First DAFNE-Light meeting

- 10:50 Introduction — Antonella Balerna 10'
- 11:00 DXR1 Soft-X beamline — Antonella Balerna 25 '
- 11:30 SINBAD-IR beamline — Mariangela Cestelli Guidi 25'
- 12:00 XUV beamlines — Roberto Cimino 25’

N.B. Talk of Emanuele Pace on the DXR2 UV-VIS beamline will be given in a next meeting.

Next dates:

2) **11 April 2019** - Aula Salvini – Ed. Alte Energie 10:50/13:00

3) **16 May 2019** - Aula Salvini – Ed. Alte Energie 10:50/13:00

4) **13 June 2019** - Aula Conversi – Ed. 57 10:50/13:00

DAΦNE-Light
INFN-LNF synchrotron radiation facility

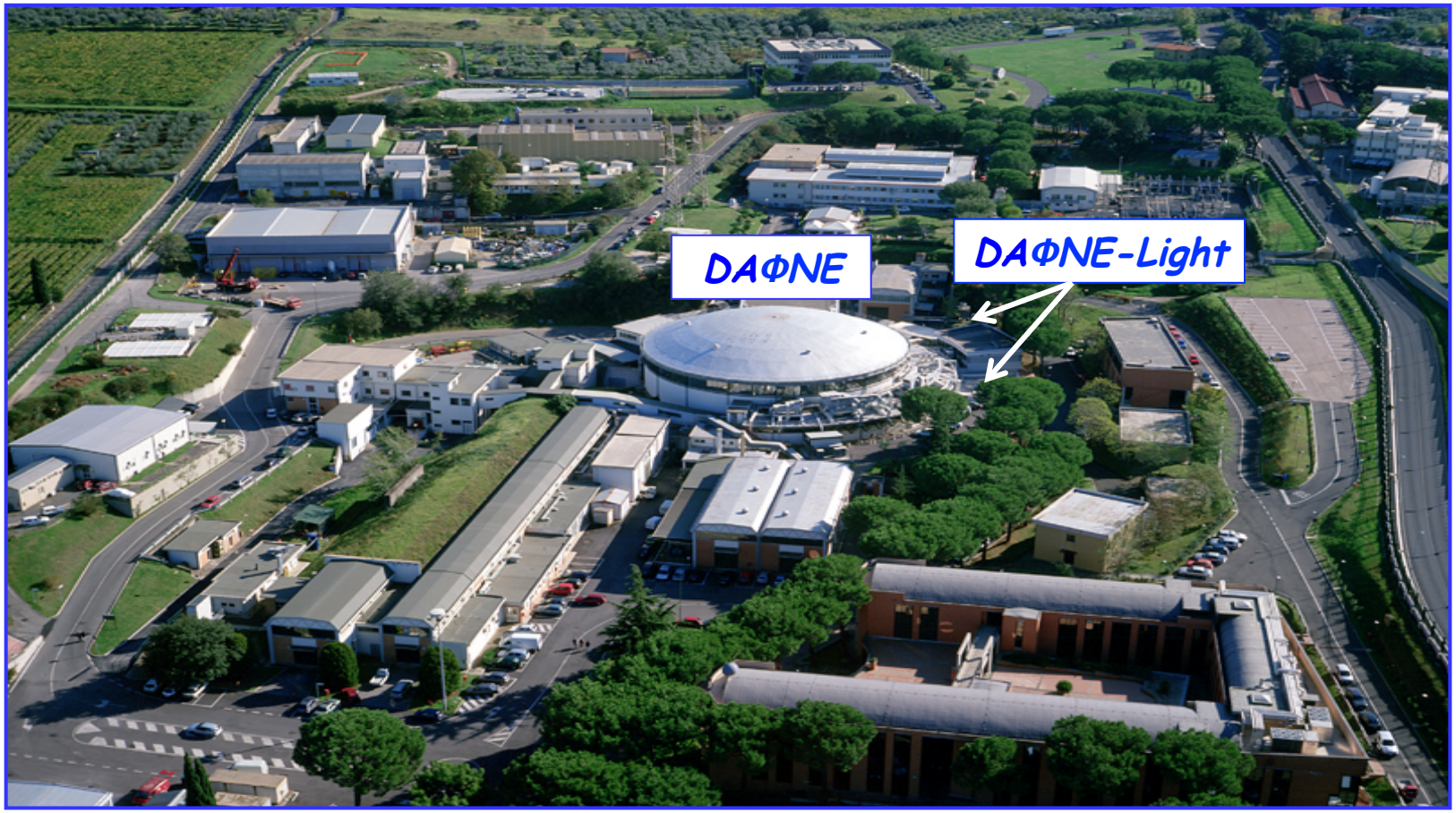
Antonella Balerna

DAΦNE-Light Facility
INFN – Frascati National Laboratory

LDS Meeting - INFN-LNF - March 14th 2019

DAΦNE-Light

INFN-LNF Synchrotron Radiation Facility

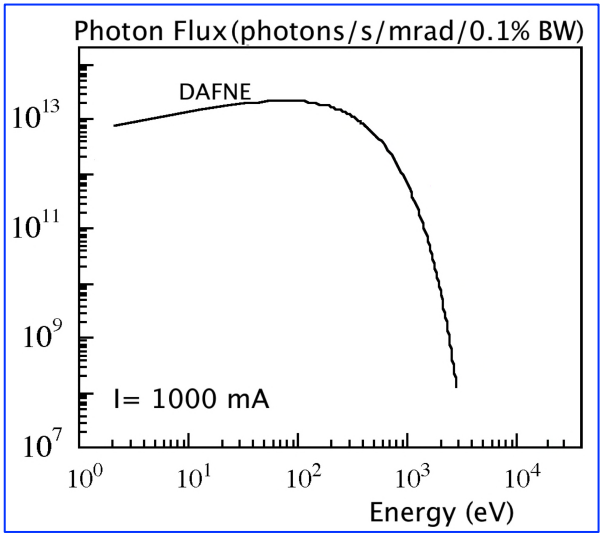


DAΦNE-Light

The **DAΦNE accelerator** complex is the largest scientific asset of the Frascati National Laboratory. ***DAΦNE-Light is a Synchrotron Light facility where MATERIAL SCIENCE STUDIES can be performed*** but also ***a laboratory where new DETECTORS and OPTICS in a wide energy range moving from IR to soft X-rays can be tested using Synchrotron Radiation but also Conventional Sources.***

Available techniques

- ***FTIR spectroscopy, IR microscopy and IR imaging***
- ***UV-VIS absorption spectroscopy***
- ***Soft x-ray spectroscopy: XANES (X-ray Absorption Near Edge Structure) light elements from Na to Cl***
- ***SEY (secondary electron yield) and XPS (X-ray photoelectron spectroscopy) – by electron and photon bombardment***



Every year about ***30 experimental teams*** get access to the **DAFNE-Light Laboratory** coming from **Italian Universities and Research Institutions, and also from EU and other Countries.**

OPEN to USERS

1) SINBAD - IR beamline (1.24 meV - 1.24 eV)

Mariangela Cestelli Guidi

2) DXR1- Soft x-ray beamline (900-3000 eV)

Antonella Balerna

3) DXR2 – UV-VIS beamline (2-10 eV)

Emanuele Pace

All open to call for proposals

XUV beamlines UNDER COMMISSIONING

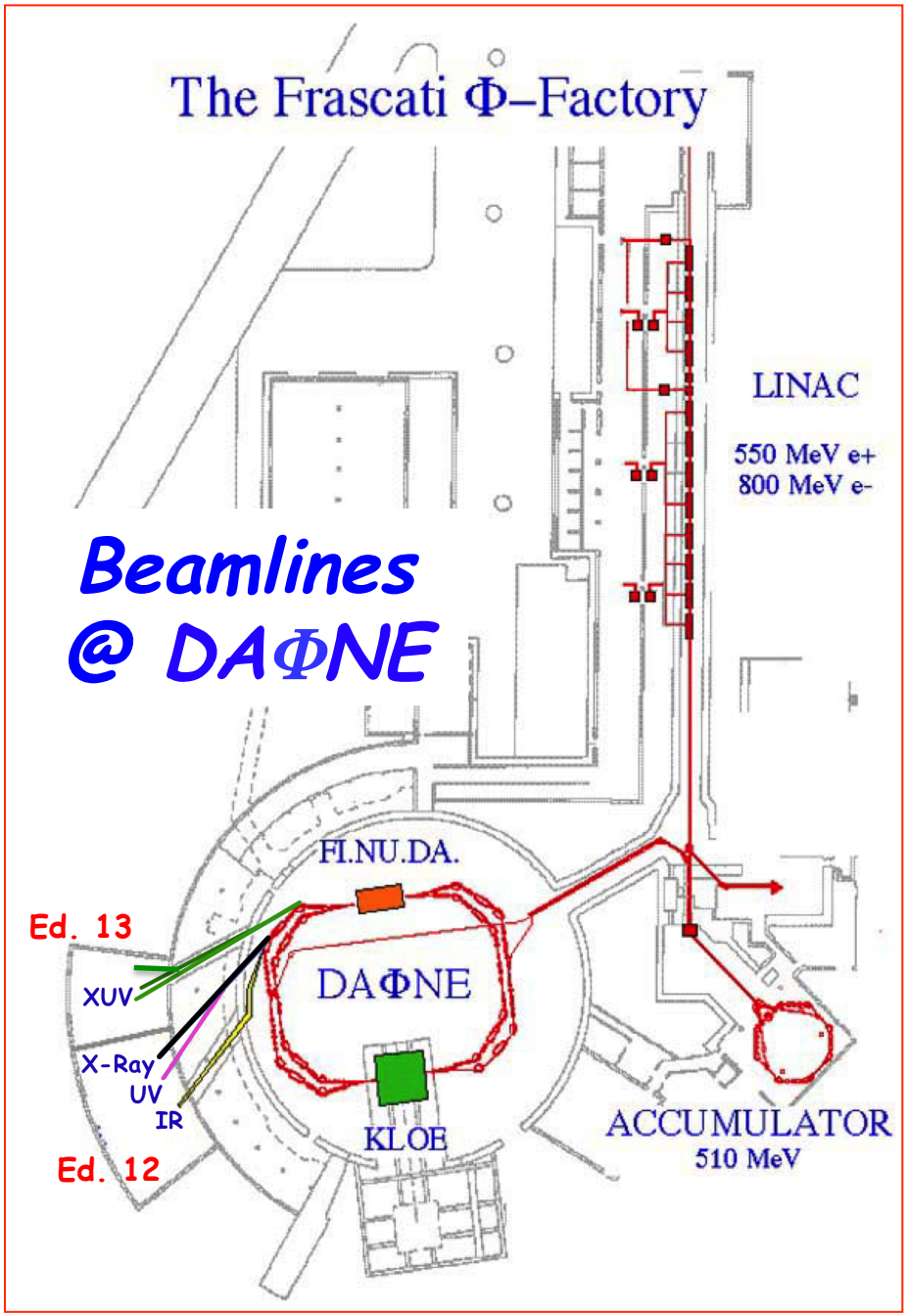
Roberto Cimino

4) LEB - Low Energy Beamline (30-200 eV)

5) HEB - High Energy Beamline (60-1000 eV)

UNDER Construction

6) New HEB Branch White Line - WINDY (MoU CERN-INFN)



Principle Beamline Scientists

SINBAD - Infrared beamline – Mariangela Cestelli-Guidi

DXR2 - UV beamline - Emanuele Pace (INFN - Univ. Fi)

DXR1 - Soft X-ray beamline - Antonella Balerna

DXUV- XUV beamlines - Roberto Cimino

DAΦNE Light

Technical Staff

Antonio Grilli, Agostino Raco, Marco Pietropaoli, Vittorio Sciarra, Vinicio Tullio and **Giacomo Viviani**

Collab. Italian Users: Silvia Colasanti **EU Users: Daniela Ferrucci**

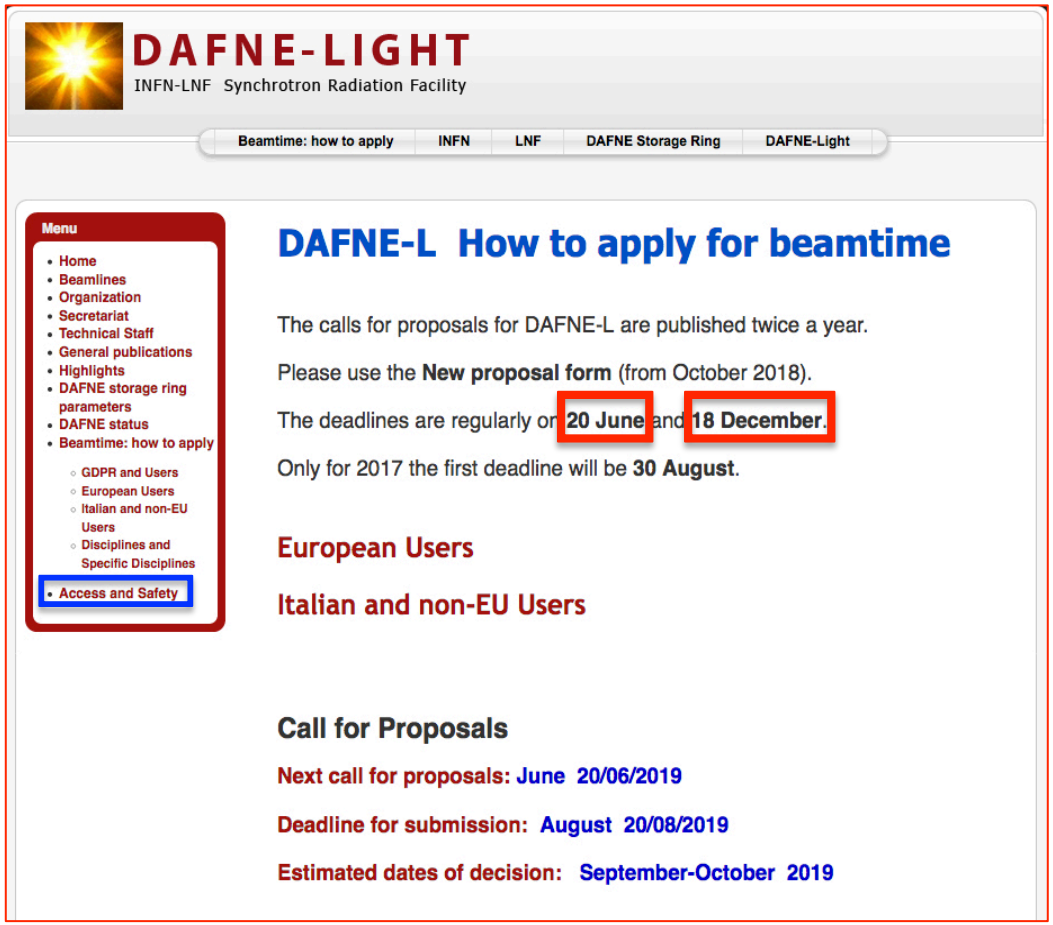
Many other researchers, professors and students involved in different experiments, thesis, PhD thesis, Italian and EU projects, MoU etc. reported in the other talks.

DAΦNE-Light Laboratory and Activity **2018**

R. Amendola (Ass.), M. Angelucci (Ass. Ric.), A. Balerna (Resp.), G. Bellisola (Ass), F. Bellatreccia (Ass.), M. Cestelli Guidi, R. Cimino, G. Della Ventura (Ass.), A. Di Trolio (Ass.), F. Galdenzi (Dott.), L. Gonzalez (Ass.), A. Grilli (Tecn.), E. La Francesca (Dott.), R. Larciprete (Ass.), A. Liedl (Ass. Ric.), E. Pace (Ass.), M. Pietropaoli (Tecn.), A. Raco (Tecn.), M. Romani (Ass. Ric.), V. Sciarra (Tecn.), L. Spallino (Ass. Ric.), V. Tullio (Tecn.), G. Viviani (Tecn.).

Activity Report 2018 - http://www.lnf.infn.it/rapatt/2018/dafne.pdf

Call for proposals: two/year
beamlines open to users



DAFNE-LIGHT
INFN-LNF Synchrotron Radiation Facility

Beamtime: how to apply | INFN | LNF | DAFNE Storage Ring | DAFNE-Light

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  - Italian and non-EU Users
  - Disciplines and Specific Disciplines
- Access and Safety

DAFNE-L How to apply for beamtime

The calls for proposals for DAFNE-L are published twice a year.

Please use the **New proposal form** (from October 2018).

The deadlines are regularly on **20 June** and **18 December**.

Only for 2017 the first deadline will be **30 August**.

European Users

Italian and non-EU Users

Call for Proposals

Next call for proposals: June 20/06/2019

Deadline for submission: August 20/08/2019

Estimated dates of decision: September-October 2019

2018 - only one call

18 October - 18 December 2018

11 Proposals

4 EU Users

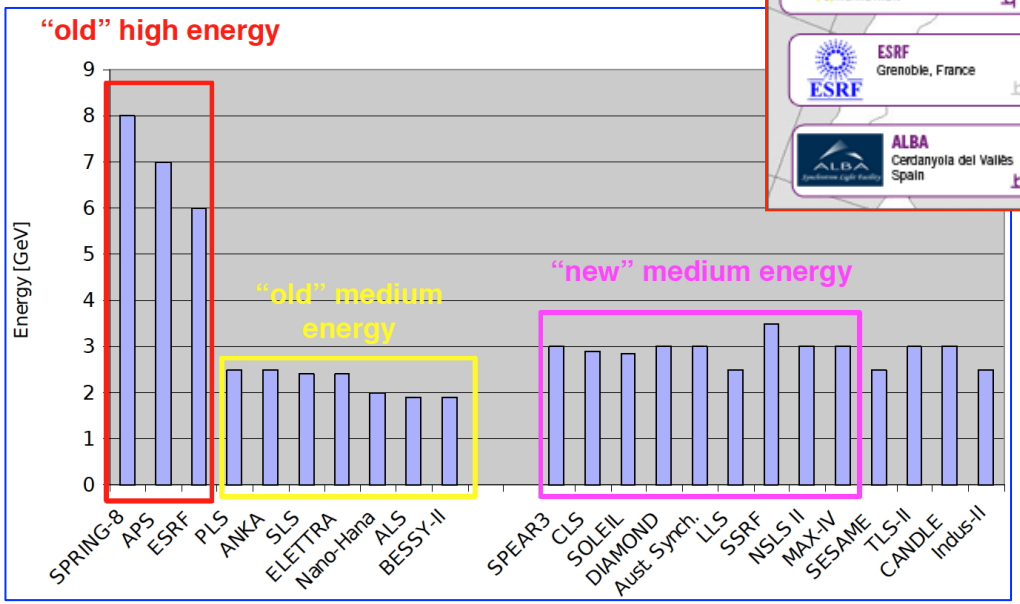
6 Italian Users

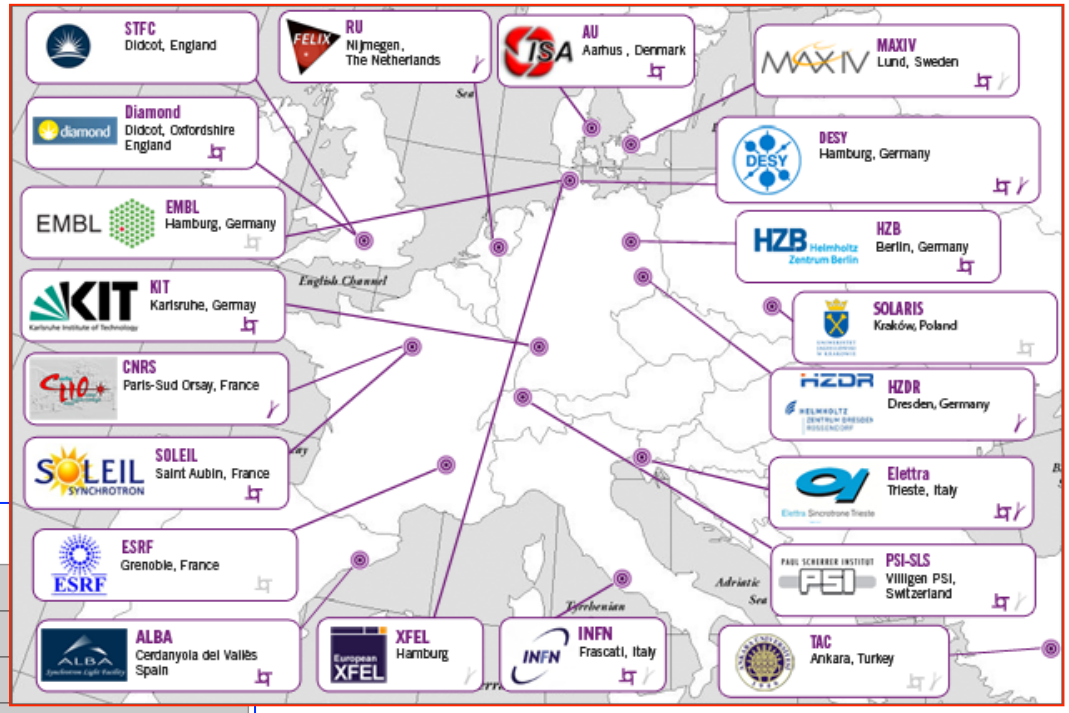
User Selection Panel - USP
President: **Paola Gianotti (INFN-LNF)**
Members:
G. Bellisola (Univ. Verona)
P. Calvani (Univ. Sapienza – Roma)
R. Fantoni (ENEA, Centro Ricerche Frascati)
C. Mariani (Univ. Sapienza – Roma)
S. Mobilio (Univ. Roma Tre).

Access instructions:
Long-term users (beamtime days > 15 days / year) **normal procedures.**
Short-term users can follow a **simplified procedure**.

Synchrotron radiation facilities

18 in America
25 in Asia
25 in Europe
1 in Oceania
including facilities under design and FELS





EUROPE

Info on European Synchrotron Radiation Facilities: www.wayforlight.eu
About 67 operational Synchrotron Radiation Facilities Around the World information on: www.lightsources.org

CALIPSOplus EU Project

Grant Agreement No 730872

CALIPSOplus Partners

DAΦNE-Light and THz
SPARC_Lab beamline included

CALIPSOplus Activities

Networking Activities

- User tools for access and data management
- Dissemination & Training (e.g. Twinning programme)
- European Light Sources for Industrial Innovation plus
- Striving for Sustainability of Photon Sources in Europe (e.g. ESUO and the wayforlight.eu portal)

Trans-national Access Activities

- Trans-national Access of the respective facility (e.g. TNA funding)
- Tailor-made for SMEs Trans-national Access (TamaTA)

Joint-Research Activities

- Metrology on One-Nanometer-Precise Optics
- Demonstrator for a Photon Science Analysis Service

www.wayforlight.eu

All information on European Light Sources

WAY FOR LIGHT

Information on the DAΦNE- Light facility

https://web.infn.it/DAFNE_Light/

DAFNE-LIGHT

INFN-LNF Synchrotron Radiation Facility

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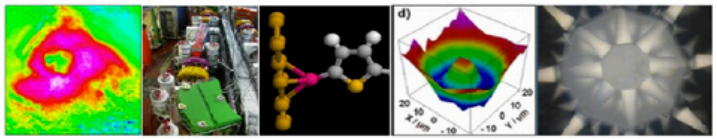


DAFNE-Light

DAFNE-Light is the Synchrotron Radiation Facility at the Laboratori Nazionali di Frascati (LNF).

Three beamlines are operational using, in parasitic and dedicated mode, the intense photon emission of DAFNE, a 0.51 GeV storage ring with a routinely circulating electron current higher than 1 Ampere. Two of these beamlines (DXR1 and DXR2) have one of the DAFNE wiggler magnets as synchrotron radiation source, while the third beamline (SINBAD-IR) collects the radiation from a bending magnet. The two XUV high and low energy bending magnet beamlines are nowadays under commissioning.

The beamlines DXR1 and SINBAD-IR are open to external users.

CALL FOR PROPOSALS IS NOW CLOSED

Next Call 20 June 2019

WAY FOR LIGHT

WayforLight the European Lightsources single entry point.

Login

Username

Username

Password

Password

Log in

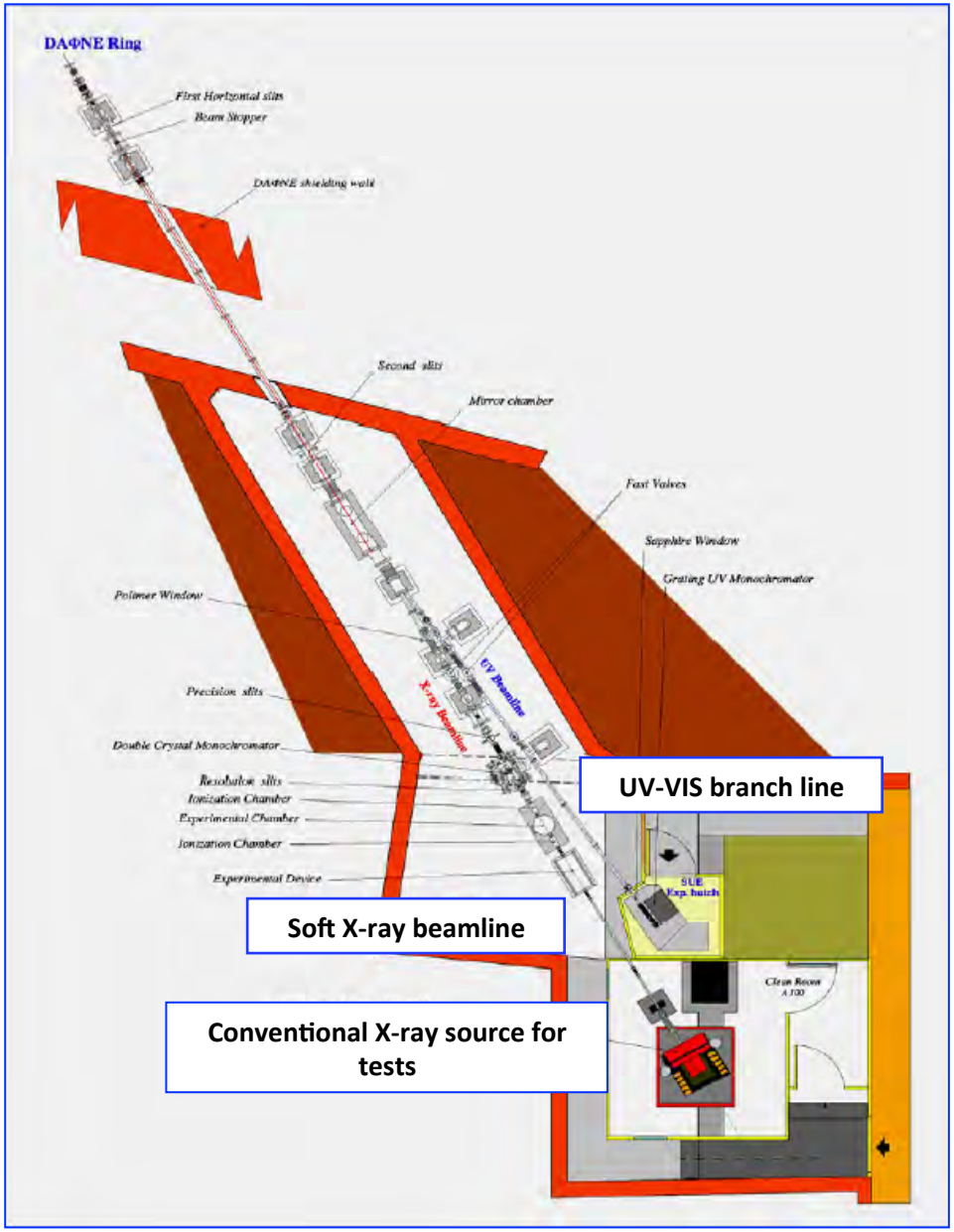
- Create an account
- Forgot your username?
- Forgot your password?

Who is online

We have 43 guests and no members online

DXR1 –Soft X-Ray Beamline

DAΦNE-Light

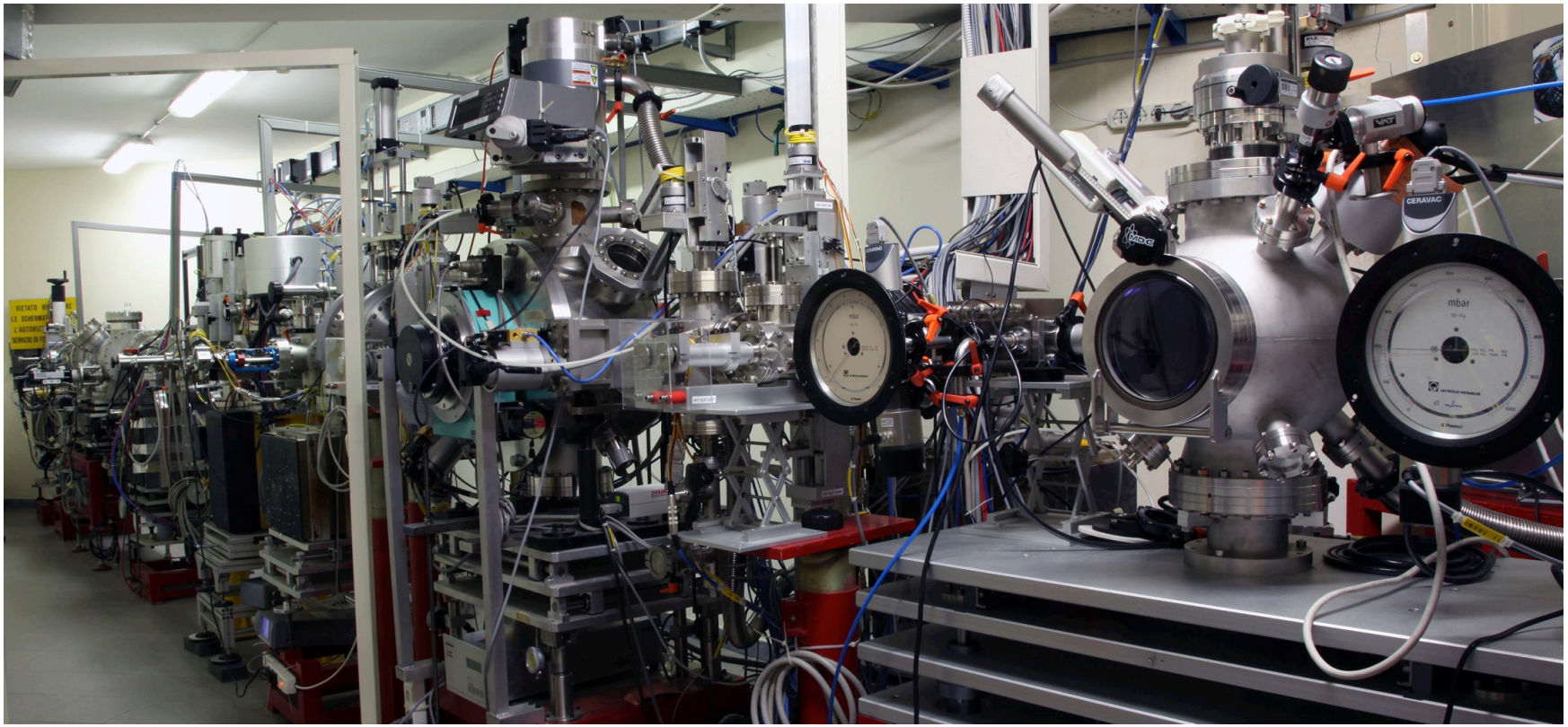


DAΦNE Soft X-ray DXR1 Beamline

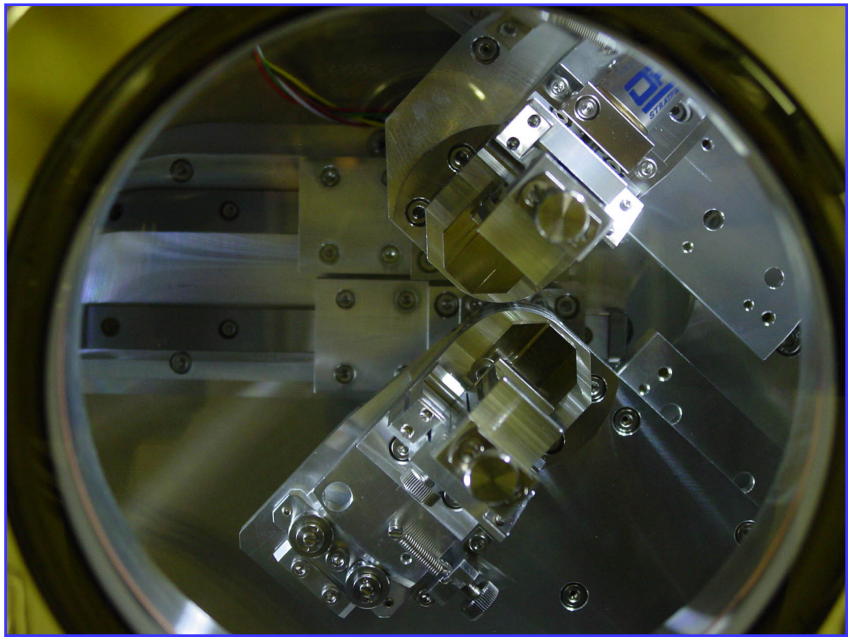
Wiggler (5 poles +2 (1/2) poles) soft x-ray beam line

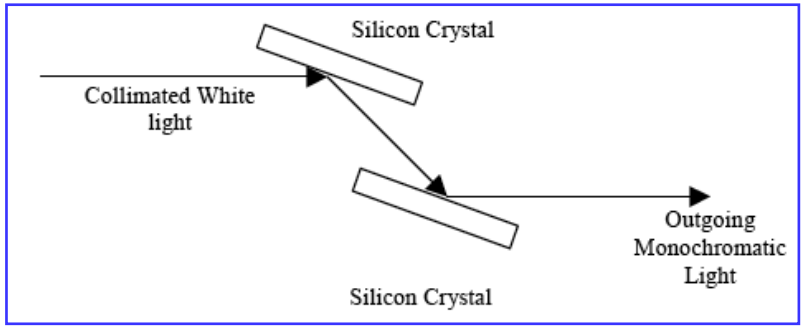
- Critical energy E_c = 284 eV
- Working range 0.9 - 3.0 keV
- TOYAMA double crystal monochromator with KTP (011), Ge (111), Si (111), InSb (111) and Beryl (10-10) crystals
- Cryostat He, LNT
- Soft X-ray absorption spectroscopy and tests of Soft x-ray optics and detectors.

DXR1 Soft X-ray Beamline



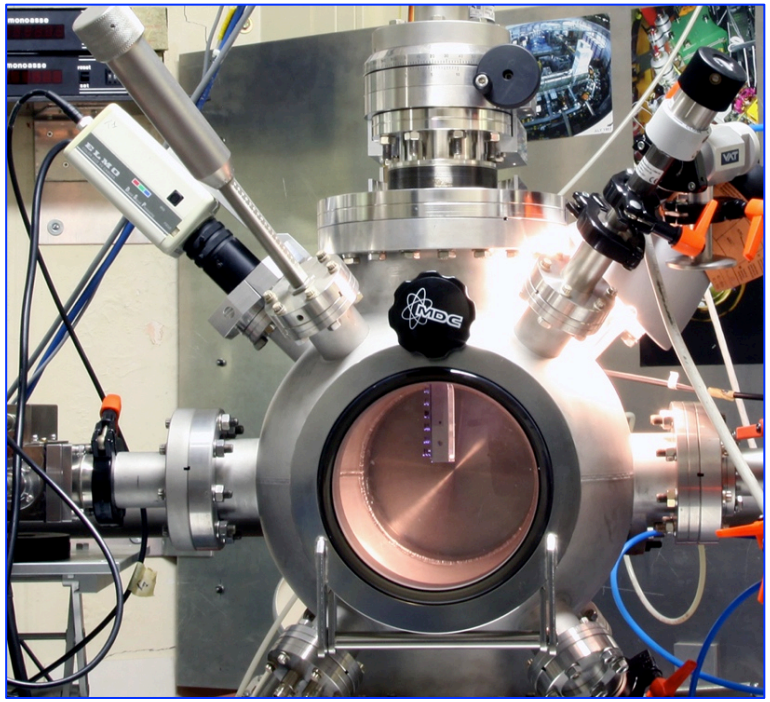
DXR1 Soft X-ray Beamline





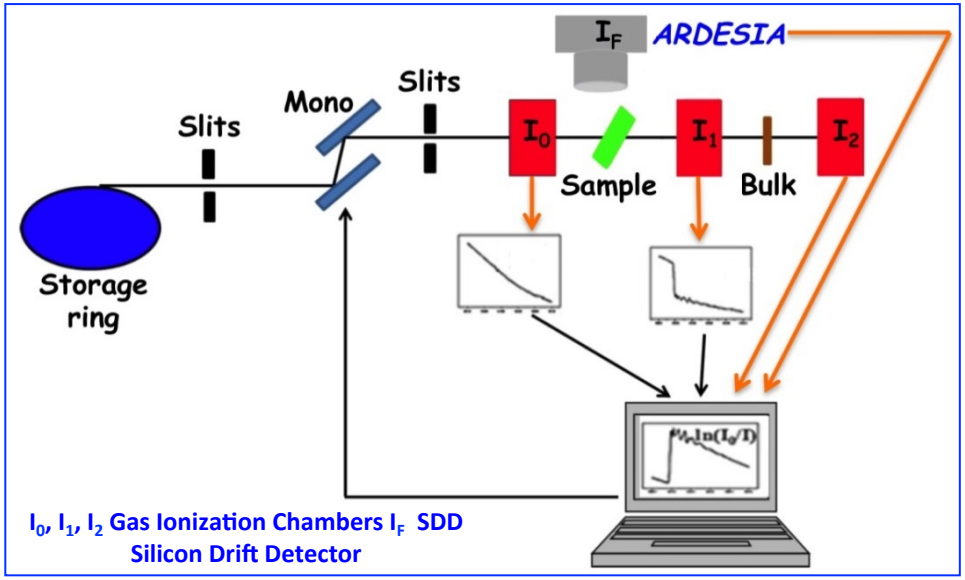
Double crystal monochromator with fixed exit boomerang mechanism.

Crystal type	2d spacing (Å)	Energy range (eV)	Absorption edges
Beryl (10-10)	15.954	1000 - 1560	Na K, Mg K, Cu L
KTP (011)	10.950	1200 - 2200	Mg K, Al K
InSb (111)	7.481	1800 - 3100	Si K, P K, S K, Cl K
Ge (111)	6.532	2100 - 3100	P K, S K, Cl K



Experimental Chamber

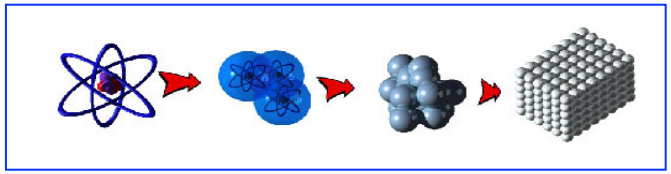
Soft X-ray spectroscopy -XAFS

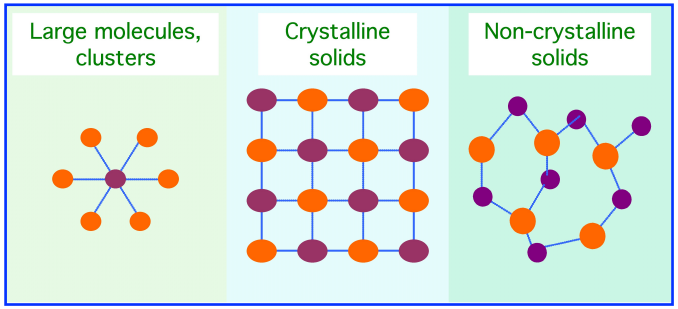


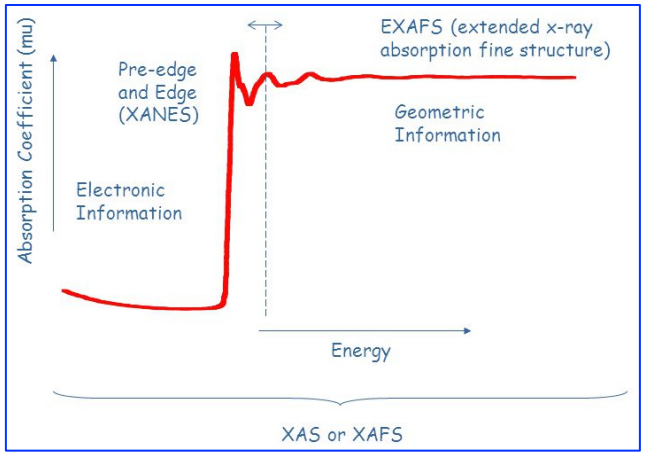
X-ray Absorption Fine Structure or XAFS spectroscopy

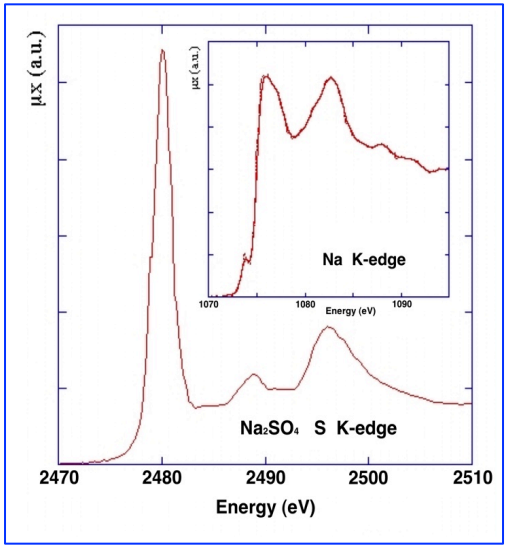
Particularly useful for investigating the electronic structure and local environment of atoms in quite different samples (solids, liquids and gasses).

At the DXR1 beamline the K absorption edges of all light elements from Na to Cl can be studied.



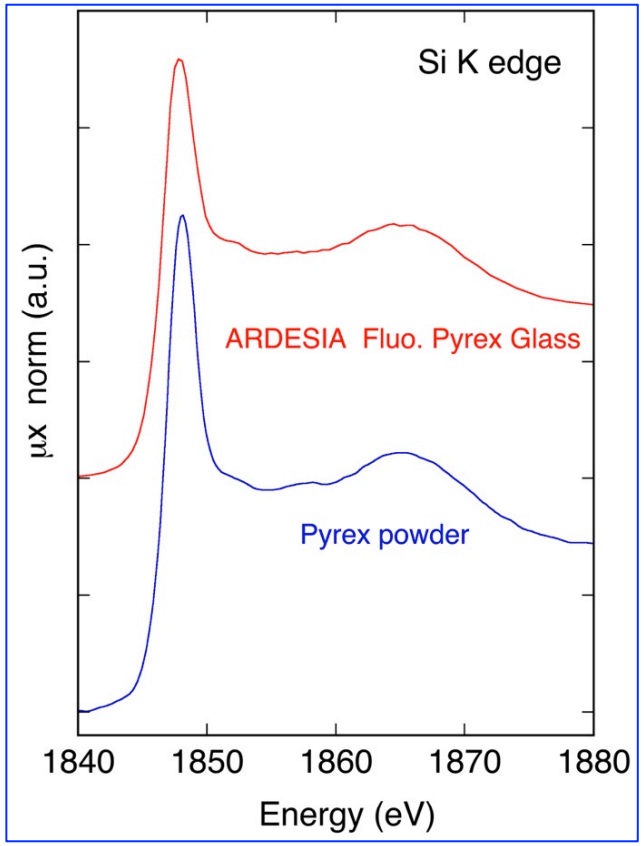




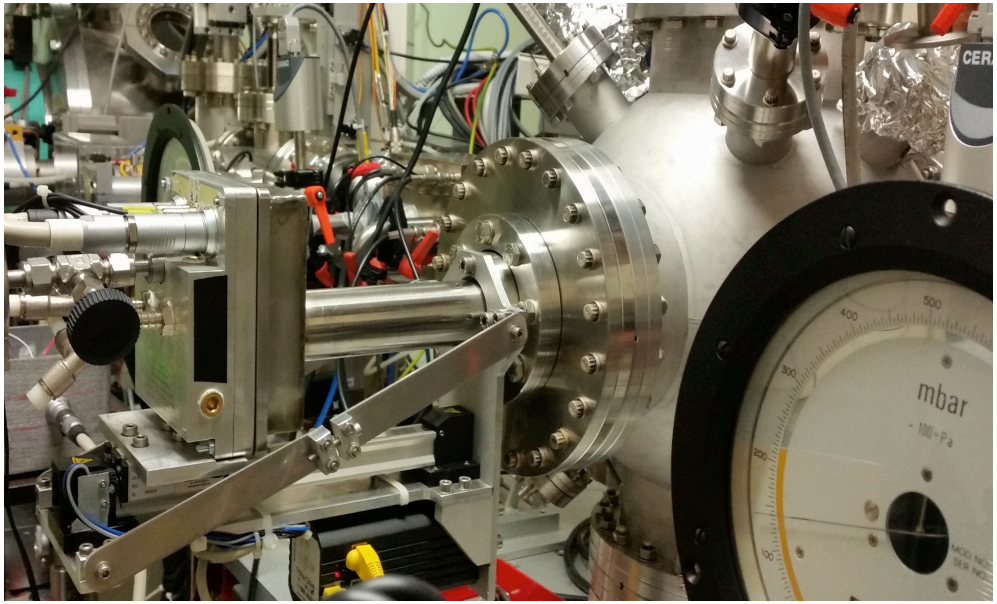


R&D – Test of X-ray Detectors

New 2x2 SDD ARDESIA 4-channel x-ray detector



Successfully tested at the DXR1 beamline - February 2018

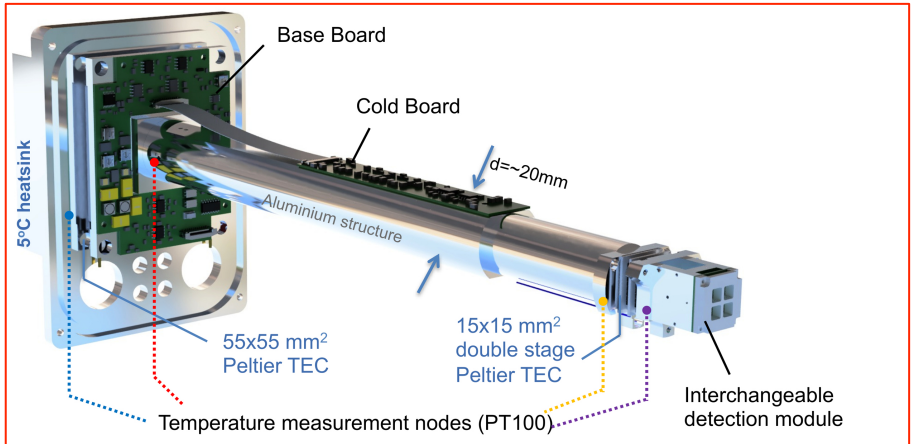


Developed thanks to a collaboration between **DAFNE-Light,** the **Politecnico di Milano** and **FBK.** Financed by **INFN through a CSN5** (**INFN National Scientific Committee 5** for technological research experiments) **ARDESIA** project (2015-2018).

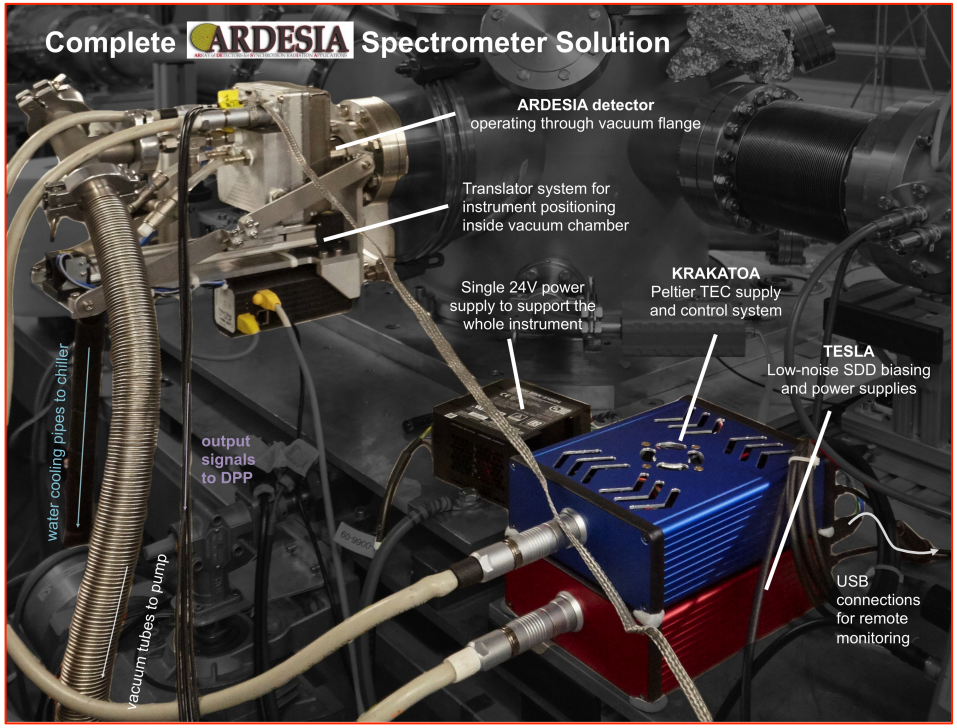
Opens at the DXR1 beamline the chance to study thin and supported samples.

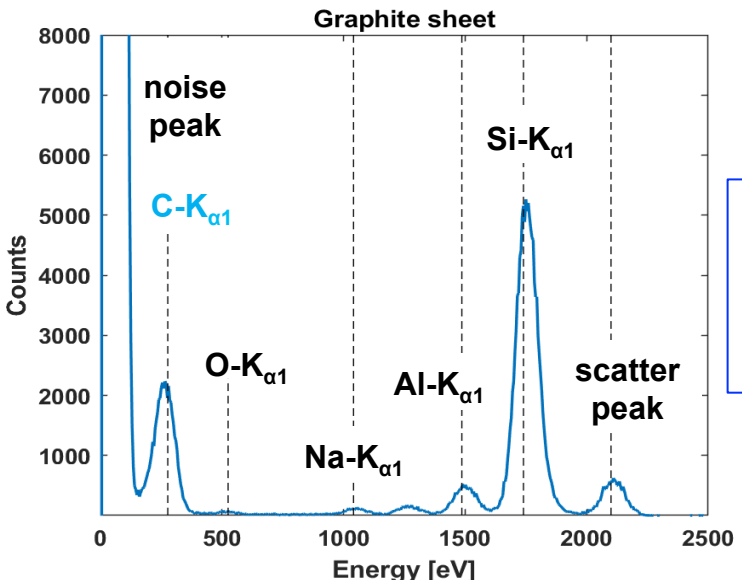
New TT project for the development of a 16 elements SDD detector and application to the ATTRACT EU project.

G.V ARDESIA 4ch-SDD detector



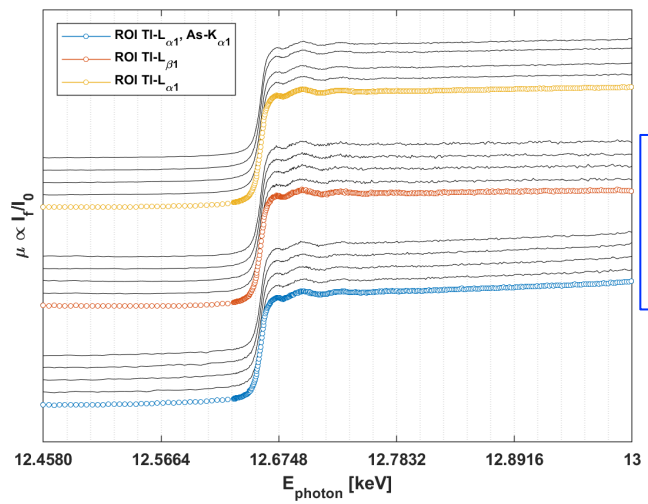
Cooling system with temperature control to achieve the desired low T for optimum measurements.





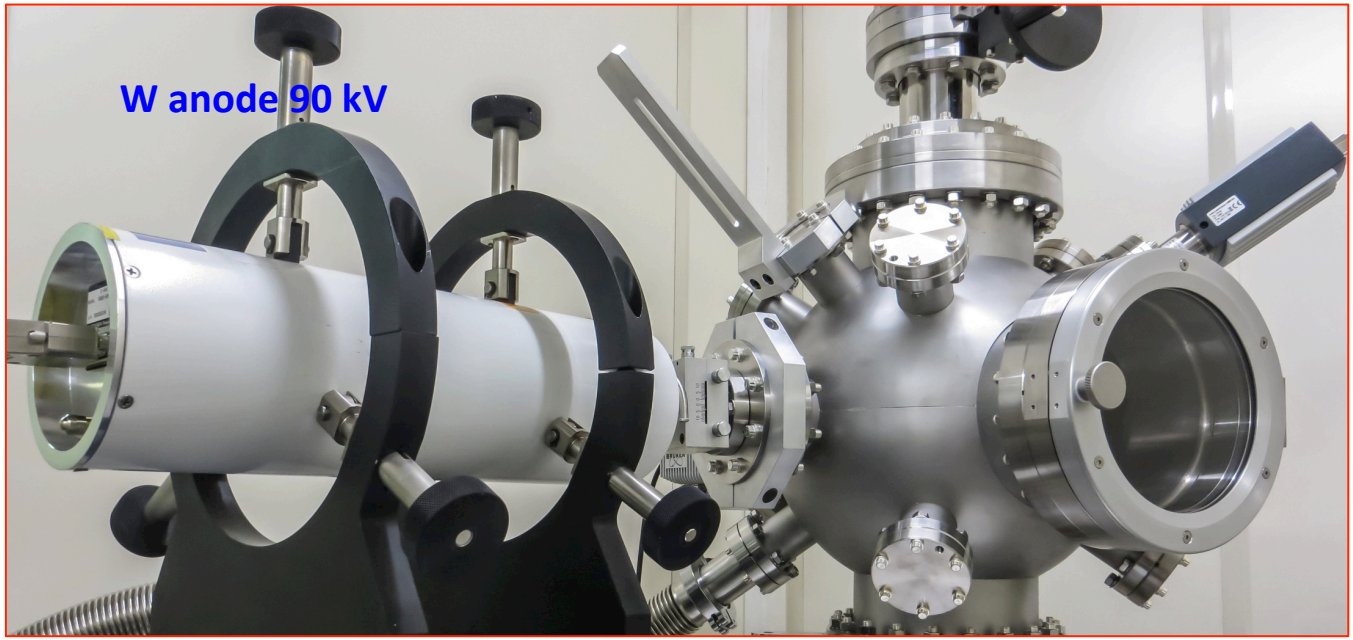
Spectra taken at the LNF DAFNE-Light DXR1 beamline

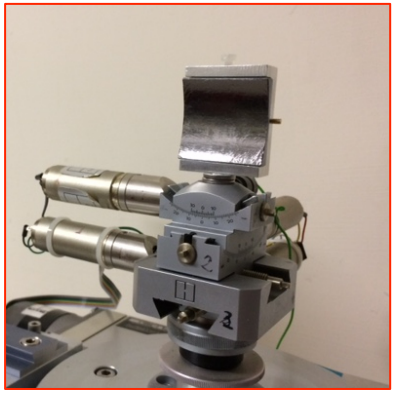
RT, E_{exc} = 2100eV, t_{meas}=900s, 4-ch spectra



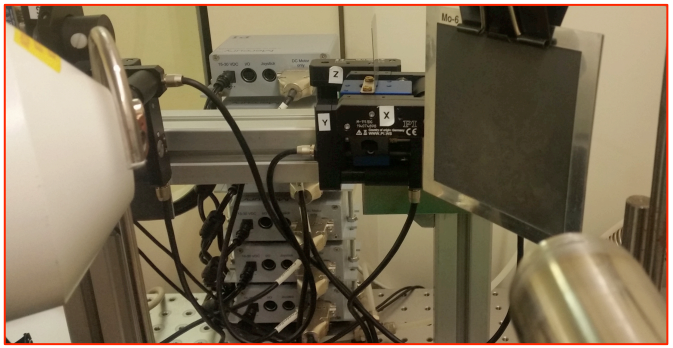
Spectra taken at the ESRF LISA CRG beamline

Test chamber and x-ray conventional source





Testing crystals

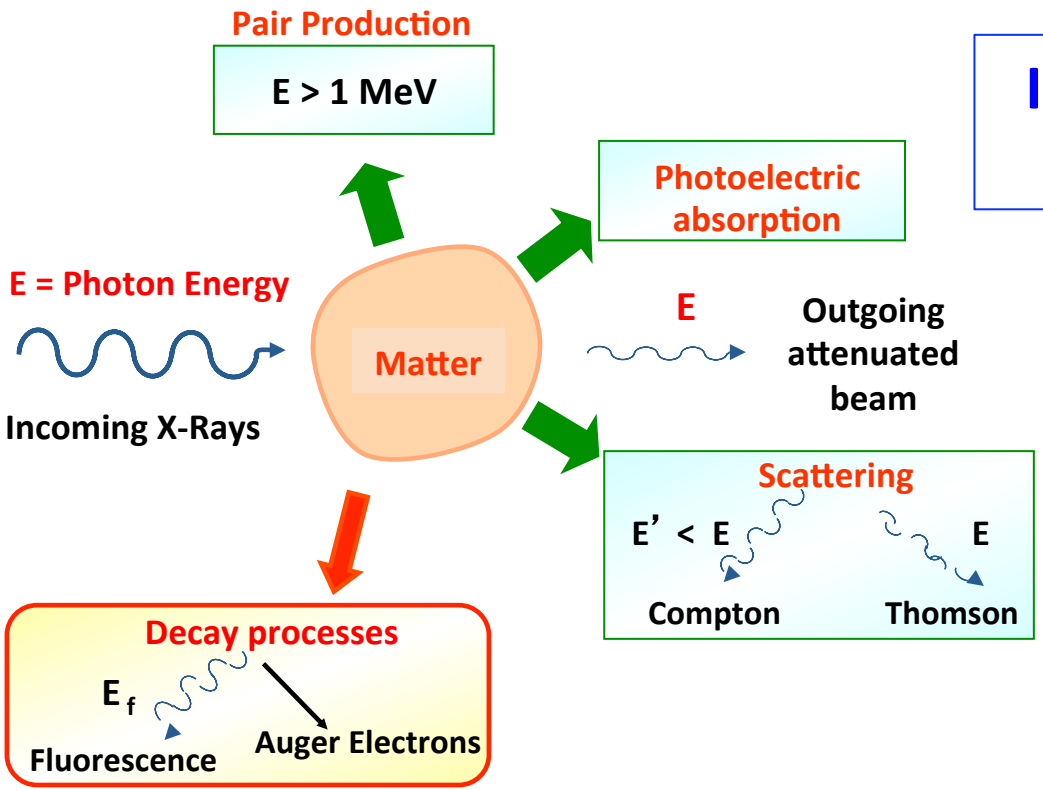


Testing detectors

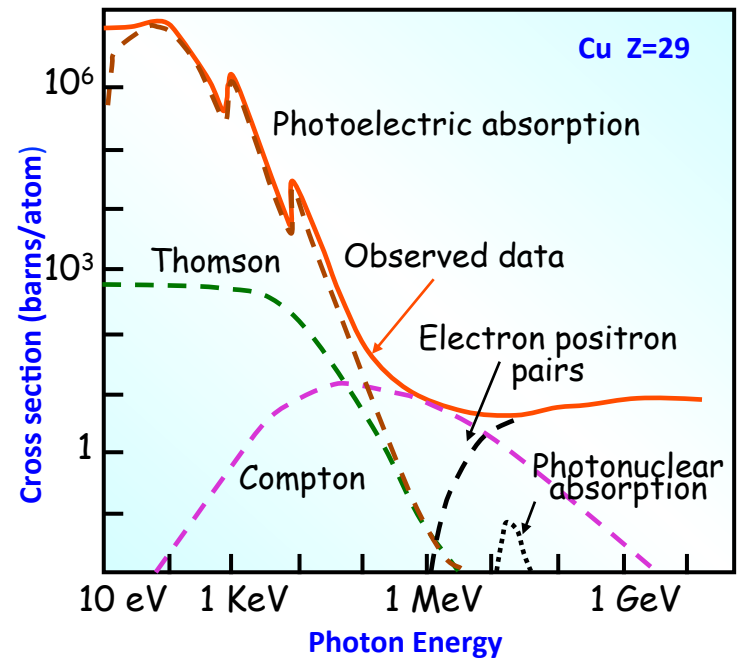
Dispersive XAFS under development

Material Science and XAFS spectroscopy

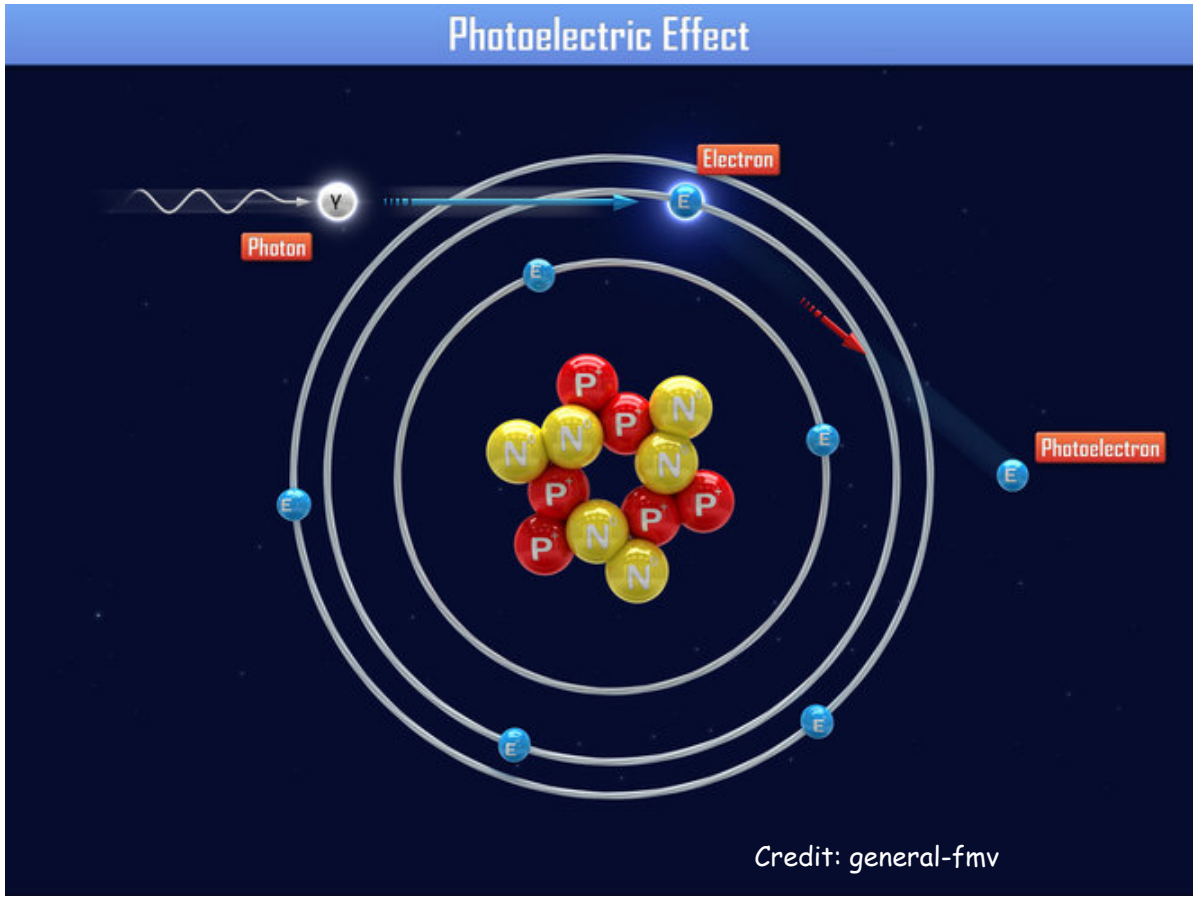
Soft X-ray beam



Interaction of X-rays with matter



Photoelectric effect



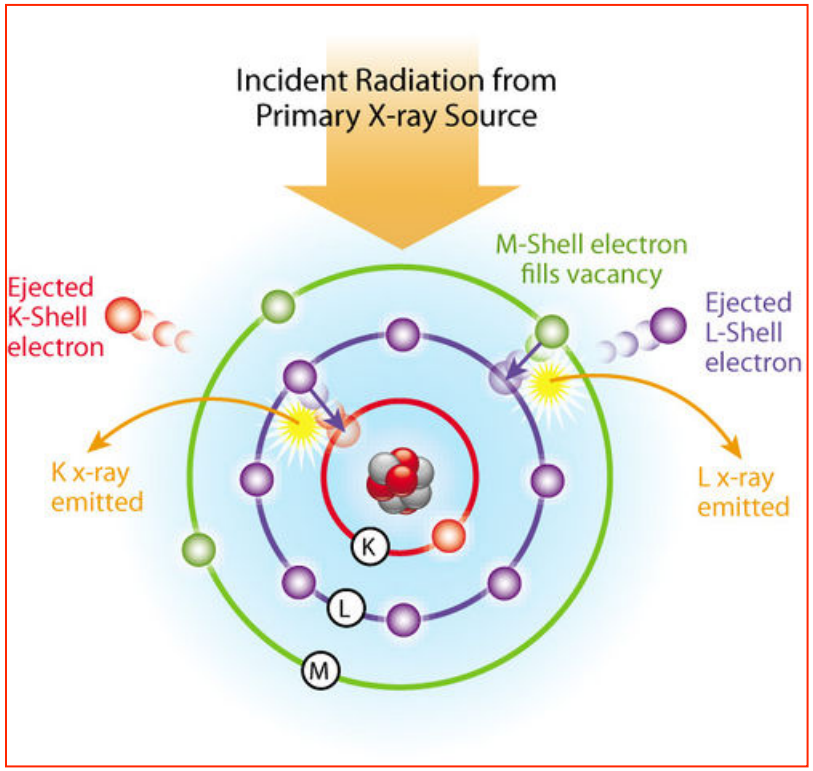
The **photoelectric effect** is the emission of electrons from matter upon the **absorption** of electromagnetic radiation, such as x-rays.

Some X-ray absorption energies

List of selected X-ray absorption edge energies (in electron volt, eV)

Element	Atomic Number	Atomic Weight	K Edge	L1 Edge	L2 Edge	L3 Edge	M1 Edge	M5 Edge
H	1	1.008	13.6	0.0	0.0	0.0	0.0	0.0
He	2	4.003	24.6	0.0	0.0	0.0	0.0	0.0
Li	3	6.941	54.8	0.0	0.0	0.0	0.0	0.0
Be	4	9.012	111.0	0.0	0.0	0.0	0.0	0.0
B	5	10.811	188.0	0.0	4.7	4.7	0.0	0.0
C	6	12.011	283.8	0.0	6.4	6.4	0.0	0.0
N	7	14.007	401.6	0.0	9.2	9.2	0.0	0.0
O	8	15.999	532.0	23.7	7.1	7.1	0.0	0.0
F	9	18.998	685.4	31.0	8.6	8.6	0.0	0.0
Ne	10	20.18	866.9	45.0	18.3	18.3	0.0	0.0
Na	11	22.99	1072.1	63.3	31.1	31.1	0.0	0.0
Mg	12	24.305	1305.0	89.4	51.4	51.4	0.0	0.0
Al	13	26.982	1559.6	117.7	73.1	73.1	0.0	0.0
Si	14	28.086	1838.9	148.7	99.2	99.2	0.0	0.0
P	15	30.974	2145.5	189.3	132.2	132.2	0.0	0.0
S	16	32.066	2472.0	229.2	164.8	164.8	0.0	0.0
Cl	17	35.453	2822.4	270.2	201.6	200.0	0.0	0.0
Ar	18	39.948	3202.9	320.0	247.3	245.2	29.3	0.0

Decay Process: X-ray Fluorescence (XRF)

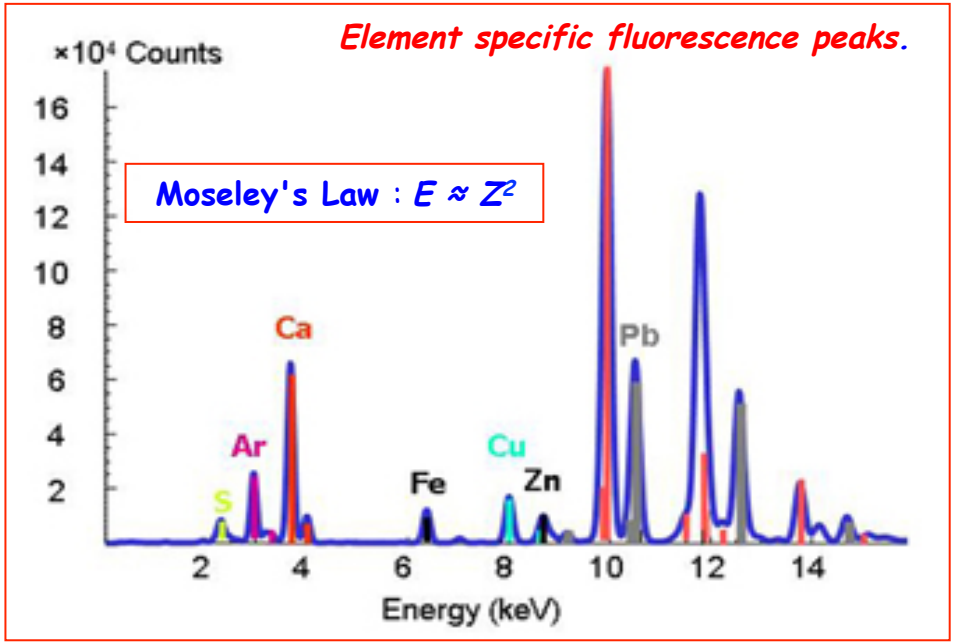


XRF can give an idea of ***the atomic composition of a material***.

If an electron is extracted from an inner shell, an electron from an outer shell will move to replace it. When the electron moves from the outer shell to the inner shell, it ***releases energy in the form of a photon (light)***.

The energy of the ***"fluorescent" photon*** released is **different for each atomic element** creating a measurable "**fingerprint**" for that element.

XRF produces a **non-destructive chemical analysis of any kind of sample.**

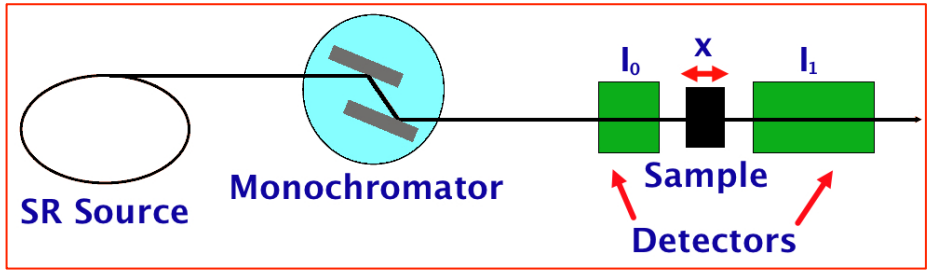


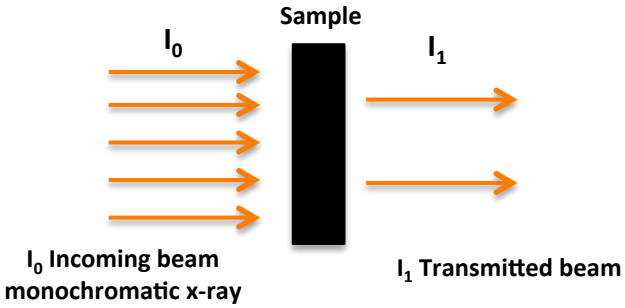
Some X-ray fluorescence energies

X-Ray Data Booklet Table 1-2. *Photon energies, in electron volts, of principal K-, L-, and M-shell emission lines.*

Element	$K\alpha_1$	$K\alpha_2$	$K\beta_1$	$L\alpha_1$	$L\alpha_2$	$L\beta_1$	$L\beta_2$	$L\gamma_1$	$M\alpha_1$
3 Li	54.3								
4 Be	108.5								
5 B	183.3								
6 C	277								
7 N	392.4								
8 O	524.9								
9 F	676.8								
10 Ne	848.6	848.6							
11 Na	1,040.98	1,040.98	1,071.1						
12 Mg	1,253.60	1,253.60	1,302.2						
13 Al	1,486.70	1,486.27	1,557.45						
14 Si	1,739.98	1,739.38	1,835.94						
15 P	2,013.7	2,012.7	2,139.1						
16 S	2,307.84	2,306.64	2,464.04						
17 Cl	2,622.39	2,620.78	2,815.6						
18 Ar	2,957.70	2,955.63	3,190.5						
19 K	3,313.8	3,311.1	3,589.6						
20 Ca	3,691.68	3,688.09	4,012.7	341.3	341.3	344.9			
21 Sc	4,090.6	4,086.1	4,460.5	395.4	395.4	399.6			

XAFS Spectroscopy





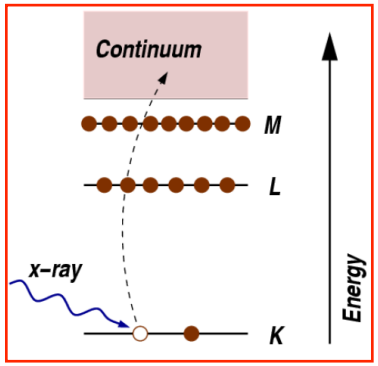
$$I_1 = I_o \exp[-\mu(E)\, x]$$

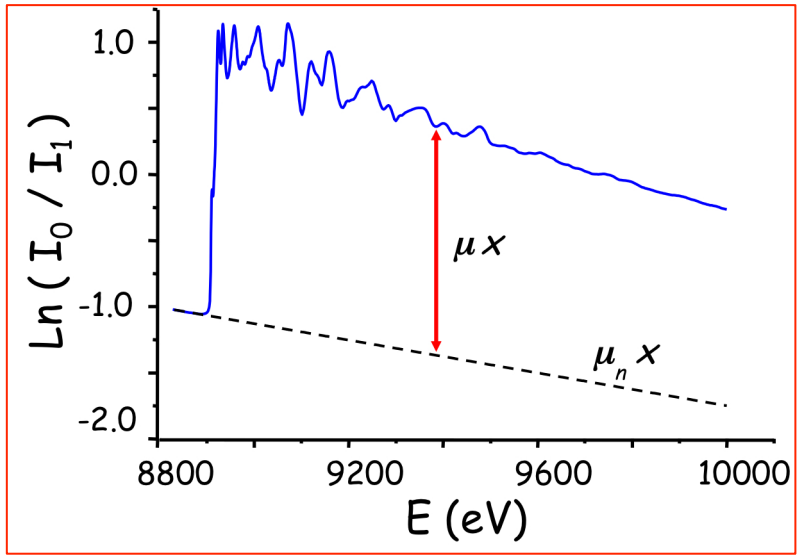
Exponential attenuation or equation of Beer-Lambert

Absorption coefficient

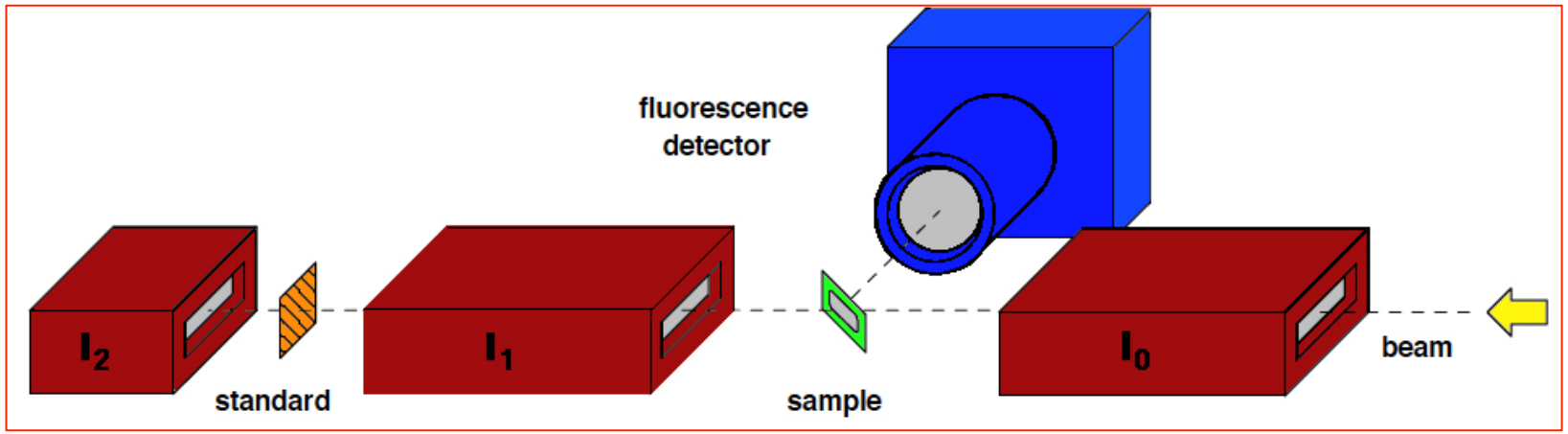
$$\mu(E) = \frac{1}{x} \ln\left(\frac{I_0}{I_1}\right)$$

Photoelectric interaction depends on the energy of the x-ray that must be equal or greater than the electron binding energy.





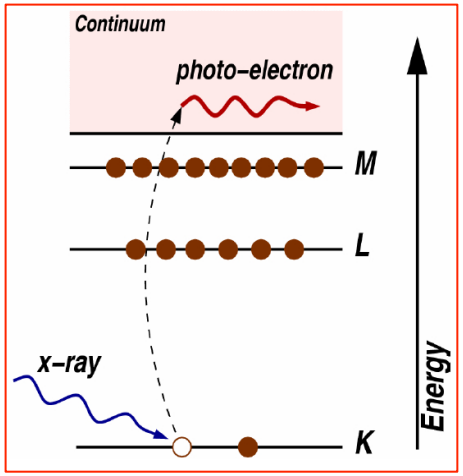
XAFS Spectroscopy modes



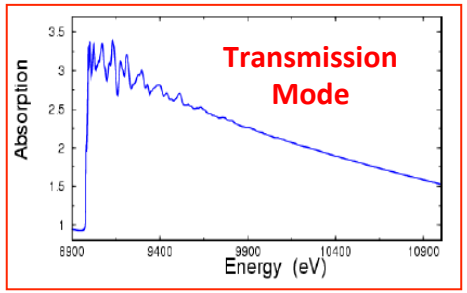
XAFS can be measured either in Transmission or Fluorescence mode.

The probability of Fluorescence emission is directly proportional to the absorption probability.

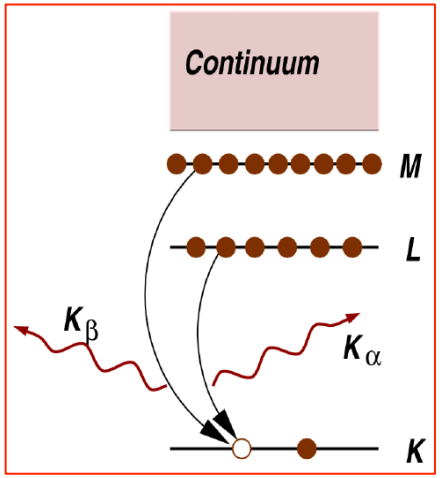
X-ray absorption



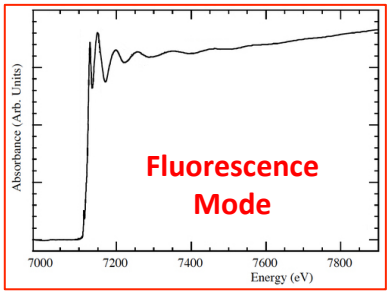
$$\mu(E)x = ln\left(\frac{I_0}{I_1}\right)$$



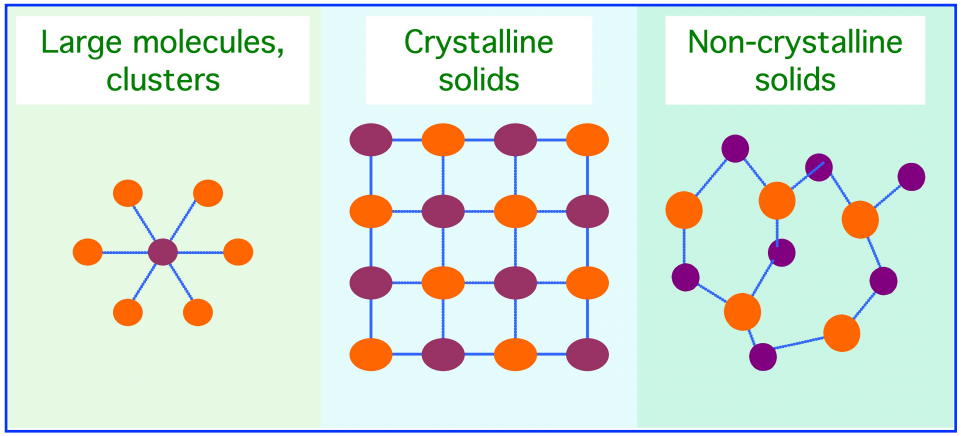
X-ray fluorescence



$$\mu(E)x = \left(\frac{I_F}{I_0}\right)$$



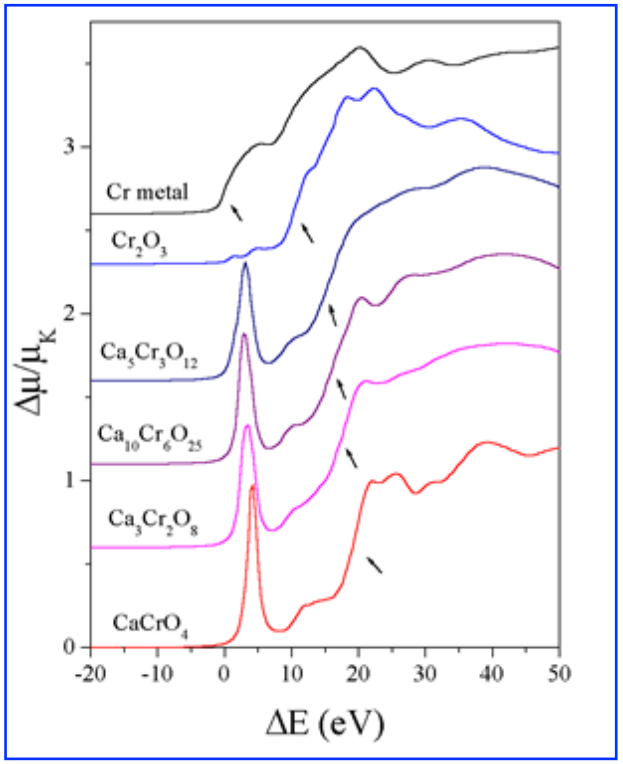
XAFS spectroscopy as a very powerful tool

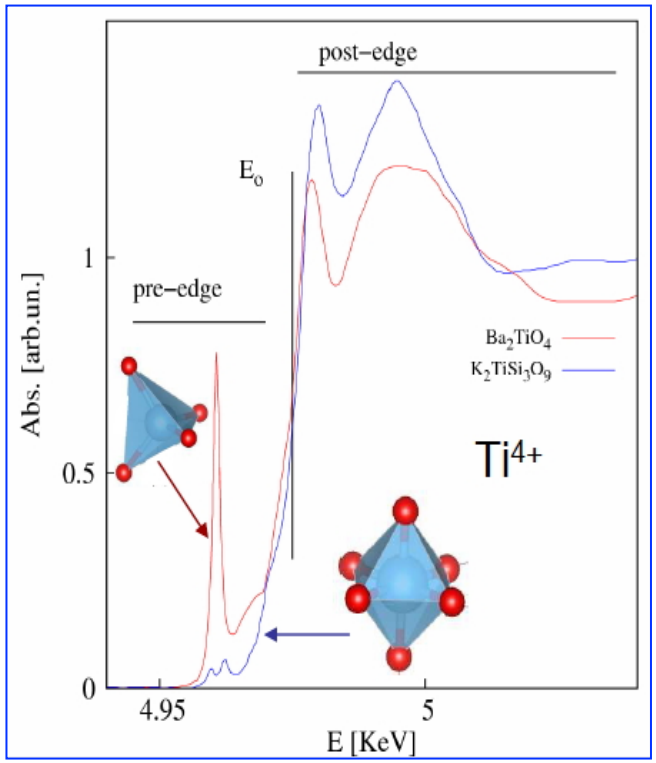


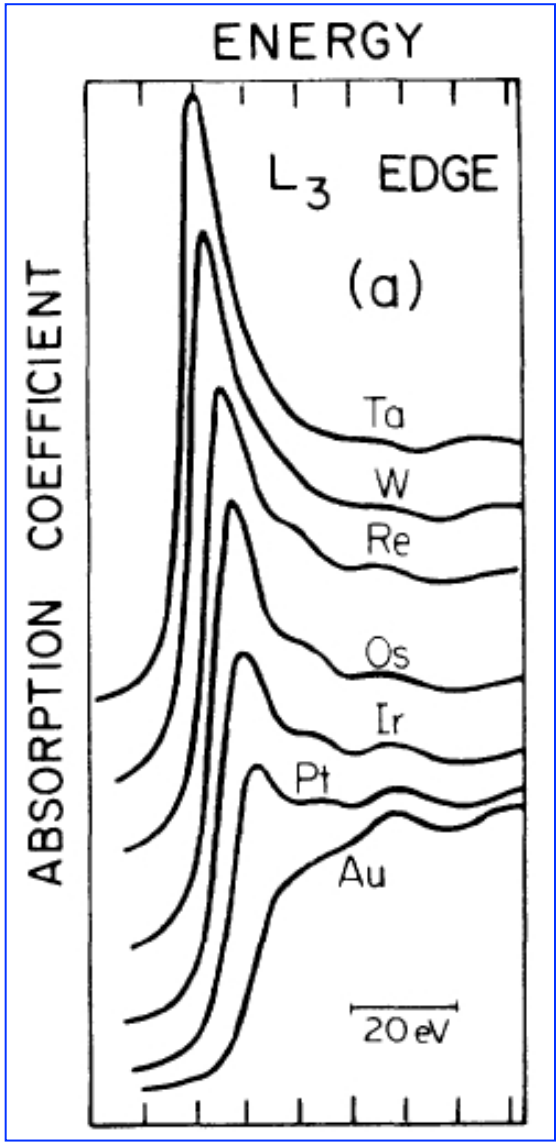
XANES Information

XANES provide quantitative information on:

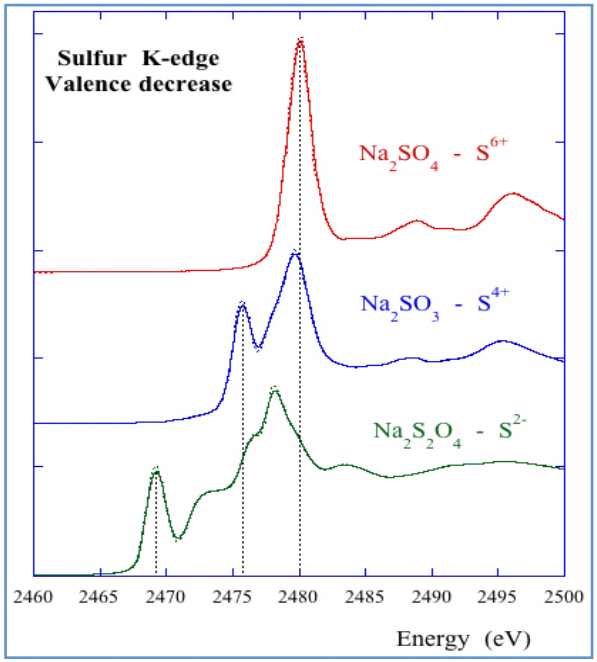
- **Average oxidation state**
- **Local coordination environment, site symmetry**
- **Electronic structure (empty density of states)**



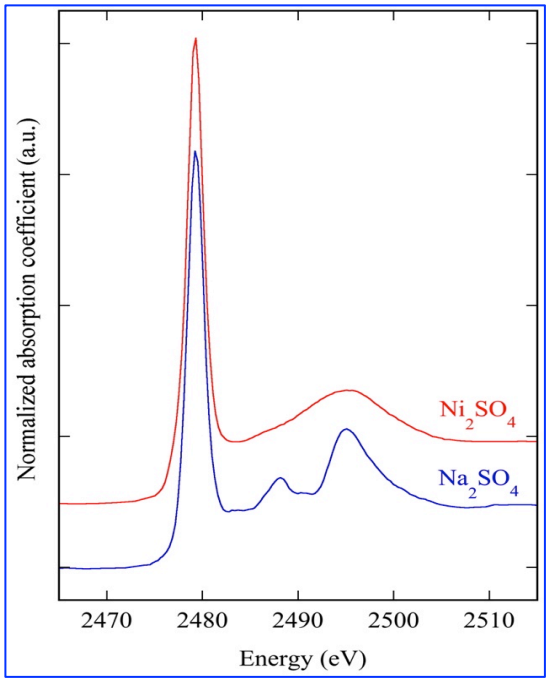




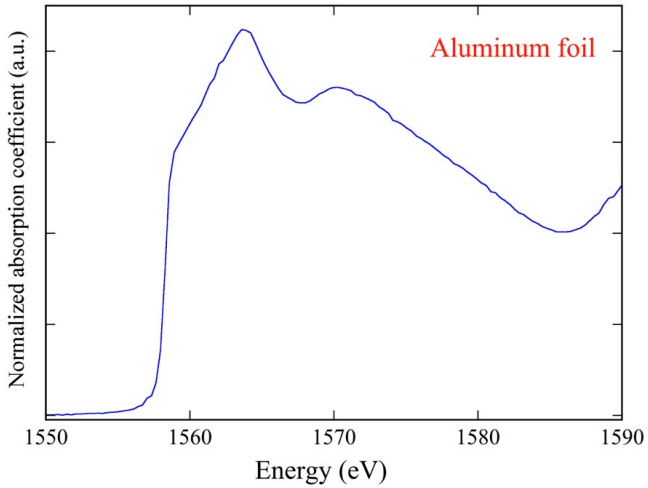
XANES DXR1

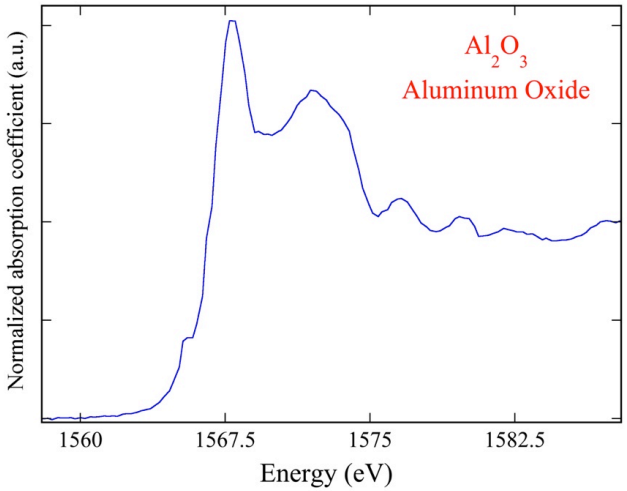


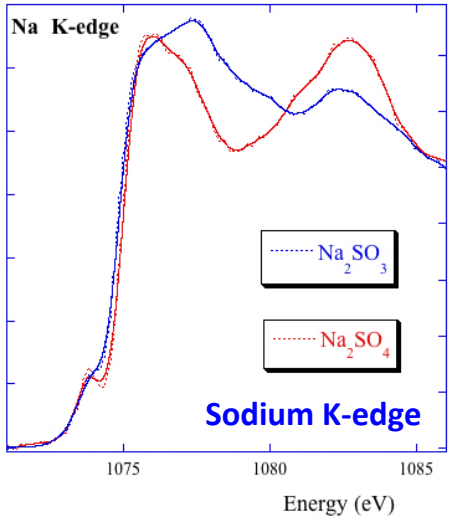
Sulfur K-edge



Aluminum K-edge

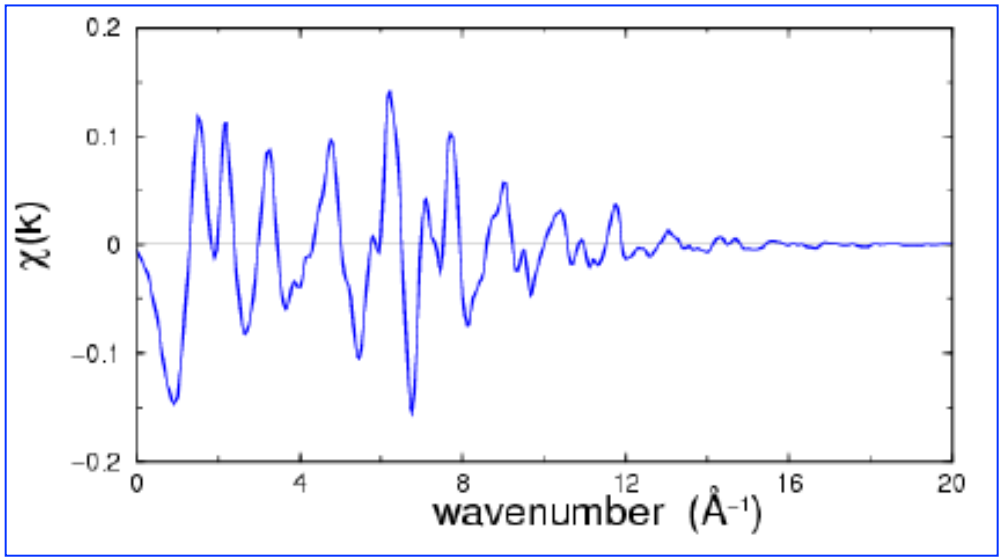




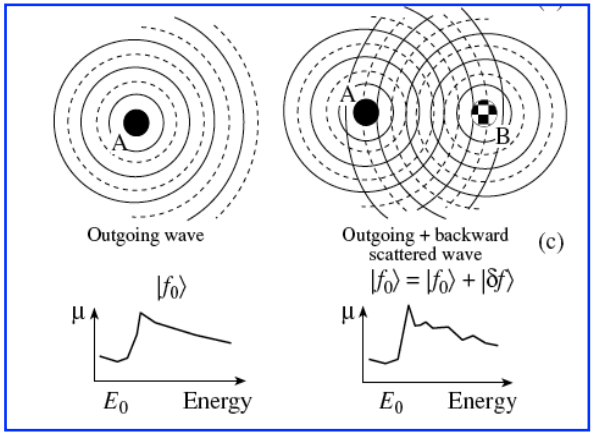


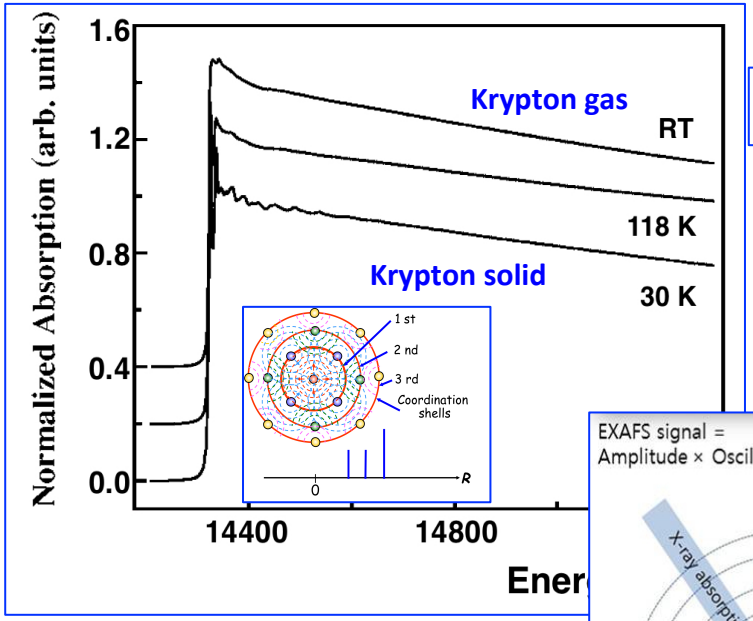
EXAFS Information

Extended X-ray Absorption Fine Structure

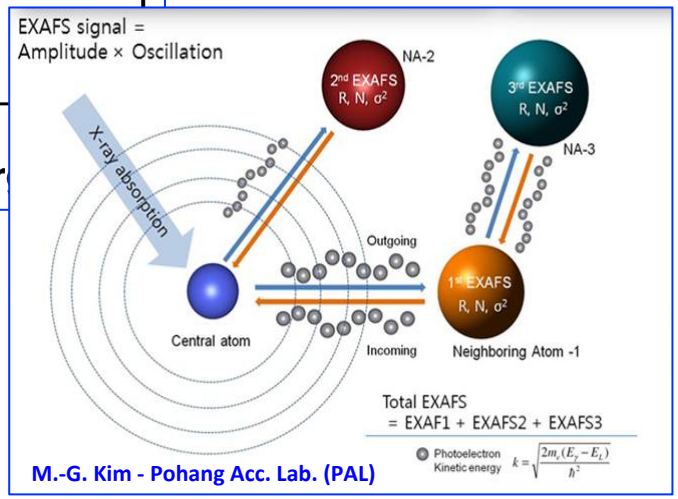


EXAFS phenomenological interpretation

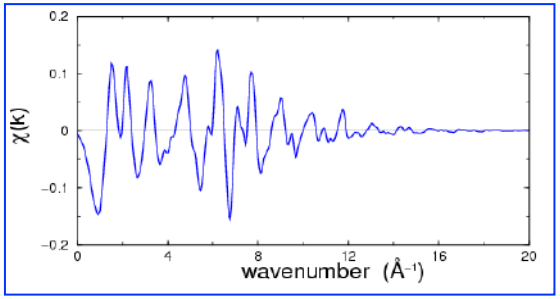




Q. Wang et al. Phys. Rev. B 72, 092202 (2005)



M.-G. Kim - Pohang Acc. Lab. (PAL)



$$\chi(k) = -\frac{S_0^2}{k}\sum_s N_s \frac{|f_s(\pi,k)|}{R_s^2} e^{-k^2/\sigma_s^2} e^{-2R_s/\lambda_s} \sin\left(2kR_s + \phi_s(k)\right)$$

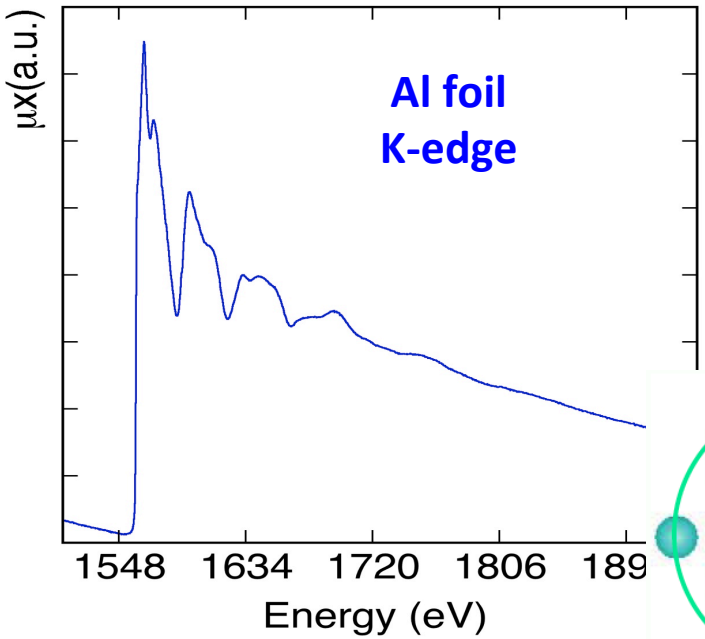
EXAFS

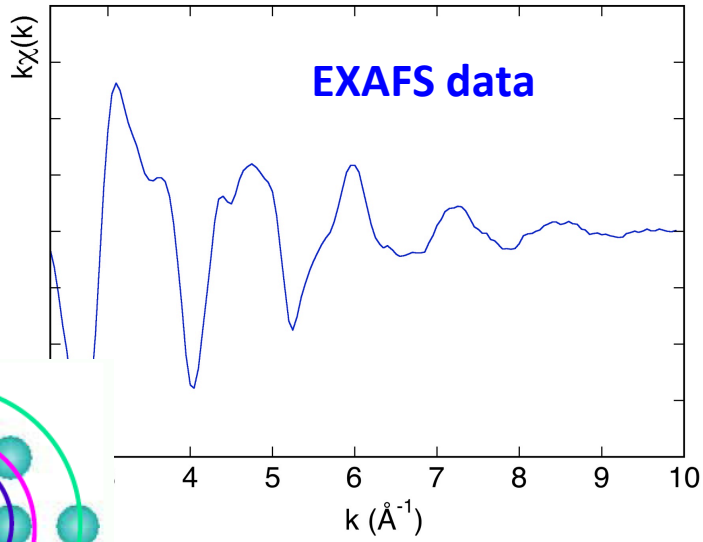
Coordination number

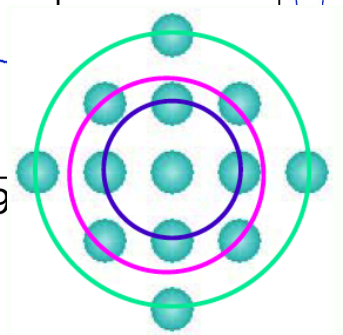
Debye Waller factor

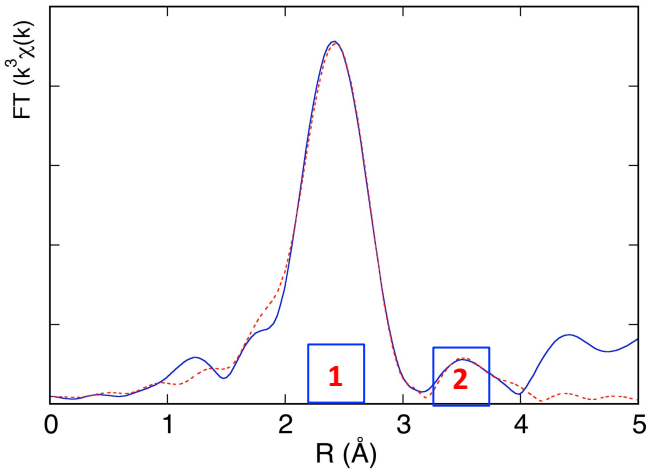
Interatomic distance

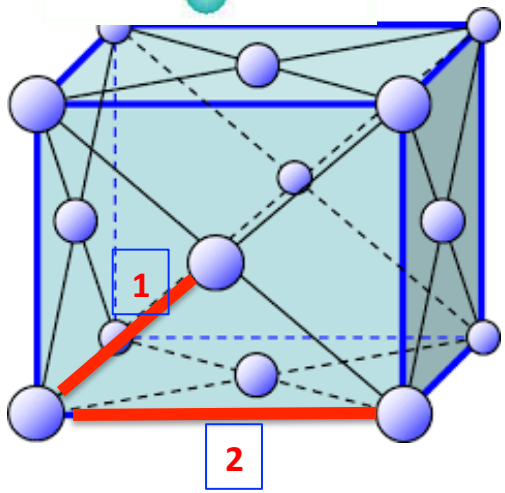
EXAFS DXR1











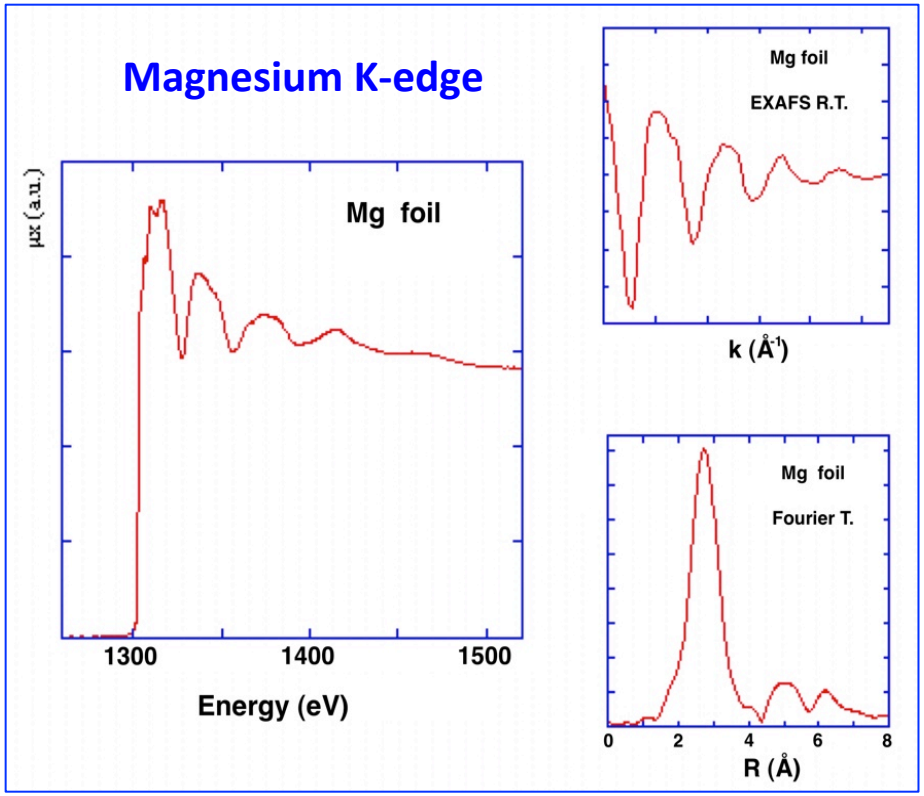
N_{1Al}= 12
R_{1Al}= 2.85 Å
$\sigma_1^2{}_{Al}$=0.014 Å^2

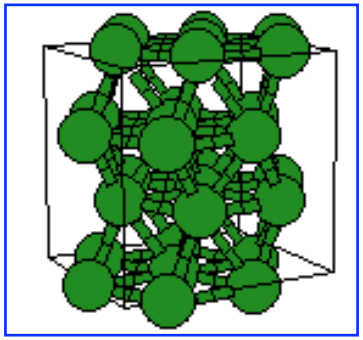
First shell

N_{2Al}= 6
R_{2Al}= 4.04 Å
$\sigma_2^2{}_{Al}$=0.027 Å^2

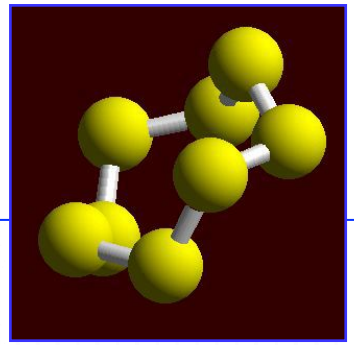
Second shell

EXAFS DXR1

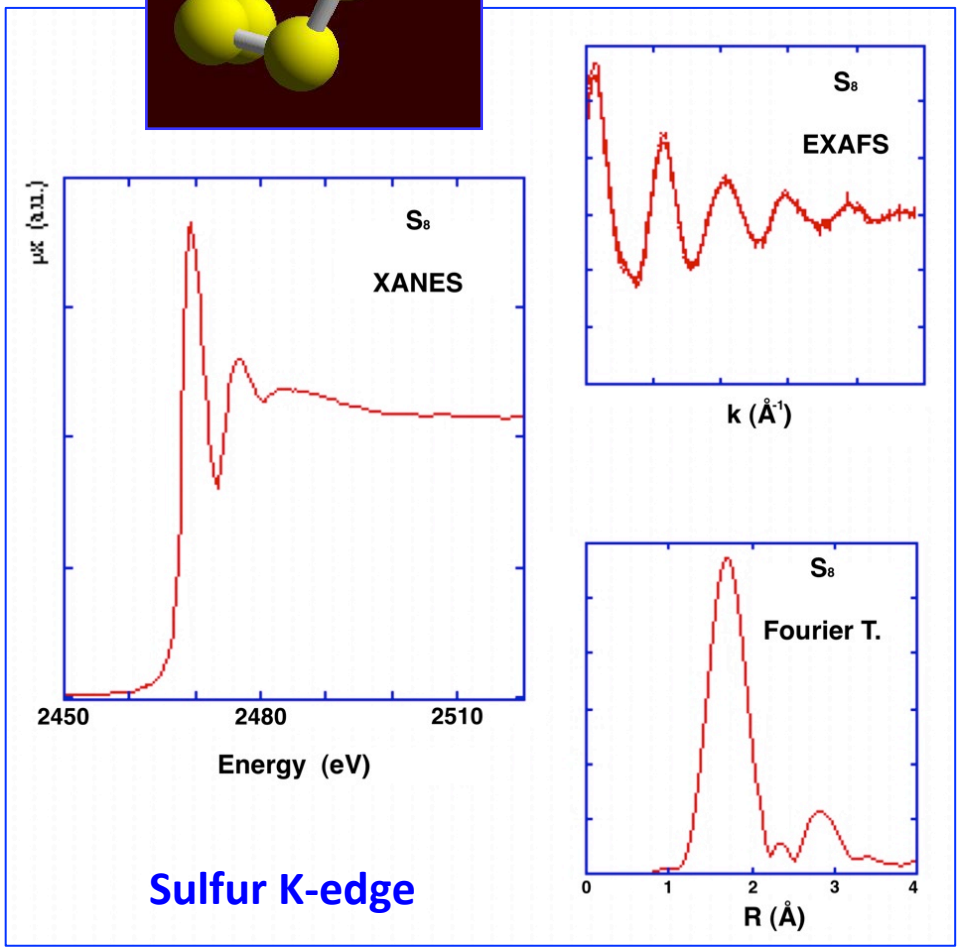




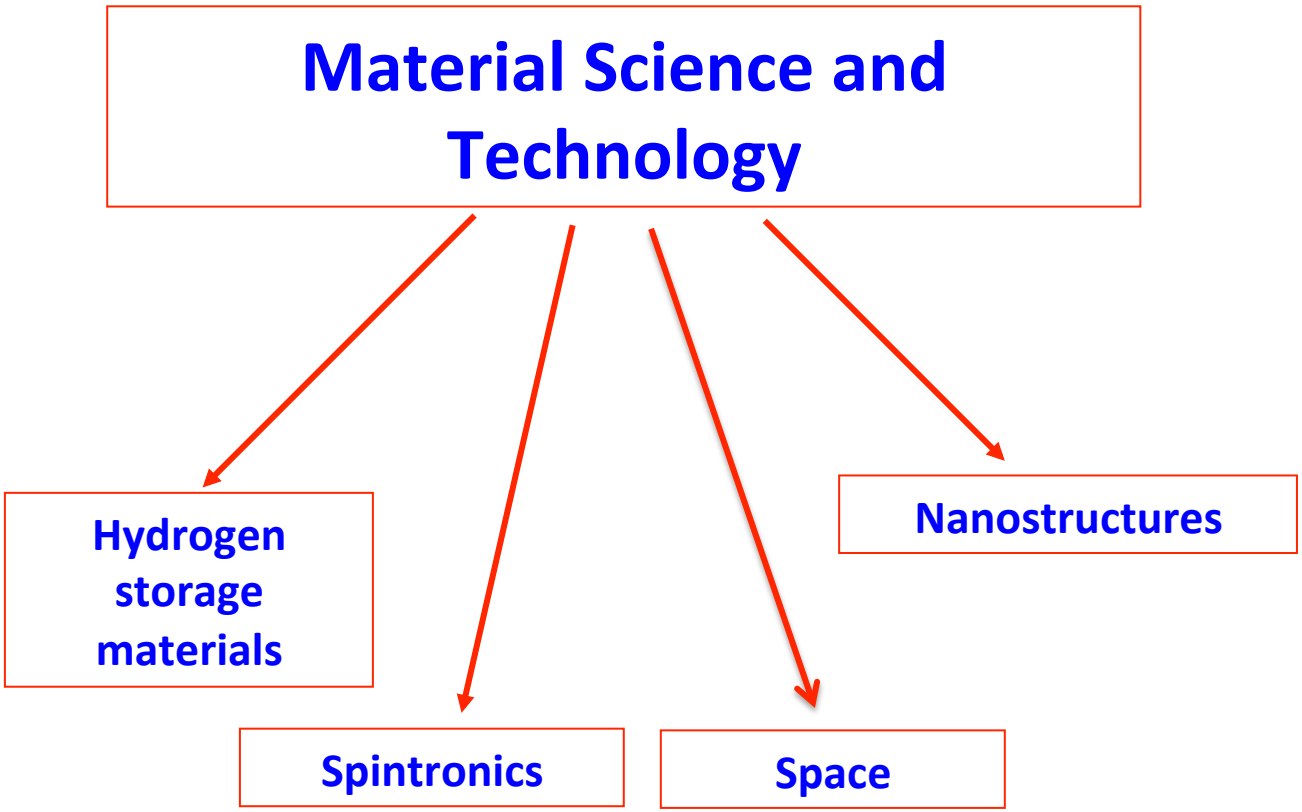
Mg metal HCP crystal structure

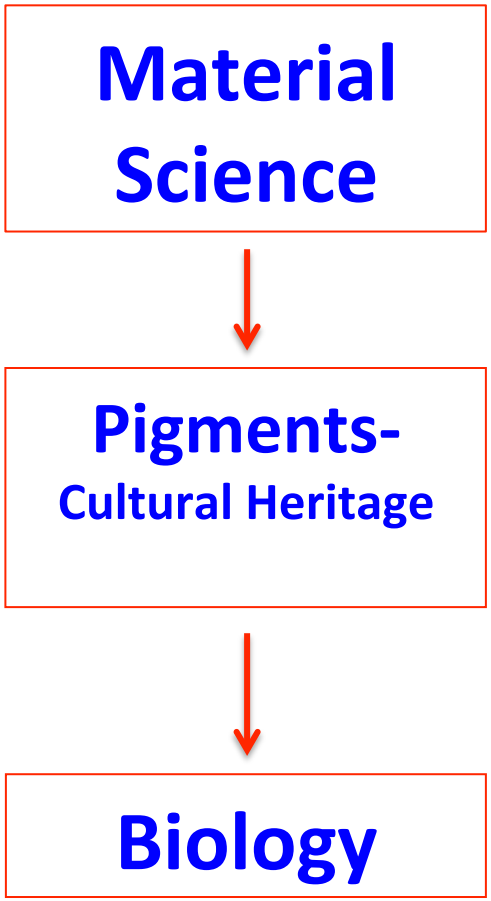


S_8



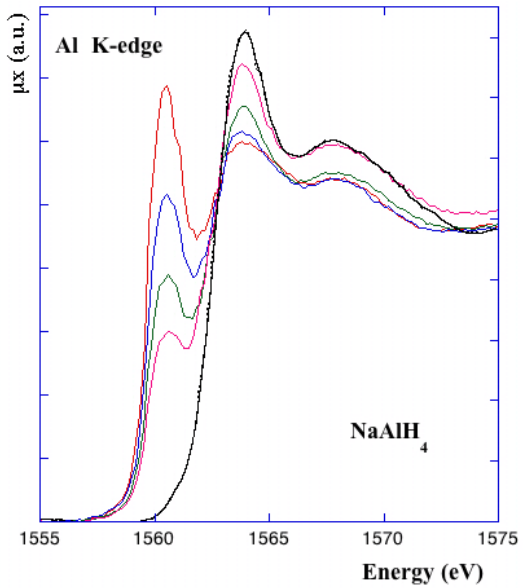
Some Applications @ DXR1





Technology

Hydrogen storage materials



Al K-edge XANES study of alkali aluminium hydrides

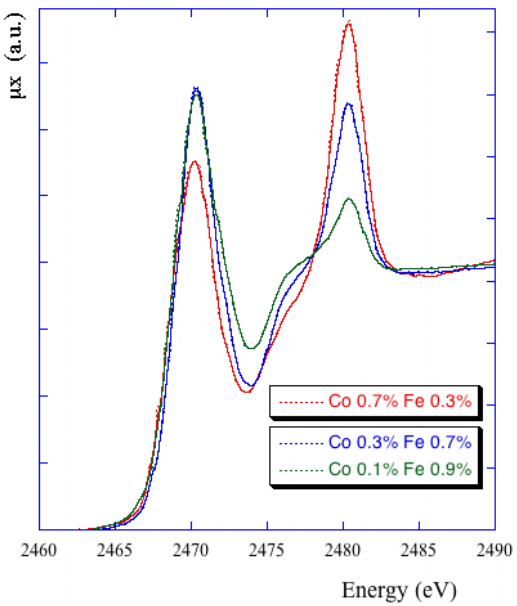
Development of higher-efficiency hydrogen storage materials

The aim of the experiments is to investigate the local structure around Al atoms in nanoscale hydrogen storage materials, **studying the alanate phase at different stages of the reaction when the material is cycled under hydrogen**.

This will allow to clarifying if a modification occurs to the alanate structure during cycling under hydrogen.

A. Leon et al. - KIT

Spintronics



Near-edge X-ray Absorption spectroscopic investigations: Fe doped CoS_2

Conventional electronics is based on the **transport of charges**. **Spintronics** or **spin-based electronics** is an **emergent technology** which **exploits the quantum spin states of electrons**. The electron spin itself is manifested as a two state magnetic energy system. The structural investigation around the S K-edge of **Fe doped CoS_2 samples** gives information on the **electronic structural changes** induced by **doping of 3d transition metal ion**, **Fe** of **$Co(1-x)Fe(x)S_2$**.

I. Zuburtikudis - Dept. of Engineering, GREECE

Technology

Space

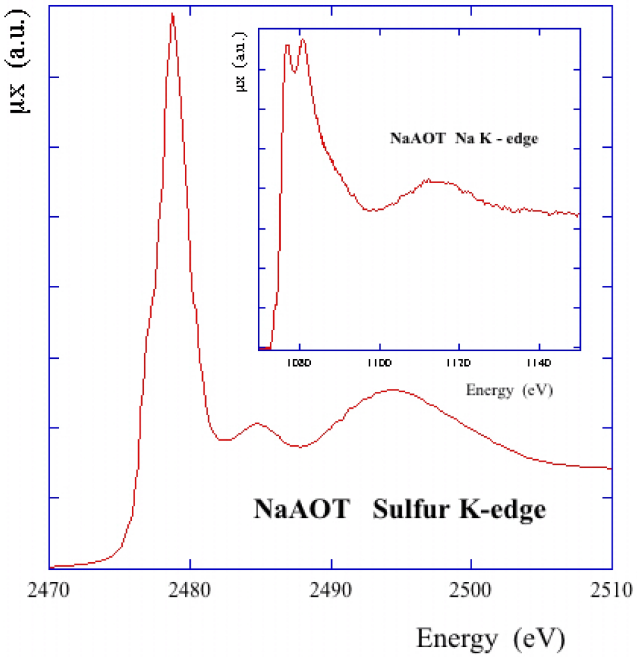
Characterization of thermal filters as a function of temperature

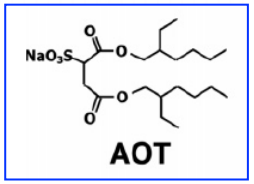
ATHENA is a large astrophysics space mission approved by the European Space Agency (launch scheduled in 2028) to address the science theme "**Hot and Energetic Universe**". The main instrument on-board ATHENA is the **X-ray Integral Field Unit (X-IFU)**, an x-ray detector with spectral, imaging and timing capabilities in the energy range 0.2÷12 keV. **Thermal filters need to be mounted to protect the detector**.

1. Measuring the **X-ray transmission curves in the energy range 1500-1750 eV** should map the regions around the absorption edges of aluminum with an energy resolution similar to that one of the X-IFU.
2. **Monitoring the temperature dependence of the Absorption Fine Structures as a function of temperature** from room temperature to LN temperature

M. Barbera and L. Sciortino, Univ. Palermo

Nanostructures





Sodium bis (2-ethylhexyl) sulfosuccinate -**NaAOT**

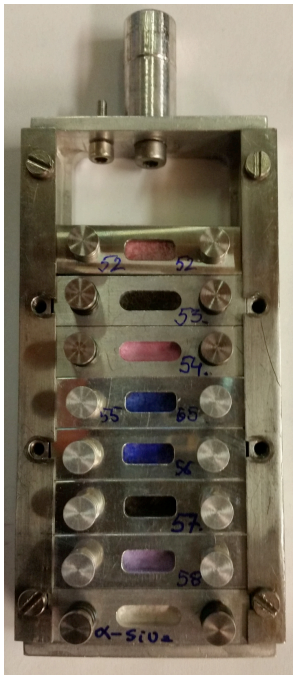
Structural investigation of water-soluble inorganic salts within reversed micelles or liquid crystals used as good templates in which nanoparticles can be trapped and stabilized.

System studied: Co, Yb and Er in NaAOT

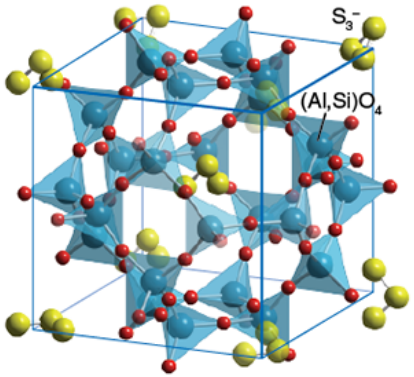
V. Turco Liveri, Univ. Palermo

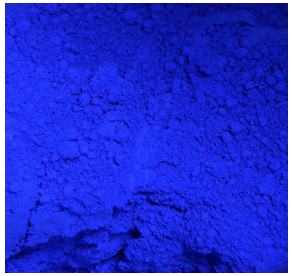
Study of Pigments

possible applications in the Cultural Heritage field



XAFS RT sample holder





Silicon K Edge XAS study of Silicate Pigments

Silicon pigments have been known since millennia. Their properties were first investigated at the beginning of the XIX century but the **exact structure** and composition of $BaCuSi_2O_6$ (Han purple), $BaCuSi_4O_{10}$ (Han blue) and $CaCuSi_4O_{10}$ (Egyptian blue) **were established over 100 years later.**

Contemporary, $CaCoSi_2O_6$ is used in industry as pigment varying in colors from blue to pink.

Zajdel P. et al. Univ. Of Silesia, Poland.

Extra framework molecules in lazurite: a combined XAS/FTIR study

Lapis lazuli is a rare and precious pigment exploited and prized for its deep blue color since, at least, the 5th century B.C.

Lapis lazuli is indeed a complex rock characterized by the **abundance of the mineral lazurite** $(Na,Ca)_8(AlSiO_4)_6(SO_4,S,Cl)_2$, which is responsible for its overall blue hue. **Lazurite belongs to the sodalite group of minerals**, which includes, along with sodalite, also nosean and hauyne.

A series of sodalite-group minerals spanning a large range of colors, have been studied as a function of T using Raman spectroscopy observing changes probably related to the S sites.

Following these results it was proposed to study using XANES spectroscopy at the S K-edge a set of samples treated at different T and for different time durations.

Della Ventura G. Univ. Roma Tre

Biology

X-Ray Absorption Spectroscopy to Characterize Metallodrug/Protein Interactions

Interactions of metal-based drugs with serum proteins have biological and pharmacological implications.
Specifically, **great attention has been given to ruthenium and gold complexes that seem to be very promising**. The mechanisms through which the **metal complexes produce their biological and pharmacological effects** are still largely unexplored and it seems that **gold and ruthenium complexes act on different targets, most likely on protein targets.**

I. Ascone et al.

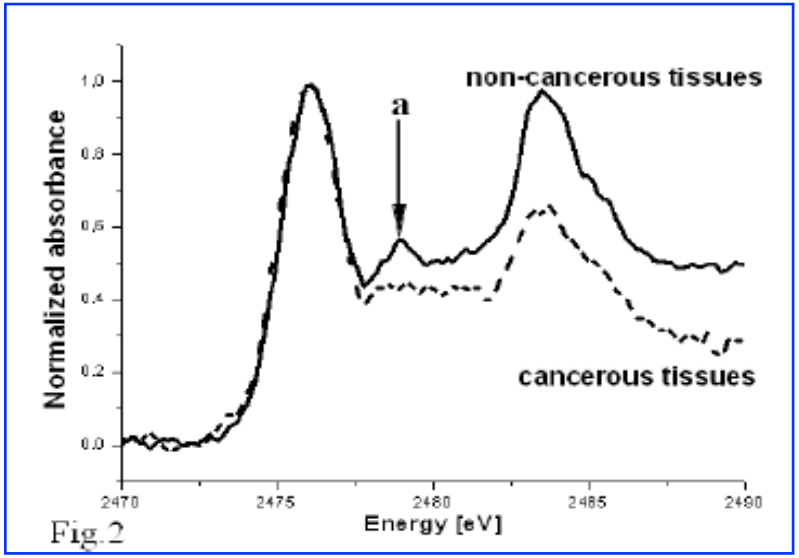
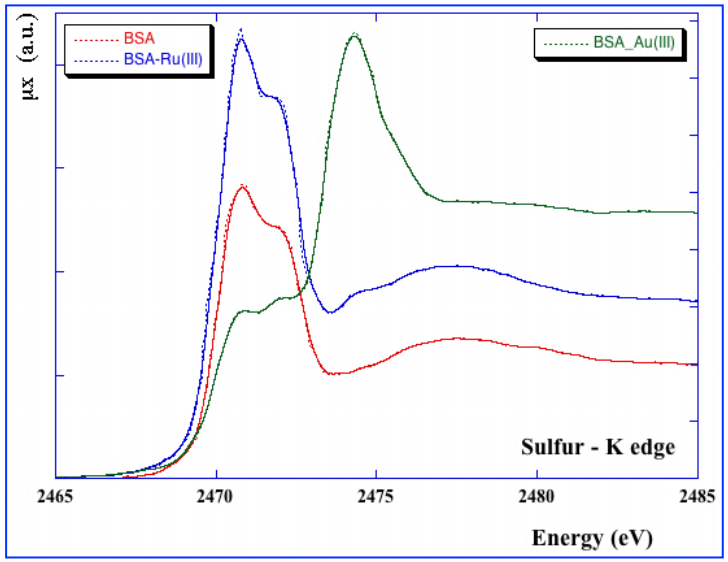


Fig. 2



Preliminary Study on Chemical Speciation of Sulfur in Cancerous Tissues

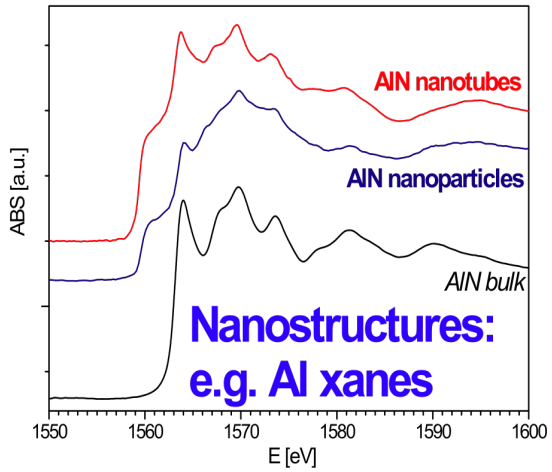
Urological cancers comprise approximately one-third of all cancers diagnosed in men worldwide and out of these, prostate cancer is the most common one.

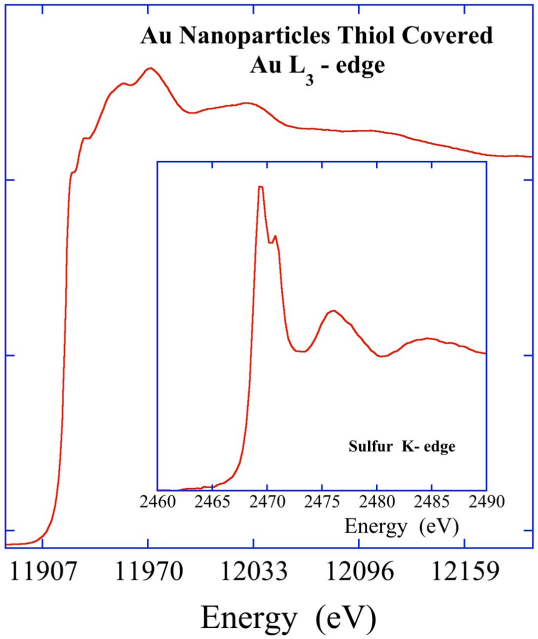
Out of many relevant factors, it is believed that **sulfur can take an important part in cancer transformations**. Prostate cancer tissues along with selected organic and inorganic compounds used as references have been investigated.

W.M. Kwiatek et al., Division of Inter. Res. IFJ PAN, Poland

Nanomaterial Science

Nanostructures





Characterization of Aluminum Nitride Nanostructures by XANES, XRD, FTIR

The atomic structure of nanotubes or particles is normally different to that in the bulk due to the reduced symmetry. **Both hexagonal (wurtzite-AlN) and cubic phases (zincblende-AlN) of aluminum nitride have found applications in a variety of technologies, ranging from cutting/grinding to corrosion or in exotic devices such as electro-acoustic systems.** The main result obtained from the analysis of the XANES is **the high sensitivity to the presence of both symmetries: hexagonal and cubic**. Indeed, all spectra are characterized by the **same features whose intensity scales proportionally with the content of the wurtzite AlN phase.**

C. Balasubramanian et al.

Structural investigation of thiol-capped gold nanoparticles

The use of **multithiolated species improves the stability of gold nanoparticle**.
Sulfur K-edge XANES spectroscopy as an attractive approach for characterizing **multithiolated or disulfide species adsorbed on gold surface nanoparticles**.

T. Comaschi et al.

Thank you for your attention