XAFS and Solid state energy dispersive detectors

E. Fonda

Synchrotron SOLEIL, L'Orme des Merisiers, BP48 Saint Aubin,
91192 Gif sur Yvette, France

An end-user point of view

What is XAFS?

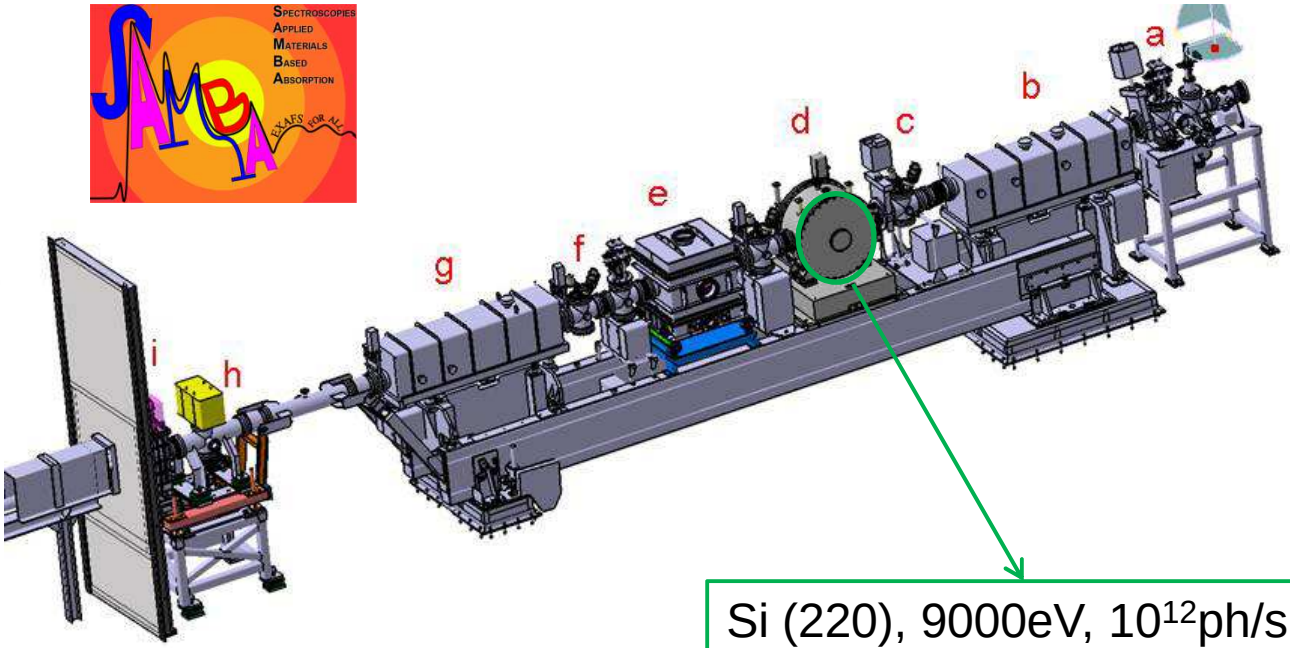
When these detectors are useful?

Different samples = Different constraints

We want more… but more what?

SAMBA: XAFS for all (4.5-43 keV)

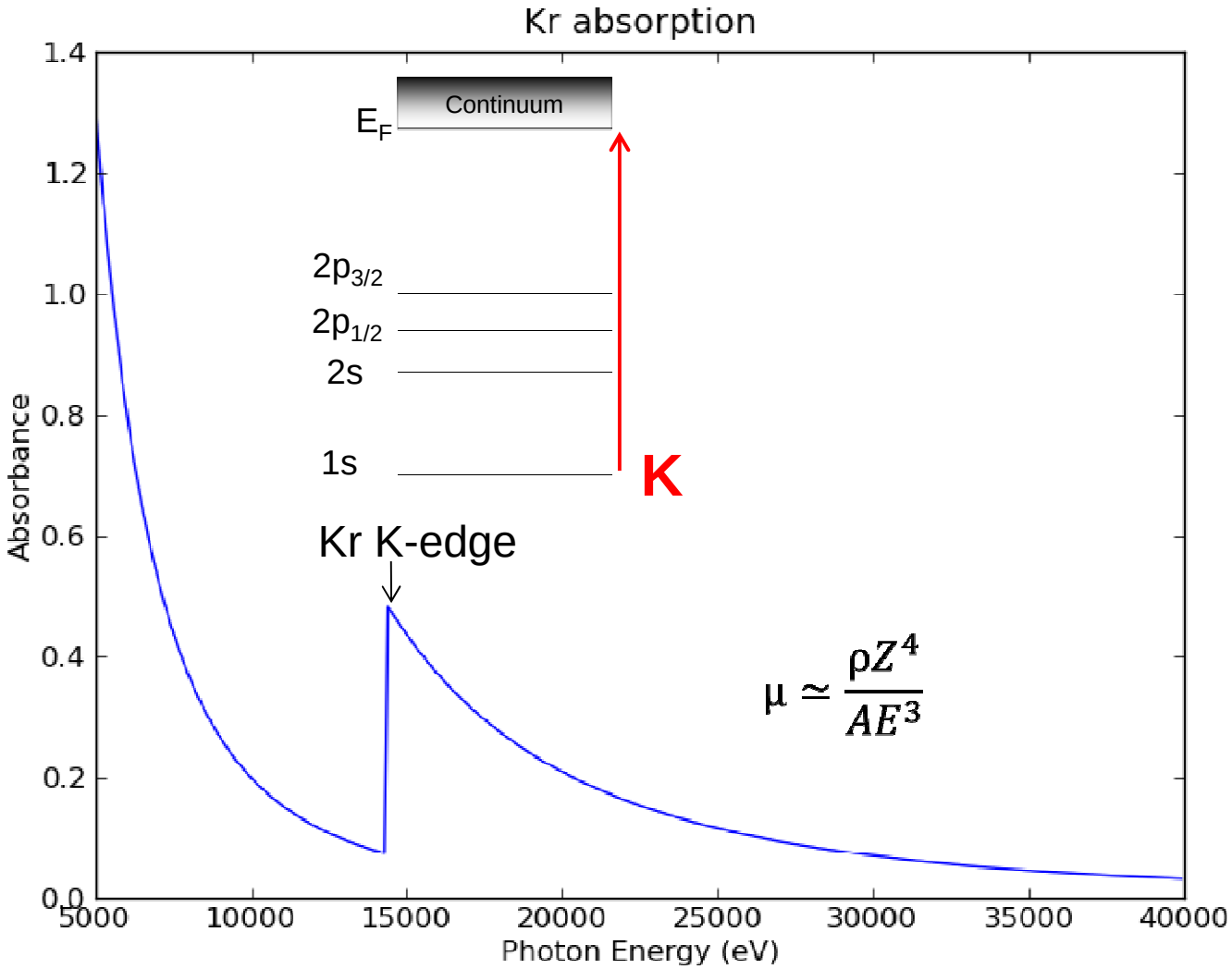
Emiliano Fonda (Sci.), Gautier Landrot (Sci.), G. Alizon (AI),
A. Zitolo (SOLEIL post-doc), A. Novikova (ANR funded PhD)

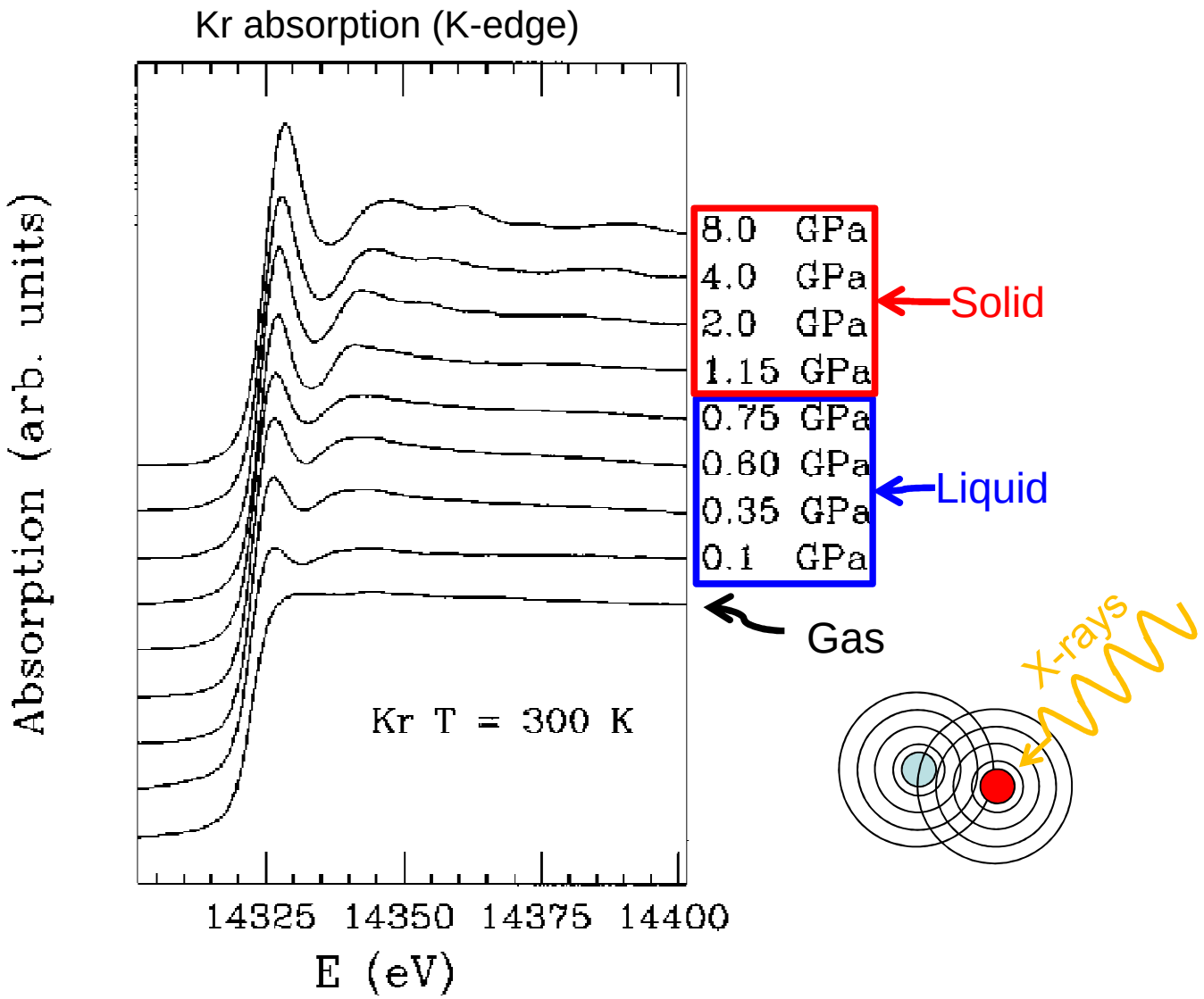


Fast scan mode:
2' → 5' XANES or 8' EXAFS

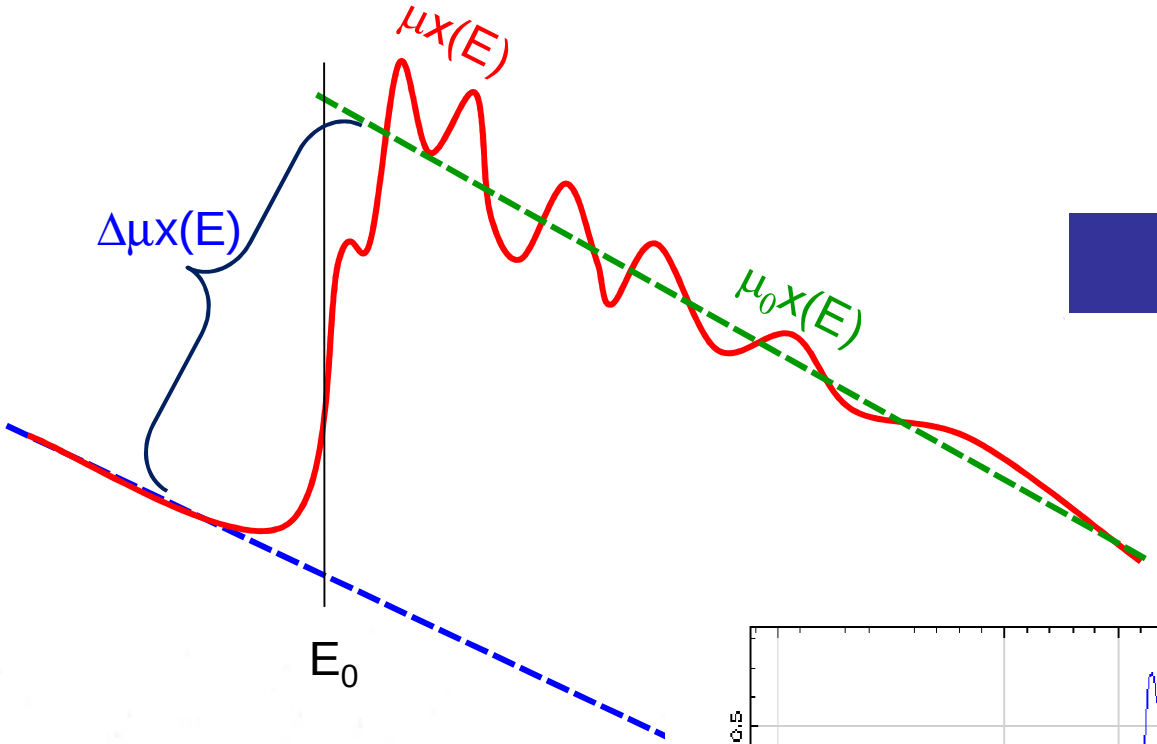
Step scan:
15' → 35' XANES + EXAFS

Horizon 2015-2016:
FlyScan for SAMBA
30" → 2' scans

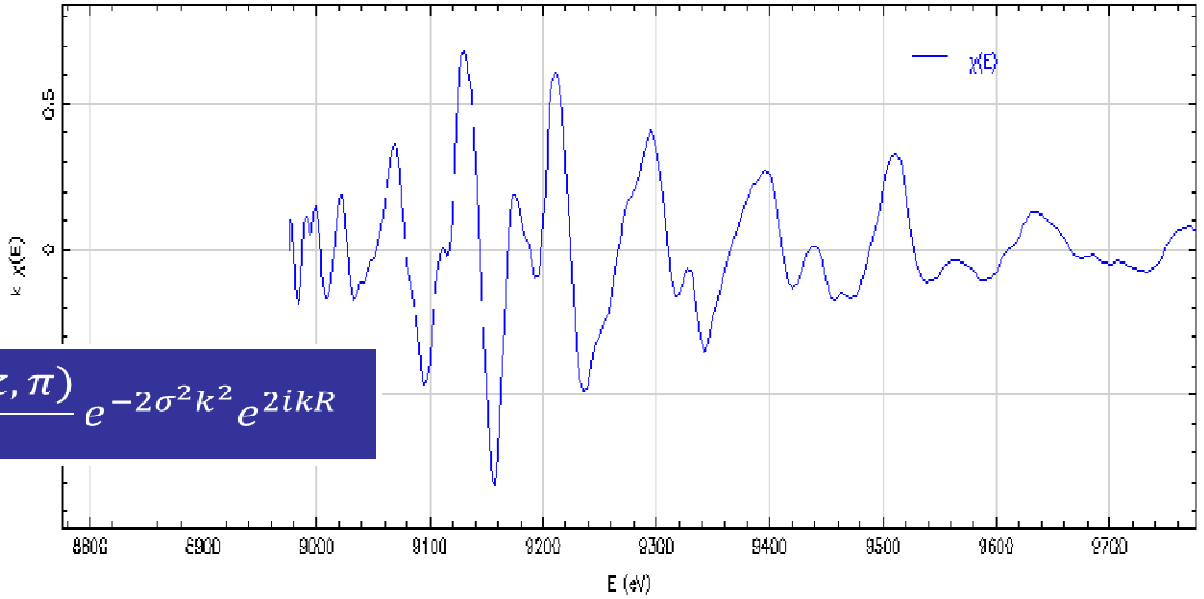

Kr absorption
Absorbance
Photon Energy (eV)
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
5000
10000
15000
20000
25000
30000
35000
40000
Continuum
E_F
$2p_{3/2}$
$2p_{1/2}$
2s
1s
K
Kr K-edge
$\mu \simeq \frac{\rho Z^4}{AE^3}$



A. Di Cicco, A. Filipponi, J. P. Itié and A. Polian, Phys. Rev. B 54 (1996) 9086-9098



$$\chi = \frac{\mu x - \mu_0 x}{\Delta \mu x}$$



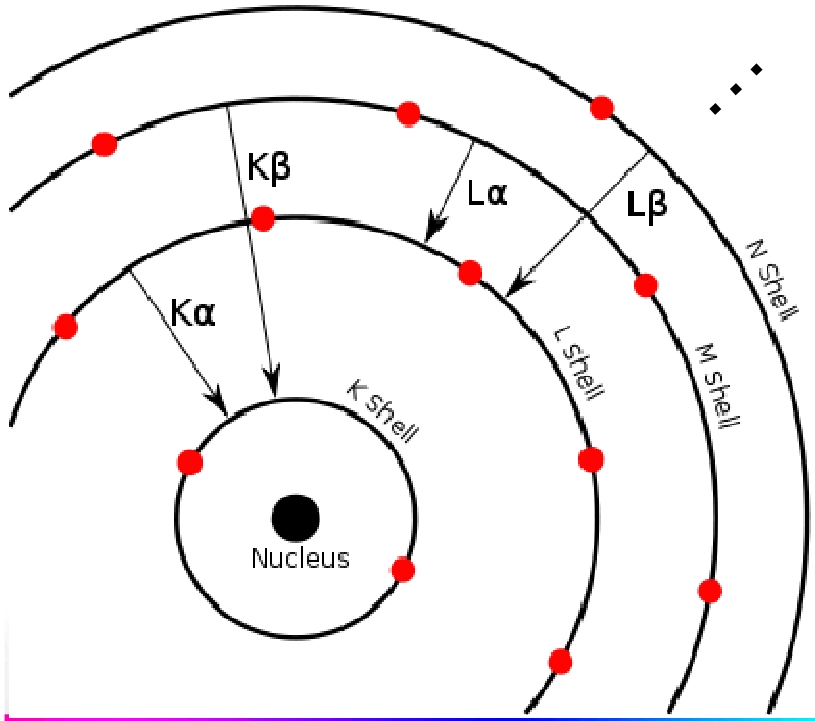
$$\chi(k) = \frac{S_0^2 N f(k,\pi)}{kR^2} e^{-2\sigma^2 k^2} e^{2ikR}$$

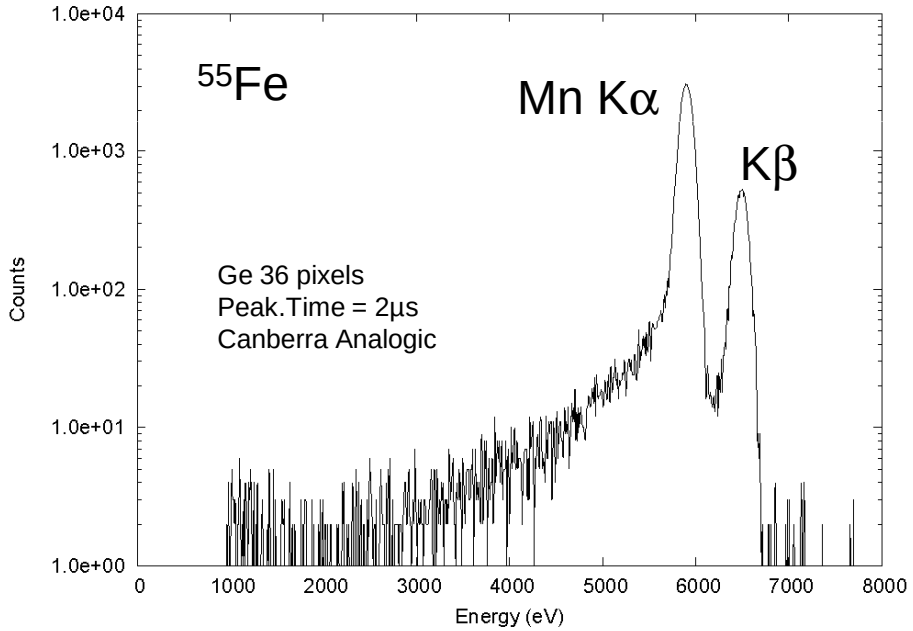
A fluorescence decay channel is not a measure of absorption jump, but its yield is proportional to absorption.

$$\text{e.g. : } \Delta\mu_K x(E)\ \alpha\ \Delta\mu_{F,K\alpha}(E)$$

We use fluorescence in case of:

- **Diluted specimens** (absorption jump is below detection noise)
- Too thick samples (μx → ∞)





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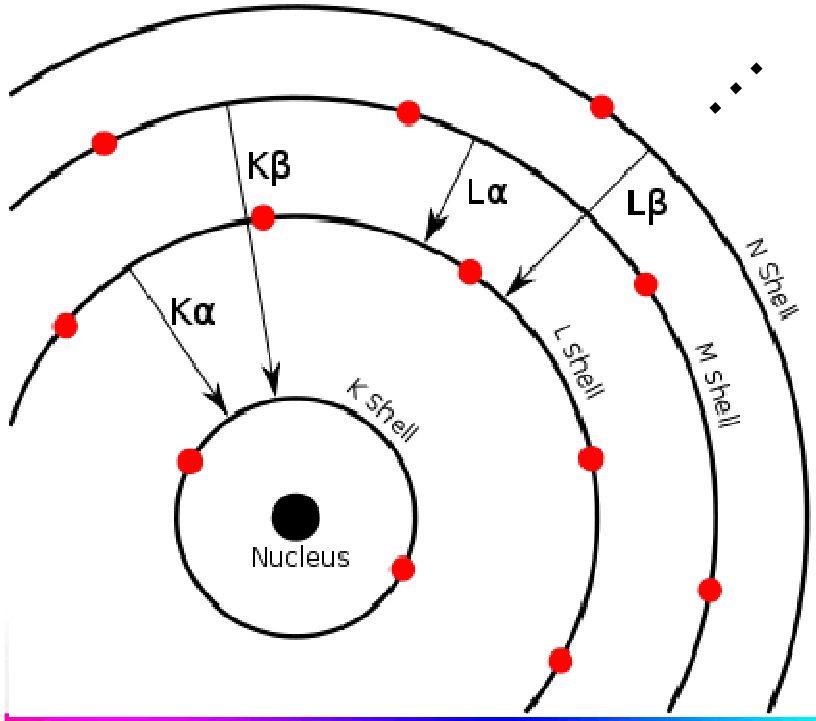


XAFS is a small modulation of absorption

Practically: $10^{-3}\Delta\mu(E) < \chi(E) < 10^{-1}\Delta\mu(E)$

$$N_{TOT} = N_f + N_b$$

b = background

$$N_{eff} = \frac{N_f}{1 + \frac{N_b}{N_f}}$$

Steve M. Heald, J. Synchr. Rad. (2015) 22, 436-445

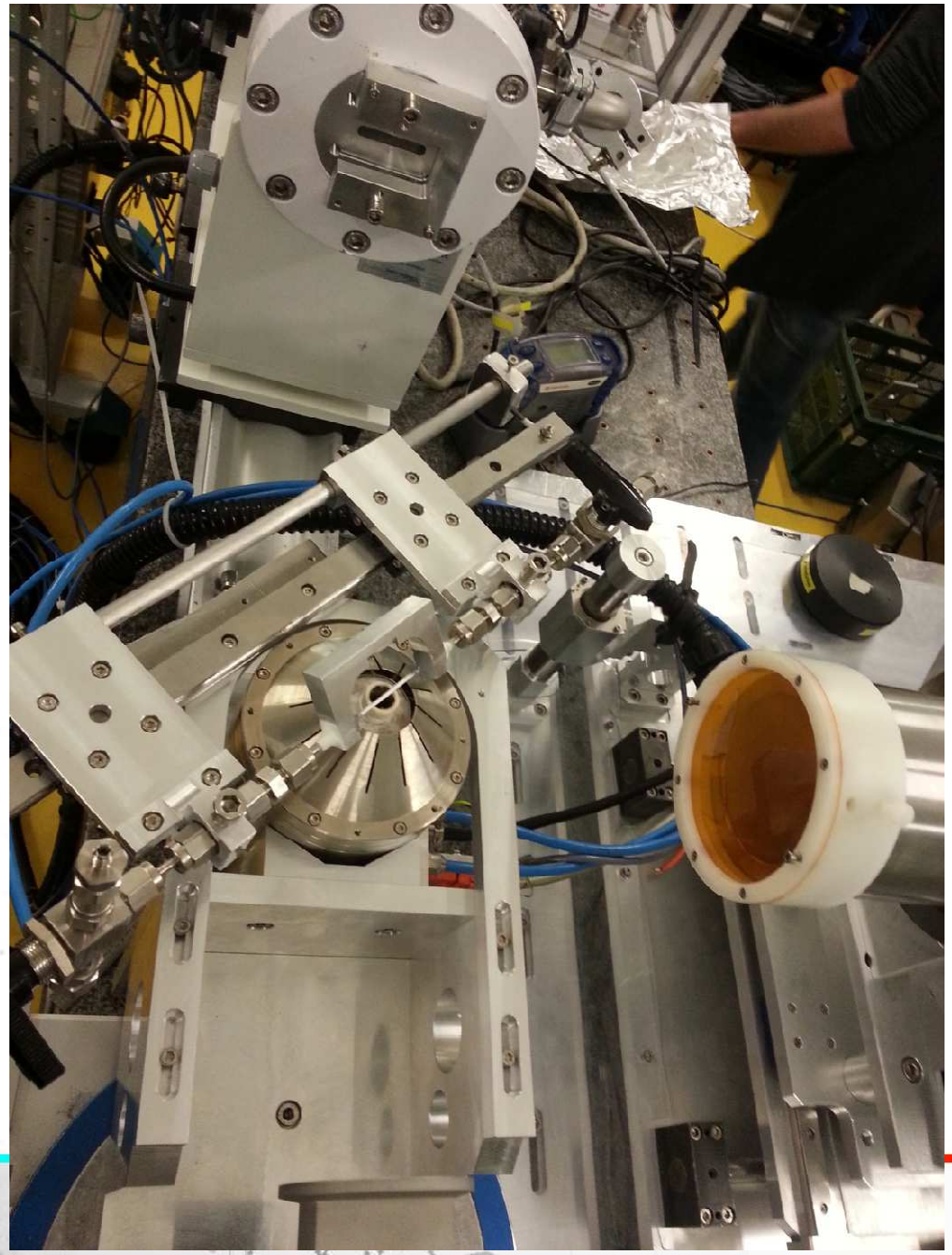
Background makes you count more time …

Real Catalysts are

Often precious
Hopefully very active

They must be diluted specimens

quartz capillary ovens
for operando measurements
up to 10-15 bars

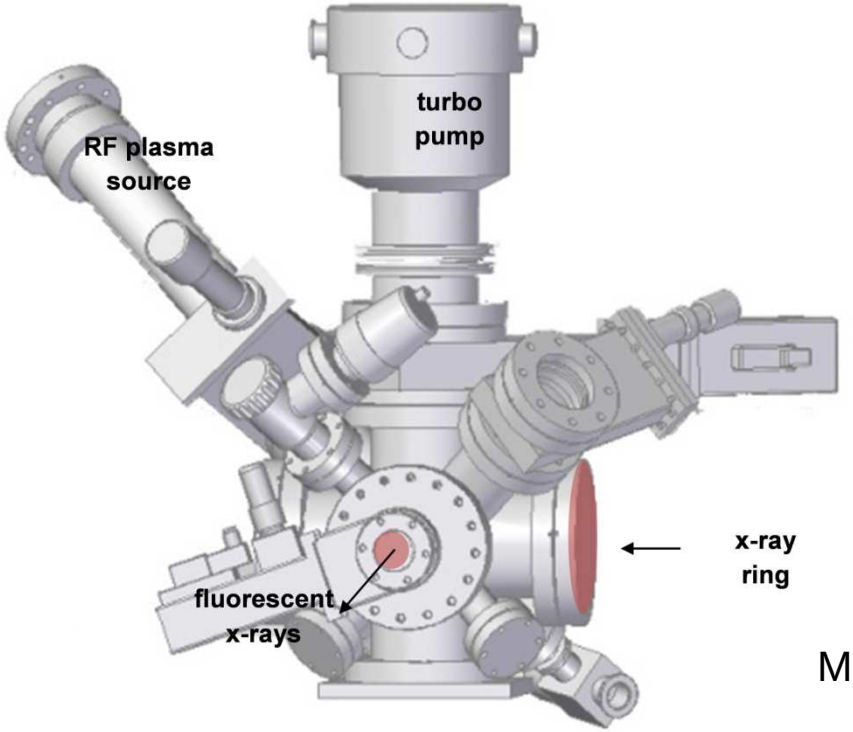


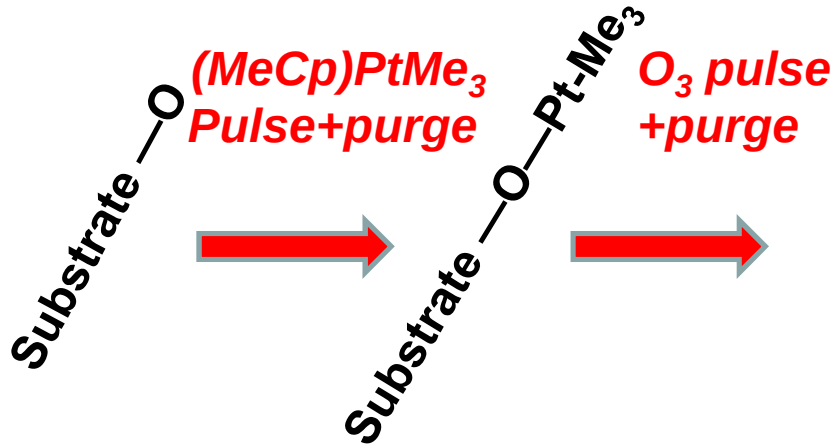
SOLEIL SYNCHROTRON

Thin films

In situ ALD growth of thin films: low temperature growth with O_3

C. Detavernier, H. Poelman, G. B. Marin (Dep. of Solid State Sciences and Lab. of Chemical Technology), Universiteit Ghent, Belgium





M. Filez et al., Catalysis Today **229** (2014), 2–13.

Dendooven et al., *J. Phys. Chem. C*, **117** (2013), 20557–20561

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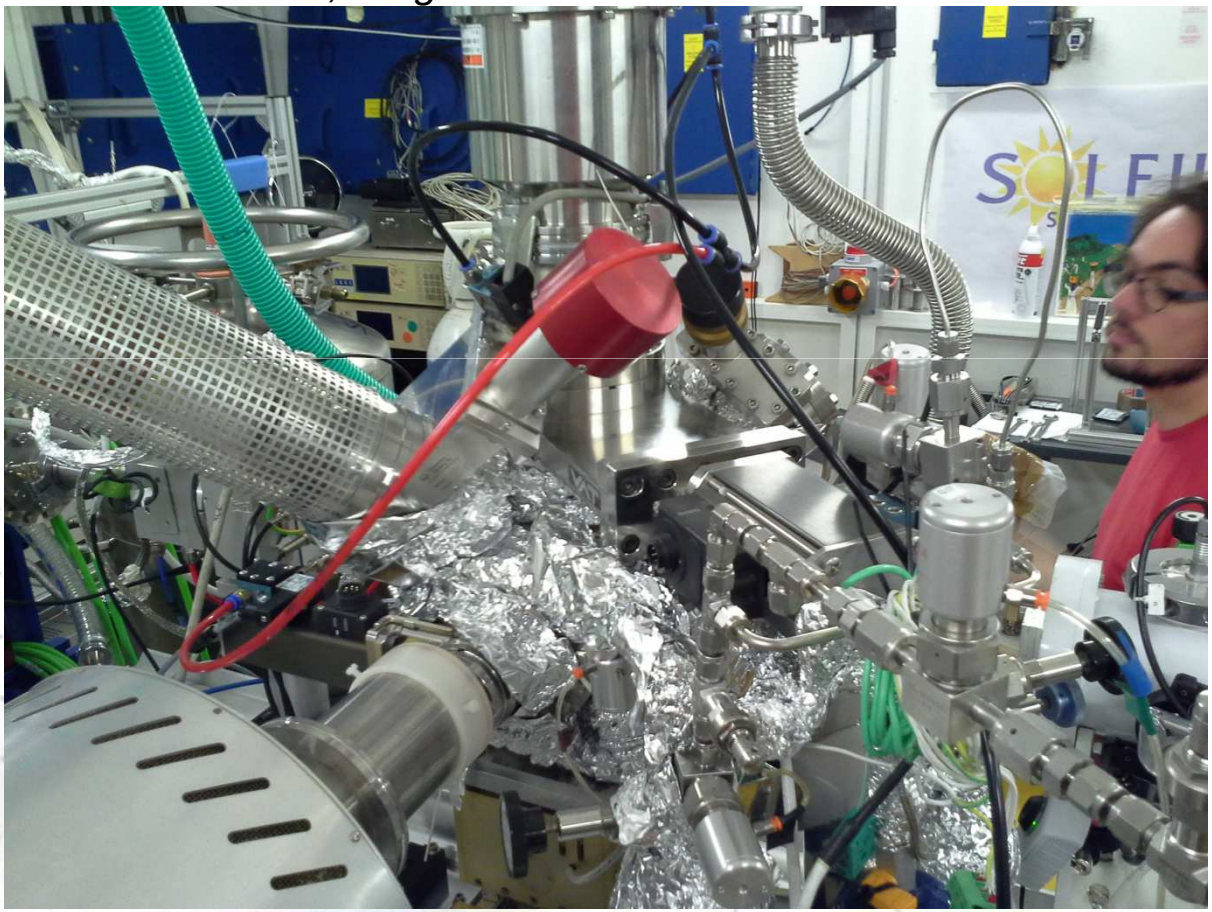
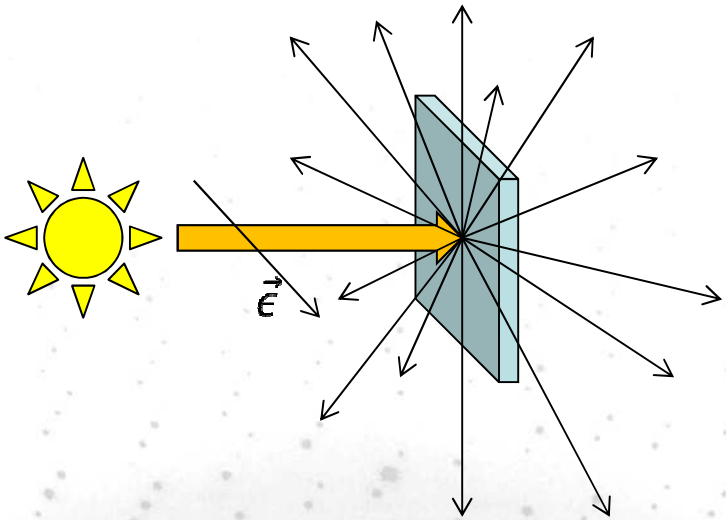
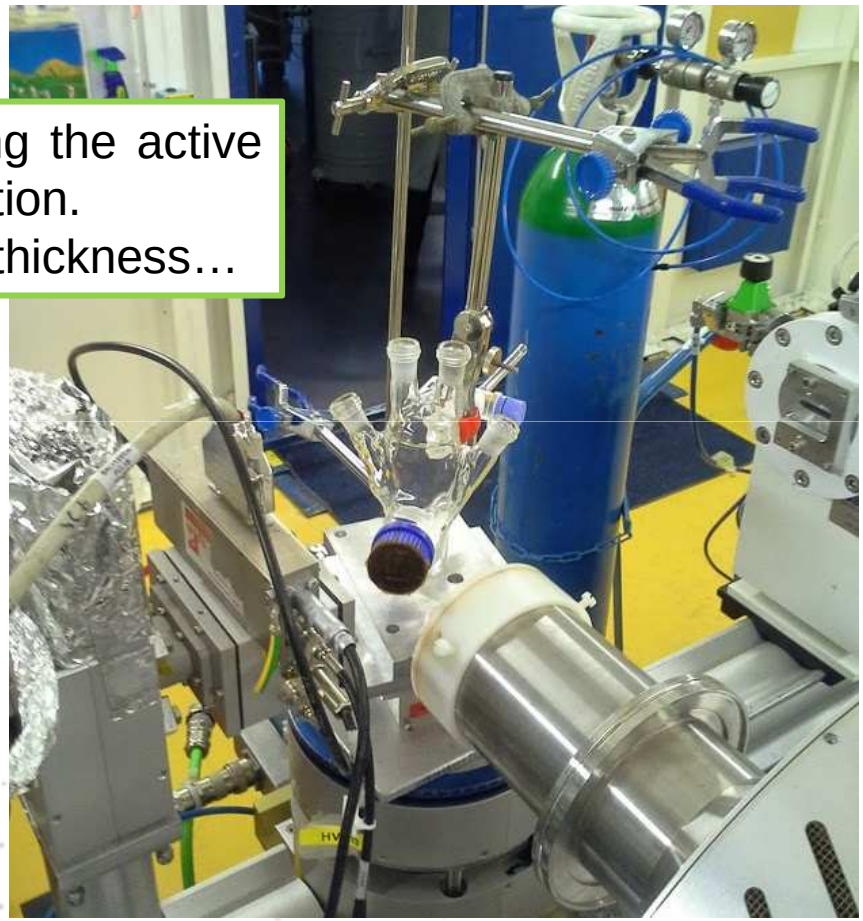


Electrochemistry (H_2 production…)

Non precious metal electrodes for H_2 production…

A carbon fiber electrode containing the active phase with back contact to the solution.
X-rays will cross the whole sample thickness…



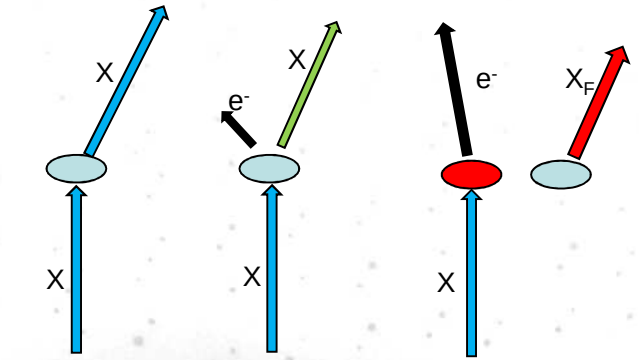


- We have to separate lines to reduce noise
- We do not want to observe modulations of another specie

We observe at nearly 90° and collinear with light polarization, but

- polarization is not 100% linear
- detector size is not negligible

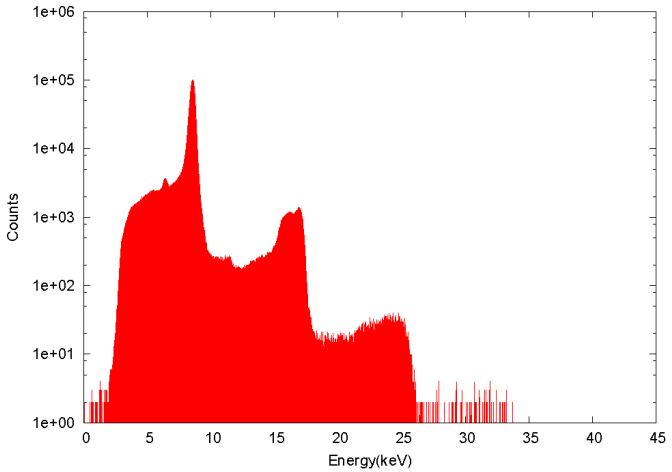
- the elastic (Rayleigh) peak moves in energy during a XAFS measurement
  - tailing of the elastic peak introduces a deformation of spectrum
  - Rayleigh scattering have a close friend: Compton scattering



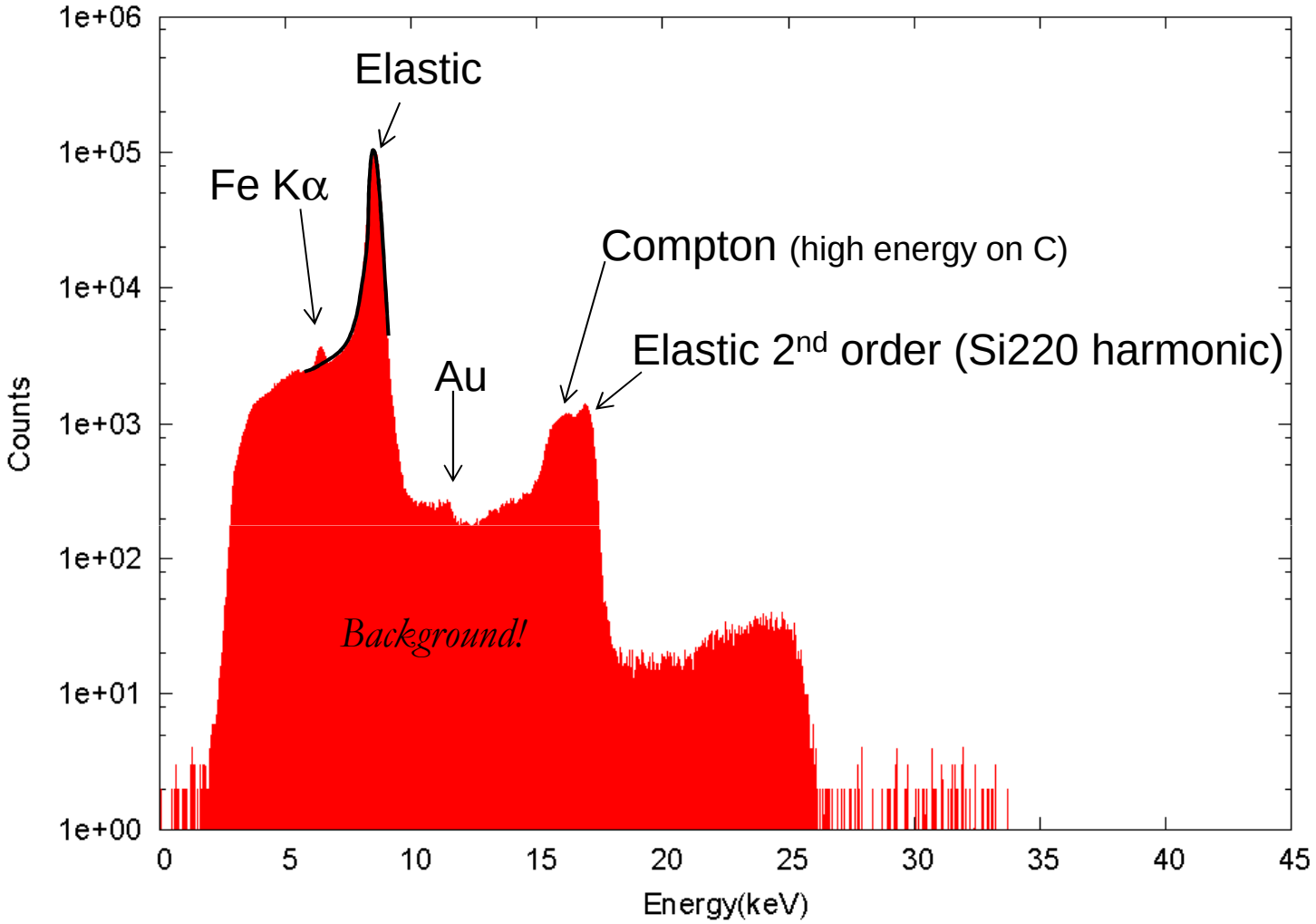
Rayleigh scattering

Compton scattering

Fluorescence: core hole creation and emission



Fe diluted in a carbon fiber electrode



Peaking Time 2µs, 100kcps, 60s (1 pixel over 36 shown)

Concluding remarks on common samples of high interest:

Thick light matrices (C, Si, Al_2O_3)

→Strong Compton at high E → Increase background counts
filters are not enough

Light crystallized matrices

→Diffraction peaks → strong modulation of ICR with E
requires very good estimate of ICR/OCR
high dynamics

Mixtures of elements… and we are interested in dopants!

→Resolution

It is often impossible to work in ideal total reflection or small emerging angles

Often samples cannot sustain beam

(many more than you can suspect of…)

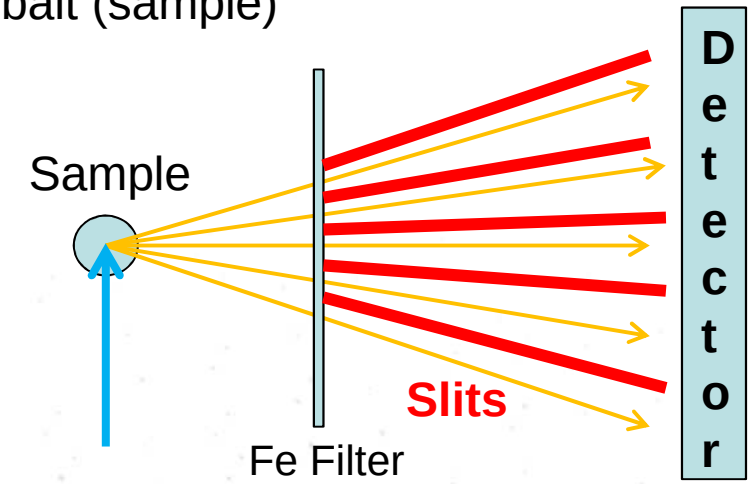
Other very simple tools in the box

"Z-1" filters

e.g.: an iron filter for cobalt to reduce elastic peak

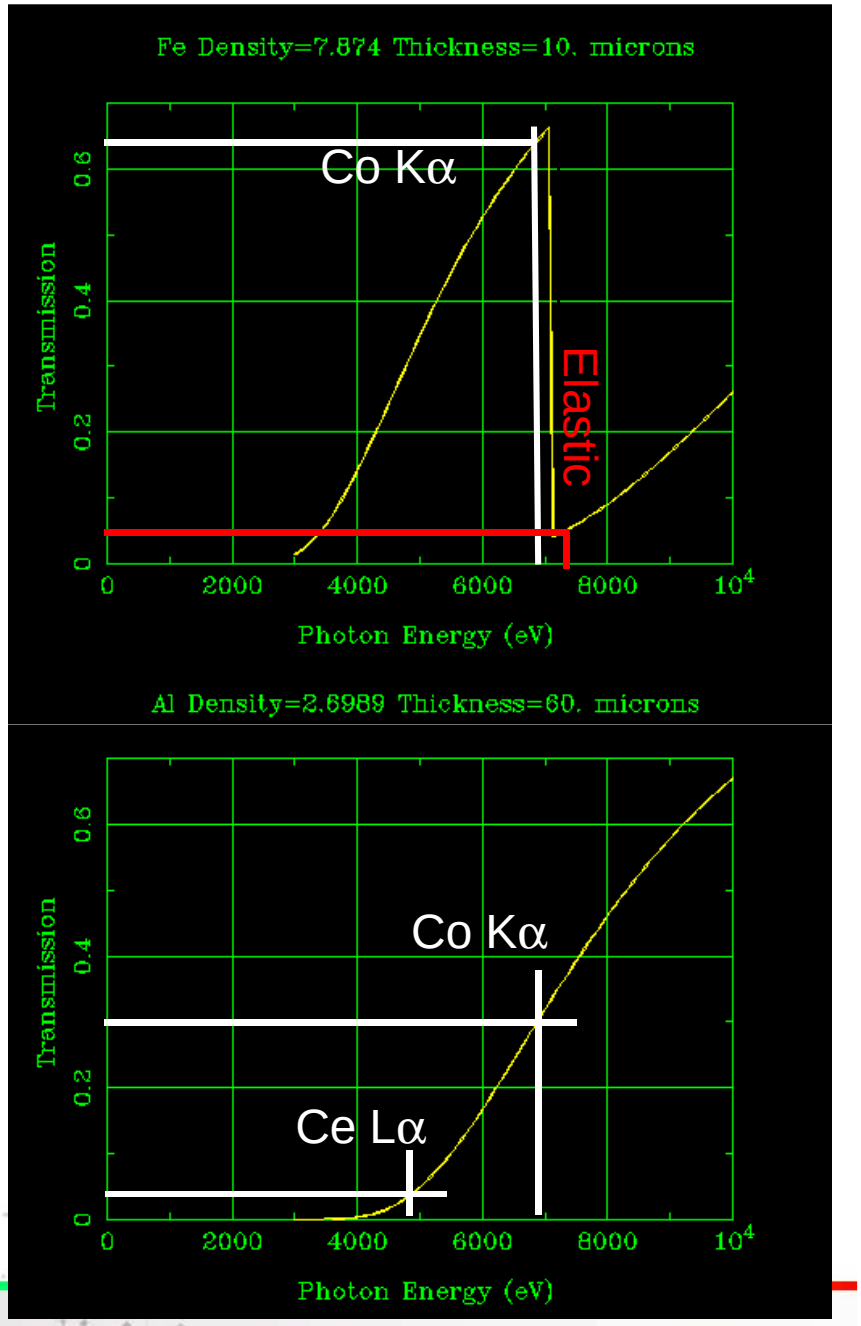
Soller slits:

minimize iron fluorescence (filter) over cobalt (sample)



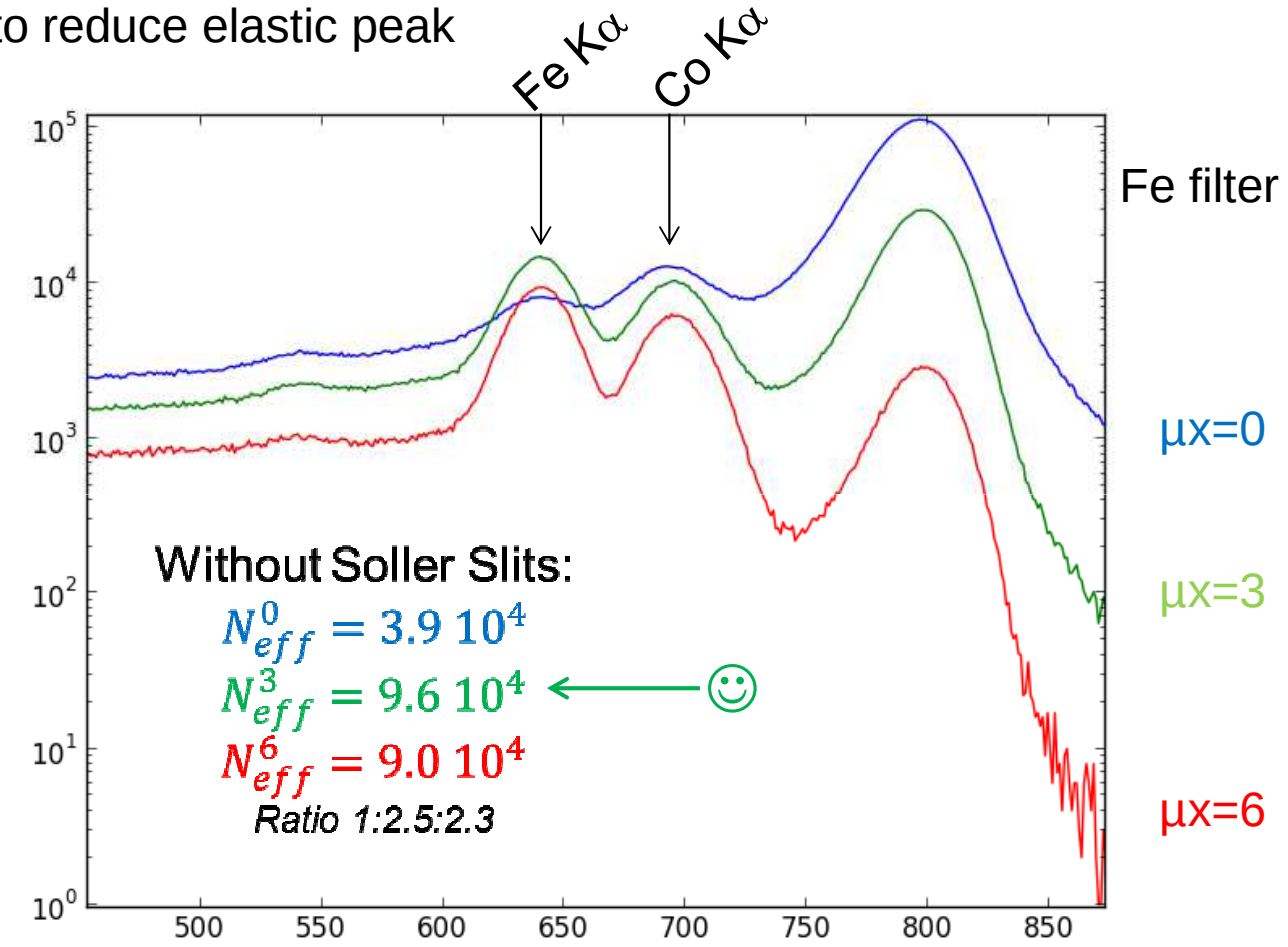
Light Z absorbers:

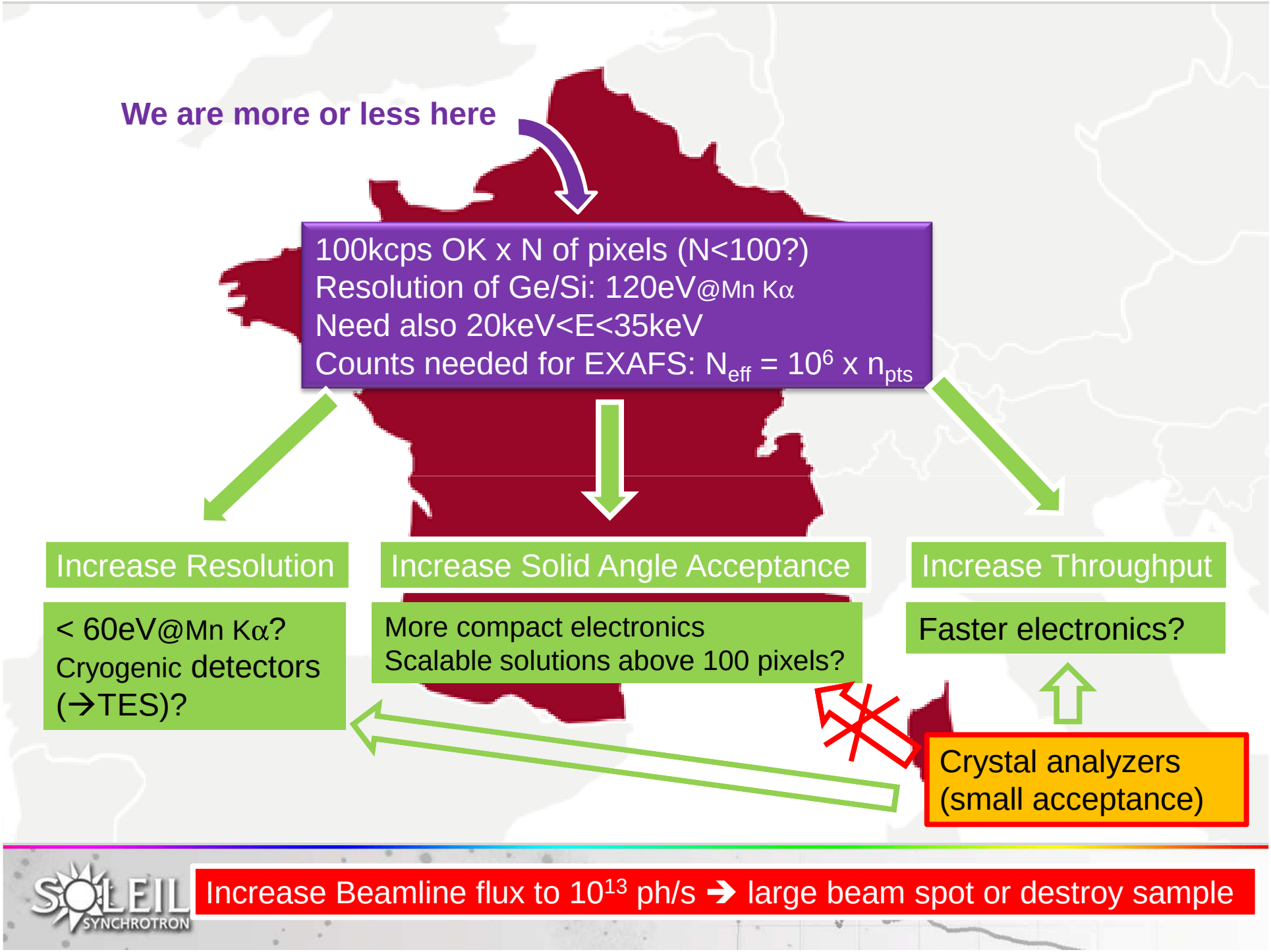
e.g.: reducing Ce L fluorescence versus Co K with a 60µm Al foil (ratio of ~6 improvement)



"Z-1" filters

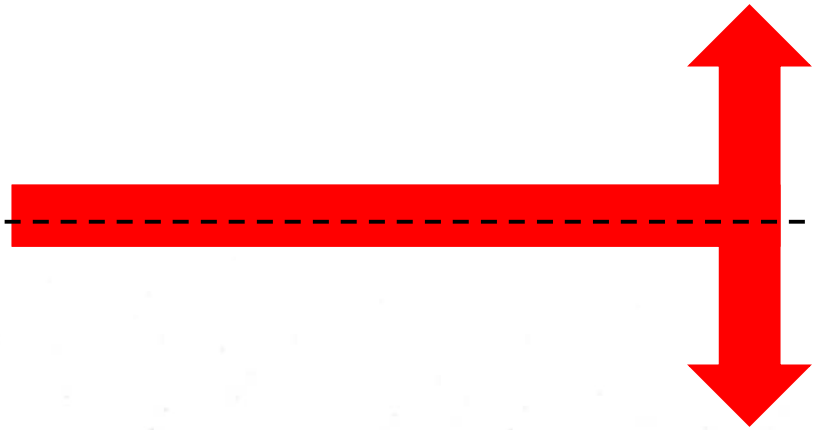
e.g.: an iron filter for cobalt
to reduce elastic peak




We are more or less here
100kcps OK x N of pixels (N<100?)
Resolution of Ge/Si: 120eV@Mn Kα
Need also 20keV<E<35keV
Counts needed for EXAFS: $N_{eff} = 10^6 \times n_{pts}$
Increase Resolution
< 60eV@Mn Kα?
Cryogenic detectors (→TES)?
Increase Solid Angle Acceptance
More compact electronics
Scalable solutions above 100 pixels?
Increase Throughput
Faster electronics?
Crystal analyzers (small acceptance)
Increase Beamline flux to 10^{13} ph/s → large beam spot or destroy sample

Objective: reduce noise by increasing acceptance, keeping background steady

Build an X-ray “camera” with (n x n) pixels and ≤120eV resolution
max n ? (MAIA has 384 channels)



Where is larger “market”?
XAFS, time resolved XAFS
Spectroscopic imaging

Where is novelty?
Time resolved RIXS

Keep actual performances, but increase resolving power
What is the limit → 30eV? 60eV?
Technology gap? STJ, TES…?

Objective: 1) reduce noise by decreasing background (<60eV, ideally 30eV)
2) opening new possibilities (ideally 1-5 eV below 6keV)