

# X-Ray Activities @ DAΦNE-Light

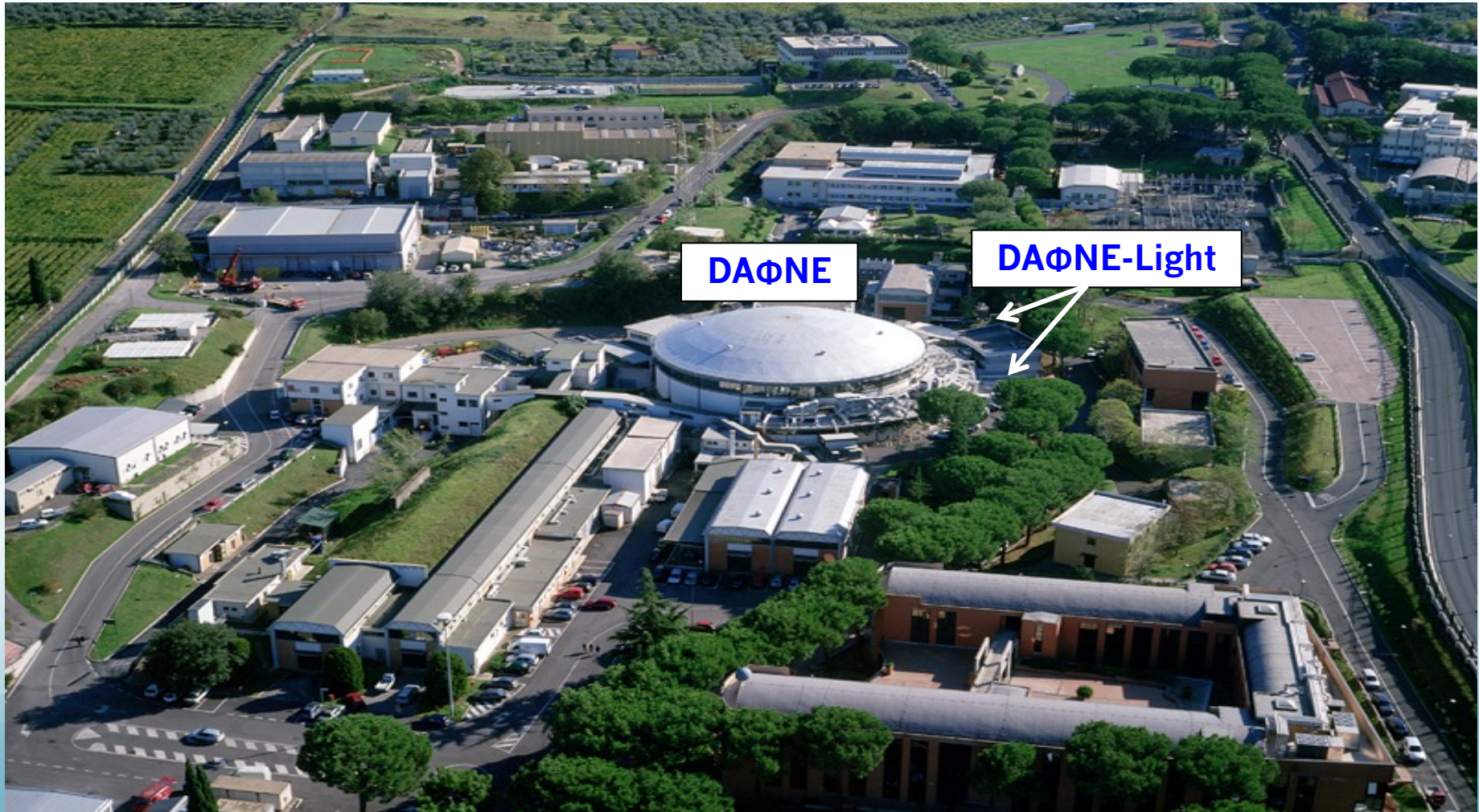
Antonella Balerna



INFN-LNF DAΦNE-Light Synchrotron Radiation Facility



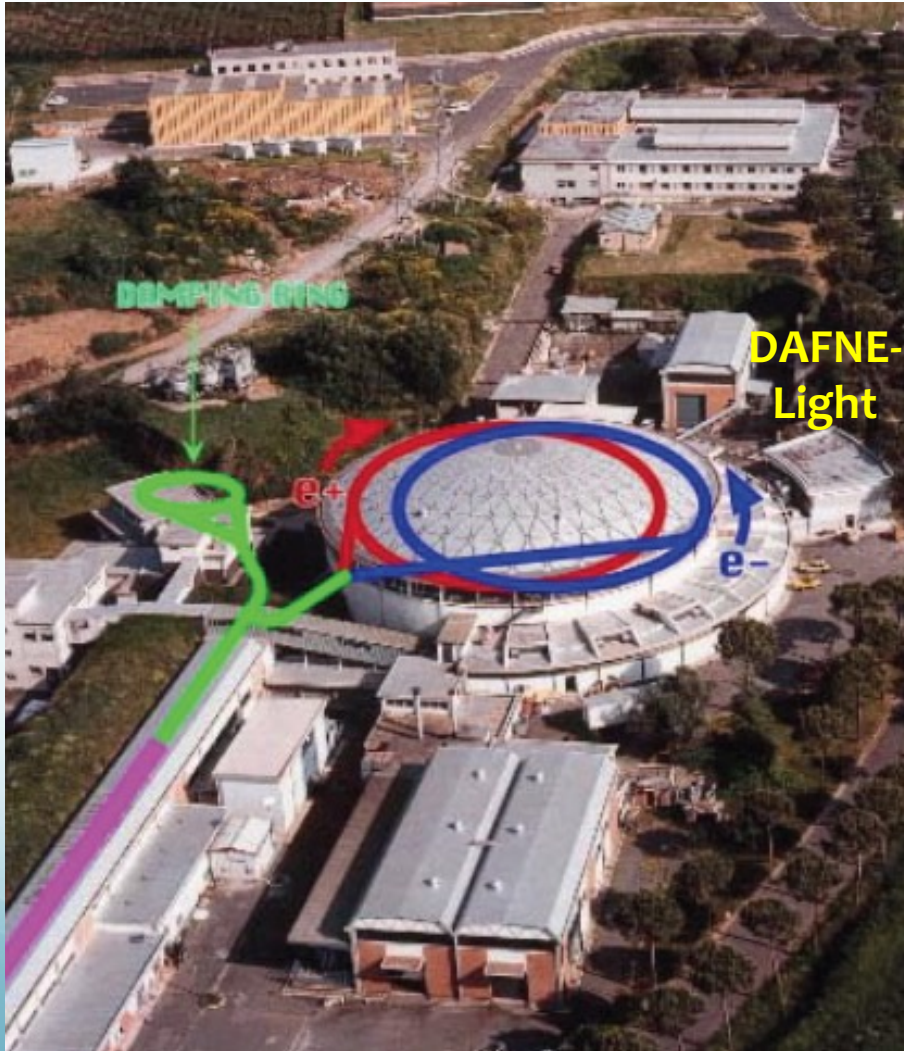
# DAΦNE-Light INFN-LNF Synchrotron Radiation Facility





# DAΦNE

## Double Annular $\Phi$ factory for Nice Experiments



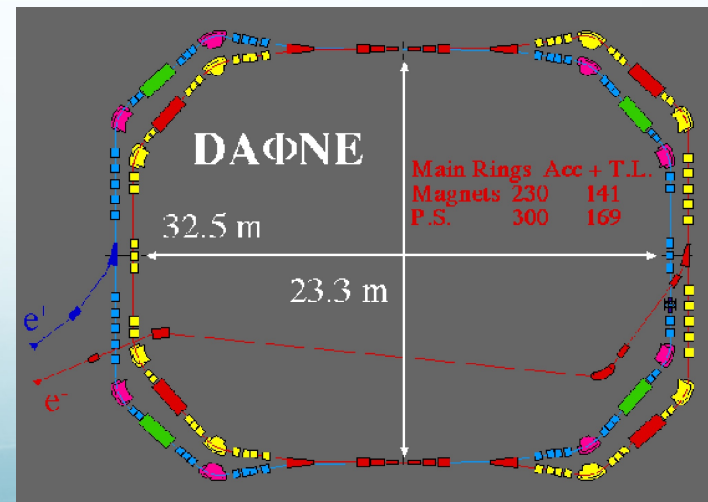
*DAΦNE is a two rings  $e^+/e^-$  collider,  
with two interaction regions.*

*Energy = 0.51 GeV*

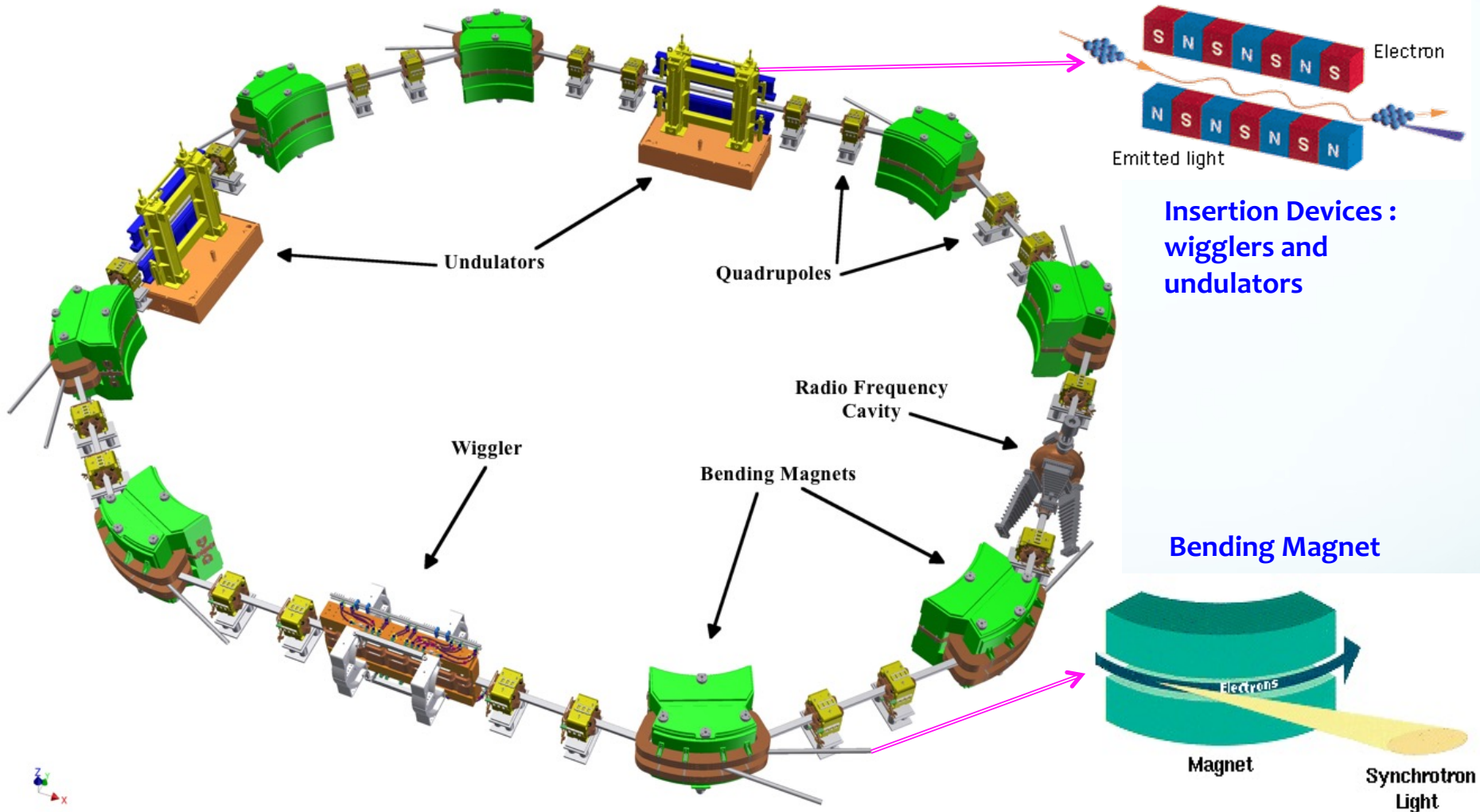
*Circumference = 97.7 m*

*$I > 1500$  mA  $e^-$*

*$I > 1000$  mA  $e^+$*



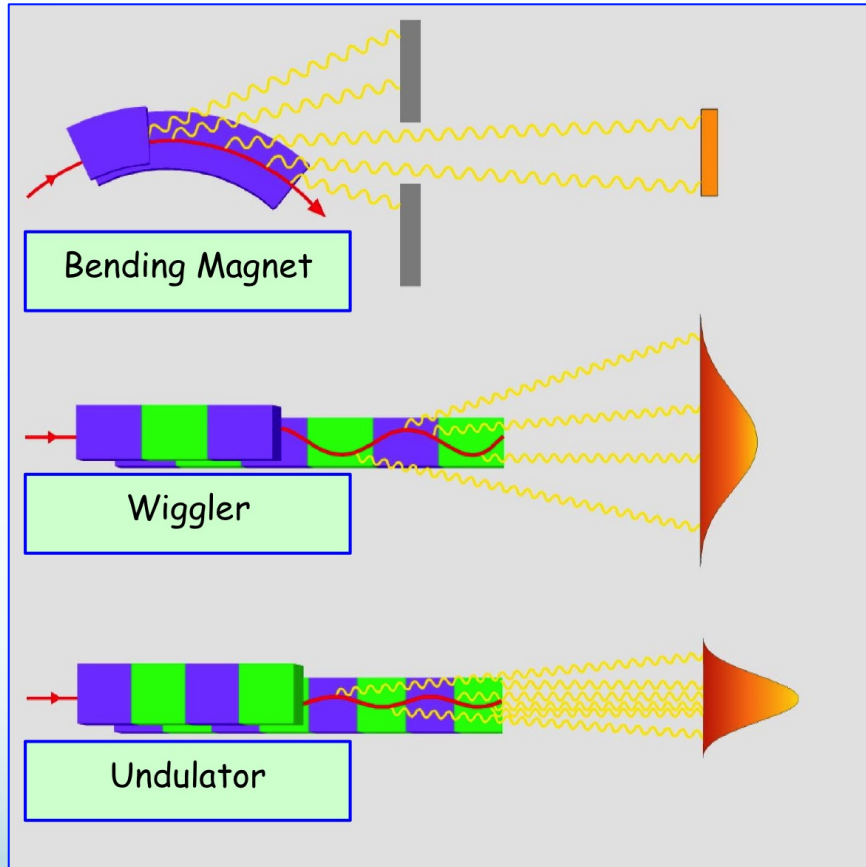
# Synchrotron light and relativistic particles accelerated in circular orbits.



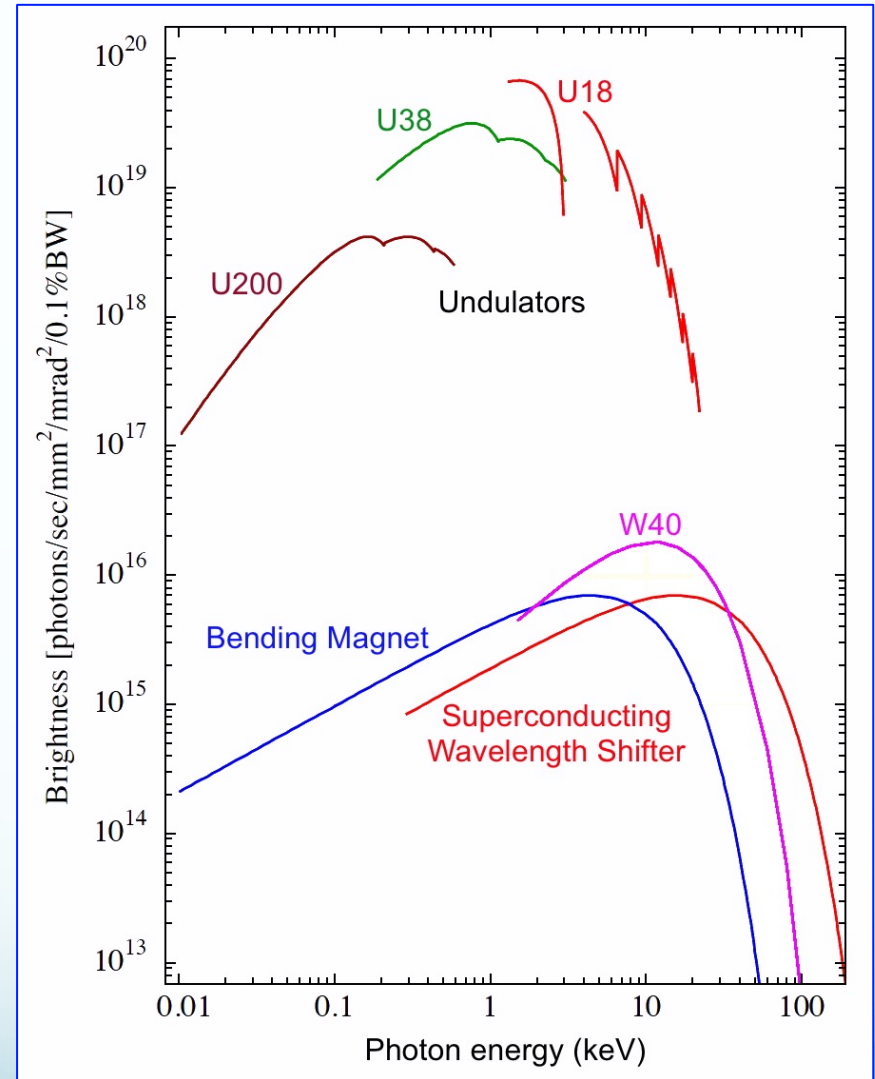
... and synchrotron radiation is also the coherent radiation emitted by the undulators of Free Electron Lasers (FELs) .



# Comparing the achievable brightness



Courtesy DESY



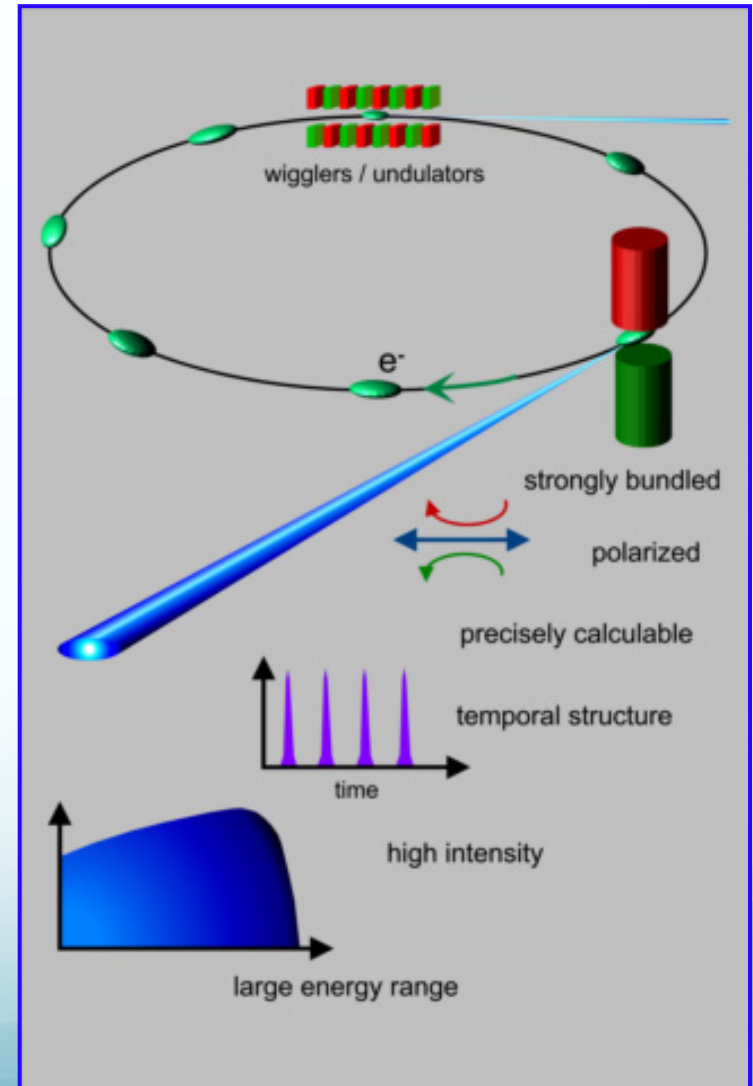
*Calculated brightness* of beams emitted by undulators (200 mm (L= 10 m), 38 mm (L= 4 m) and 18 mm (L= 2 m) periods), wigglers (wavelength shifter and 40 mm (L= 2 m) period) and bending magnets for a 2.4 GeV storage ring with 400 mA circulating current (**M. Boge**

<http://accelconf.web.cern.ch/Accelconf/e98/PAPERS/MOP28G.PDF>

# Synchrotron Radiation Properties

What makes synchrotron radiation interesting and powerful?

- **Tunability** or large energy range from **IR to X-rays**: users can select the wavelength required for their experiments - **continuous** (Bending Magnet/Wiggler) - **quasi-monochromatic** (Undulator) emission
- **Source in a clean UHV environment**
- **Very high flux** and **brightness** (with undulators) highly **collimated** photon beams generated by a **small divergence** and **small size sources**.
- **Highly Polarized**
- **Pulsed time structure**
- **High stability** (submicron source stability)





# Beamlines @ DAΦNE

## Building 12

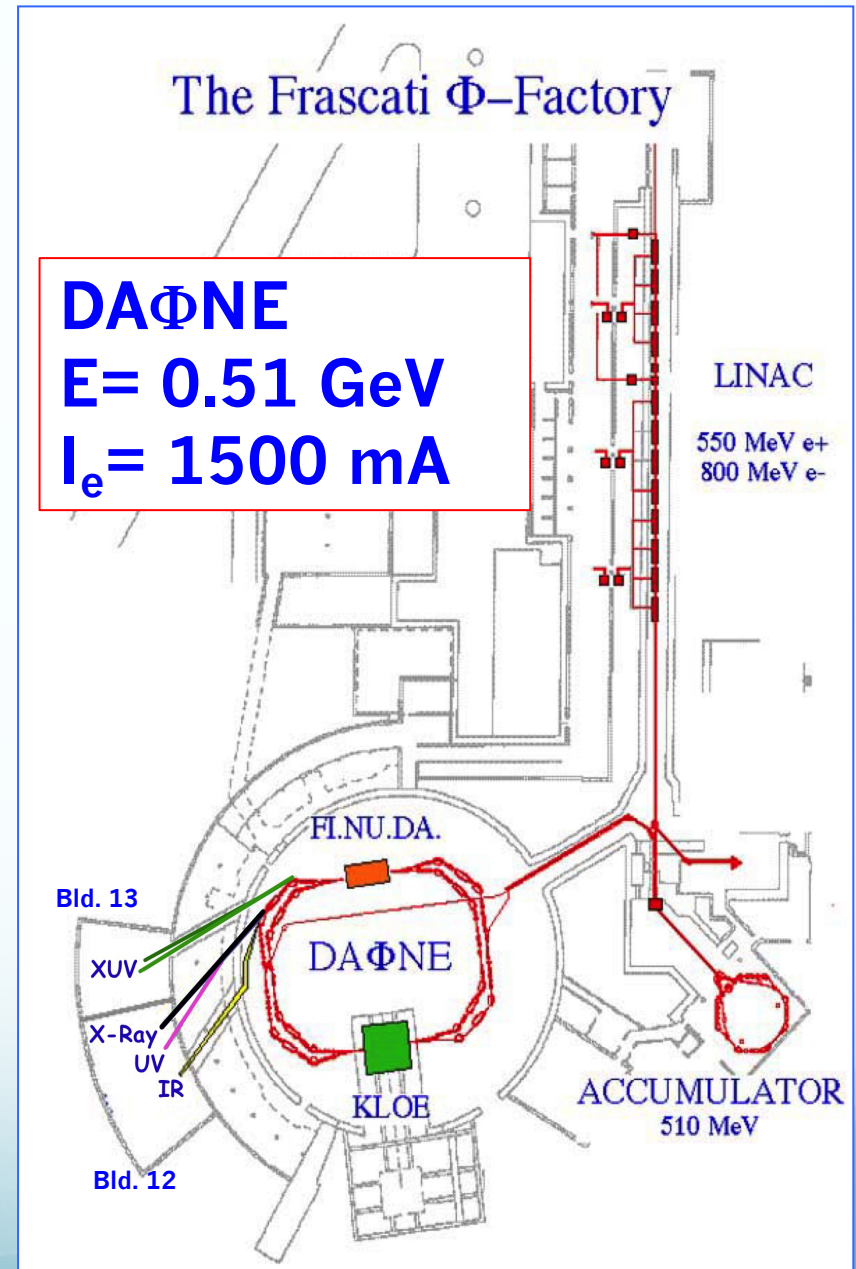
**OPEN to USERS**

- 1) SINBAD - IR beamline (1.24 meV - 1.24 eV)
- 2) DXR1- Soft x-ray beamline (900-3000 eV)
- 3) DXR2 – UV-VIS beamline (2-10 eV)

## Building 13

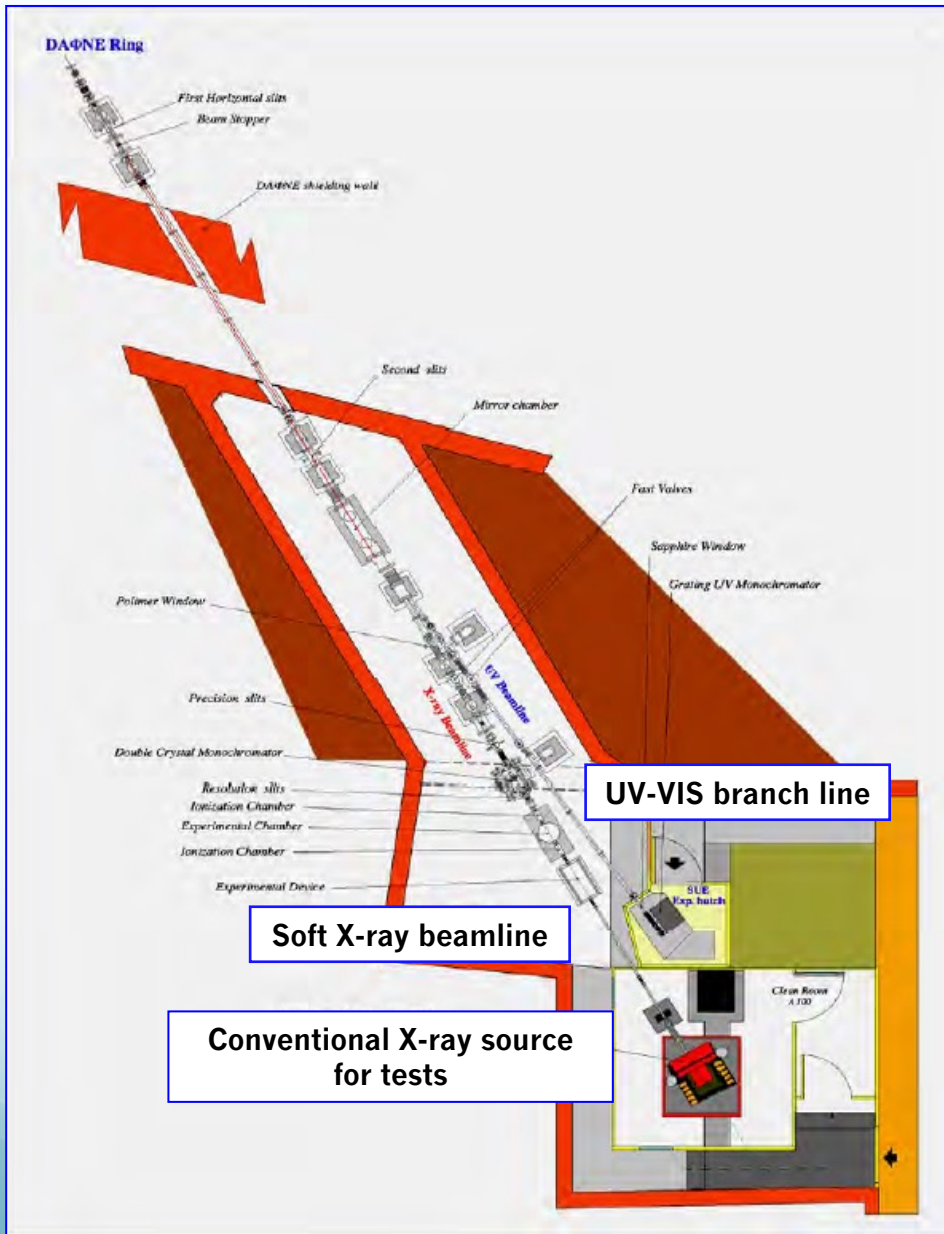
**XUV beamlines**

- 4) XUV1 - Low Energy Beamline (30-200 eV)
- 5) XUV2 - High Energy Beamline (60-1000 eV)
- 6) New XUV2 Branch White Line



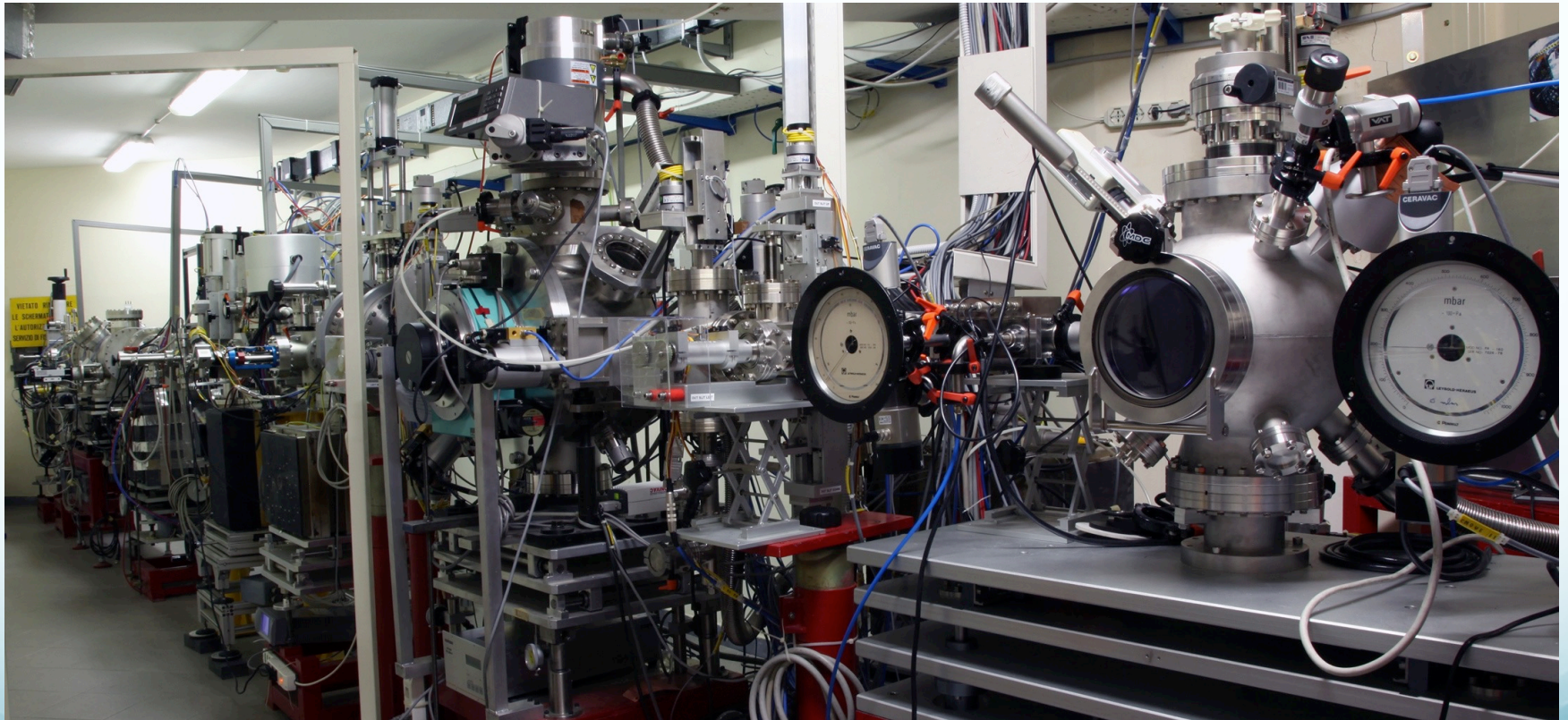
# DAΦNE Soft X-ray DXR1 Beamline

- 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 LNT
- Soft X-ray Absorption Spectroscopy and tests of Soft x-ray optics and detectors.



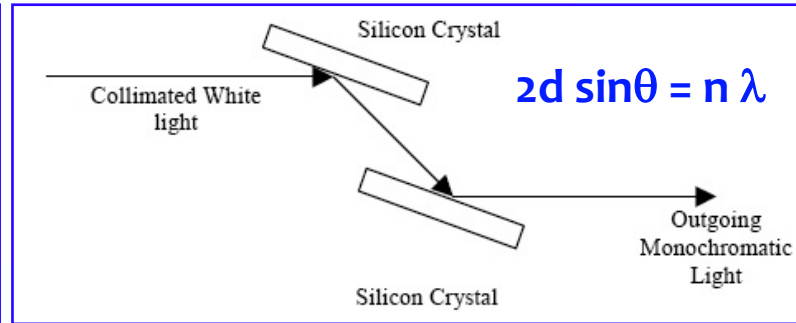
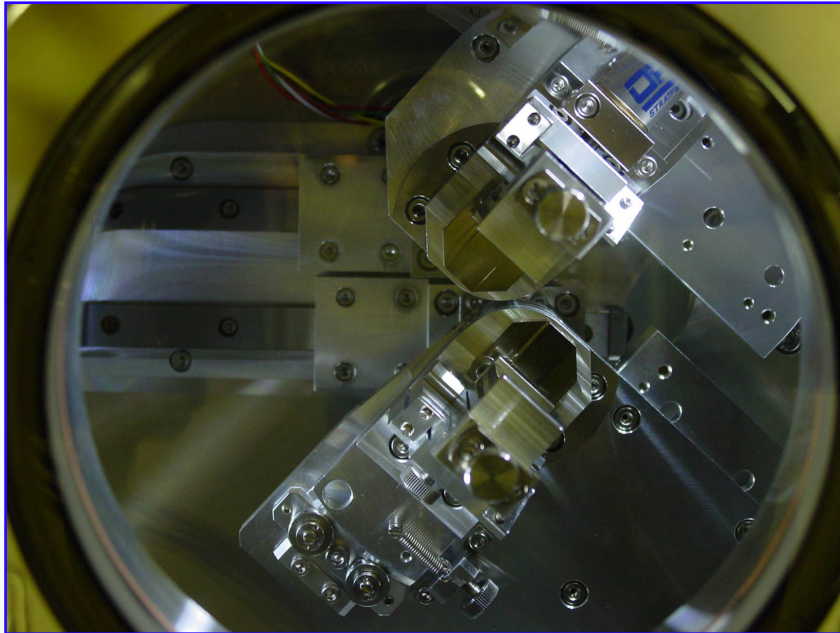


# DXR1 Soft X-ray Beamline



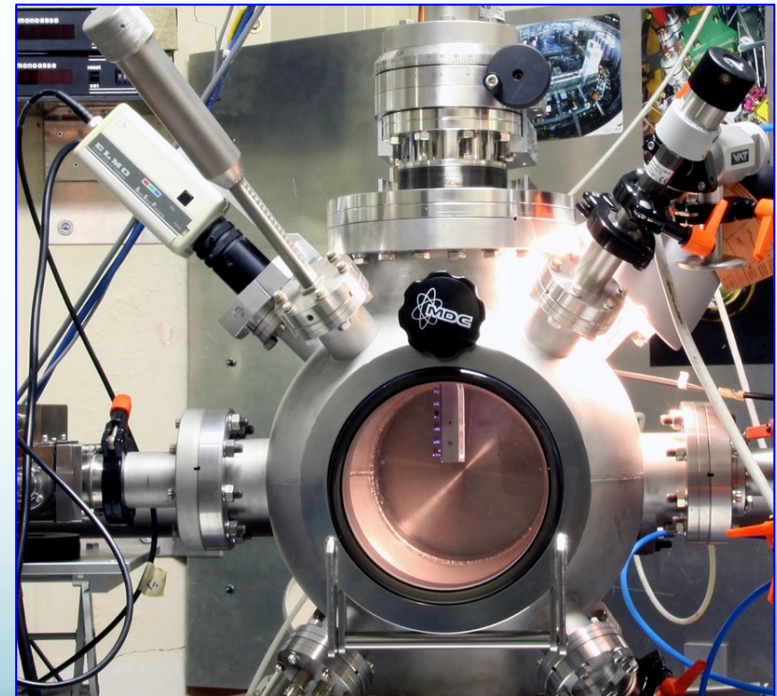


# DXR1 Soft X-ray Beamline



**Double crystal monochromator with fixed exit boomerang mechanism.**

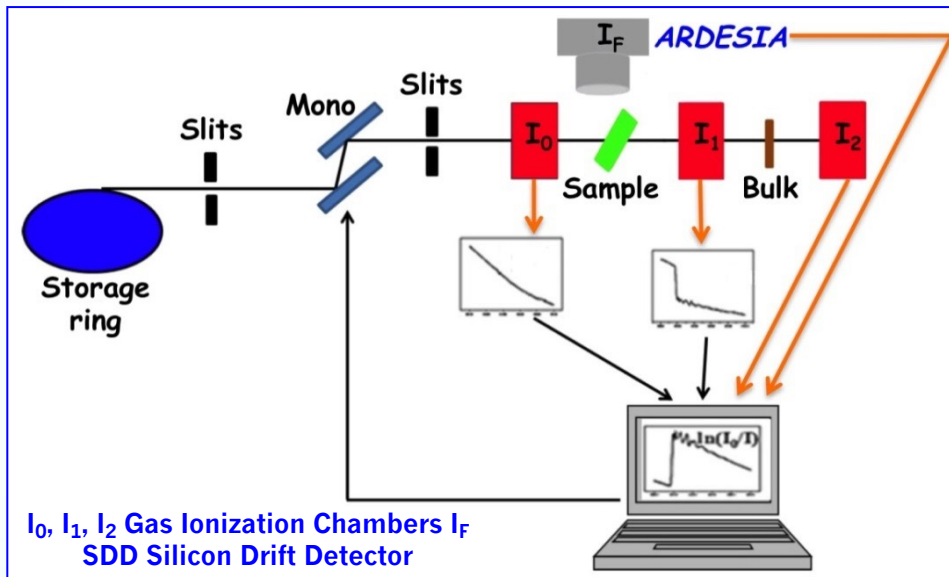
Crystal type	2d spacing (Å)	Energy range (eV)	Absorption edges
Beryl (10-10)	15.954	1000 - 1560	Na K, Mg K, Cu L
KTP (011)	10.950	1200 - 2200	Mg K, Al K
InSb (111)	7.481	1800 - 3100	Si K, P K, S K, Cl K
Ge (111)	6.532	2100 - 3100	P K, S K, Cl K



**Experimental Chamber**



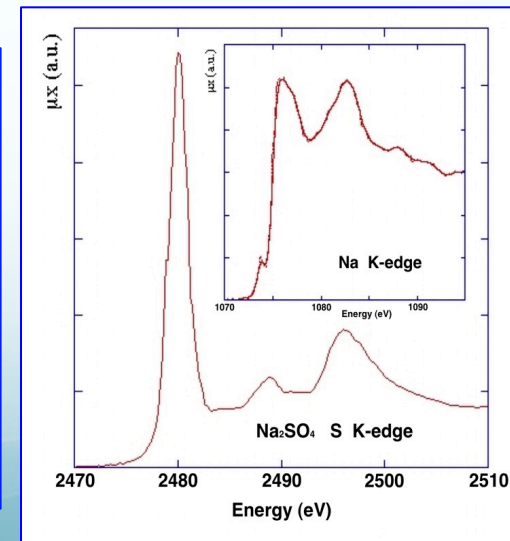
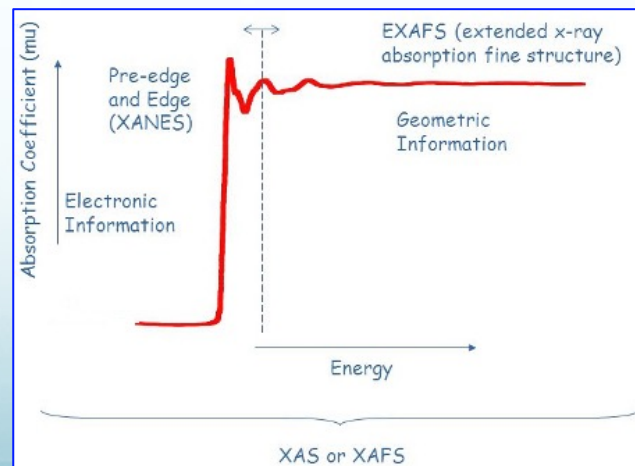
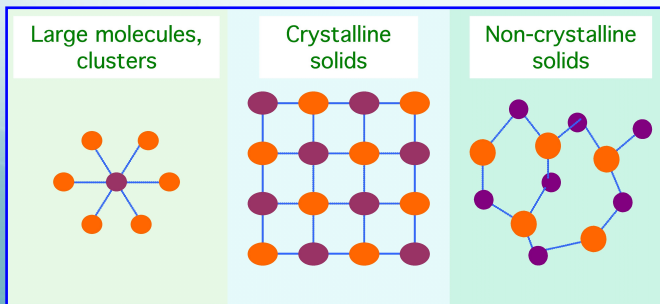
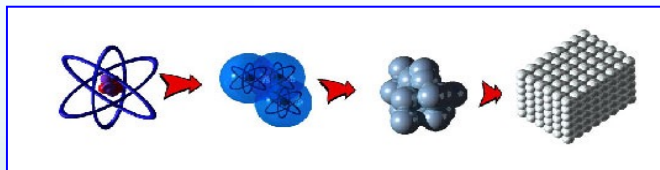
# Soft X-ray spectroscopy -XAFS



## X-ray Absorption Fine Structure or XAFS spectroscopy

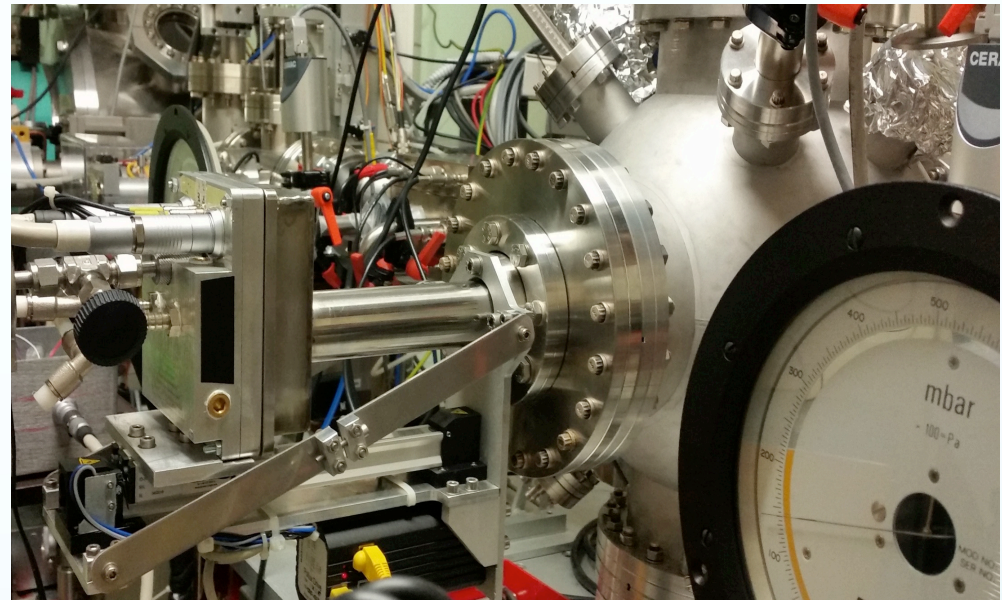
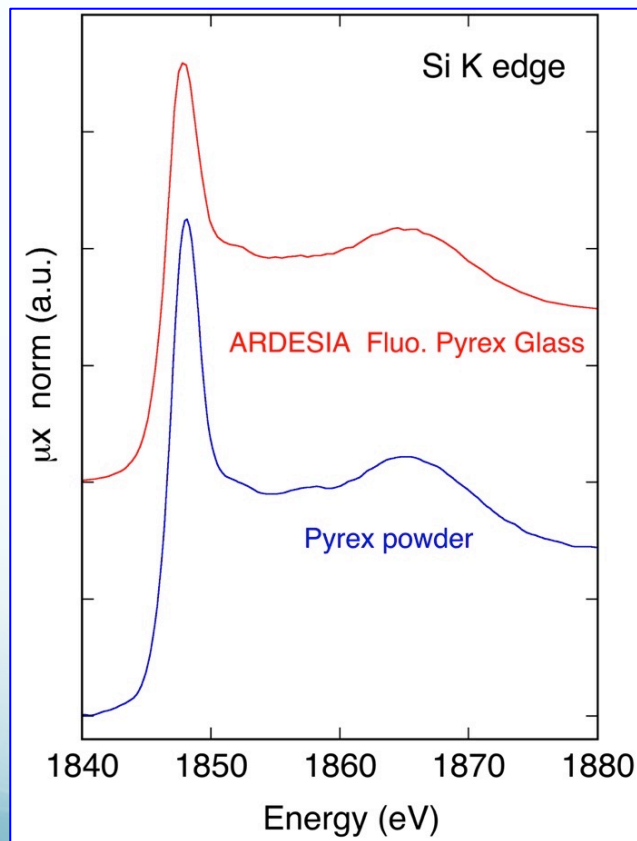
Particularly useful for investigating the electronic structure and local environment of atoms in quite different samples (solids, liquids and gasses).

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



# ARDESIA SDD detector

## ARDESIA 4-channel X-ray XRF detector

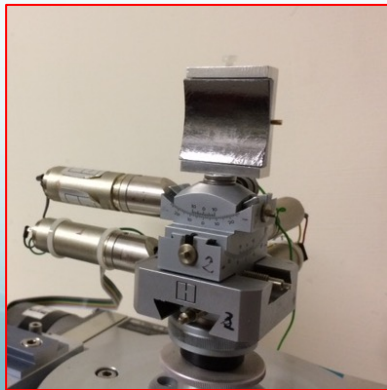
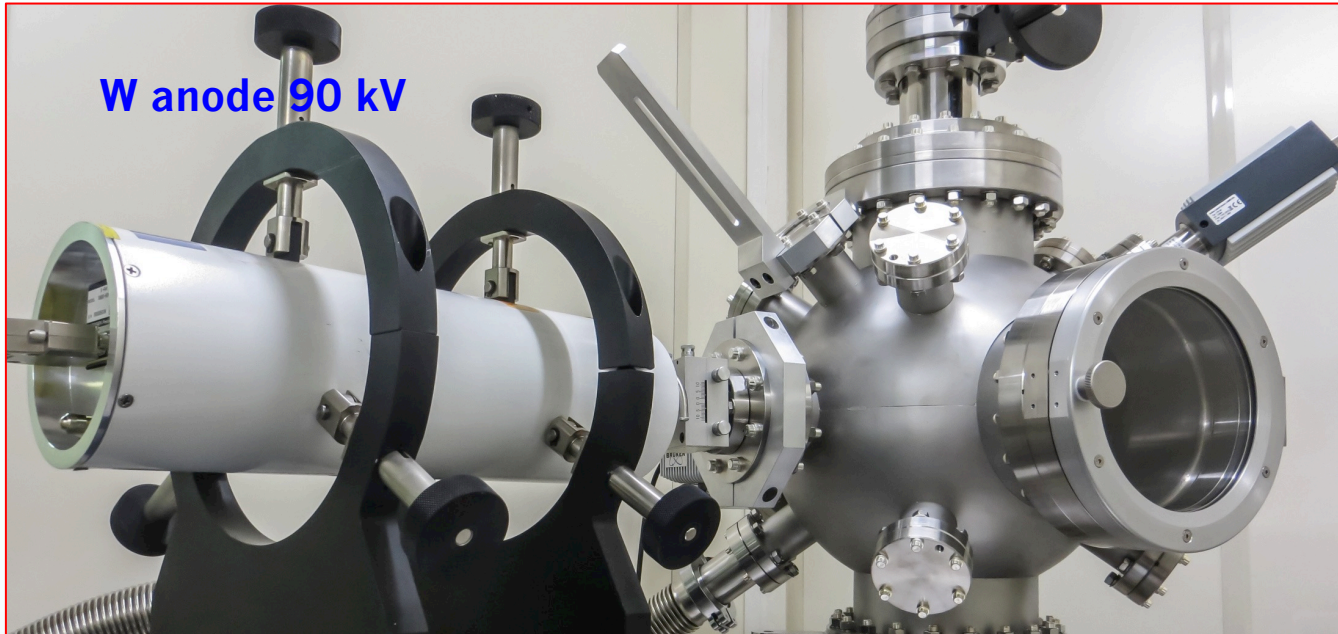


Developed thanks to a collaboration between **DAFNE-Light** and the **Politecnico di Milano**. Financed by **INFN** through a **CSN5** (INFN National Scientific Committee 5 for technological research experiments) **ARDESIA** project.

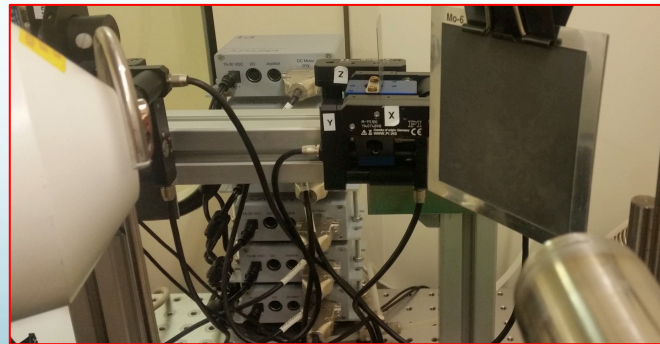
**XAFS-XRF studies of thin and supported samples.**

**First XRF-XAFS @ DXR1 beamline - February 2018**

# Test chamber and x-ray conventional source



Testing crystals



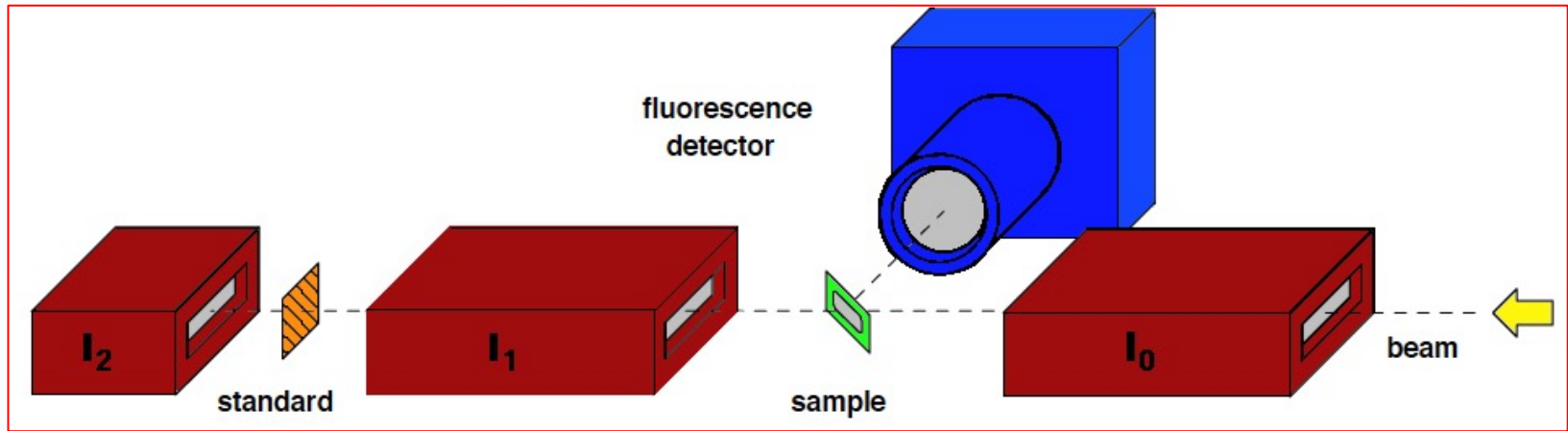
Testing detectors

Dispersive  
XAFS under  
development



# **Material Science and XAFS spectroscopy**

# XAFS Spectroscopy

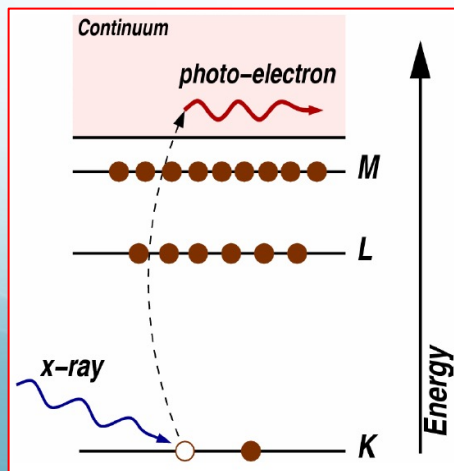


XAFS can be measured either in Transmission or Fluorescence mode.

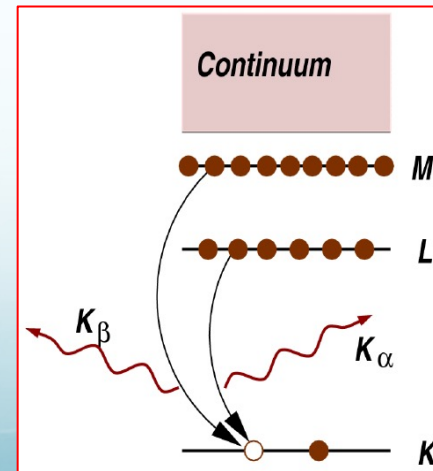
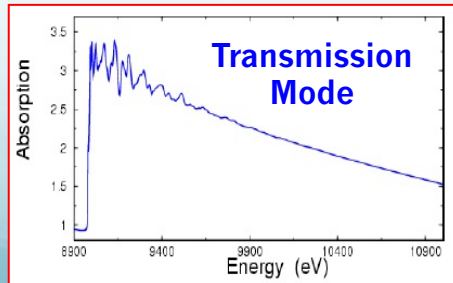
The probability of fluorescence emission is directly proportional to the absorption probability.

## X-ray absorption

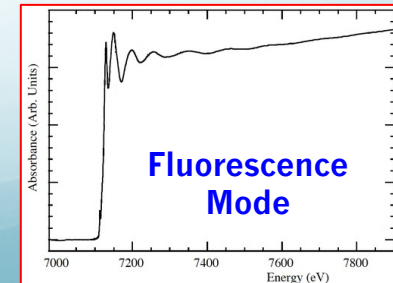
## X-ray fluorescence



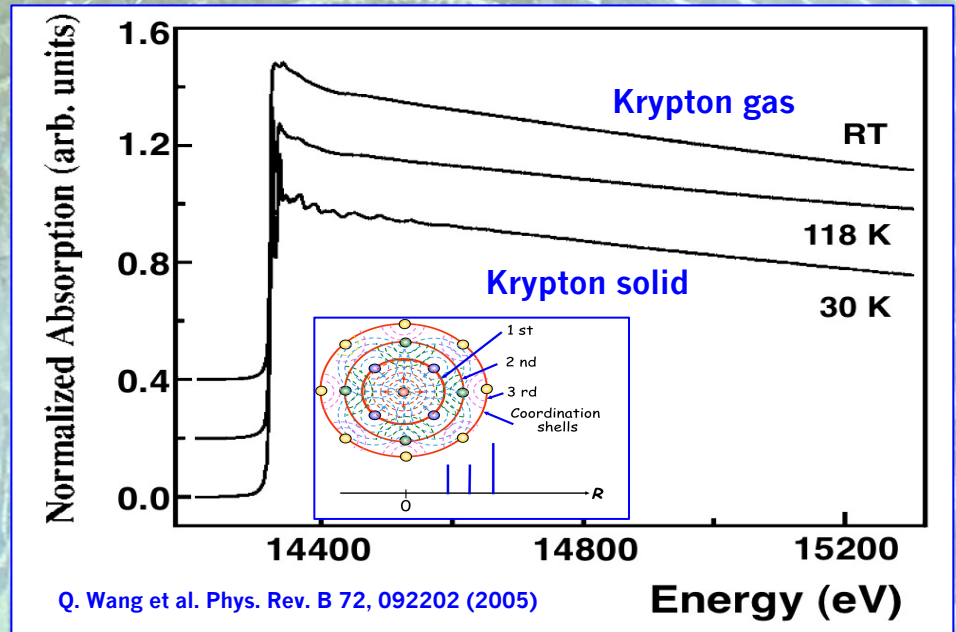
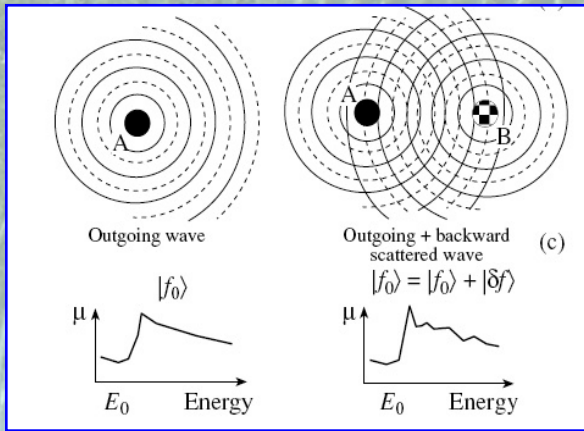
$$\mu(E)x = \ln\left(\frac{I_0}{I_1}\right)$$



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



# XAFS spectroscopy



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

**EXAFS**

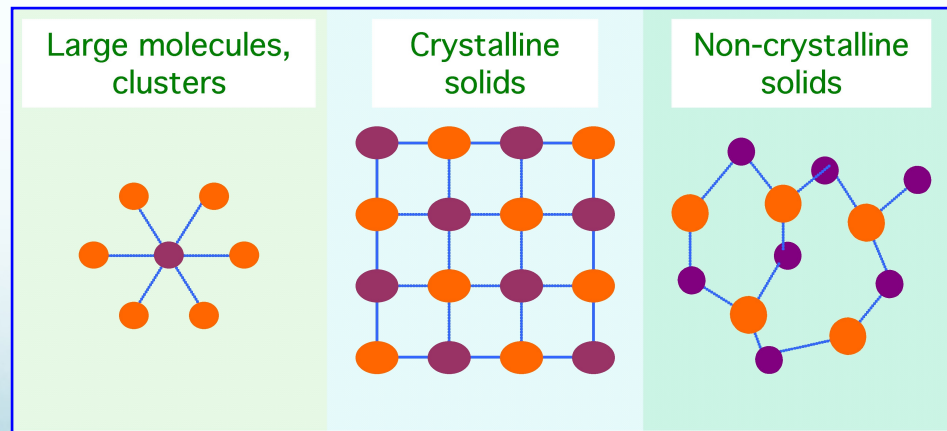
Coordination number

Debye Waller factor

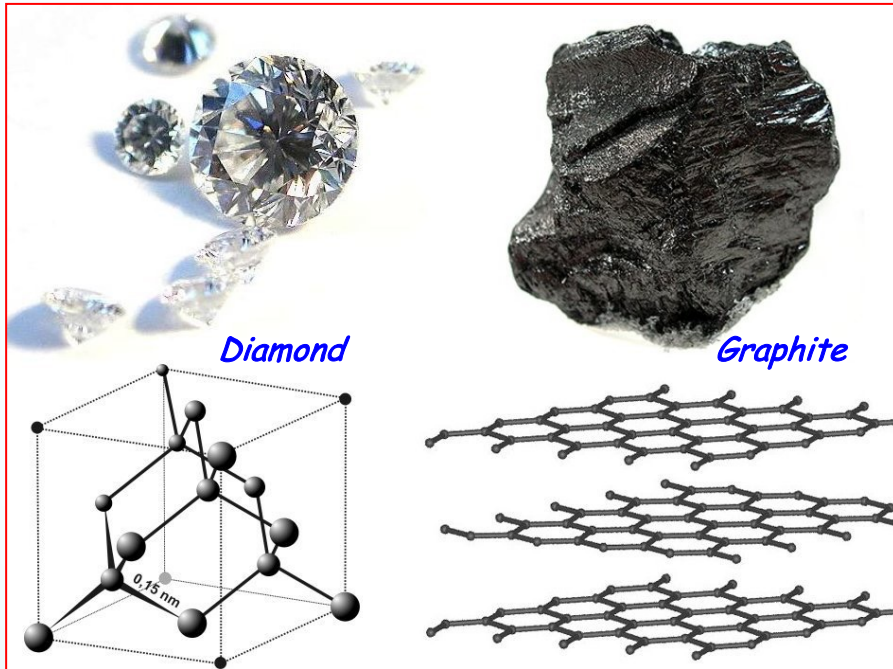
Interatomic distance



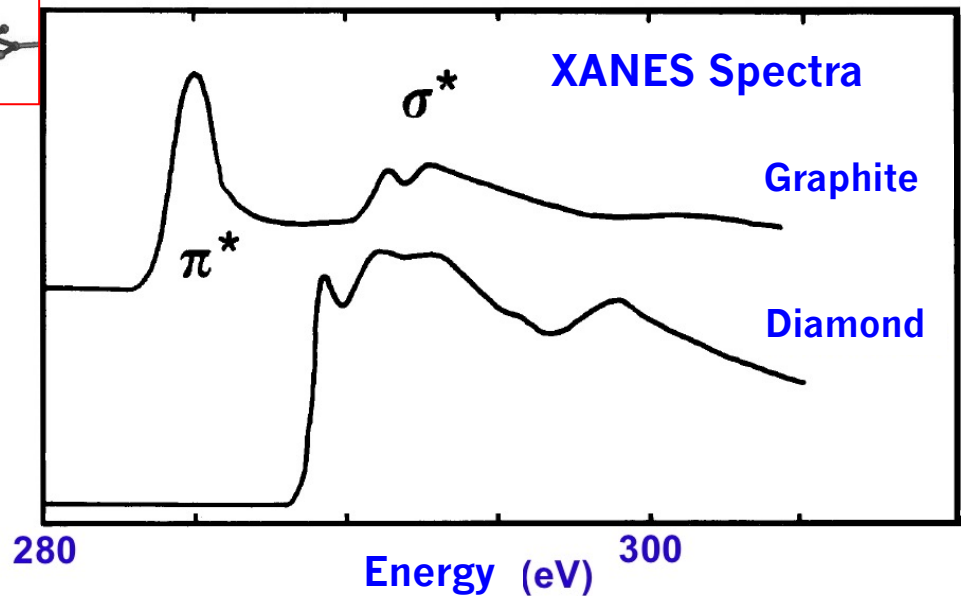
# XAFS spectroscopy as a very powerful tool



# XAFS - Carbon K edge

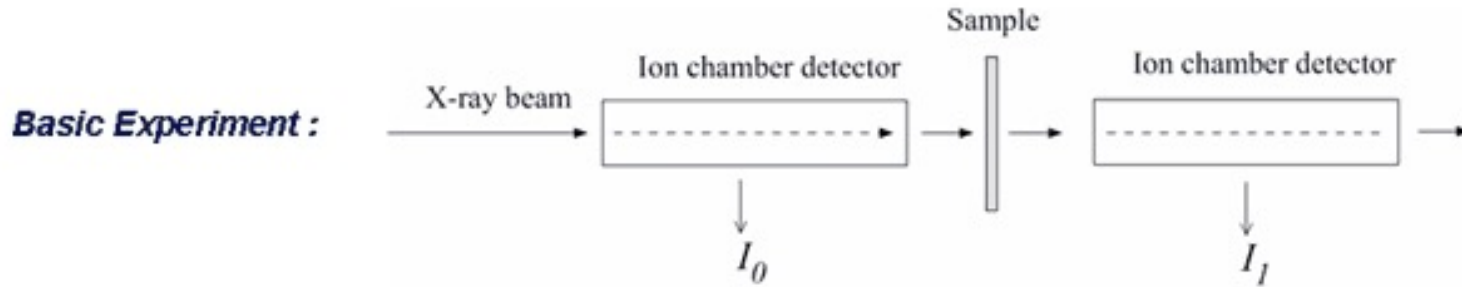


The quite different XANES spectra for Graphite and Diamond due to their different atomic structures.



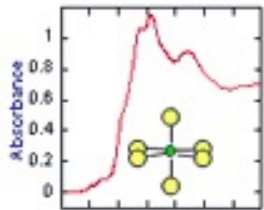
J. Robertson, Prog Solid St. Chem 21, 199 (1991)

# XAFS information

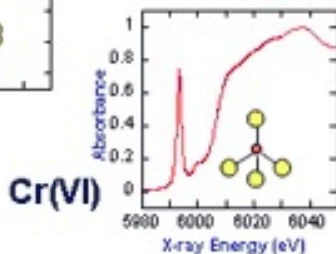


## XANES

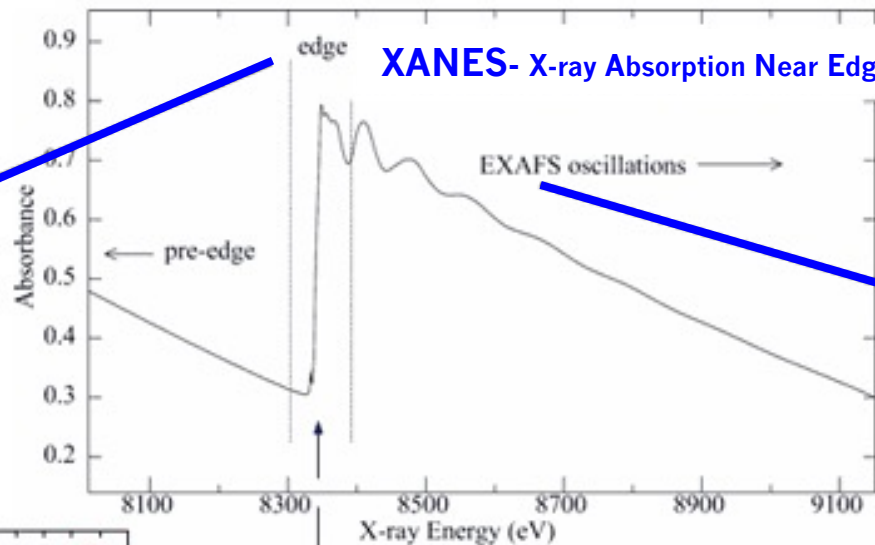
Oxidation state,  
Molecular composition,  
structure.



Cr(III)



Cr(VI)

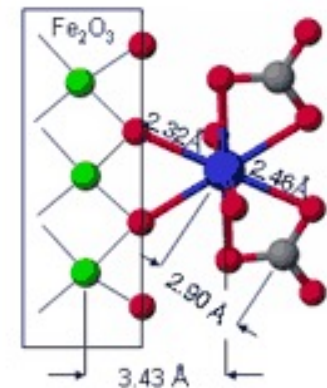


XANES- X-ray Absorption Near Edge Structure

EXAFS- Extended X-ray  
Absorption Fine Structure

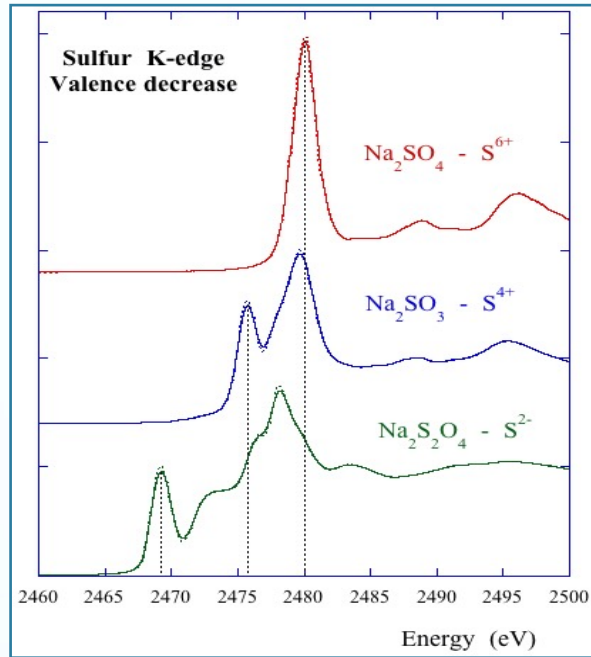
## EXAFS

Quantitative Local Structure.

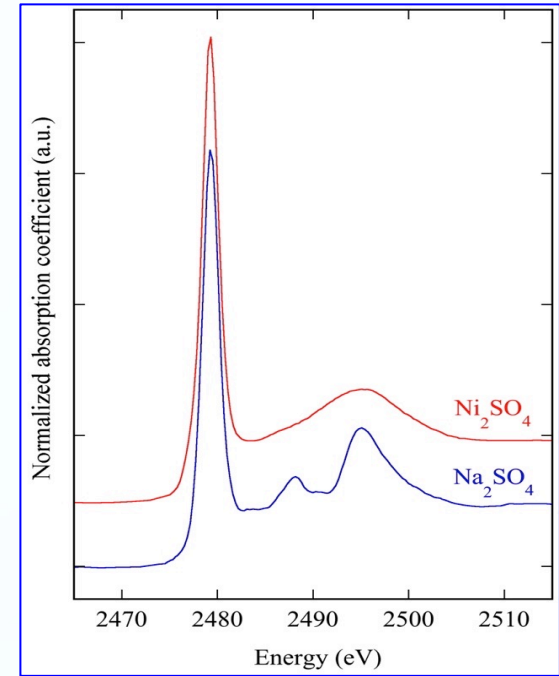




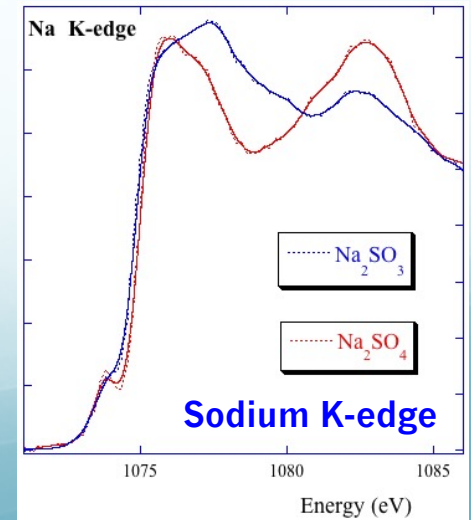
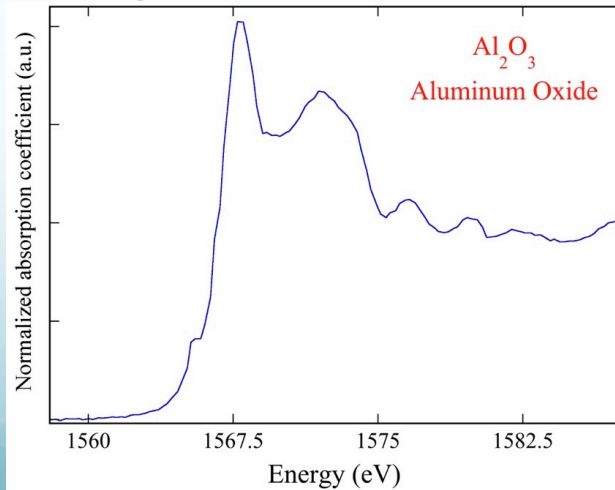
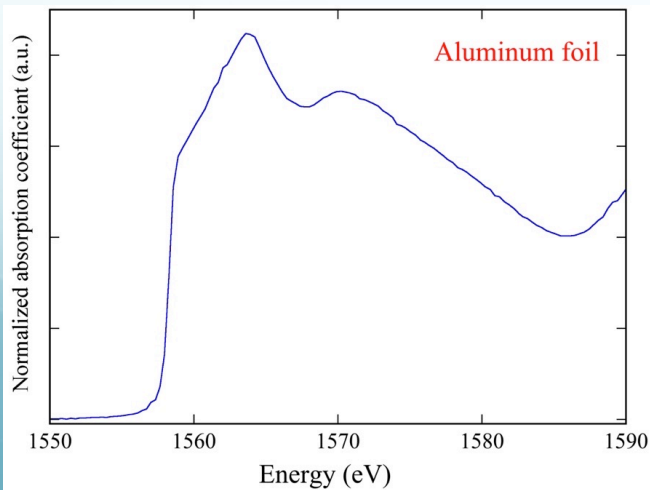
# XANES DXR1



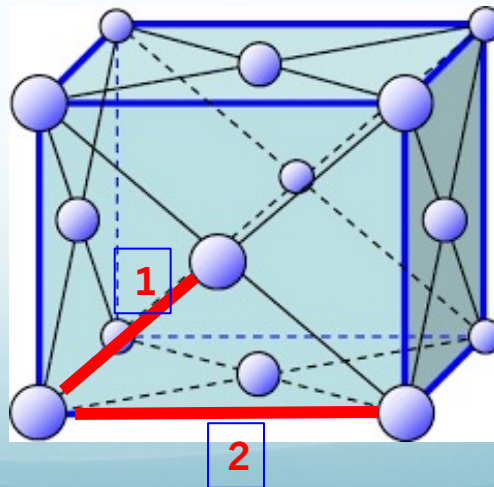
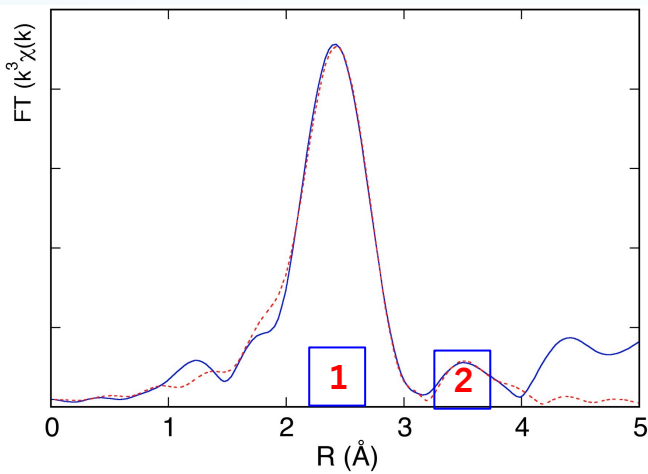
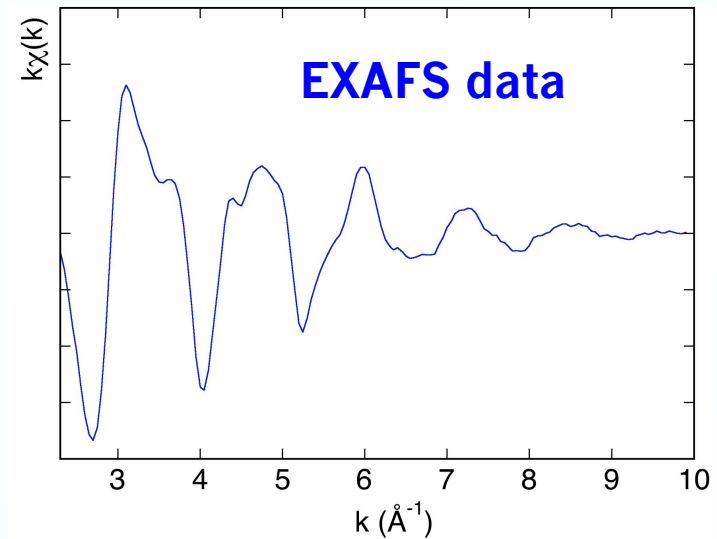
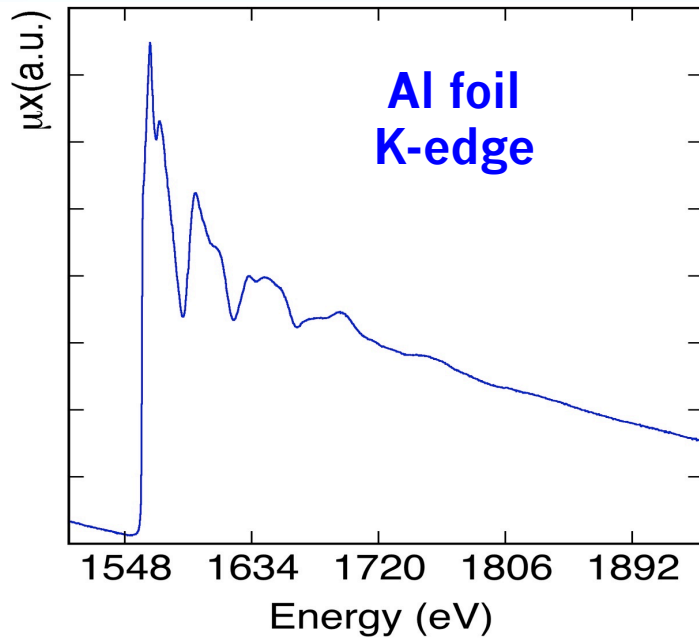
Sulfur K-edge



Aluminum K-edge



# EXAFS DXR1



$$N_{1\text{Al}} = 12$$
$$R_{1\text{Al}} = 2.85 \text{ \AA}$$
$$\sigma_{1^2\text{Al}} = 0.014 \text{ \AA}^2$$

First shell

$$N_{2\text{Al}} = 6$$
$$R_{2\text{Al}} = 4.04 \text{ \AA}$$
$$\sigma_{2^2\text{Al}} = 0.027 \text{ \AA}^2$$

Second shell

# Some DXR1 XAFS Applications

**Material Science and Technology**

```
graph TD; A[Material Science and Technology] --> B[Hydrogen storage materials]; A --> C[Spintronics]; A --> D[Space]; A --> E[Nanostructures]; F[Material Science] --> G[Pigments-Cultural Heritage]; G --> H[Biology]
```

**Hydrogen  
storage  
materials**

**Spintronics**

**Space**

**Nanostructures**

**Material  
Science**

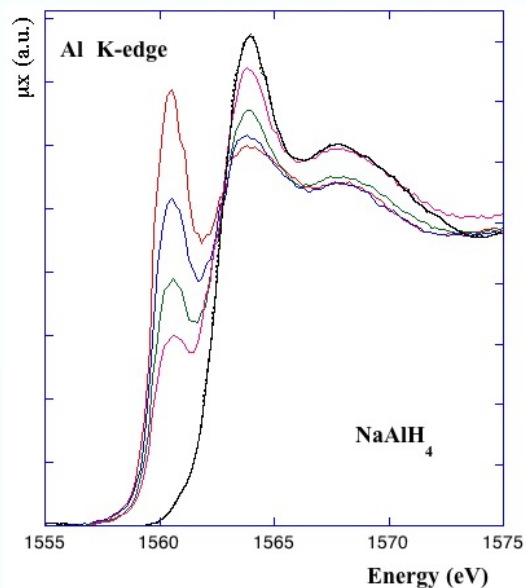
**Pigments-  
Cultural Heritage**

**Biology**



# Technology

## Hydrogen storage materials



Al K-edge XANES study of alkali aluminium hydrides

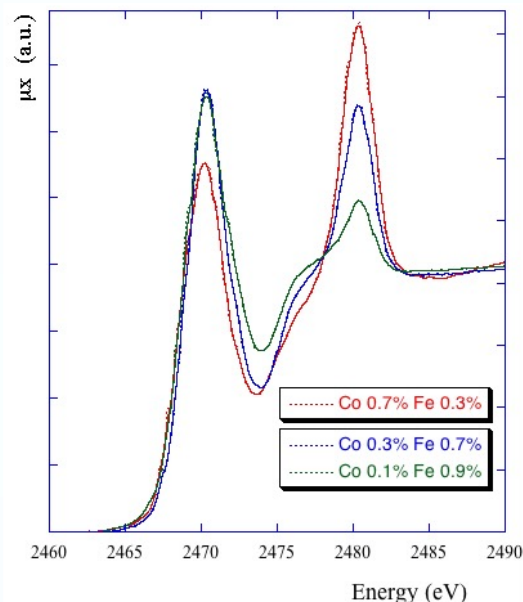
### Development of higher-efficiency hydrogen storage materials

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

This study shows the structural changes of the alunate structure upon cycling under hydrogen.

A. Leon et al. - KIT

## Spintronics



Near-edge X-ray Absorption spectroscopic investigations: **Fe doped CoS<sub>2</sub>**

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<sub>2</sub> samples** gives information on the **electronic structural changes** induced by **doping of 3d transition metal ion, Fe of Co<sub>1-x</sub>Fe<sub>x</sub>S<sub>2</sub>**.

I. Zuburtikudis - Dept. of Engineering, GREECE

# Technology

## Space



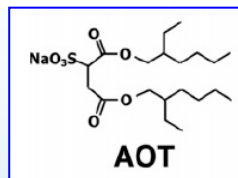
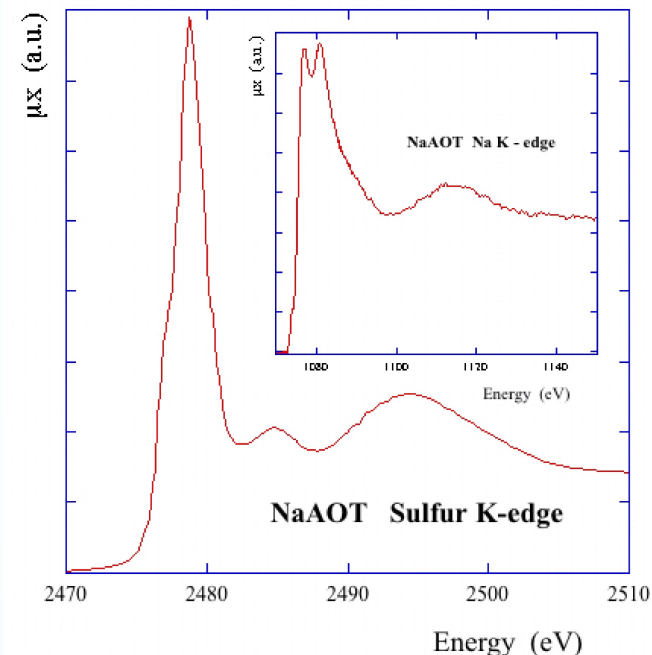
### Characterization of thermal filters as a function of temperature

**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 like that 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

## Liquid crystals



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

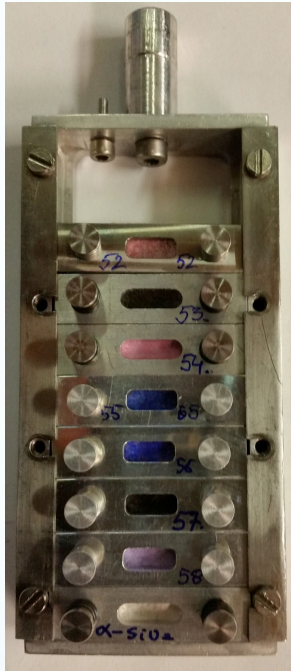
**Structural investigation of water-soluble inorganic salts within reversed micelles or liquid crystals used as good templates in which nanoparticles can be trapped and stabilized.**

**System studied: Co, Yb and Er in NaAOT**

V. Turco Liveri, Univ. Palermo

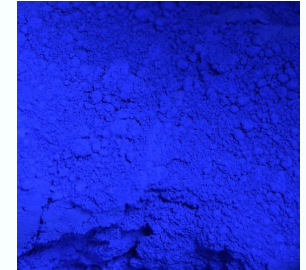
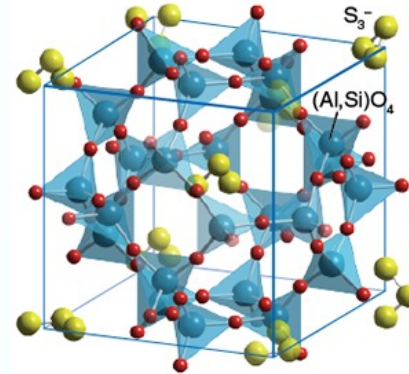
# Study of Pigments

possible applications in the Cultural Heritage field



XAFS RT  
sample holder

Revisiting properties  
of  $\text{CaCoSi}_n\text{O}_{2n+2}$   
Crystal and  
electronic structure  
M. Szubka et al.  
*Journal of Magnetism  
and Magnetic  
Materials* (2022)



## 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  $(\text{Na,Ca})_8(\text{AlSiO}_4)_6(\text{SO}_4,\text{S,Cl})_2$ , 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

## 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  $\text{BaCuSi}_2\text{O}_6$  (Han purple),  $\text{BaCuSi}_4\text{O}_{10}$  (Han blue) and  $\text{CaCuSi}_4\text{O}_{10}$  (Egyptian blue) were established over 100 years later.

Contemporary,  $\text{CaCoSi}_2\text{O}_6$  is used in industry as pigment varying in colors from blue to pink.

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



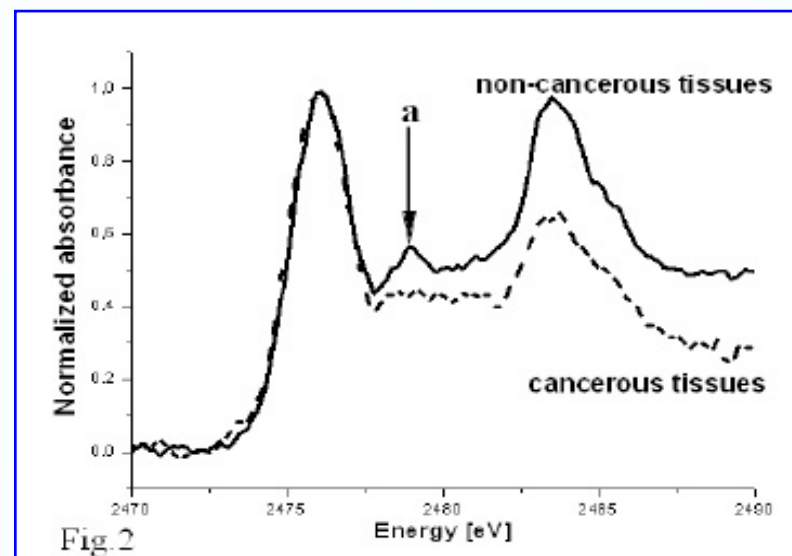
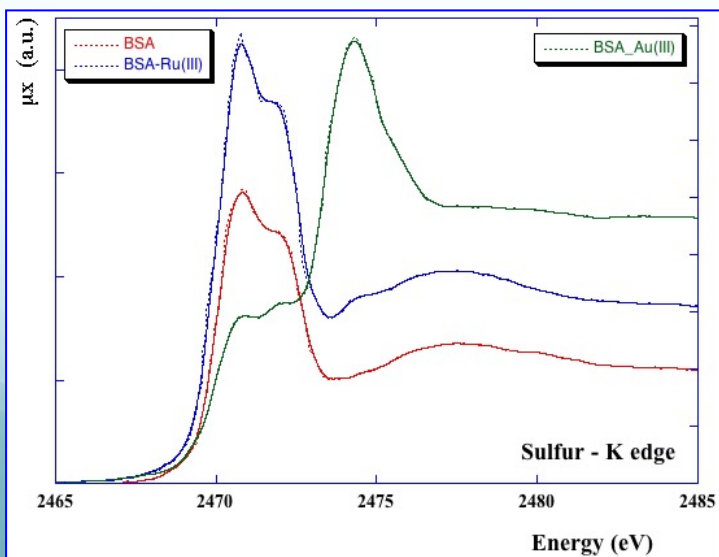
# Biology

## X-Ray Absorption Spectroscopy to Characterize Metallo-drug/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.



## Preliminary Study on Chemical Speciation of Sulfur in Cancerous Tissues

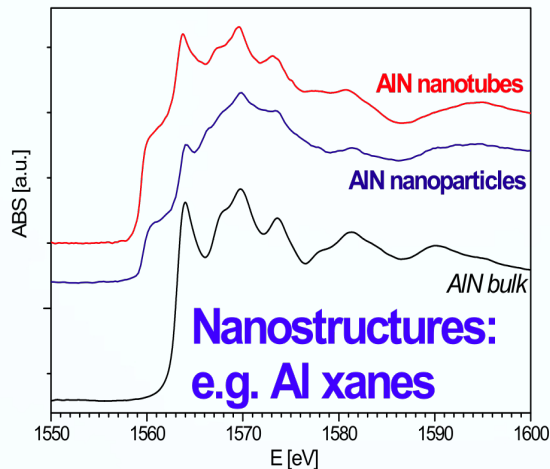
**Urological cancers comprise approximately one-third of all cancers diagnosed in men** worldwide and out of these, prostate cancer is the most common one.

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

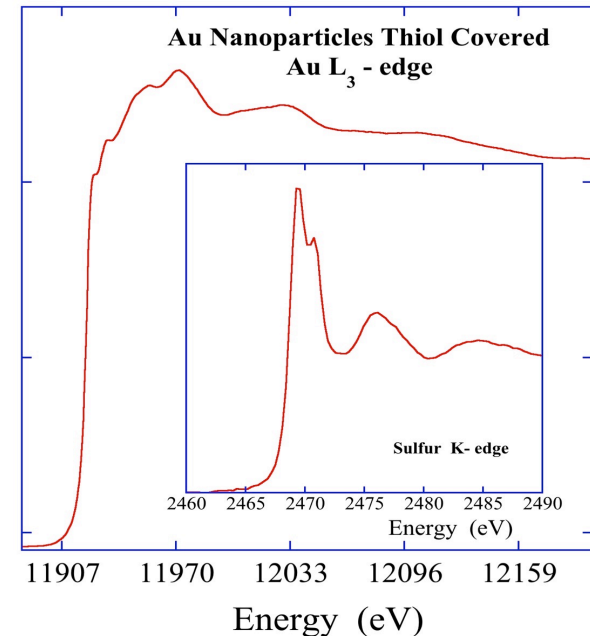
## Nanostructures



### 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.

# **From synchrotron radiation to a conventional X-ray source**



# ARTEMISIA Project



## ARTEMISIA

**ART**ificial intelligence **Ext**ended-**M**ultispectral Imaging **S**canner for **In**-situ **Art**work analysis

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G. Viviani<sup>1</sup>, V. Sciarra<sup>1</sup>, G. Verona Rinati<sup>2</sup>, G. Bonifazi<sup>3</sup>, G.  
Capobianco<sup>3</sup>, S. Serranti<sup>3</sup>, F. Aramini<sup>4</sup>, E. Gianì<sup>4</sup>, M. Ioele<sup>4</sup>, B.  
Lavorini<sup>4</sup>, S. Brandalesi<sup>5</sup>, A. Mantoan<sup>5</sup>, S. Tamascelli<sup>5</sup>, M. Simeone<sup>6</sup>

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**2 INFN Sezione di Roma II**

**3 DICMA Sapienza University of Rome**

**4 Central Institute of Restoration, Rome**

**5 XTeam Software Solutions s.r.l., Rovigo RO**

**6 Vianet slr, Rome.**

**5 and 6 COMPANIES OPERATING IN THE FIELD OF CULTURAL HERITAGE**

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SAPIENZA  
UNIVERSITÀ DI ROMA



ARTEMISIA  
Artificial Intelligence Extended-Multispectral Imaging Scanner  
for In-situ artwork analysis

Credits Image: XTeam Software Solutions srl

# ARTEMISIA Project

**ARTEMISIA** is an Experimental project for the in-situ identification of pictorial materials through **an integrated multi-modal approach for assessing the state of conservation of pictorial artworks.**

## GOALS OF THE PROJECT:

- The enhancement of imaging diagnostics through the extension of the spectral range of analysis
- The development of artificial intelligence algorithms dedicated to the automatic recognition and monitoring of the state of conservation of artwork's materials.

## THE DEVELOPED PROTOTYPE INVOLVES THE COMBINATION AND THE DATA FUSION OF TWO TECHNOLOGIES:

- **Hyperspectral imaging** (in the VIS-NIR spectral range)
- **FT-IR spectroscopy in reflection** (Medium IR spectral range)

## FOUNDING

The project was among the winners of the public notice of LAZIO INNOVA

**Financing entities:** Lazio Region and MUR

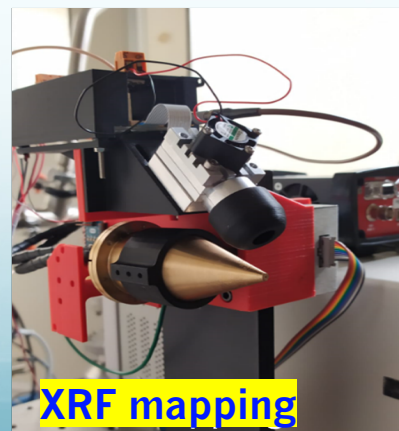
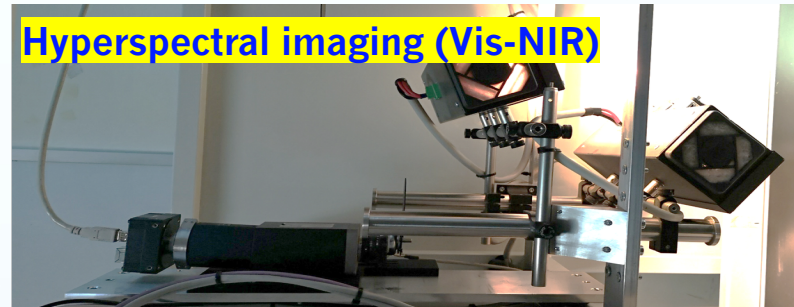
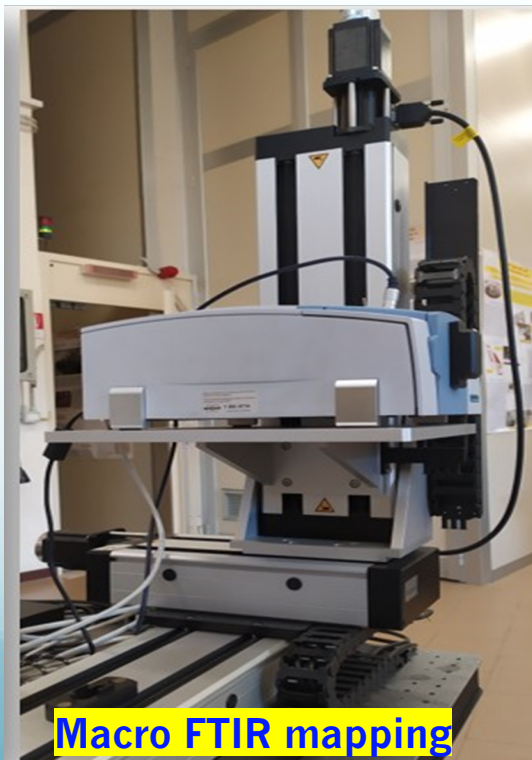
**Grant awarded:** € 149,102.48

## DURATION

22 June 2021 – 22 September 2023

# TECHNOLOGICAL DEVELOPMENT

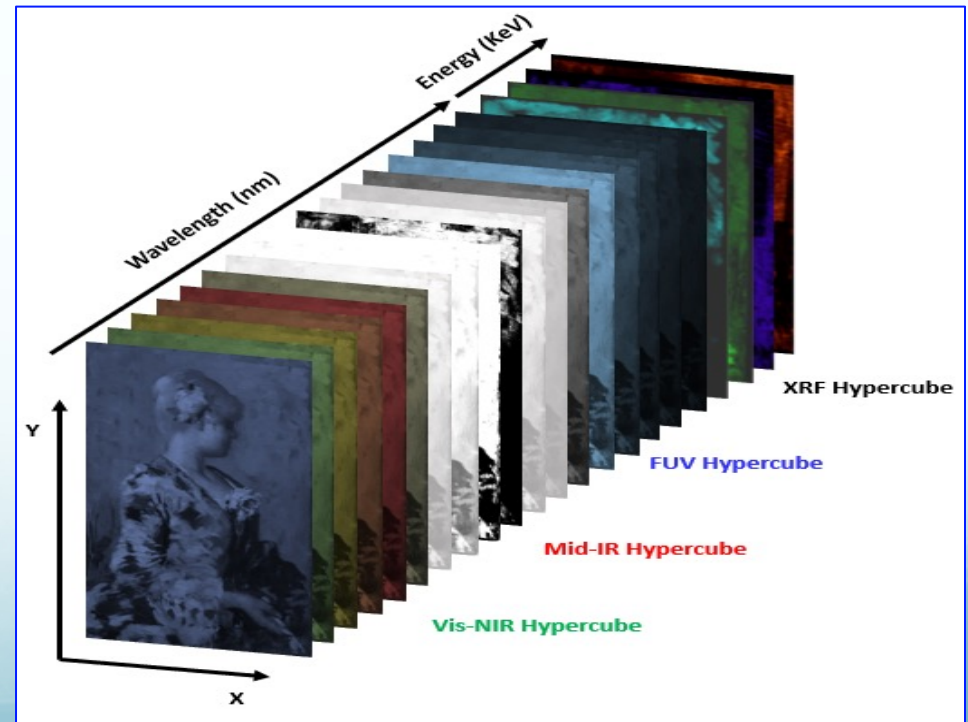
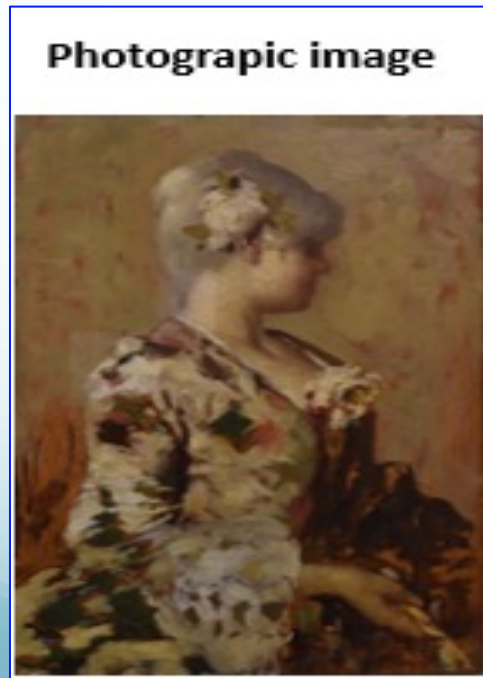
- Development of a scanning system to **motorized the FT-IR spectrometer** in order to perform **macro-FTIR mapping**.
- Integration of the macro-FTIR mapping with the **hyperspectral imaging (Vis-NIR)** technique to extend the spectral range from visible to mid-infrared range of the hyperspectral images acquired.
- **Feasibility study** for the integration in the mapping system of the **UV Fluorescence** and **XRF** spectroscopies (in **collaboration with CHNet LABEC-Fi**).



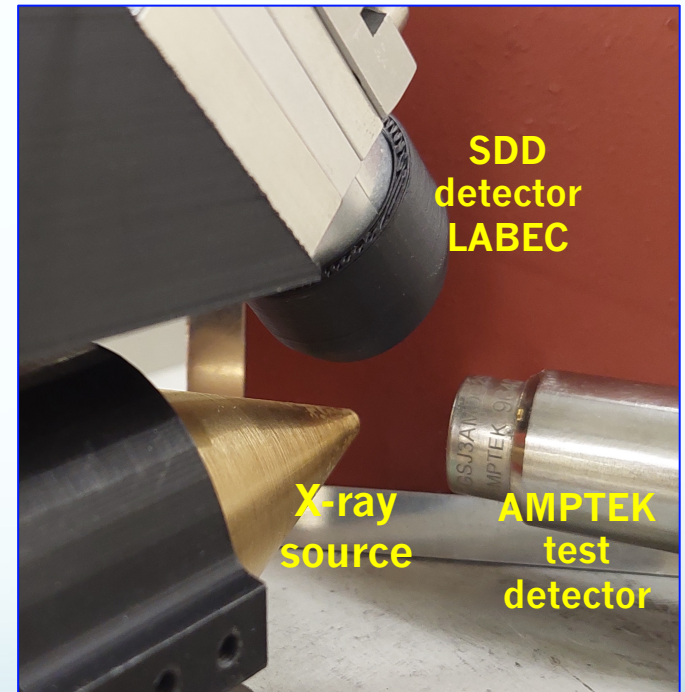
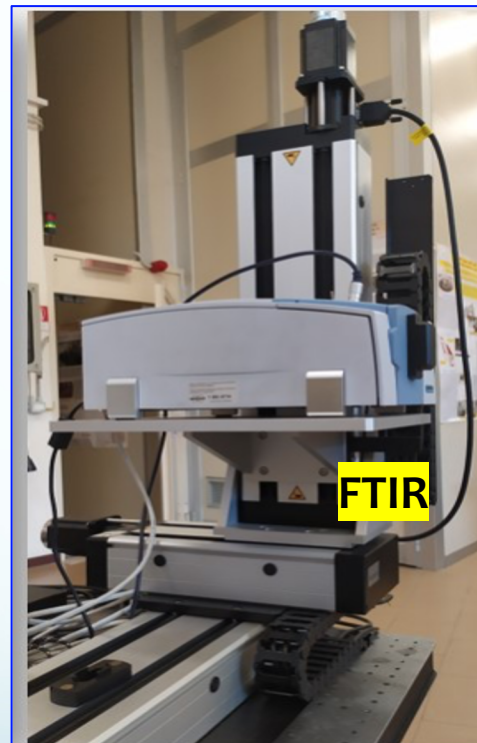
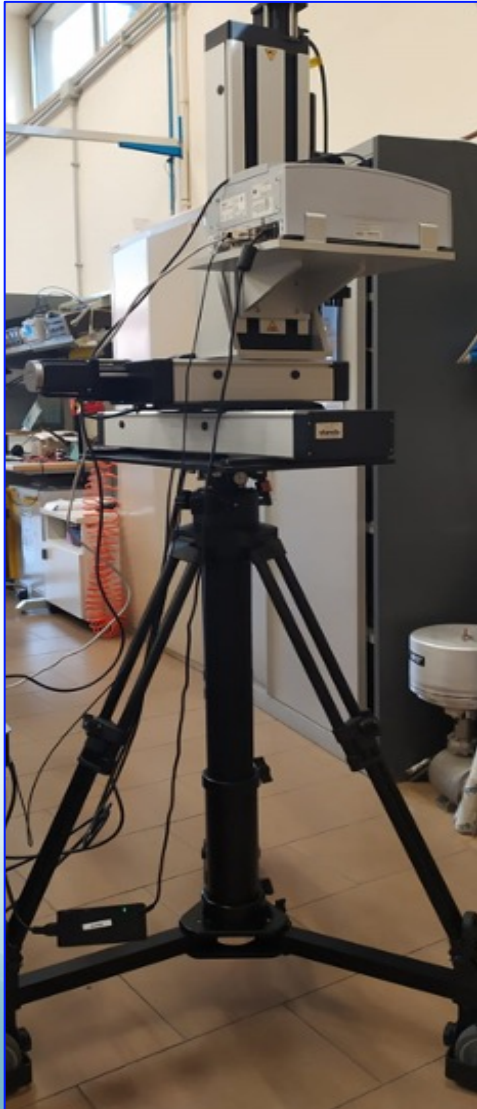


# DATA FUSION AND MACHINE LEARNING

1. **Development of a dedicated software for the registration of the hyperspectral images** including the photographic image of the artwork and application of data fusion methods.
2. Creation of a **database of hyperspectral images of pictorial materials** and development of **machine learning methods for their automatic recognition on artworks**.
3. Development of a **User-Friendly graphical interface** to show the distribution of the constituting materials as multi-level information.



# Present Status of ARTEMISIA



The Vis-NIR and FTIR XY mapping has been tested on artworks in a Museum: we are now working on the integration of the XRF and UVF systems.

# **DAΦNE-Light Facility and [www](http://www)**



# More information

## <https://dafne-light.Inf.infn.it/>

The screenshot shows the website's navigation bar with links: HOME, BEAMLINES, TEAM, USERS, RESEARCH, EDUCATION, GALLERY, NEWS, COVID-19. The main header features the logo 'DAΦNE-Light' and the 'About us' section. The 'About us' text describes the laboratory's multidisciplinary focus and the use of the DAFNE collider. A blue box indicates that the 'CALL FOR PROPOSALS IS NOW CLOSED'. Below this are four promotional images: 'APPLY FOR BEAMTIME' (a synchrotron chamber), 'ELECTRODYNAMICS OF MNB12TE4' (a diagram of Dirac surface states and bulk bands on an HRFZ-Si substrate with a graph of transmittance vs. wavenumber), 'NEW DIAGNOSTIC TOOLS' (a close-up of a diagnostic component), and 'ANNUAL ACTIVITY REPORTS' (a laboratory setup).

HOME BEAMLINES TEAM USERS RESEARCH EDUCATION GALLERY NEWS COVID-19

## DAΦNE-Light

### About us

DAFNE-Light is a multidisciplinary synchrotron radiation laboratory specialized in materials science, life science, medicine, surface science, cultural heritage and other different fields. One of the key features of synchrotron light is that the range of the emitted radiation extends from Infrared (IR) to X-rays. At the Frascati National Laboratories, the presence of **DAΦNE**, the collider currently in operation, allows to use the synchrotron light transported to the DAFNE-Light laboratory through 5 different beam lines and a branch line, built for different applications ranging from IR spectroscopy to UV-Vis, XUV and Soft-X ones.

CALL FOR PROPOSALS IS NOW CLOSED

**APPLY FOR BEAMTIME**

**ELECTRODYNAMICS OF MNB12TE4**

Dirac Surface States Bulk Bands

HRFZ-Si

Transmittance

Wavenumber

Bi Te Mn

HRFZ-Si

HRFZ-Si

**NEW DIAGNOSTIC TOOLS**

**ANNUAL ACTIVITY REPORTS**

**Next Call for Proposals: 20 June 2023 – 20 August 2023**

# Thank you for your attention

