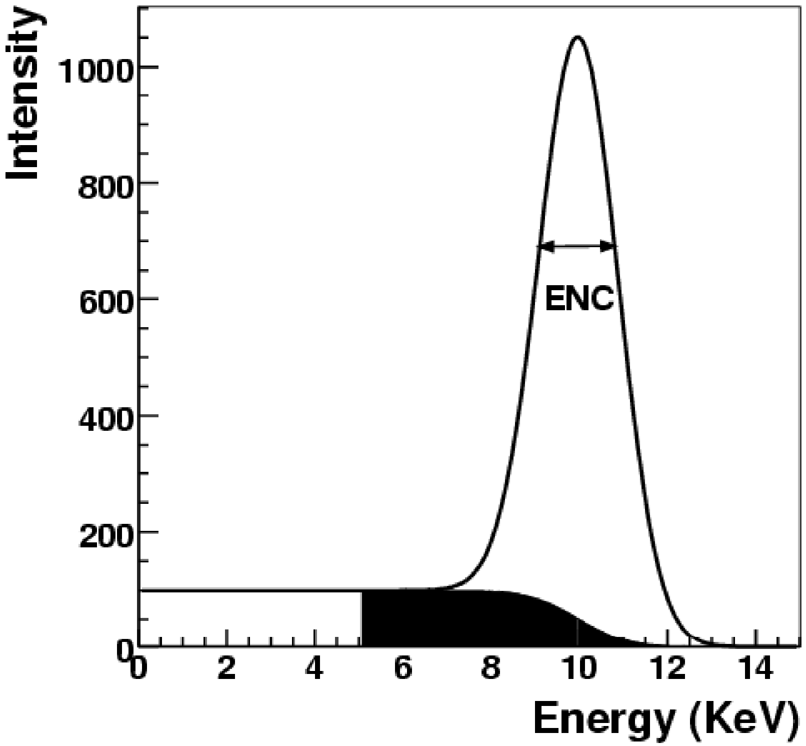




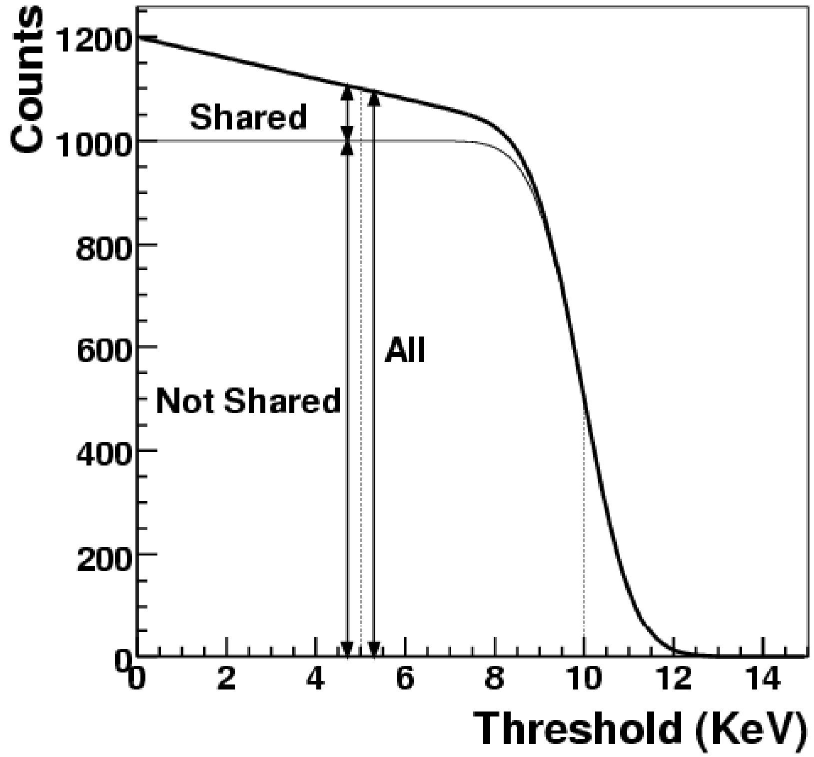
- ➔Faster frame rate
  - ➔Up to 600 fps 1 module - 4 bits

Expected spectra

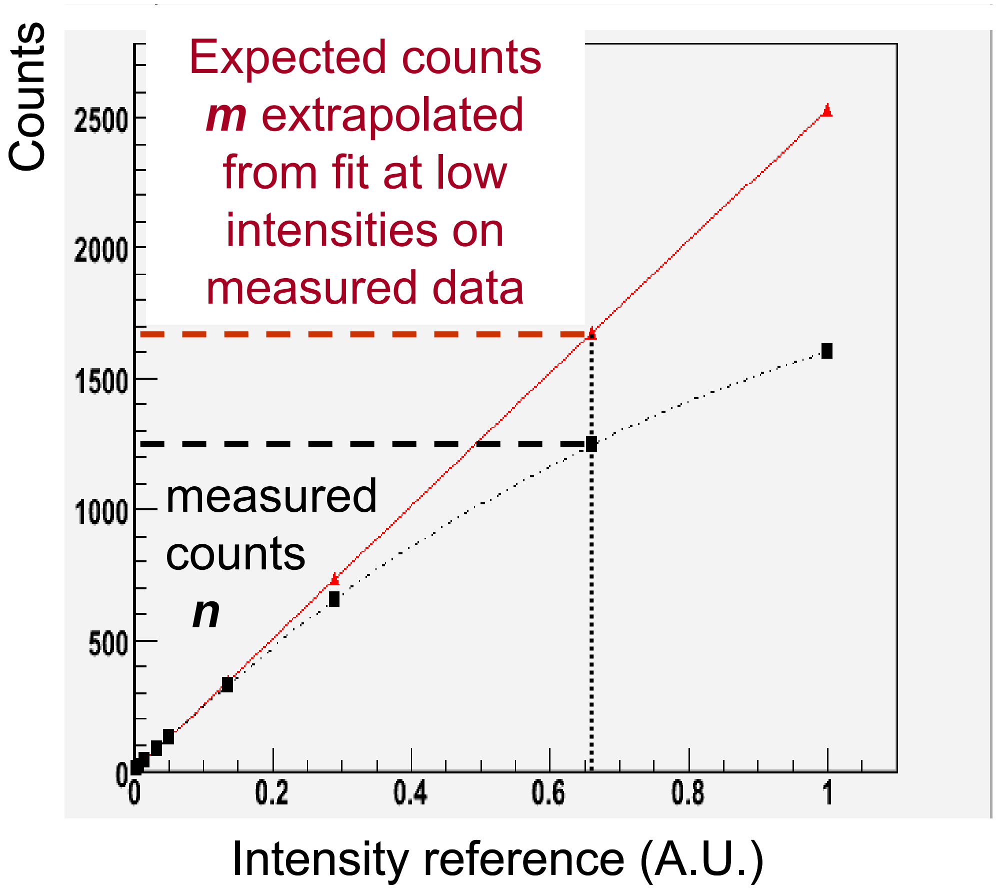
Spectrum



Threshold scan

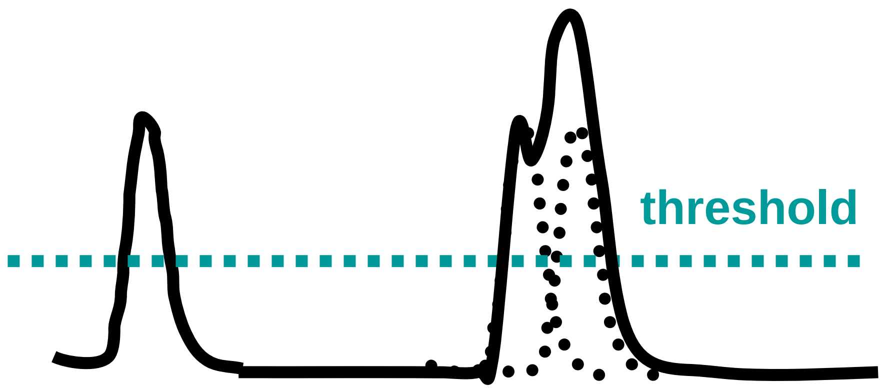


Loss of efficiency at high rates



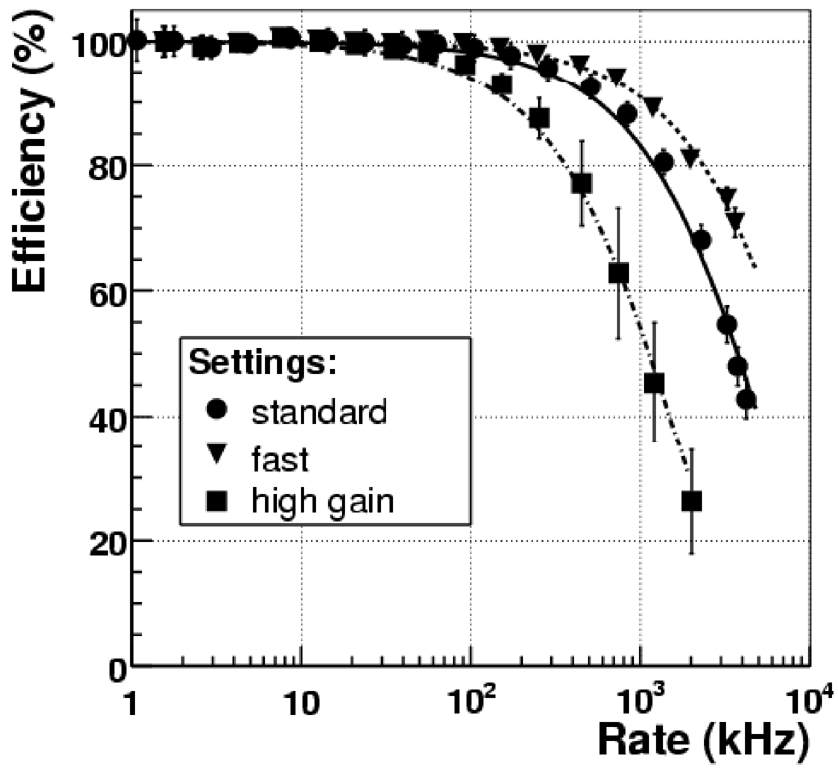
➔Reference for X-ray intensity given by a IC or by the detector background counts

Efficiency vs. rate

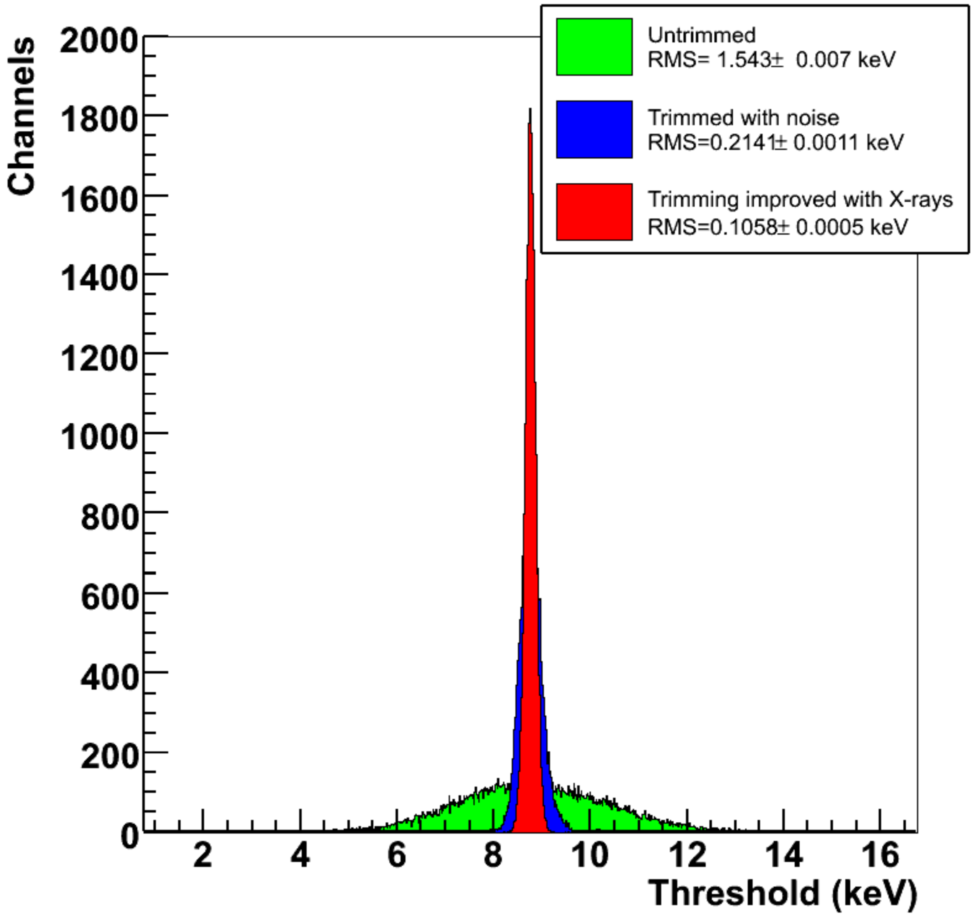


➔Negligible efficiency loss up to **200 kHz** (fast)

- τ_{fast} ~ 90 ns
- $\tau_{standard}$ ~ 170 ns
- $\tau_{high\ gain}$ ~ 600 ns



Trimming with X-rays



- ➔ X-ray energy set at the threshold level
  - Uniform illumination of the whole detector by scanning in front of an aperture
- ➔ Starting from the noise settings the trimbits are changed in order to equalize the number of counts for each module
  - 45 minutes due to the detector movement (5 steps)
- ➔ The threshold dispersion is reduced of a factor 15
  - A further optimization of the DAC dynamic range is possible

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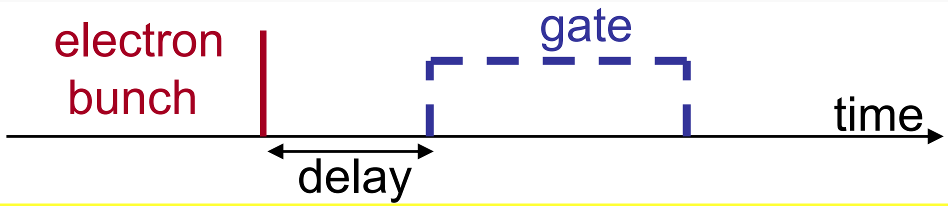
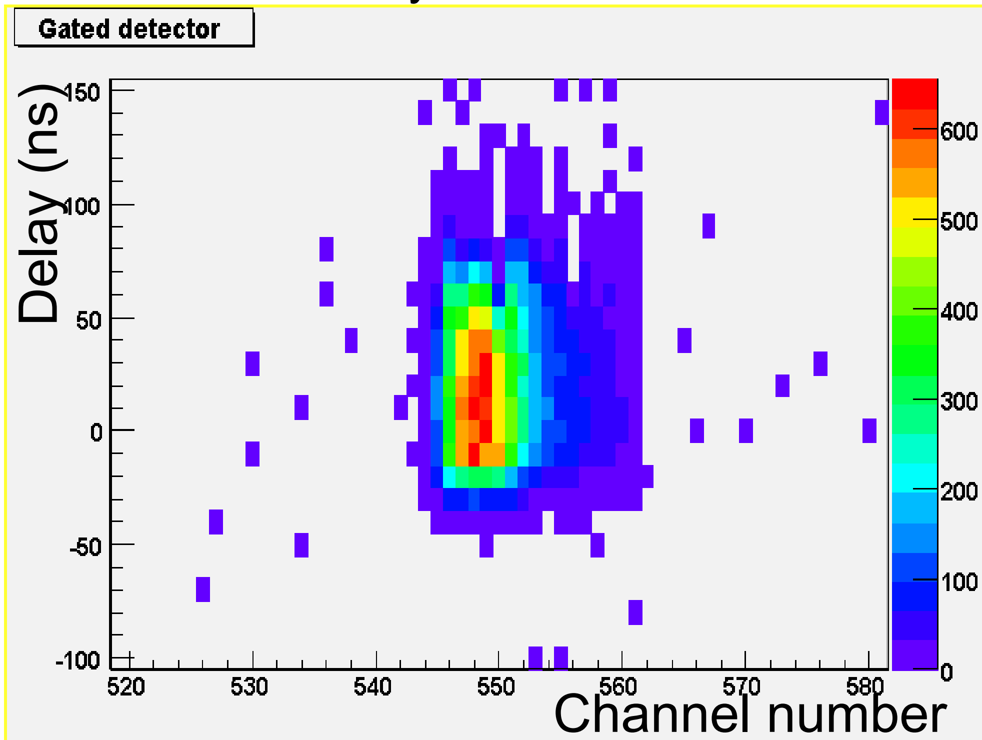
FEMTO commissioning

- ➔ The gating is needed only to separate the single bunch from the halo
- ➔ The time resolution is defined by the length of the bunch
- ➔ Each channel can count maximum 1 photon/bunch
- ➔ Need for normalization between pumped and unpumped measurements





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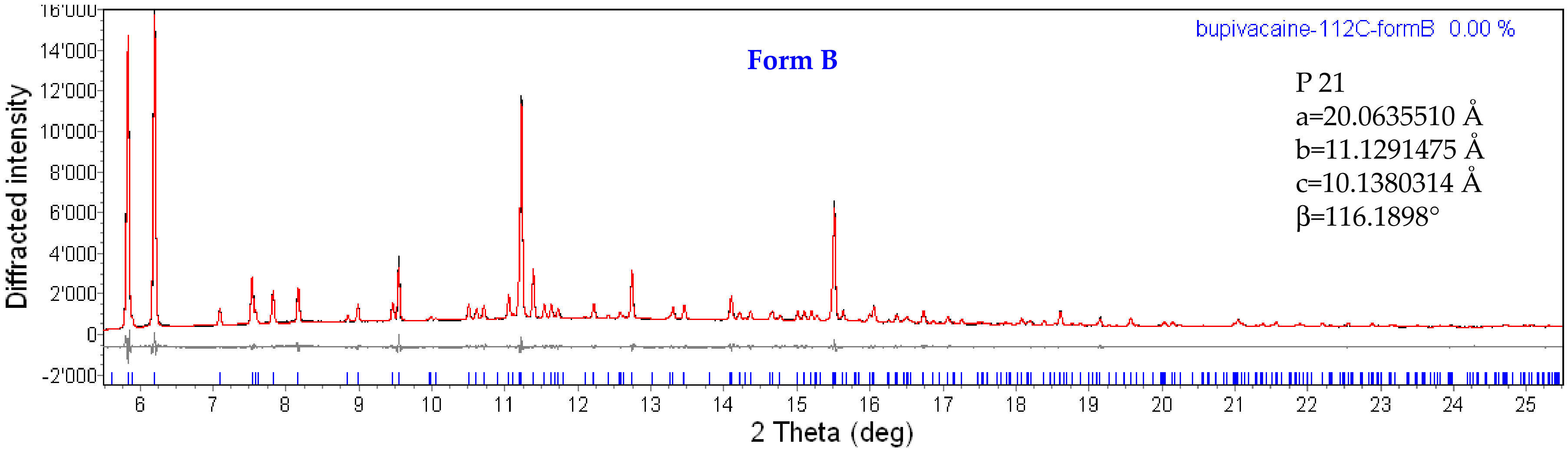
Sample in a variable enviroment

- ➔Structural solution of Bupivacaine (local anesthetic)
- ➔In-situ measurements in the furnace
- ➔Several cycles to check the reversibility of forms



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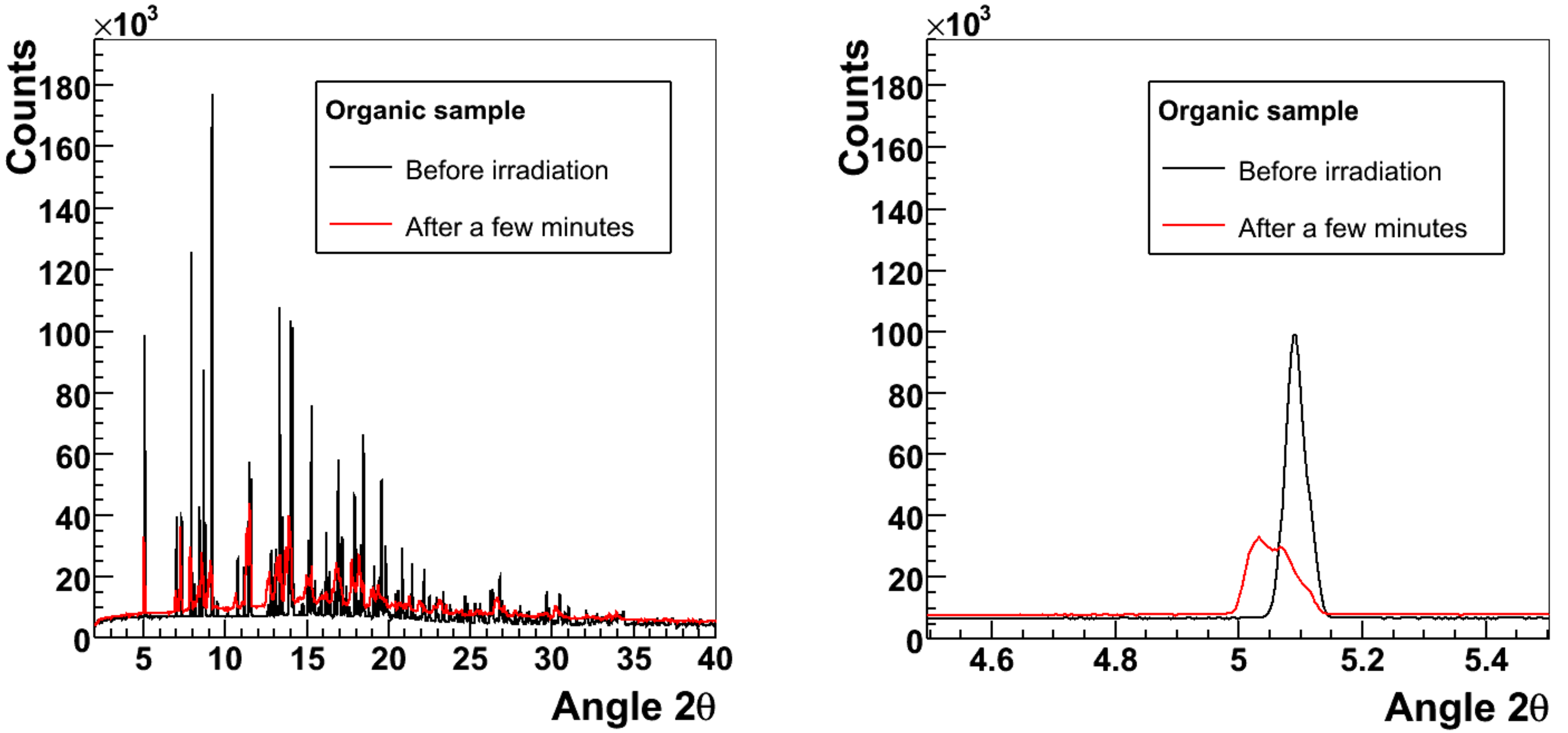
Radiation damage

Example of an organic sample from and industrial user before and after irradiation



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