

SOLEX: a tunable monochromatic X-ray source tool for X-ray detectors characterization

Yves Ménesguen, Marie-Christine Lépy

CEA, LIST, Laboratoire National Henri Becquerel, F-91191 Gif-sur-Yvette, France

Metrological context

The LNE-LNHB is the French metrology institute for ionizing radiation: One of its missions is to provide accurate radionuclide decay data to the users by:

- accurately measuring photon emission intensities,
- evaluating radionuclide decay data,
- publishing recommended data (Nucleide).



X-ray emission intensities of radionuclides ($P_K \omega_K$) [1] ←

→ Energy-dispersive (ED) detector efficiency characterization

↓
Need of detector calibration independent from radionuclide decay data

Available atomic data and associated uncertainties

All uncertainties are given for one standard deviation

Fe55 → **Mn55**

	Energy keV	Relative Probability %	Photons per decay x 100	
K α 2	5.88765	51	8.45 ± 0.14	} K α
K α 1	5.89875	100	16.57 ± 0.27	
K β 3	6.49045	20.5	3.4 ± 0.07	} K β 1
K β 1				
K β 5''	6.5352			
K β 5'				

U = 1.64 %

U = 2.06 %

Table 4. Estimated Percentage Uncertainties for Fluorescence and Coster-Kronig Yields

Z(range)	ω_K	ω_{L_1}	ω_{L_2}	ω_{L_3}	f_{12}	f_{13}	f_{23}
5-10	40-10 ^a						
10-20	10-5	>30 ^a	>25 ^a	>25 ^a	10 ^a	5 ^a	
20-30	5-3	30 ^a	25 ^{a,b}	25	15 ^a	10 ^a	40 ^a
30-40	3	30 ^b	25	20	15	10	30-20
40-50	2	30-20 ^b	25-10	20-10	20 ^b	10 ^b	20
50-60	2-1	20-15	10	10-5	20	15	20
60-70	1	15	10-5	5	15	10	20-15 ^a
70-80	1	15 ^b	5	5-3	20 ^b	10-5 ^b	15
80-90	<1	15	5	3	10	5	15
90-100	<1	15-20	10 ^b	3-5	10-50	5-10	15 ^b
100-110	1	20	10	5	50-100	15	20

Fluorescence yields : ω

Most of the available experimental data are older than 30 years [2] and measured with less accurate setup than nowadays capabilities

- ω_K uncertainties are larger than 3 % for most elements with Z < 30
- ω_L uncertainties are larger than 15 % for all elements [3,4]

[2] W. Bambynek, et al. *X-ray fluorescence yields, Auger, and Coster-Kronig transition probabilities*. Rev. of Mod. Physics, **44**, (716-813), 1972

[3] M. O. Krause. *Atomic Radiative and Radiationless Yields for K and L Shells*. J. Phys. Chem. Ref. Data, Vol **8**, No 2, 1979

[4] E. Schönfeld, H. Janssen. *Evaluation of atomic shell data*. Nuclear Instruments & Methods in Physics Research A, **369**, (527-533), 1996

[5] M. O. Krause. *Atomic Radiative and Radiationless Yields for K and L Shells*. J. Phys. Chem. Ref. Data, Vol **8**, No 2, 1979

Outline

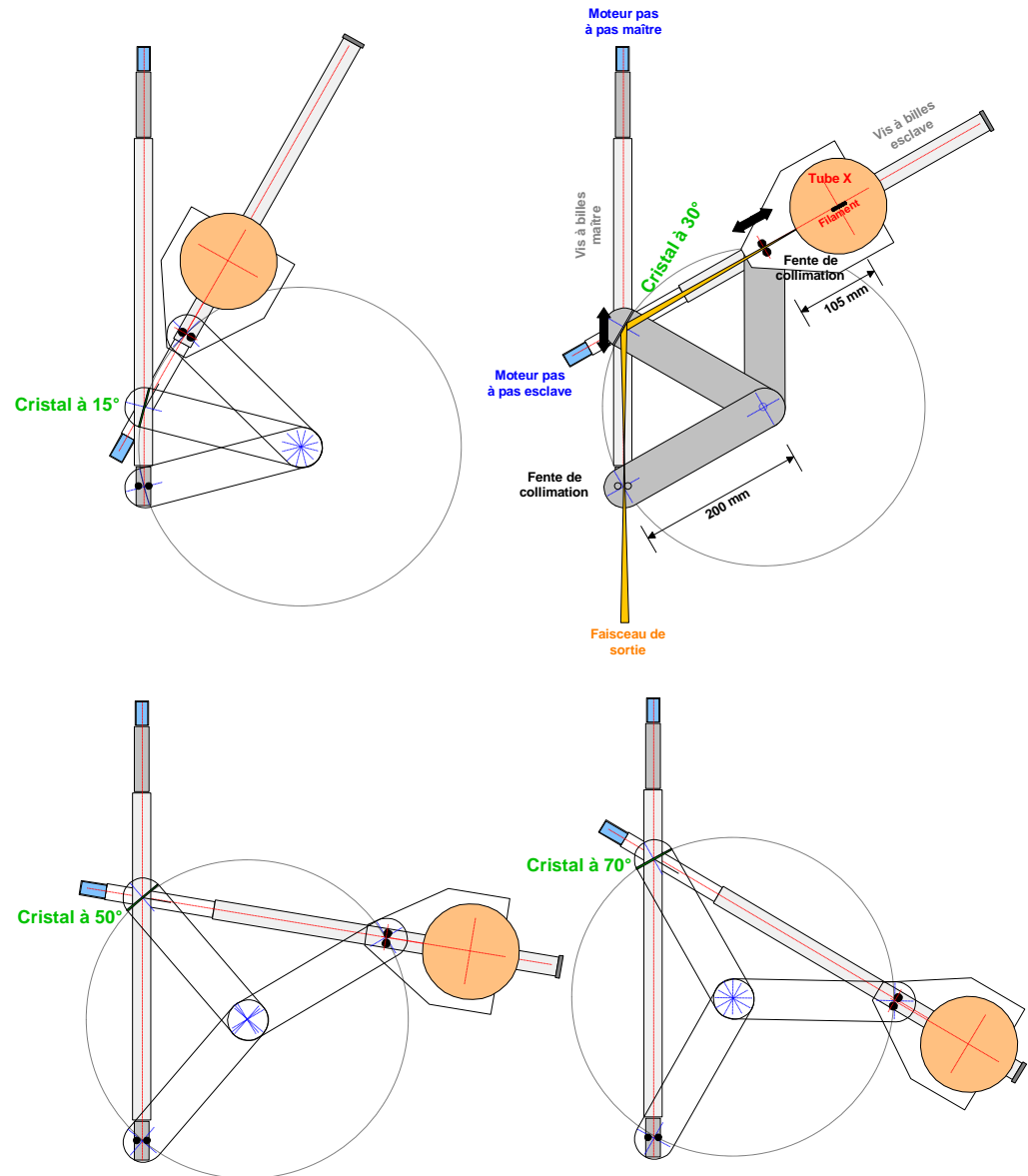
1. SOLEX : The monochromatic & tunable X-ray source
 - 1.1 The source setup
 - 1.2 The source technical properties
 - 1.3 Reference detector

2. Energy-dispersive X-ray spectrometer (EDS) characterization
 - 2.1 Surface Map
 - 2.2 EDS response function
 - 2.3 EDS efficiency calibration

3. Other capabilities of SOLEX
 - 3.1 Total mass attenuation coefficients measurements
 - 3.2 Fluorescence yields

1.1 SOLEX, a tunable monochromatic X-ray source: principles

- X-ray tube
- Dispersive crystal
- Johann geometry [6]
- Fixed output: X-ray tube & crystal are moving
- LabVIEW™ interfaced



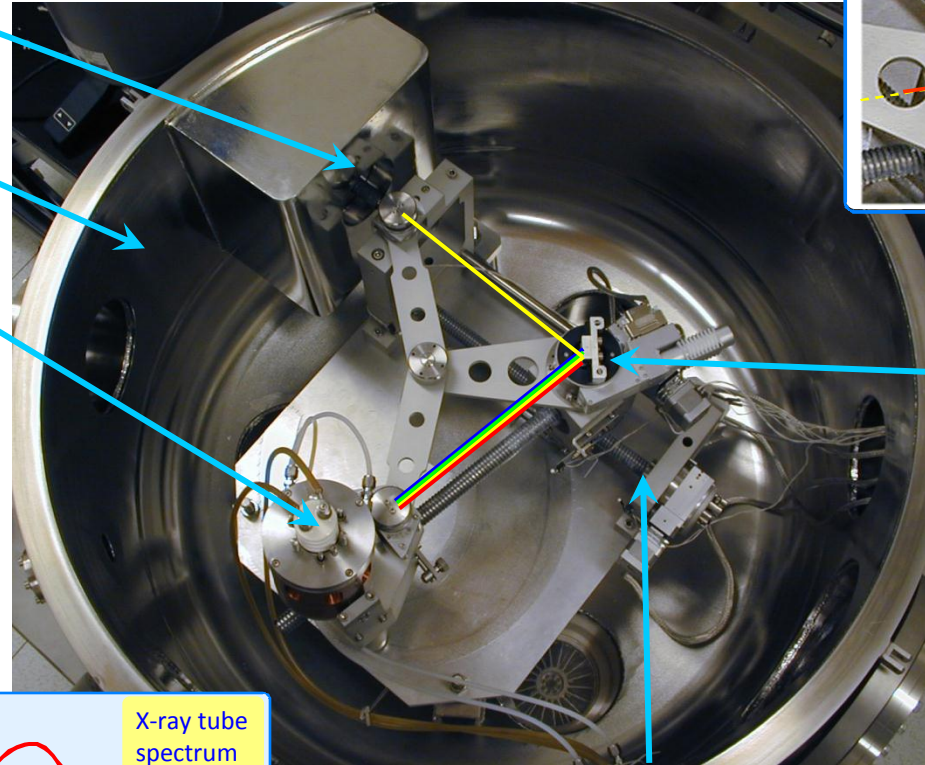
1.2 SOLEX: a tunable monochromatic X-ray source [7]

Output 1: X-ray Detector (HPGe, Si(Li), SDD...)

Vacuum chamber (10^{-7} hPa)

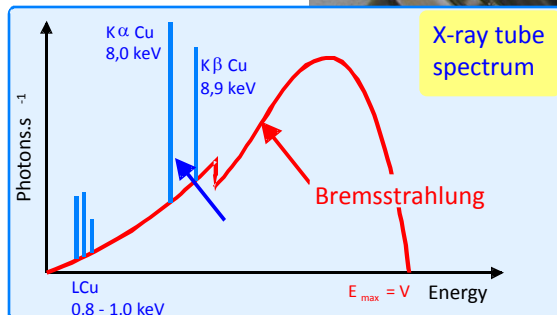
X-ray tube

- Windowless, water-cooled
- Several anodes materials : (Cu, Au, Ag Hastelloy...)
- HV up to 50 kV
- Controllable intensity up to 100 mA
- Intensity fluctuations <1%



Dispersive Crystal : Bragg law

- Emitted X-rays : FWHM \sim some eVs
- Different Crystals (InSb, Beryl, LiF, Quartz...)
- $0.6 \text{ keV} \leq E \leq 28 \text{ keV}$

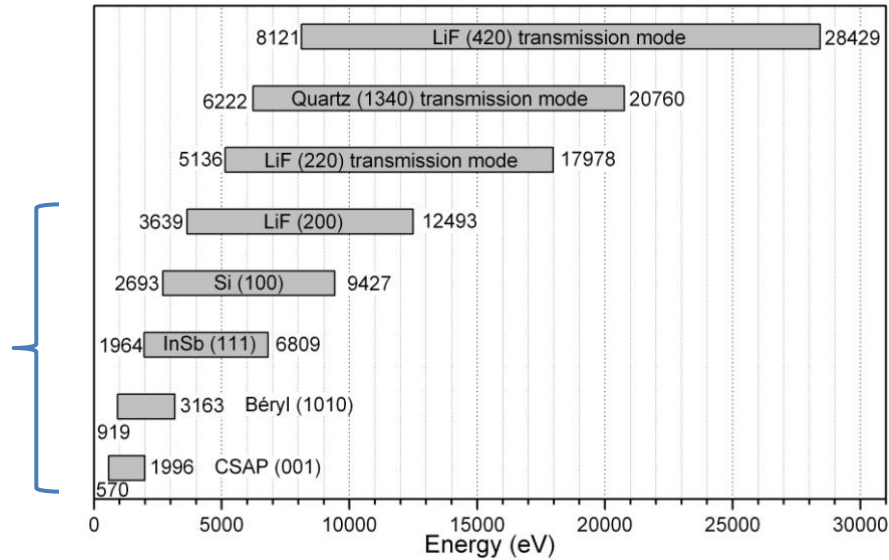


Options

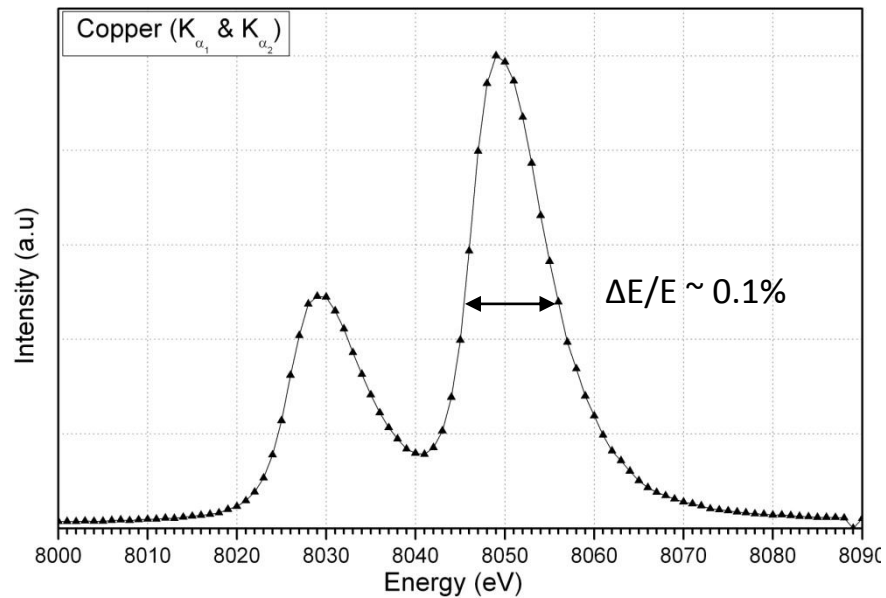
- Removable filter holder
- Proportional counter for absolute flux measurements

1.2 SOLEX: characteristics

Good energy resolution
& reproducible energy-
calibration



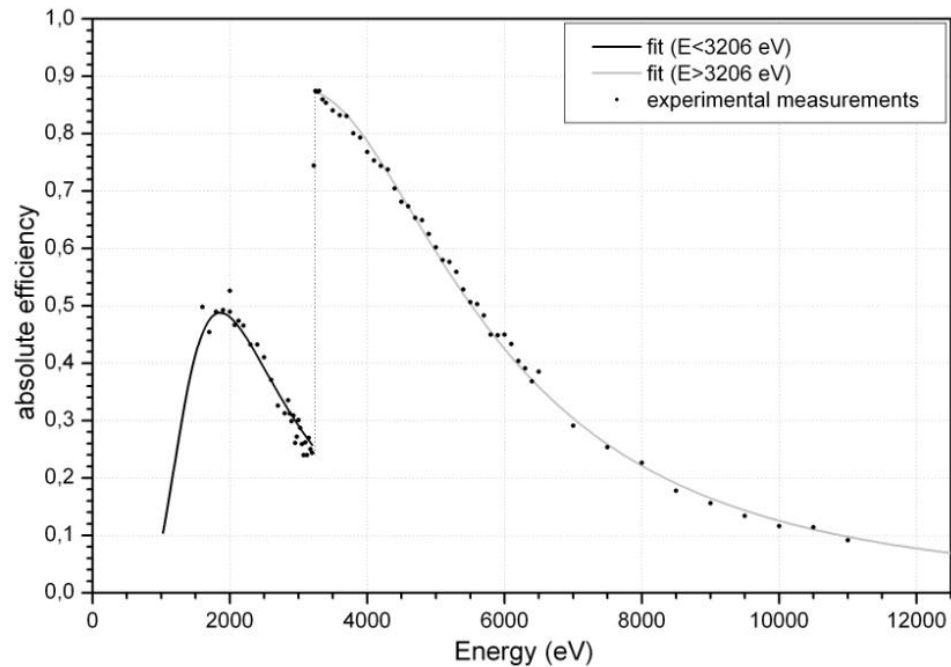
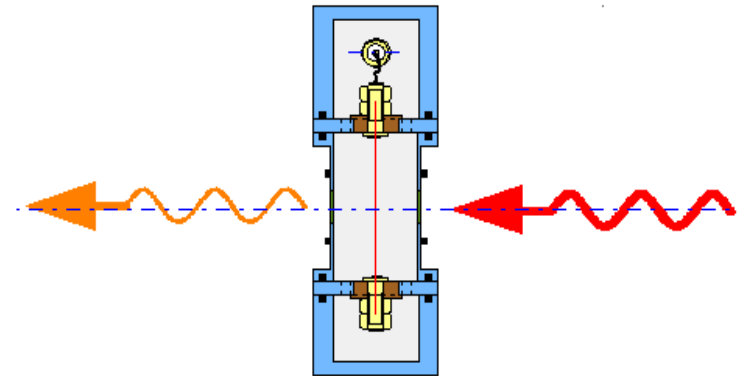
Worse spectral
resolution



1.3 Gaseous Proportional Counter as reference detector

- Mounted on the 2nd output
- Two beryllium windows
- Full transmission/absorption characterization

- Dedicated to full-energy peak efficiency calibration



Outline

1. SOLEX : The monochromatic & tunable X-ray source
 - 1.1 The source setup
 - 1.2 The source technical properties
 - 1.3 Reference detector

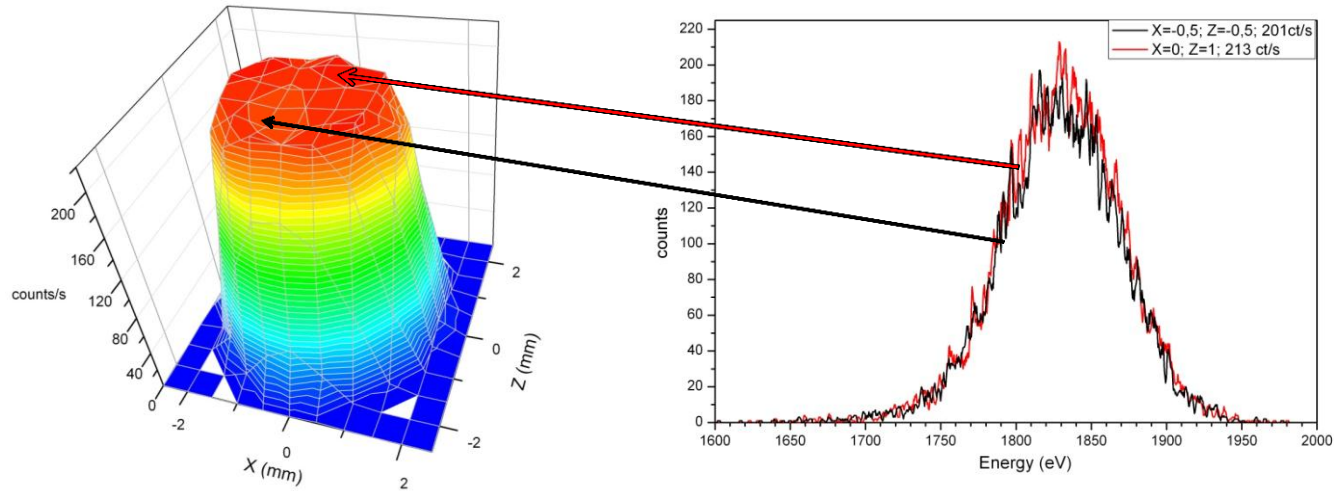
2. Energy-dispersive X-ray spectrometer (EDS) characterization
 - 2.1 Surface Map
 - 2.2 EDS response function
 - 2.3 EDS efficiency calibration

3. Other capabilities of SOLEX
 - 3.1 Total mass attenuation coefficients measurements
 - 3.2 Fluorescence yields

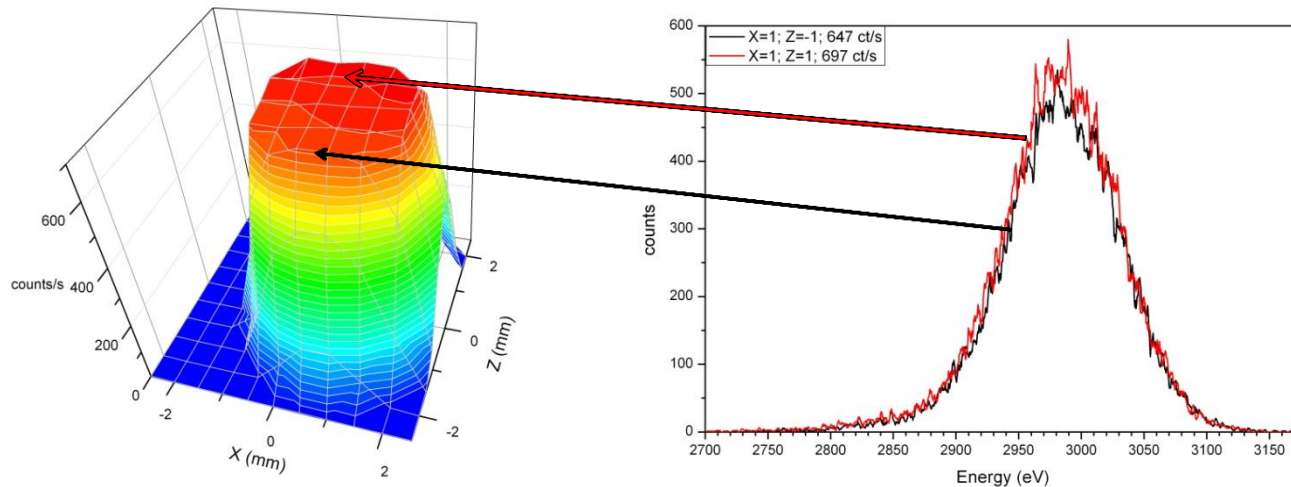
2.1 EDS characterization: surface map

- 2D scan (40 x 40 mm)
- Beam size about 0,5 x 0,5 mm
- Spectrum acquisition and automated treatment

1830 eV



2984 eV



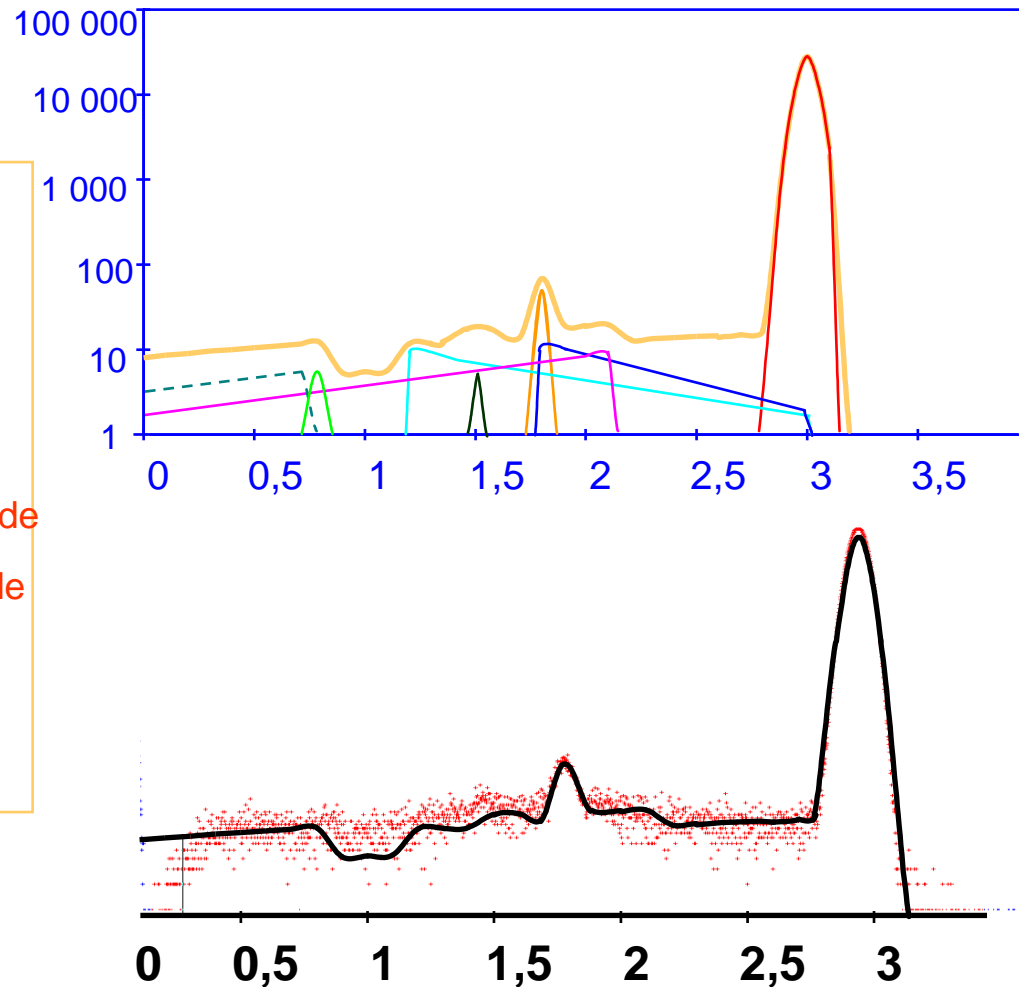
2.2 EDS characterization: response function

Si(Li) EDS

Monochromatic photons

Energy = 3 keV

- Total Spectrum
- Full-energy peak (total absorption)
- Escape peak
- Photoelectrons escape
- Auger electrons escape
- - - Interaction of Auger electrons of the electrode
- Interaction of photoelectrons of the electrode
- L- fluorescence peak of the electrode
- K- fluorescence peak of aluminum



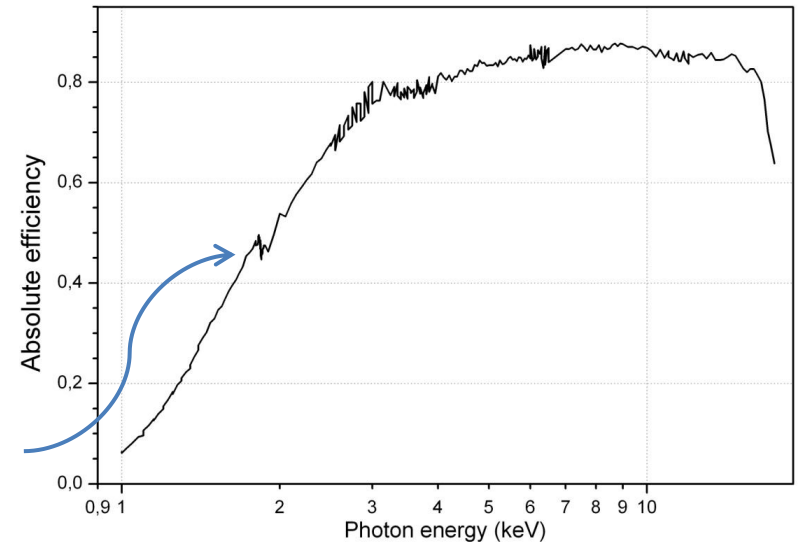
2.3 EDS full-energy peak efficiency calibration

Full-energy peak efficiency of a Si(Li) detector

Comparison with the PC:

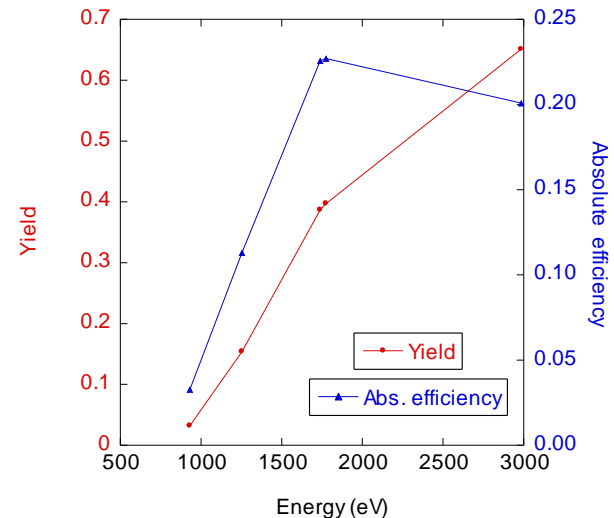
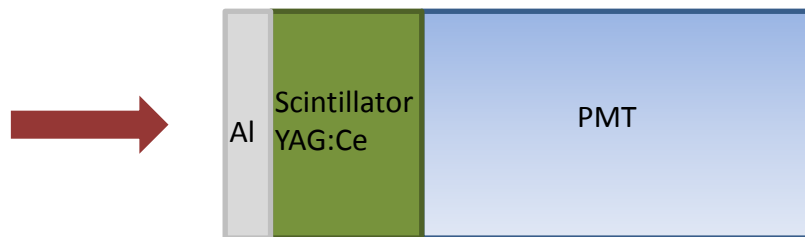
$$\eta_{\text{det}} = \frac{N_{\text{det}} / t_{\text{det}}}{N_{\text{PC}} / t_{\text{PC}}} \times \eta_{\text{PC}}$$

Si K-edge → dead layer



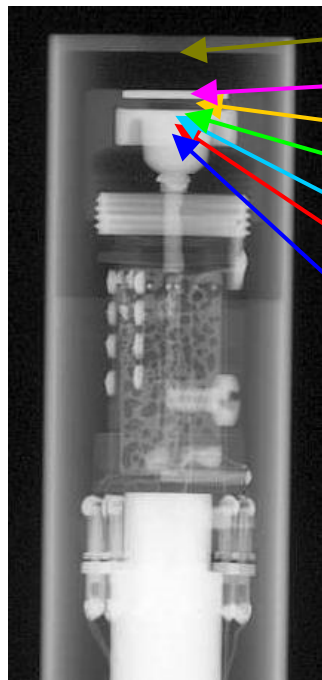
X-ray counting system based on YAG scintillator and a PM [8]

DUVEX: association of a YAG and a PM

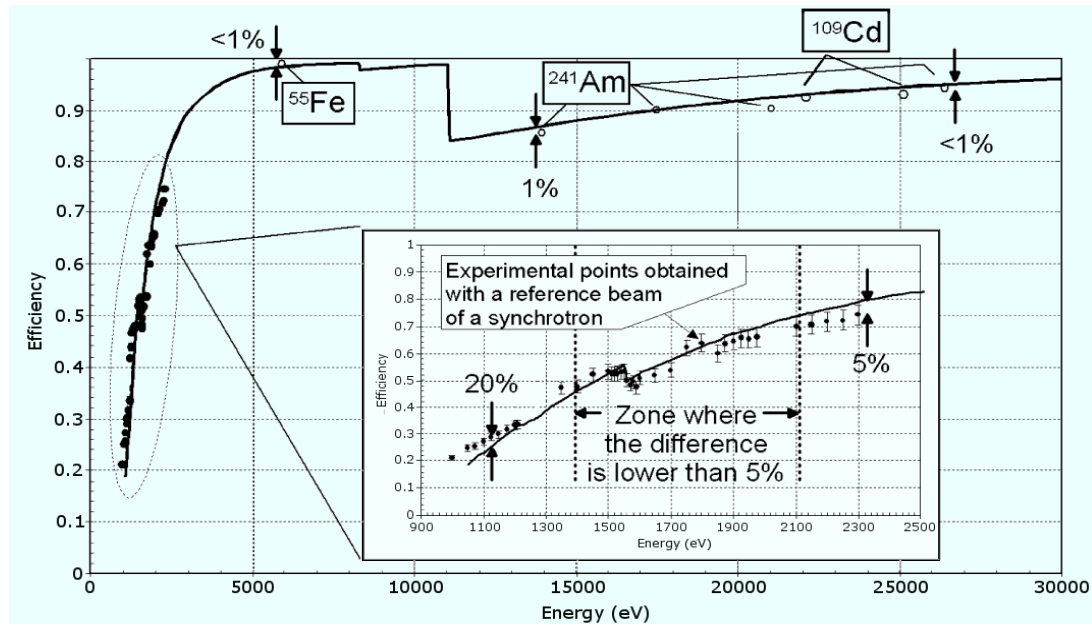
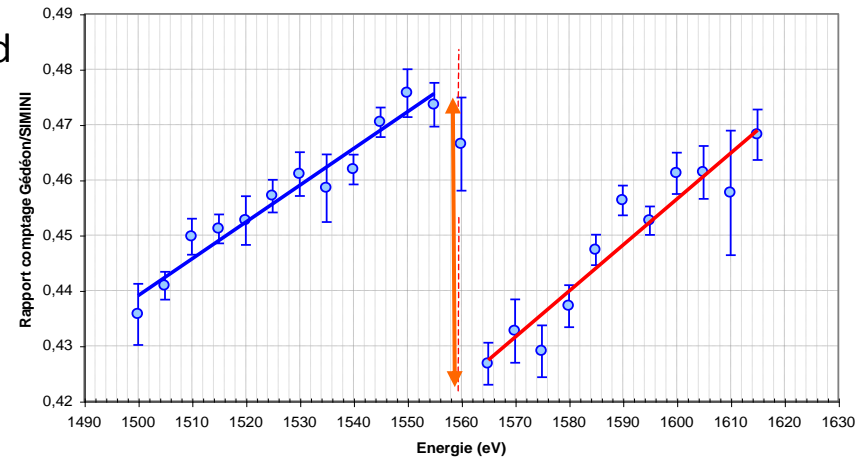


2.3 EDS full-energy peak efficiency calibration [9]

- Measurement of screen thicknesses : (scanning around the binding energy)
- Germanium partial active layer : peak shape analysis



- Be window 10 μ m
- Collimator
- Al infra-red screen 0.113 (18) μ m
- Ni electrode 51 (20) nm
- Ge dead layer 11.6 (61) nm
- Ge partial active layer : 1.27(4) μ m
- Ge crystal : 4 mm



3. Other capabilities of SOLEX

Mass attenuation coefficients measurements / Filter transmission

NIST XCOM database [10]

Select by: (only elements 1 - 100) Atomic Number: <input type="text"/> or Symbol: <input type="text"/>	Options for output units: <input checked="" type="radio"/> All quantities in cm^2/g <input type="radio"/> All quantities in $barns/atom$ <input type="radio"/> Partial interaction coefficients in $barns/atom$ and total attenuation coefficients in cm^2/g
Graph options: <input checked="" type="checkbox"/> Total Attenuation with Coherent Scattering <input type="checkbox"/> Total Attenuation without Coherent Scattering <input type="checkbox"/> Coherent Scattering <input type="checkbox"/> Incoherent Scattering <input type="checkbox"/> Photoelectric Absorption <input type="checkbox"/> Pair Production in Nuclear Field <input type="checkbox"/> Pair Production in Electron Field <input type="checkbox"/> None	Additional energies in MeV (optional) (up to 75 allowed) Note: Energies must be between 0.001 - 100000 MeV (1 keV - 100 GeV) (only 4 significant figures will be used). One energy per line. Blank lines will be ignored. <input type="text"/> <input checked="" type="checkbox"/> Include the standard grid Energy Range: Minimum: <input type="text"/> MeV Maximum: <input type="text"/> MeV

CXRO database [11]

<h3>Filter Transmission</h3> <ul style="list-style-type: none">Choose from a list of common materials: <input type="text" value="Enter Formula"/>Chemical Formula: <input type="text" value="Si3N4"/>Density: <input type="text" value="-1"/> gm/cm^3 (enter negative number to use tabulated values.)Thickness: <input type="text" value=".2"/> micronsPhoton Energy (eV): <input type="text"/> Range from <input type="text" value="10"/> to <input type="text" value="1000"/> in <input type="text" value="100"/> steps (< 500). <p>(NOTE: Photon Energy must be in the range $10\text{ eV} < E < 30,000\text{ eV}$ and Wavelengths in the range of $.041\text{ nm} < \text{Wavelength} < 124\text{ nm}$.)</p> <p>To request a <input type="text" value="Linear"/> <input type="text" value="Plot"/> press this button: <input type="button" value="Submit Request"/></p> <p>To reset to default values, press this button: <input type="button" value="Reset"/></p>

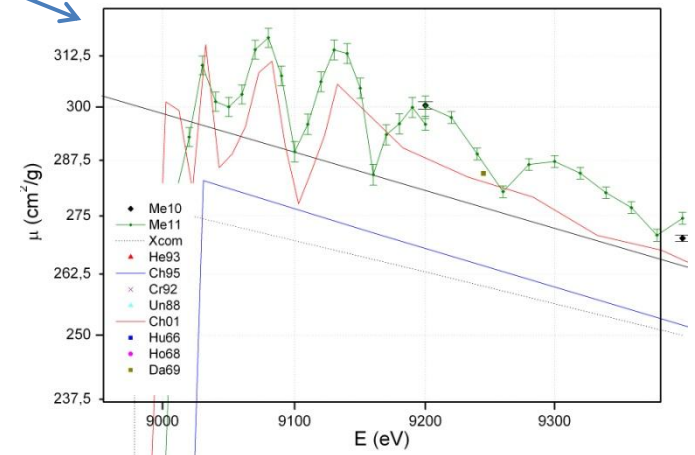
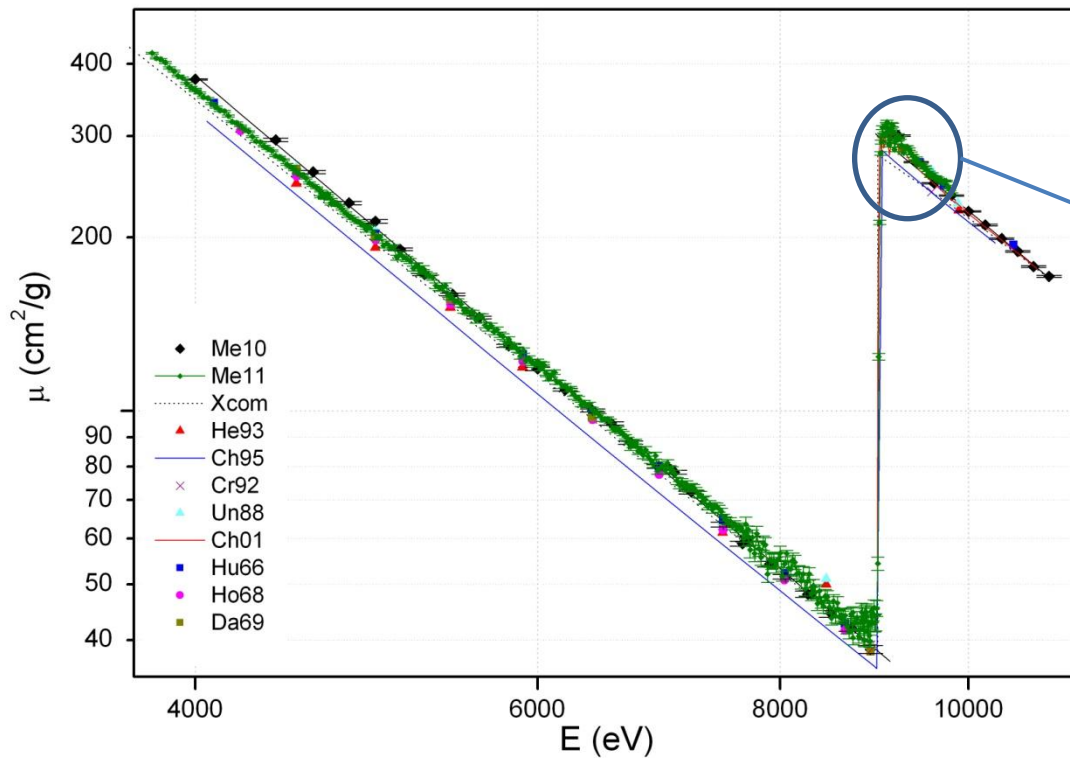
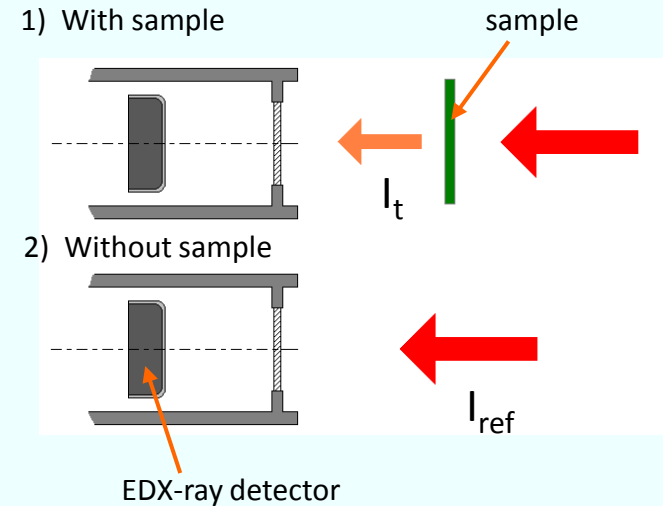
[10] M.J. Berger, J.H. Hubbell, J. Seltzer, S.M. Chang, J.S. Coursey, R. Sukumar, D.S. Zucker, XCOM: Photon Cross Sections Database. Available online: <http://physics.nist.gov/PhysRefData/Xcom/Text/XCOM.html>

[11] B.L. Henke, E.M. Gullikson, and J.C. Davis. *X-ray interactions: photoabsorption, scattering, transmission, and reflection at E=50-30000 eV, Z=1-92*, Atomic Data and Nuclear Data Tables Vol. **54** (no.2), 181-342 (July 1993). Available online: http://henke.lbl.gov/optical_constants/

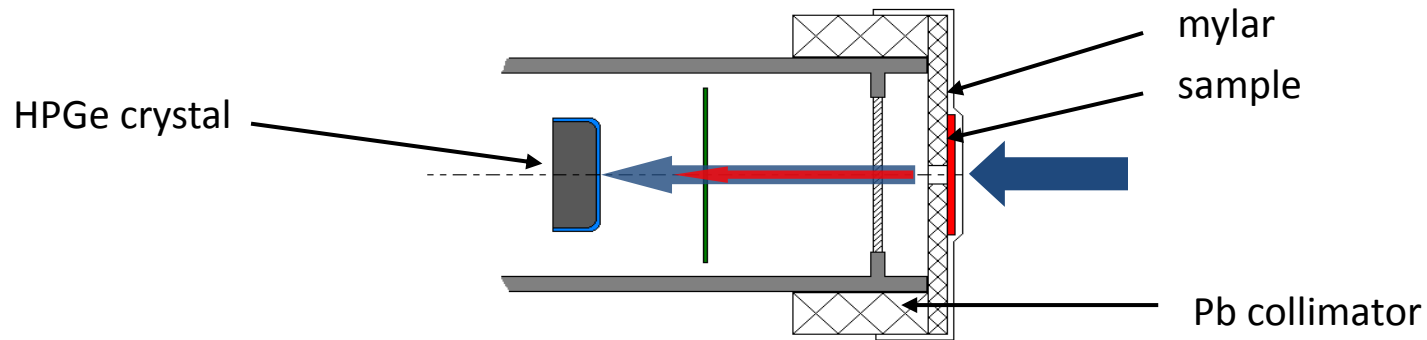
3.1 Total mass attenuation coefficients (example of Cu)

$$\frac{\mu}{\rho} = \frac{\tau_{K,L,M\dots}}{\rho} + \frac{\tau_{cs}}{\rho} + \frac{\tau_{ics}}{\rho}$$

$$\frac{\mu}{\rho} = -\frac{A}{M} \times \text{Ln} \left(\frac{I_t}{I_{ref}} \right)$$

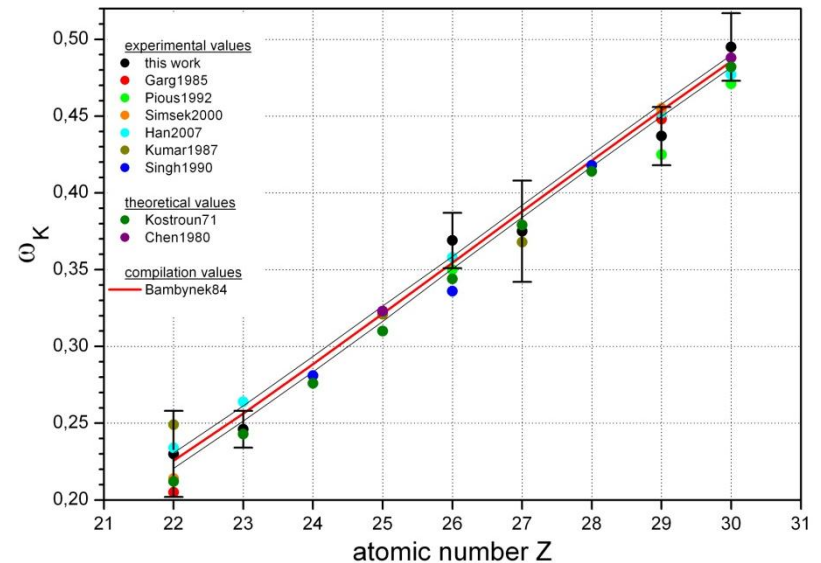


3.2 Fluorescence yields



- ☺ Only one solid angle to measure
- ☺ Simultaneous measurement of the input beam and the fluorescence lines
- ☹ Low input rate

Z	element	$\omega_{K\alpha}$	$\omega_{K\beta}$	ω_K
22	Ti	0.206(23)	0.0233(55)	0.230(28)
23	V	0.219(9)	0.0269(32)	0.246(12)
26	Fe	0.323(16)	0.0452(24)	0.369(18)
27	Co	0.332(29)	0.0427(38)	0.375(33)
29	Cu	0.384(16)	0.0535(30)	0.437(19)
30	Zn	0.427(19)	0.0682(33)	0.495(22)



[12] Y.Ménesguen et al. *Mass attenuation coefficients in the range 3.8<E<11 keV, K fluorescence yield and K_{β}/K_{α} relative X-ray emission rate for Ti, V, Fe, Co, Ni, Cu and Zn measured with a tunable monochromatic X-ray source.* Nuclear Instruments & Methods In Physics Research B, 2010

Conclusion

- SOLEX
 - Laboratory tool : permanent access
 - Flexible
 - Easier than synchrotron, however with less flux
- Tool for accurate characterization of detectors in the 1-20 keV energy range
 - Detector response homogeneity (mapping)
 - Detector response function
 - Efficiency calibration (independent of radionuclides decay data)
- Tool for accurate characterization of materials
 - Improved facility (monochromatic photons, EDS, tuneability)
 - Transmission (filters, sample)
 - Attenuation coefficients
 - Fluorescence yields
 - Etc.