

Atoms, X-rays and Synchrotron Radiation

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INFN - Frascati National Laboratory*



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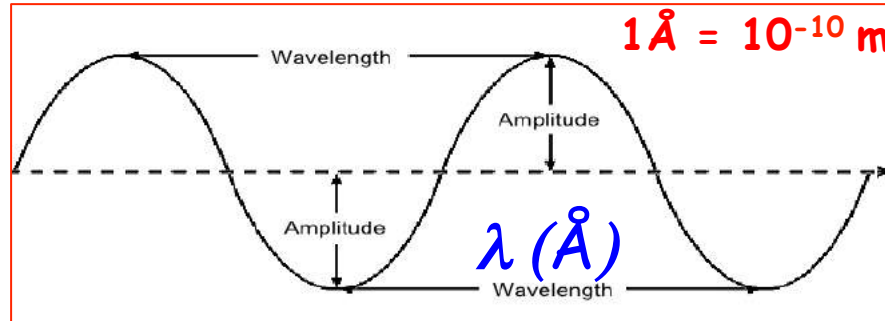
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Outline

- *Light*
- *X-rays and Synchrotron radiation sources*
 - *X-rays and atoms*
 - *X-ray sources*
- *Detectors Basic Principles*
 - *Interactions of x-rays with matter*
 - *XAFS: EXAFS and XANES*
- *Gas Detectors*
 - *Ionization chambers*
- *Conclusions*

Reminder on some units used

- Unit of Wavelengths (λ): Angstrom - \AA



$$1 \text{ nm} = 10^{-9} \text{ m}$$

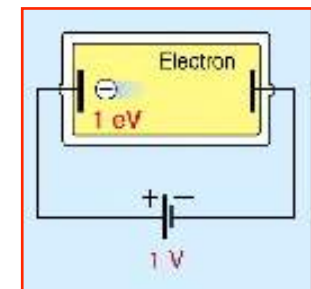
$$1 \text{\AA} = 0.1 \text{ nm}$$

- Unit of Energy: electronvolt - eV

$$1 \text{ eV} = 1.602 \times 10^{-19} \text{ Joules}$$

Energy gained (or lost) by the charge of a **single electron** moving moving across an **electron potential difference of one volt**.

$$\text{GeV} = 10^9 \text{ eV} \quad \text{MeV} = 10^6 \text{ eV}$$



- Unit of time: seconds - s ps = 10^{-12} s fs = 10^{-15} s



Light

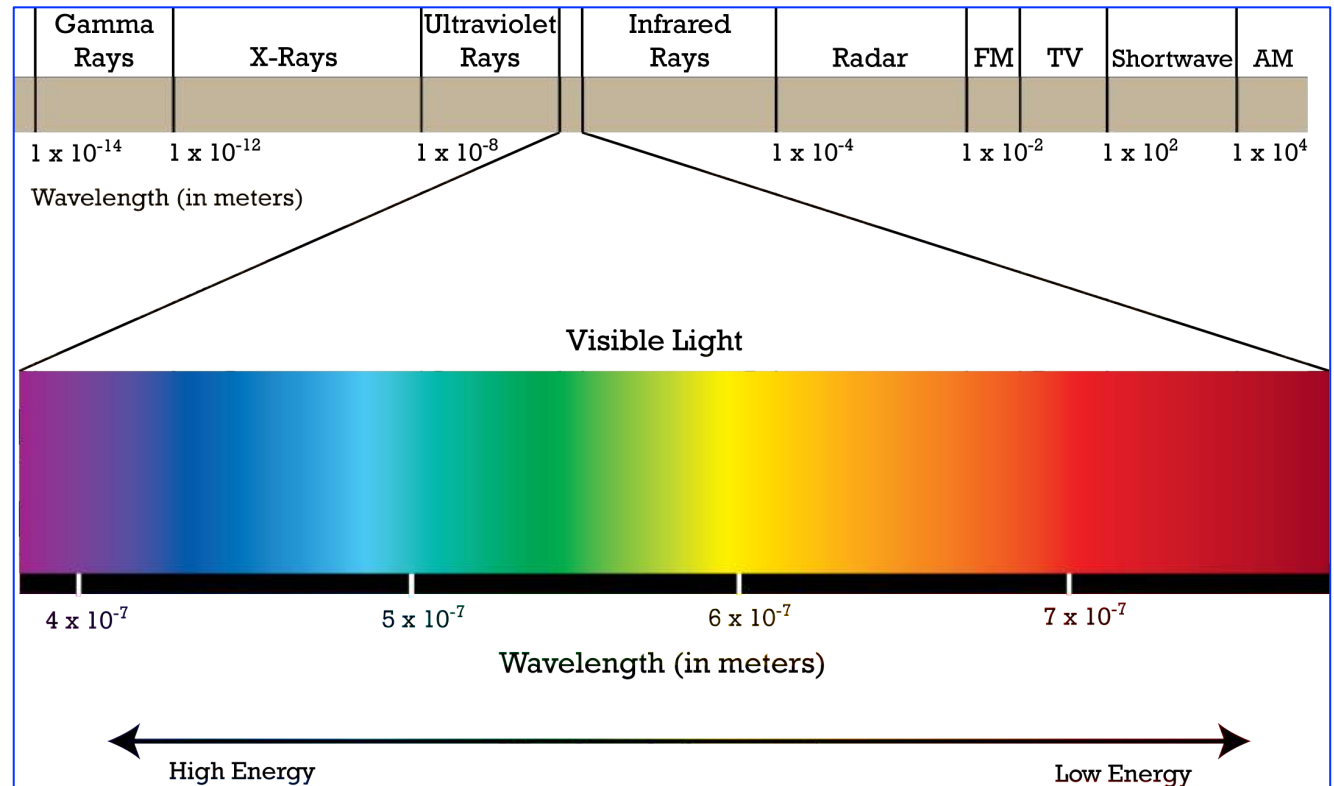


Full featured double rainbow in Wrangell-St. Elias National Park, Alaska.

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Light

Visible light is only a *tiny slice of the electromagnetic spectrum*. The entire electromagnetic spectrum of light is huge, spanning from *gamma rays on one end to radio waves*.

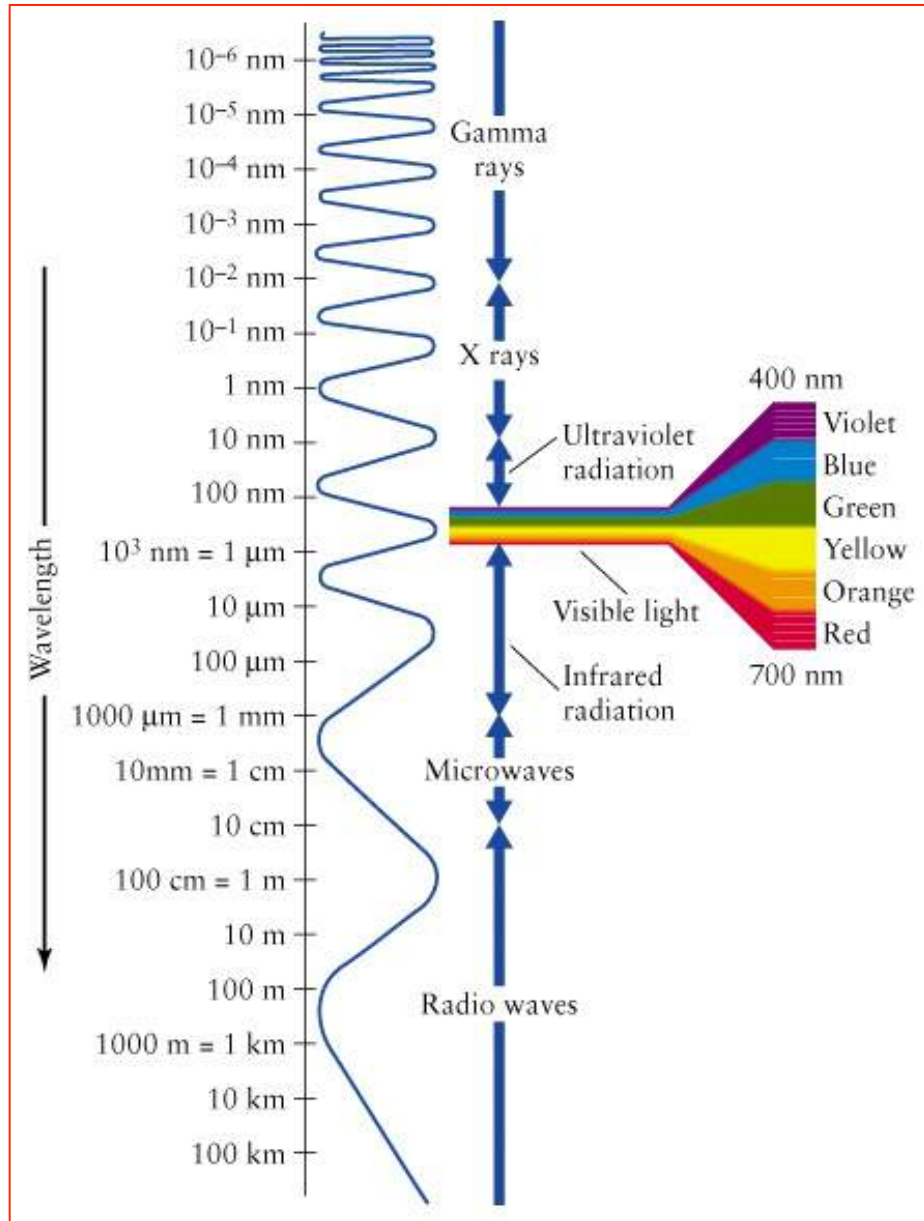
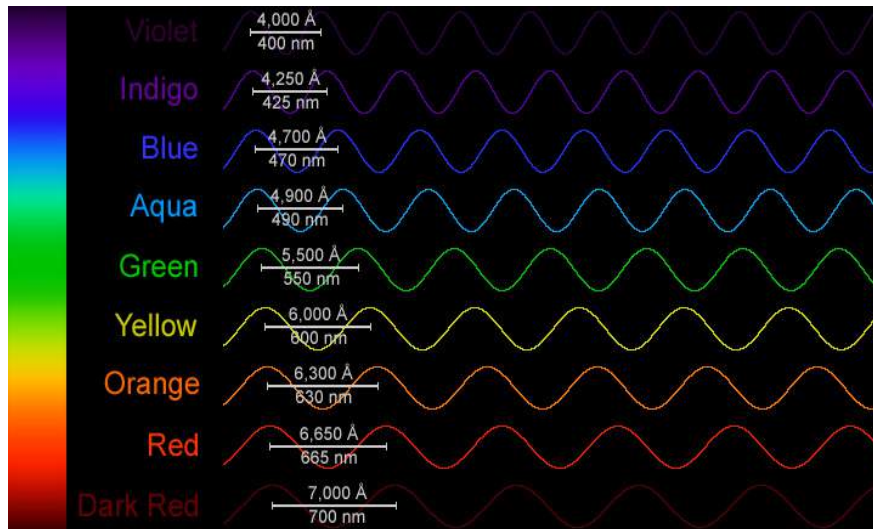
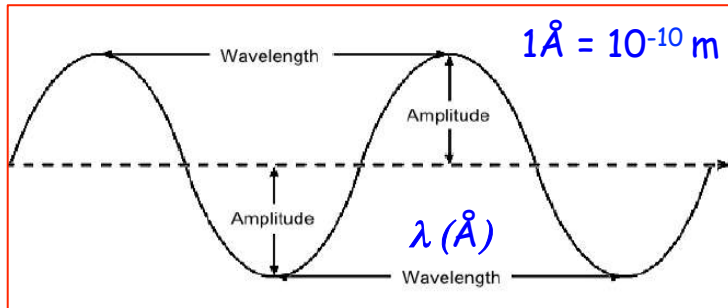


Visible Light is the light we can see using our eyes. This tiny human spectrum encompasses a very specific range of wavelengths from about **380 nm to 780 nm**.

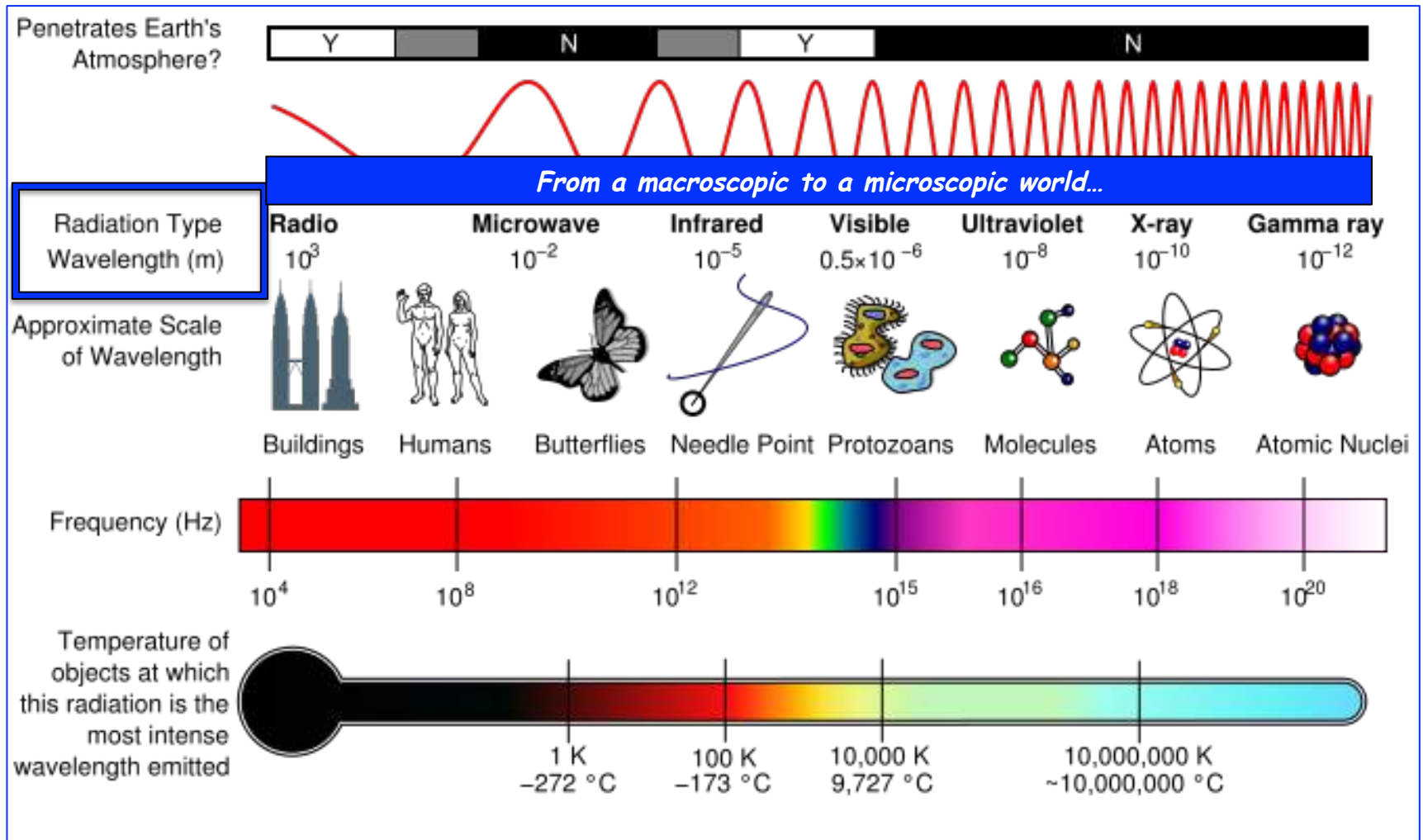
Physiologically we see these frequencies because the photoreceptors in our retinas are sensitive to them. When photons of light hit the photoreceptors this creates an electrochemical signal which is the first step in a fascinating process which ultimately results in us seeing colors.

Light and waves

Light travels as waves of energy.
Waves of light have different **wavelengths** (the distance between the top of one wave and the top of the next). **Different colors of visible light have different wavelengths.**



Electromagnetic Spectrum



The **wavelength** (λ) and **frequency** (ν) of light are strictly related: the higher the frequency the shorter the wavelength! This is because all light waves move through vacuum at the same speed (c = speed of light) and the equation that relates wavelength and frequency for electromagnetic waves is: $\lambda \nu = c$ $E = h \nu$

X-rays

X-rays discovery

EINE NEUE ART VON STRAHLEN.

VON
DR. W. RÖNTGEN,
O. O. PROFESSOR AN DER K. UNIVERSITÄT WÜRZBURG.

WÜRZBURG.
VERLAG UND DRUCK DER STAHEL-SCHEN K. Hof- und UNIVERSITÄTS-
BUCH- und KUNSTHANDLUNG.
Jede 1895.

60 S.

On a New Kind of Rays

While **Wilhelm Roentgen** was working on the effects of **cathode rays** during **1895**, he discovered X-rays. His experiments involved the passing of electric current through gases at extremely low pressure. On **November 8, 1895** he observed that certain rays were emitted during the passing of the current through discharge tube. His experiment that involved working in a totally dark room with a well covered discharge tube resulted in the **emission of rays which illuminated a barium platinocyanide screen. The screen became fluorescent even though it was placed two meters away from discharge tube.**



Wilhelm Conrad Roentgen

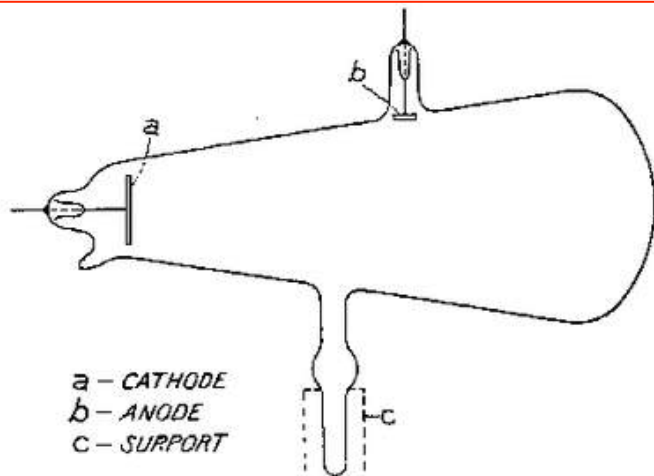


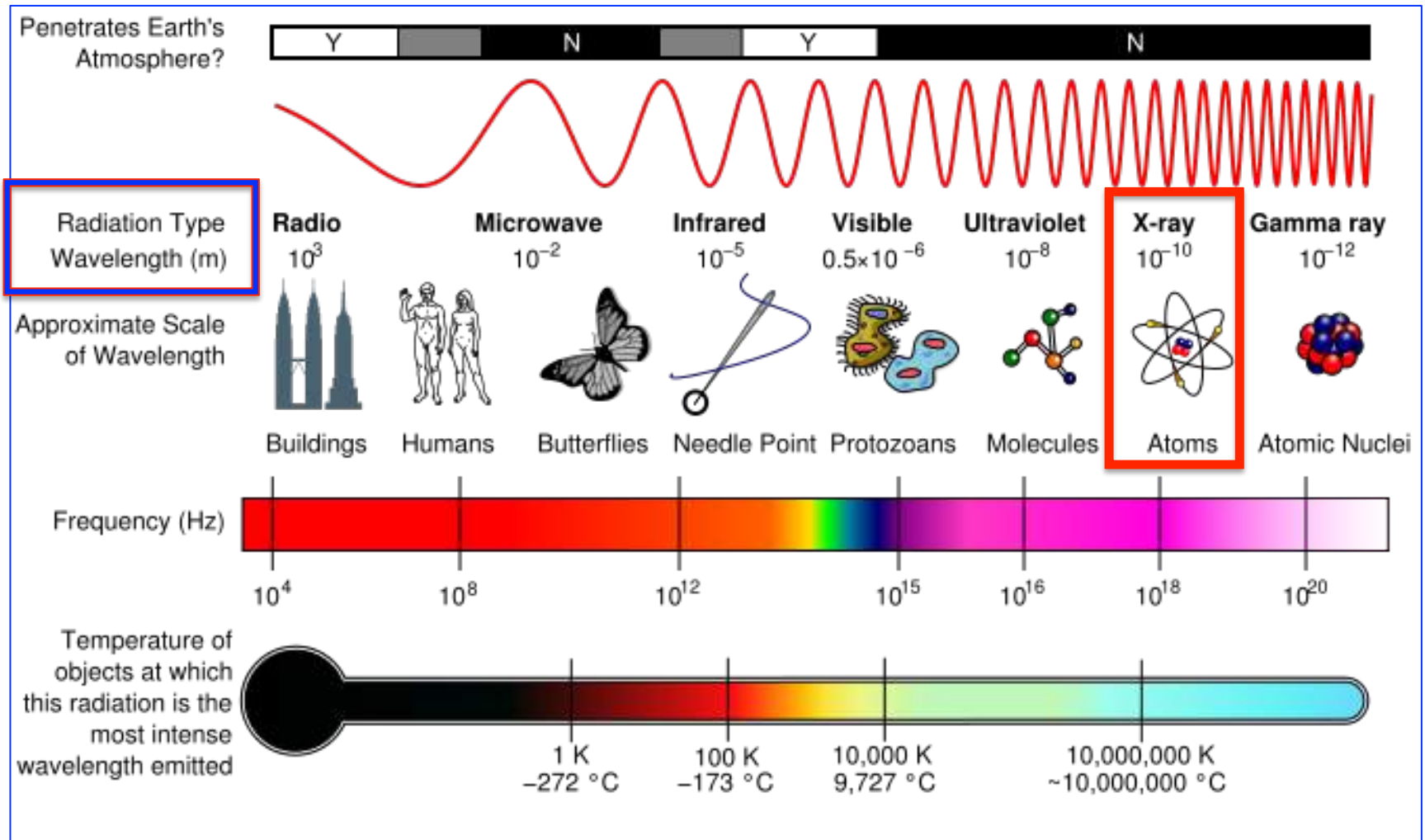
Fig. 1. Earliest type of roentgen tube.

Gas tube: electrons are freed from a cold cathode by positive ion bombardment, thus necessitating a certain gas pressure.

He continued his experiments using **photographic plates** and generated the very **first "roentgenogram"** by developing the image of **his wife's hand** and analyzed the variable transparency as showed by her bones, flesh and her wedding ring.



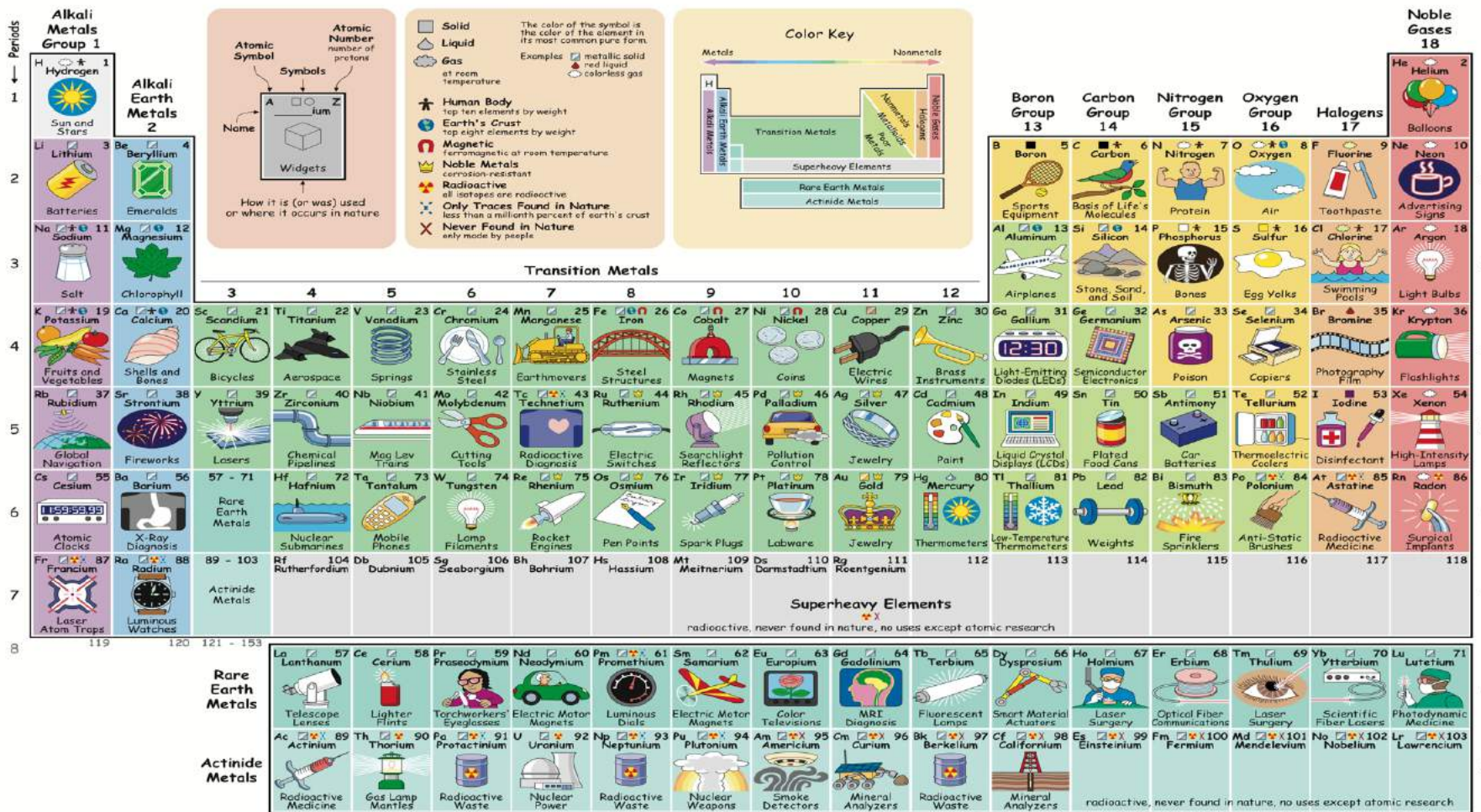
Electromagnetic Spectrum and X-rays



Atoms

Matter is everything around us! All matter such as solids, liquids, and gases, is composed of atoms. Therefore, atoms are considered to be the basic building block of matter. From the periodic table, it can be seen that there are only about 100 different kinds of atoms. These same 100 atoms form thousands of different substances ranging from the air we breathe to the metal used to support tall buildings.

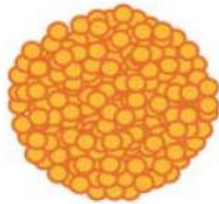
The Periodic Table of the Elements, in Pictures



Atoms and physical properties



Bulk



Nanoparticle
 $< 10 \text{ nm}$



Cluster
 $< 2 \text{ nm}$



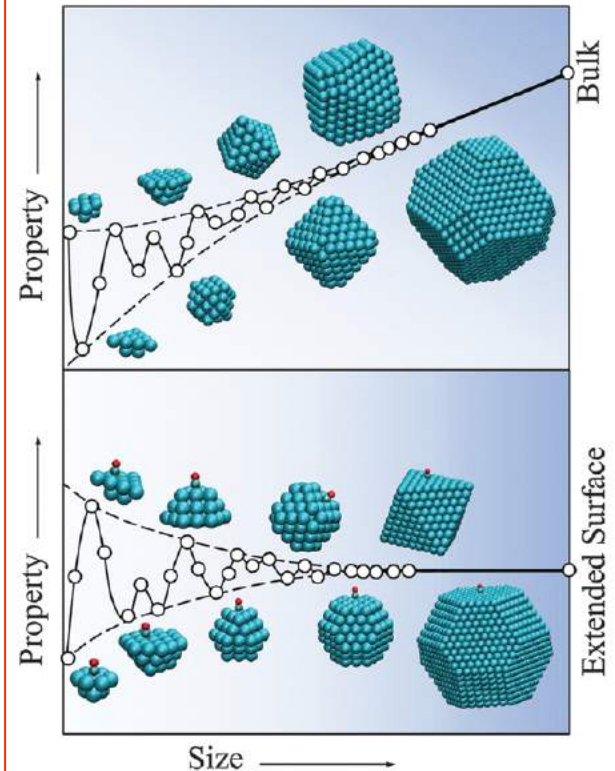
Atom



continuous

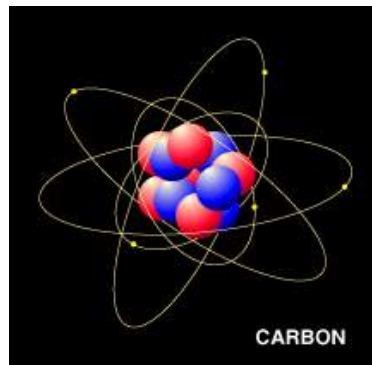


discrete



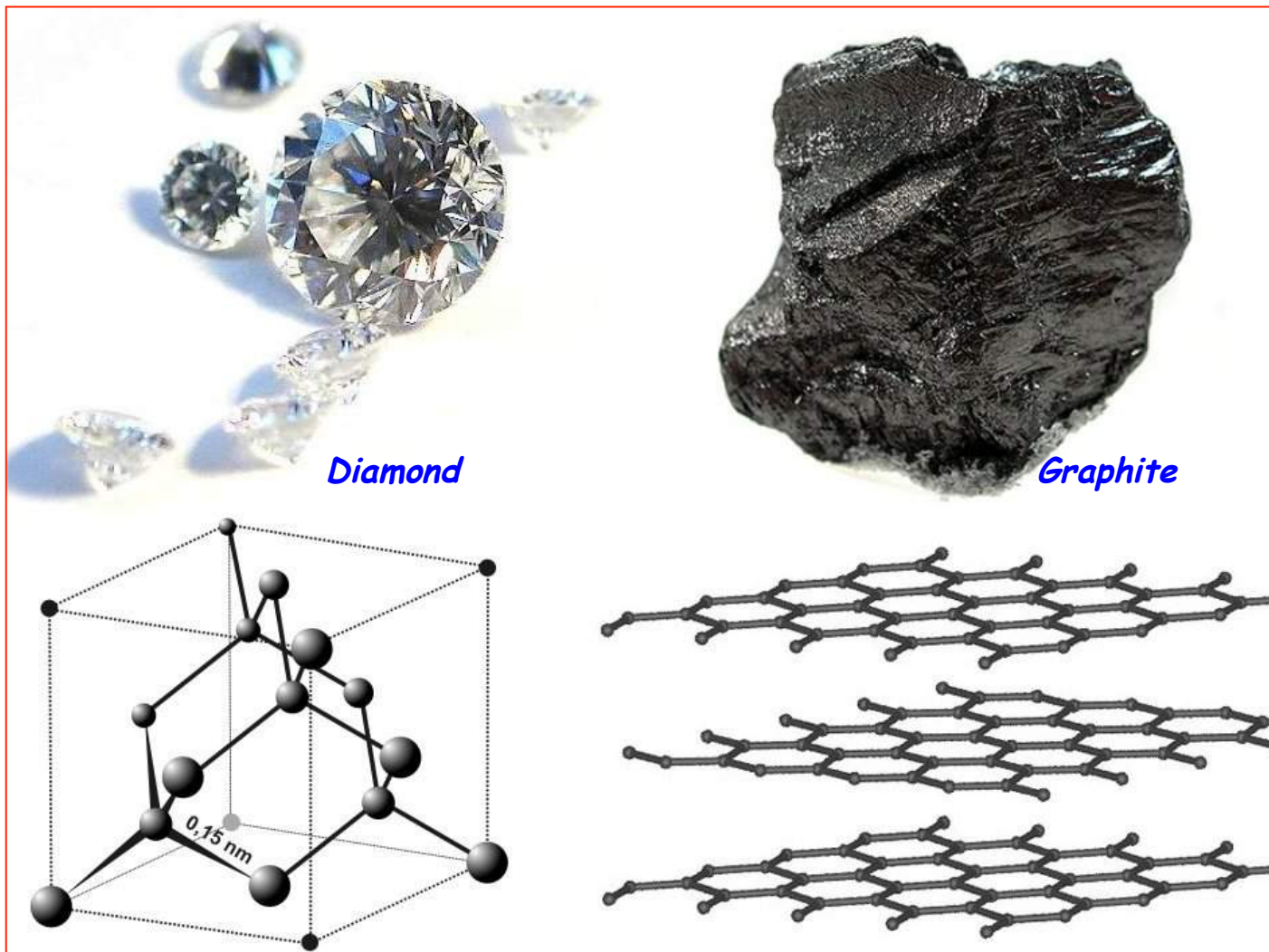
Gold coins and ingots have been highly prized for millennia. But scientists have realized that nanoparticles of this metal could have also quite important properties. In labs around the world, gold nanoparticles are being tested as components in technology and medicines. *Gold nanoparticles could be used to kill cancer cells, improve the efficiency of solar cells and catalyze chemical reactions.*

X-rays and Atoms



Both diamond and graphite are made entirely out of carbon!

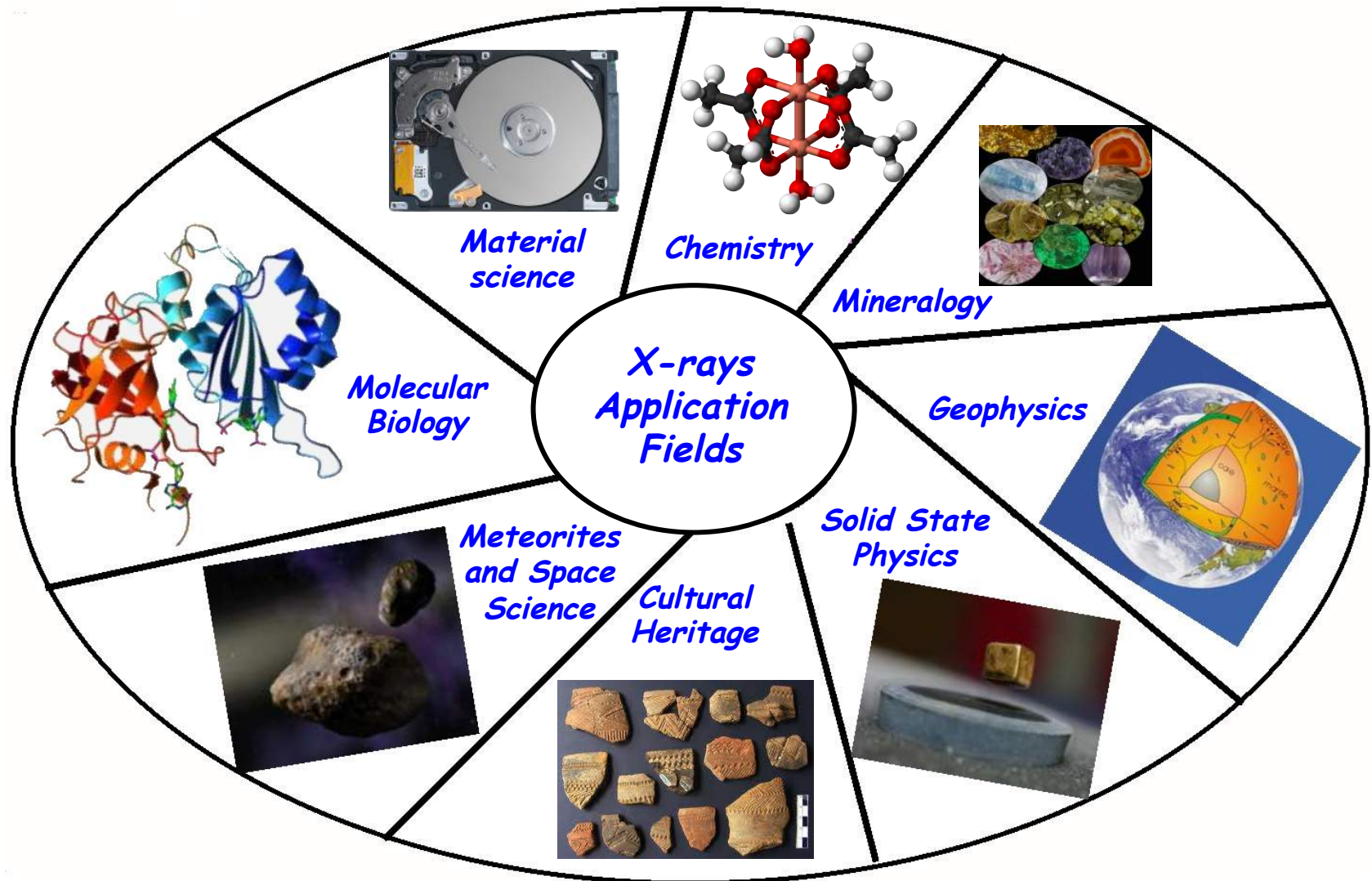
Using x-rays to reveal the atomic structure of materials



Graphite is opaque and metallic- to earthy-looking, while diamonds are transparent and brilliant.

The different properties of graphite and diamond arise from their distinct crystal structures.

X-rays application fields



Synchrotron radiation sources

Light sources

Fire is not a very useful light source to see small details because its emitted power is spread in all directions!



A torchlight is more adequate because due to its small size the emission is concentrated within a narrow angular spread: this a "bright" source!



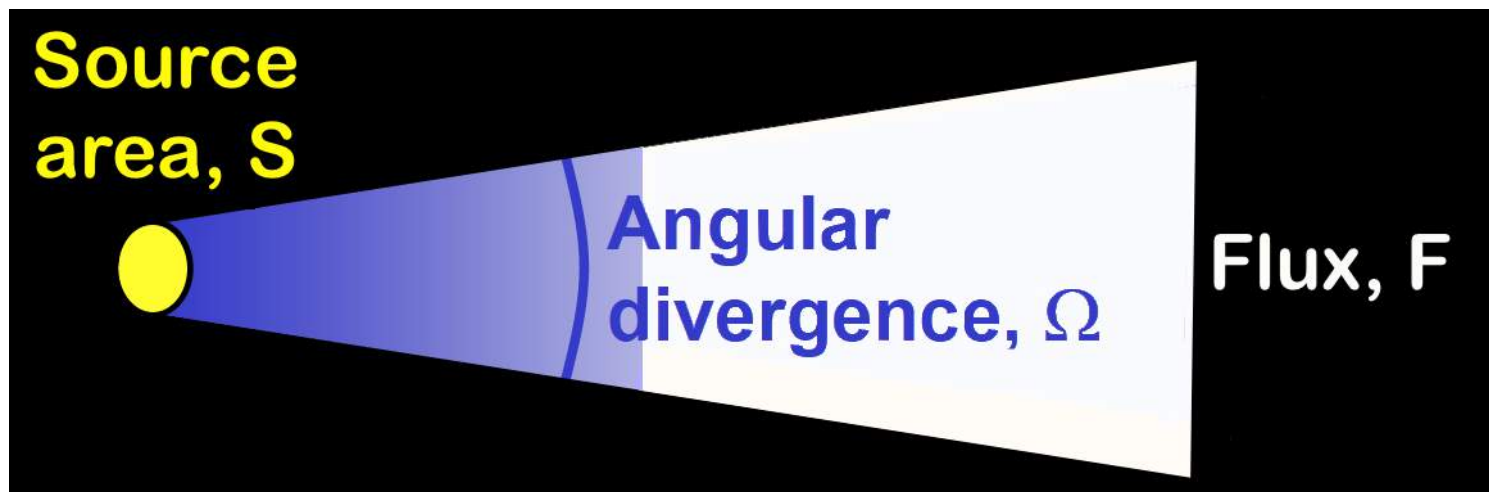
Synchrotron radiation is a very bright light source that, as will be shown, gives us the chance to study also things that we cannot "see" with our eyes using not visible light but X-rays!



Light sources and brightness

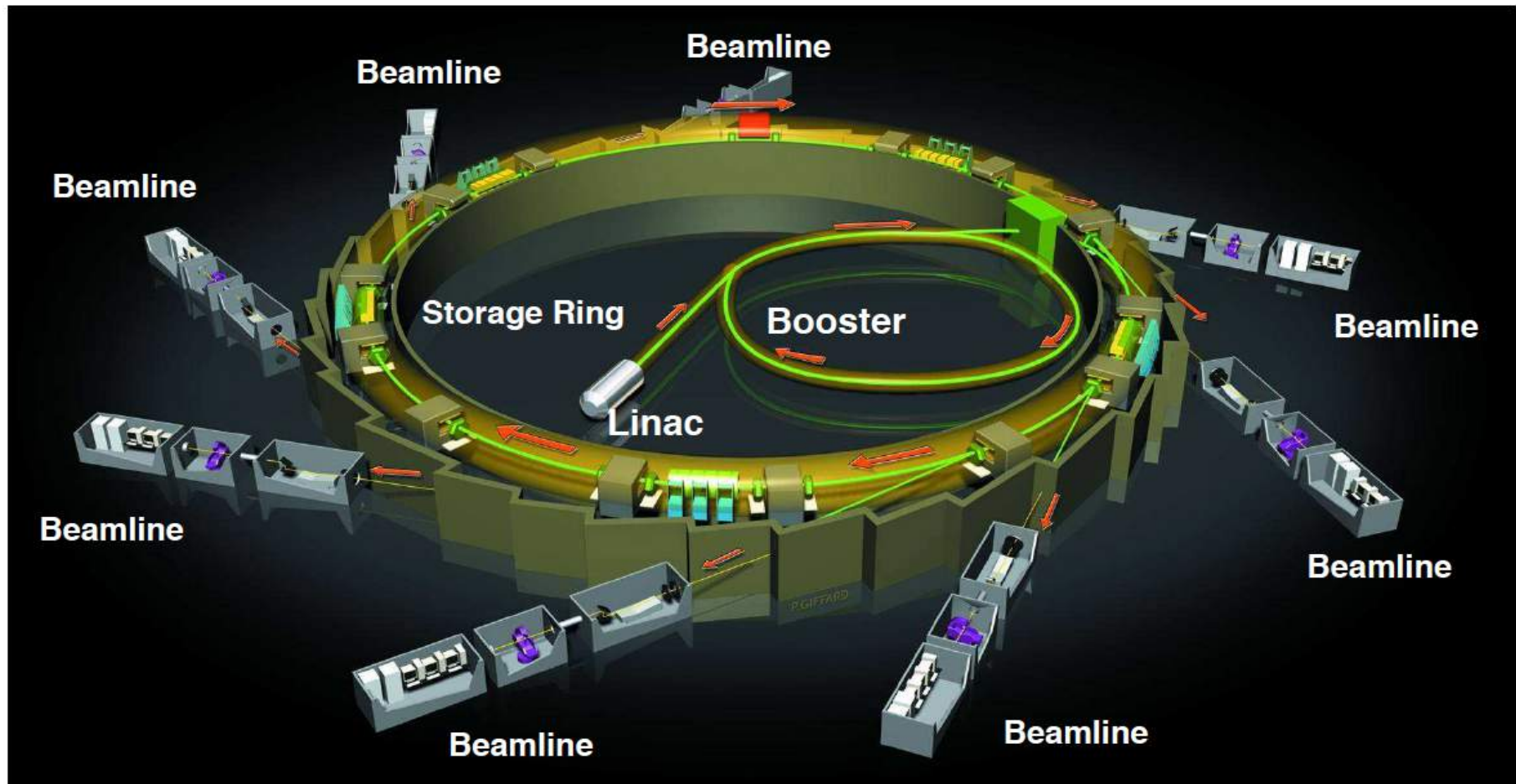
When interested in nm scale details: brightness becomes fundamental.

A **bright source** is the one **very effective in illuminating a specific target**.
If the specific target is small a **bright source** is a **small size source with emission concentrated within a narrow angular spread**.



$$\text{Brightness} = \text{constant} \frac{F}{S \times \Omega}$$

Accelerators as Bright Sources of Synchrotron Radiation



Bright X-ray sources?

HE particle accelerators

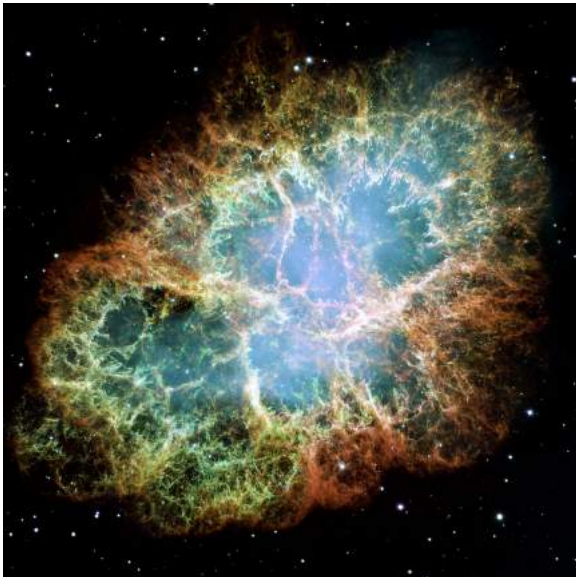


Relativistic effects



Synchrotron light

Synchrotron light is present in nature!



Synchrotron radiation is a very important emission process in astrophysics!

Crab Nebula: remnant of a supernova explosion seen on earth by Chinese astronomers in 1054, at about 6500 light years from Earth in the constellation Taurus !

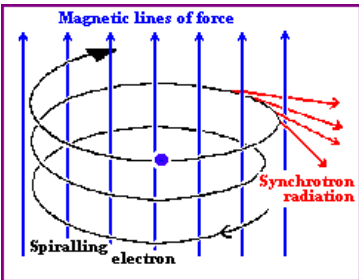
SR emission is produced by high energy electrons whirling around the magnetic fields lines originating from a Pulsar

The heart of the nebula is a rapidly-spinning neutron star, a pulsar, that powers the strongly polarised bluish 'synchrotron' nebula.

The Crab pulsar is slowing at the rate of about 10^{-8} sec per day, and the corresponding energy loss agrees well with the energy needed to keep the nebula luminous. Some of this luminosity takes the form of synchrotron radiation, requiring a source of energy for accelerating charged particles.

Composite image data from three of NASA's Great Observatories. The Chandra X-ray Observatory image is shown in blue, the Hubble Space Telescope optical image is in red and yellow, and the Spitzer Space Telescope's infrared image is in purple. The X-ray image is smaller than the others because extremely energetic electrons emitting X-rays radiate away their energy more quickly than the lower-energy electrons emitting optical and infrared light. The Crab Nebula is one of the most studied objects in the sky, truly making it a cosmic icon.

NASA Hubble Space Telescope image of the Crab Nebula (NASA, ESA and Allison Loll/Jeff Hester (Arizona State University)).

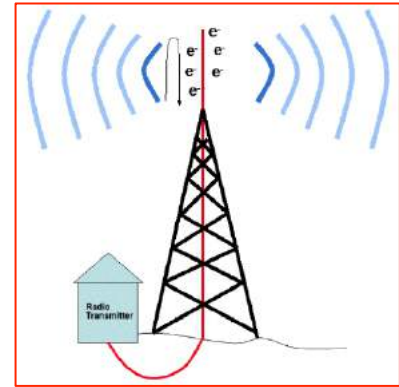


NASA's Great Observatories' View of the Crab Nebula X-Ray-blue: NASA/CXC/J.Hester (ASU); Optical-red and yellow: NASA/ESA/J.Hester & A.Loll (ASU); Infrared-purple: NASA/JPL-Caltech/R.Gehrz (Univ. Minn.)

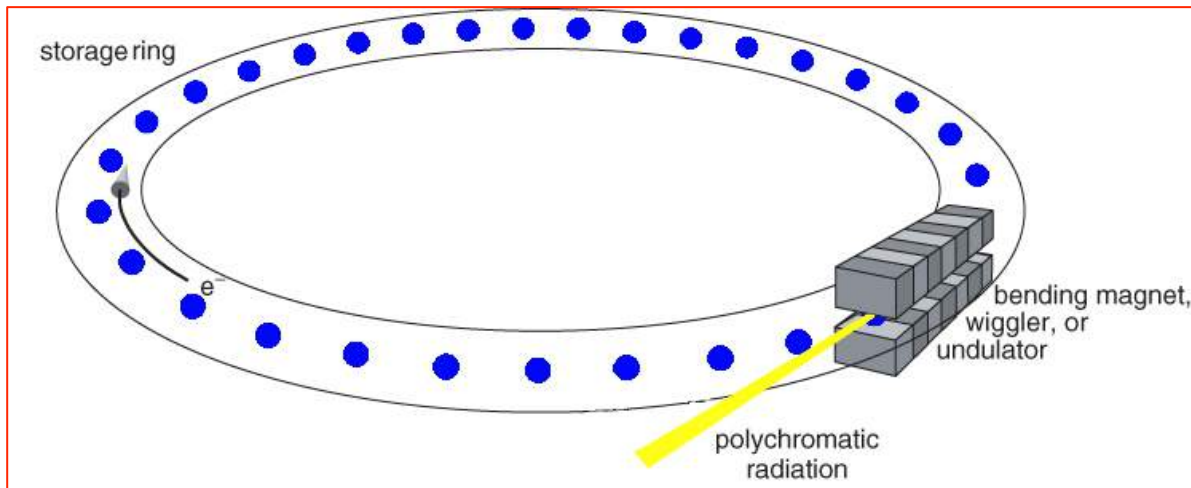
Synchrotron radiation

Accelerated NON relativistic charged particle, e^+ , e^- and ions, emit electromagnetic radiation like electric charges forced to oscillate along an antenna.

$$v \ll c \text{ or } \beta = v/c \ll 1$$

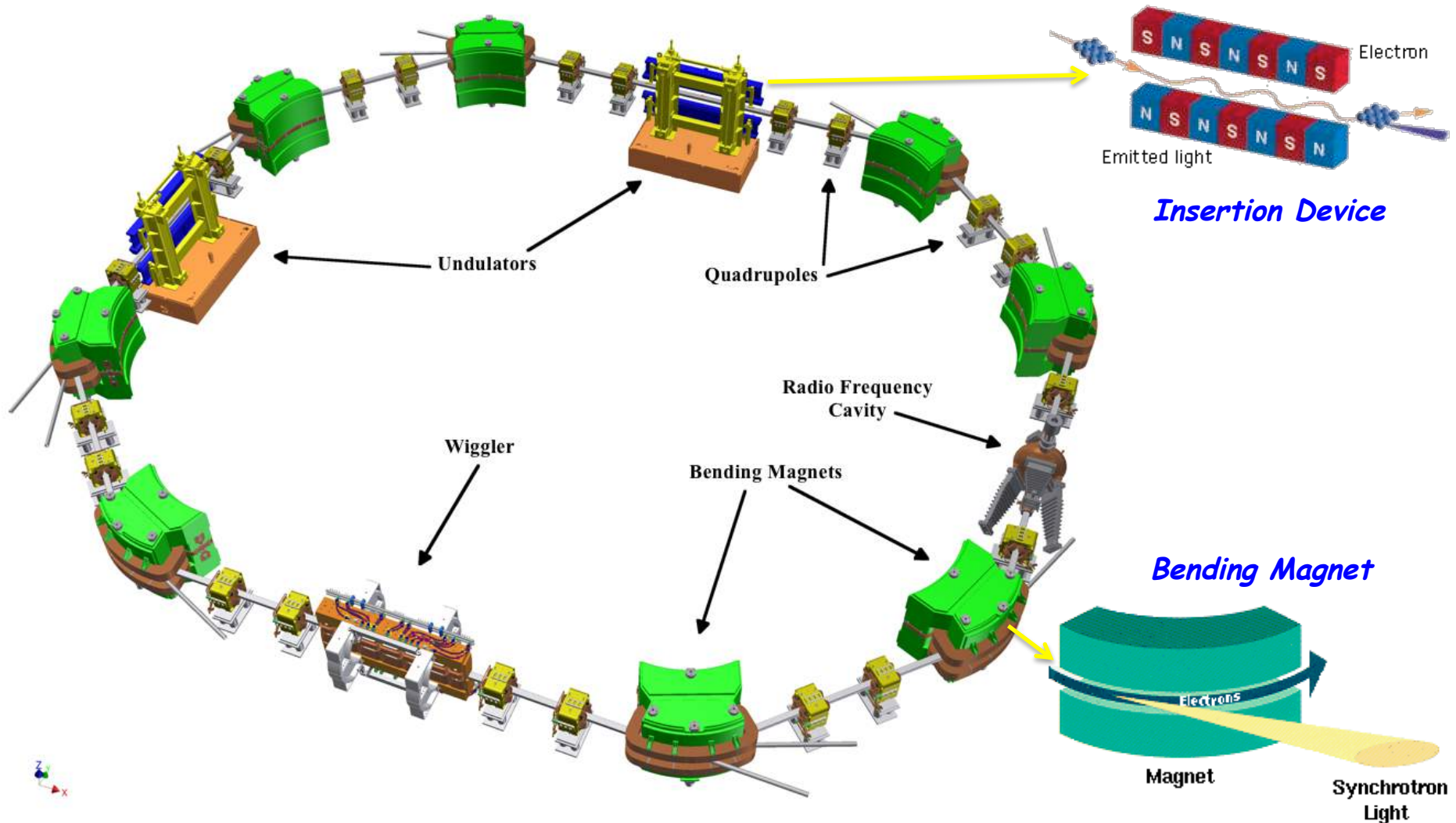


*When **charged particles**, moving at **RELATIVISTIC speeds** ($v \approx c$), are forced to **change the direction of their motion** (acceleration), under the effect of magnetic fields, in circular particle accelerators, like synchrotrons, the radiation produced is called **Synchrotron Radiation**.*



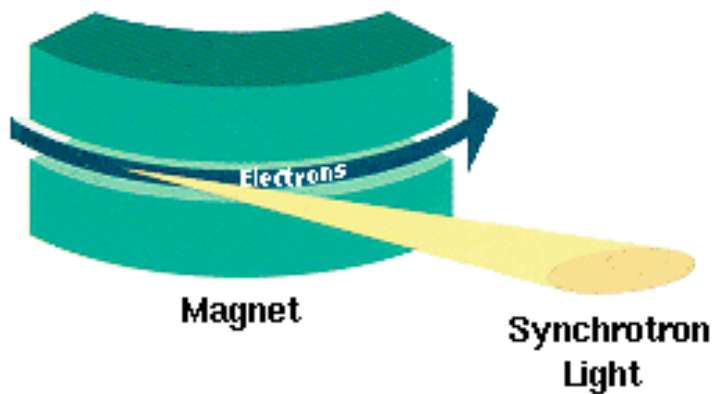
$$v \approx c \text{ or } \beta = v/c \approx 1$$

Synchrotron light is artificially produced by relativistic particles accelerated in circular orbits.

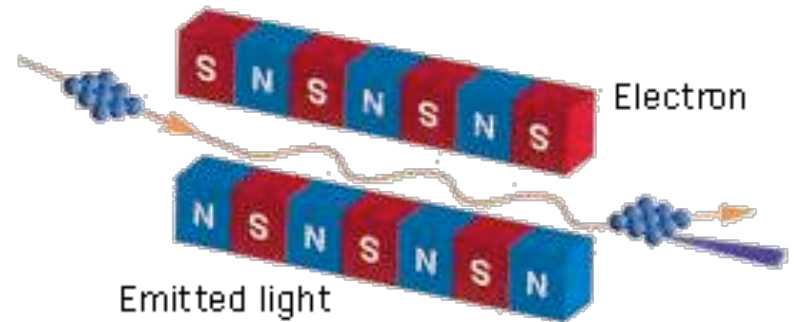


... and synchrotron radiation is also the coherent radiation emitted by the undulators of Free Electron Lasers.

Radiation sources



Bending magnet

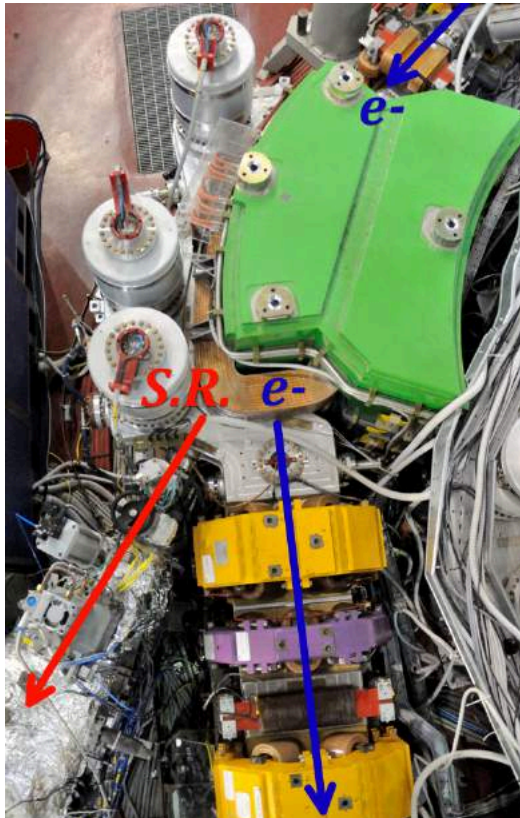


Undulator

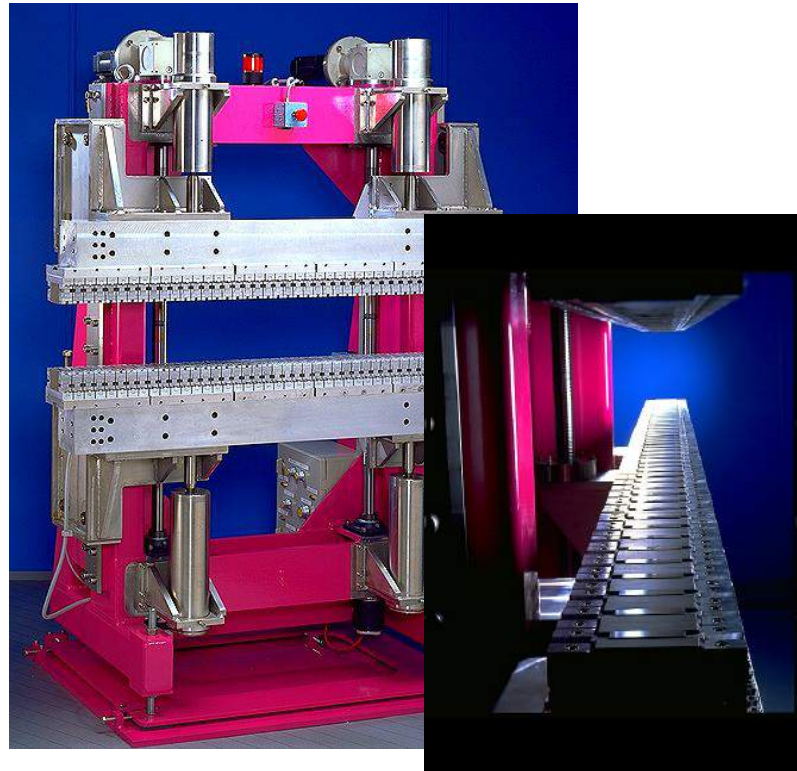
Radiation Sources

There are two different sources of radiation in a storage ring:

- bending magnets (BMs)*
- insertion devices (IDs) or periodic arrays of magnets inserted between the BMs (wigglers and undulators)*



DAΦNE bending magnet (BM)

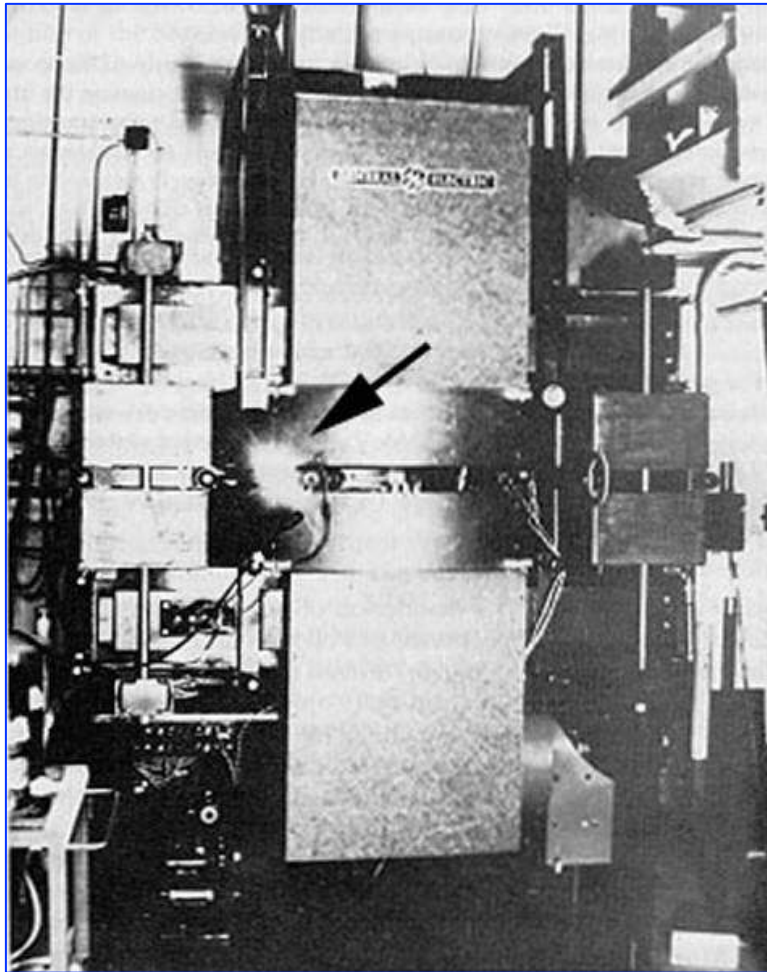


ESRF Insertion Device (ID) - Undulator

BM and ID have different characteristics concerning, spectral distribution, flux, brightness and polarization.

Short History

Synchrotron radiation: history



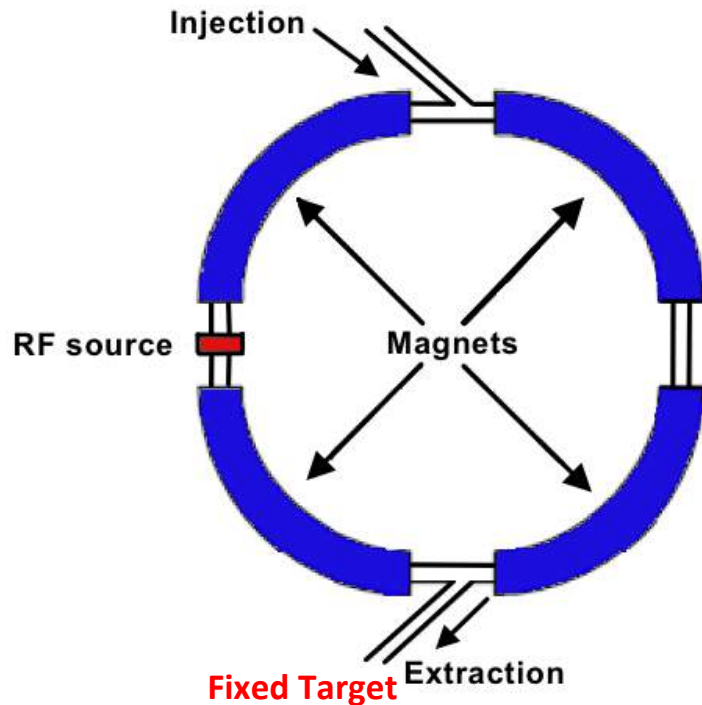
Starting point: Proof of concepts, tests of theories!

- In the 50s and 60s machines built for High Energy Physics: synchrotrons (*1947 First 'visual observation of synchrotron radiation'*).
- Synchrotron radiation was considered a *nuisance by particle physicists: unwanted but unavoidable loss of energy!*
- 1961 US National Bureau of Standards (now NIST) modified their electron synchrotron: *access to the synchrotron radiation users*.
- Synchrotron radiation scientists became *parasites* of nuclear physics experiments. (*1961 Frascati - CNEN Electrosynchrotron - (0.4-1.1) GeV*)
- 1968 *First storage ring dedicated* to synchrotron radiation research: *Tantalus* (University of Wisconsin) only *bending magnets*.

1947 General Electric Res. Lab. - 70 MeV Electron Synchrotron - N.Y. USA

*F.R. Elder, A.M. Gurewitsch, R.V. Langmuir, and H.C. Pollock, Radiation from Electrons in a Synchrotron, Phys. Rev. 71, 829 (1947)
G. C. Baldwin and D.W. Kerst, Origin of Synchrotron Radiation, Physics Today, 28, 9 (1975)*

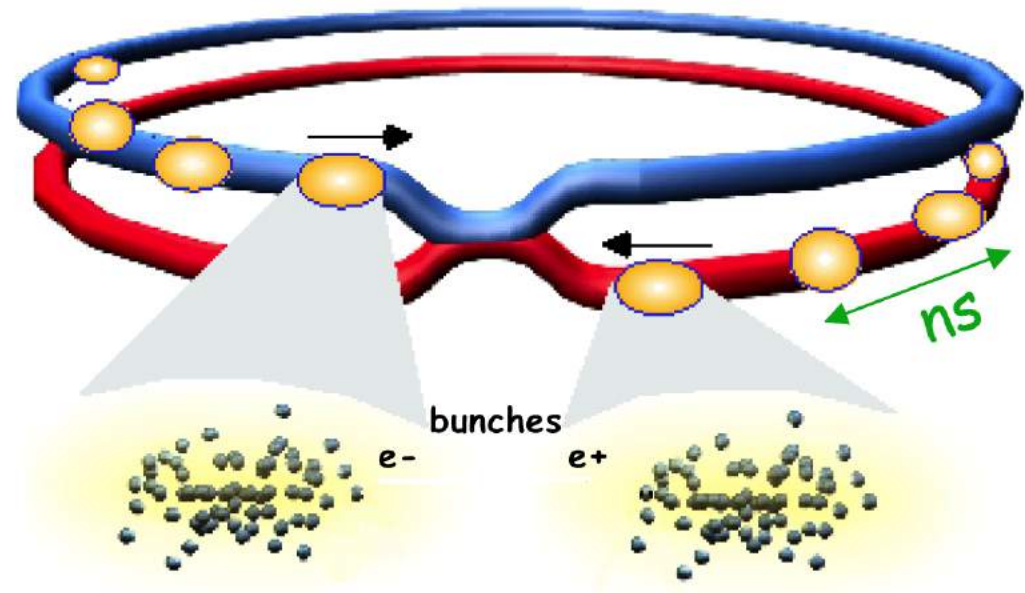
Synchrotrons and Storage Rings



Synchrotron

Particle beam on fixed target

$$E_{CM} = (mE)^{1/2}$$



Storage rings

Colliding particle beams

$$E_{CM} = 2E$$

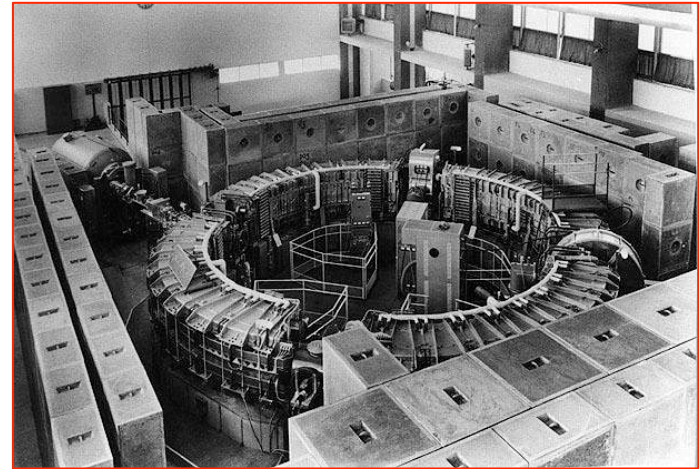
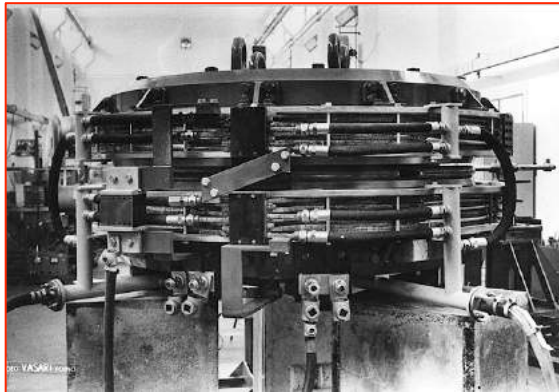
Colliding beams more efficient

$E = \text{particle energy} \gg m_0 c^2$ $E_{CM} = \text{center-of-mass energy}$

Synchrotron radiation: short history

Frascati: ElettroSynchrotron, ADA and ADONE

*Frascati - CNEN (Comitato Nazionale Energia Nucleare)
Laboratory **ElettroSincrotrone** - (0.4-1.1) GeV, $C = 28$ m
(1959-1975)*



*LNF **ADA** (Anello Di Accumulazione) - first electron-positron
storage ring (proposed by B. Touschek) 0.25 GeV, $C = 5$ m
(1961-1964)*

*LNF **ADONE** (big ADA) electron-positron storage ring 1.5
GeV per beam, $C = 105$ m
(1969-1993)*

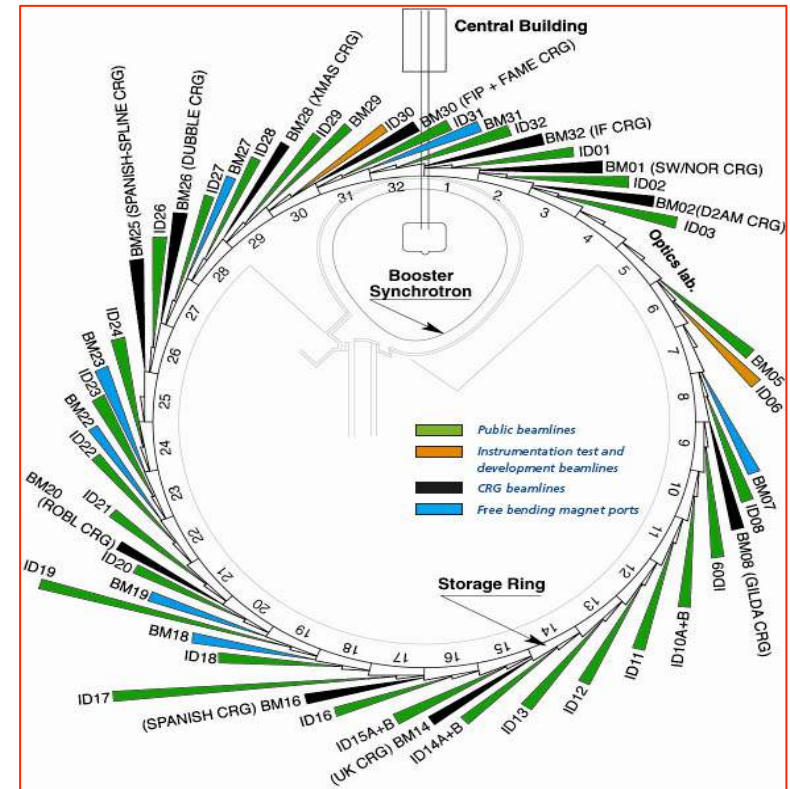
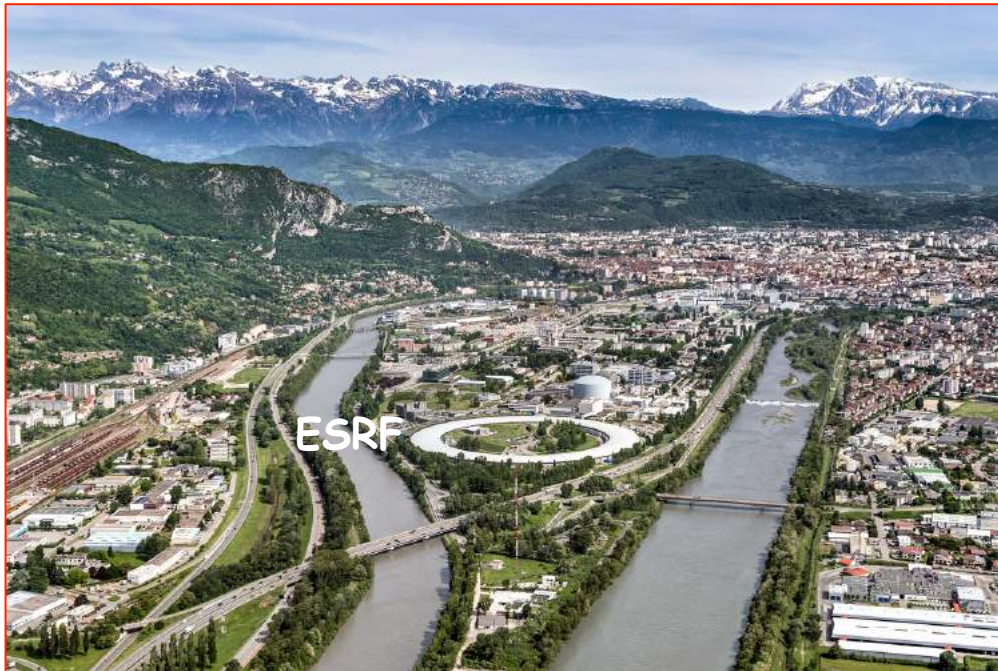


*1976-1993 LNF ADONE 1.5 GeV parasitic/dedicated use
for SR experiments after its use for HE experiments.*

Synchrotron radiation: short history

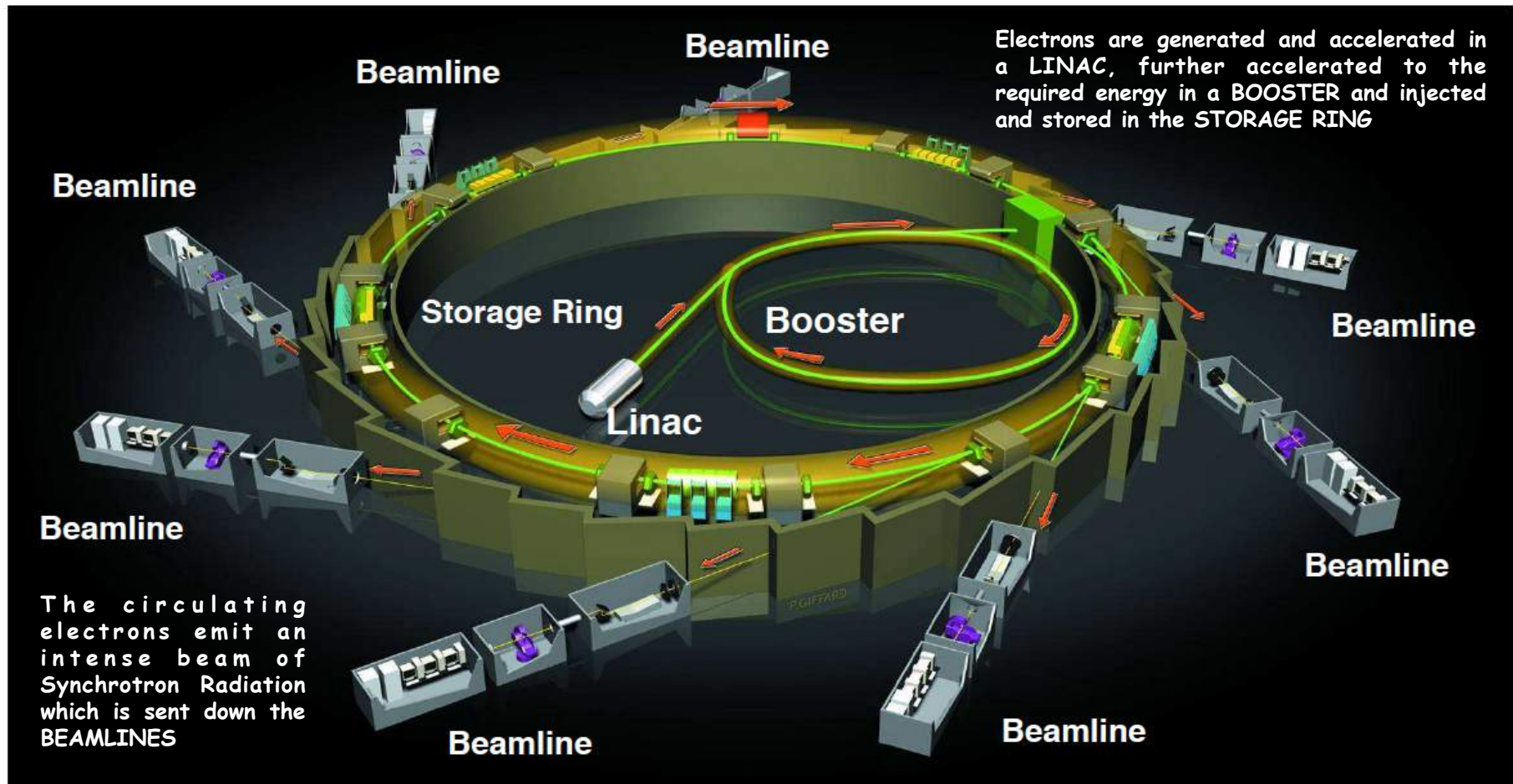
Third generation: optimized sources

Synchrotron light is now a unique tool for science!



ESRF, Grenoble - France 6 GeV, $C = 844$ m opened to users in 1994

Schematic view of a Synchrotron Radiation facility



As a function of the energy range to be used each beamline must be optimized for a particular field of research.

Beamline schematic composition:

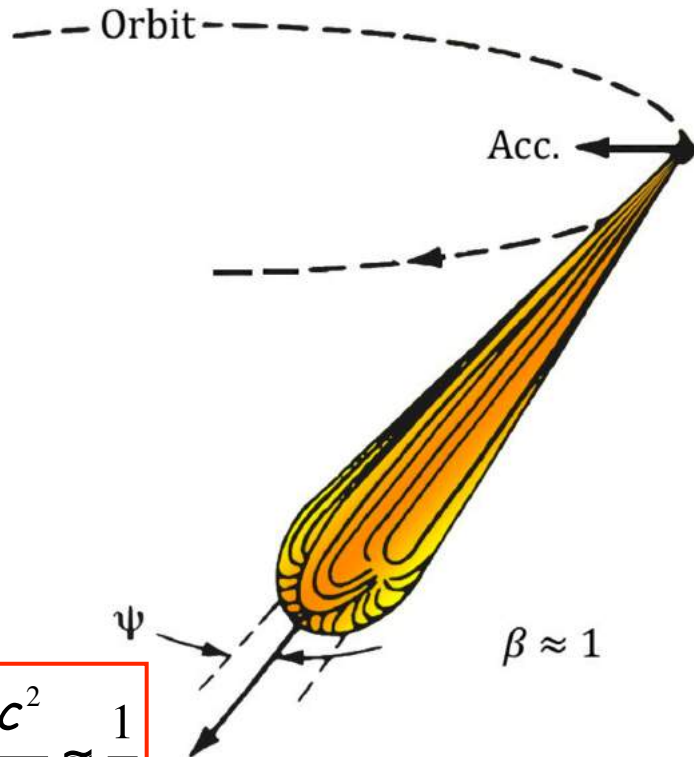
- *Front end*
- *Optical hutch*
- *Experimental hutch*
- *Control and computing*

The *front end* isolates the beamline vacuum from the storage ring vacuum; defines the angular acceptance of the synchrotron radiation via an aperture; blocks (beam shutter) when required, the x-ray and Bremsstrahlung radiation during access to the other hutches.

Synchrotron radiation properties

Synchrotron radiation: physics

Relativistic focusing of Synchrotron Radiation



$$\psi \approx \frac{m_0 c^2}{E} \approx \frac{1}{\gamma}$$

$$v \approx c \text{ or } \beta = v/c \approx 1$$

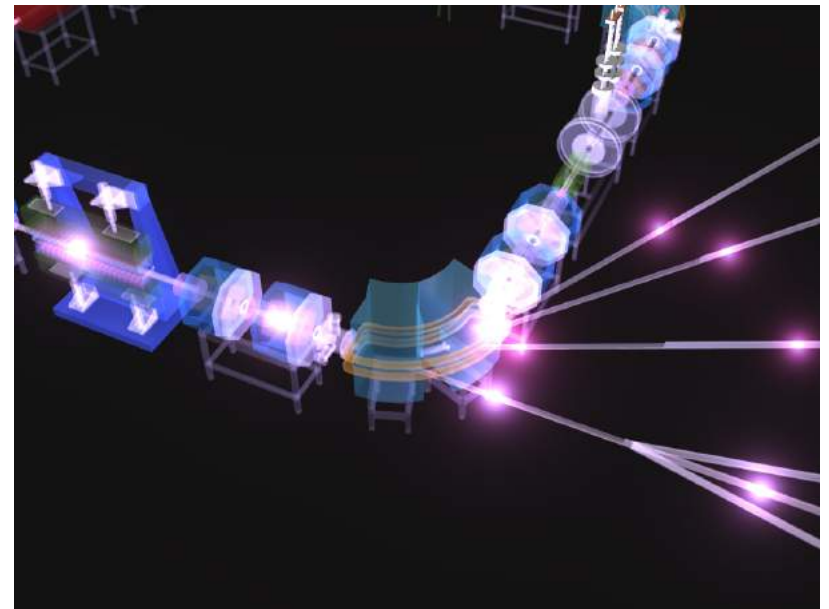
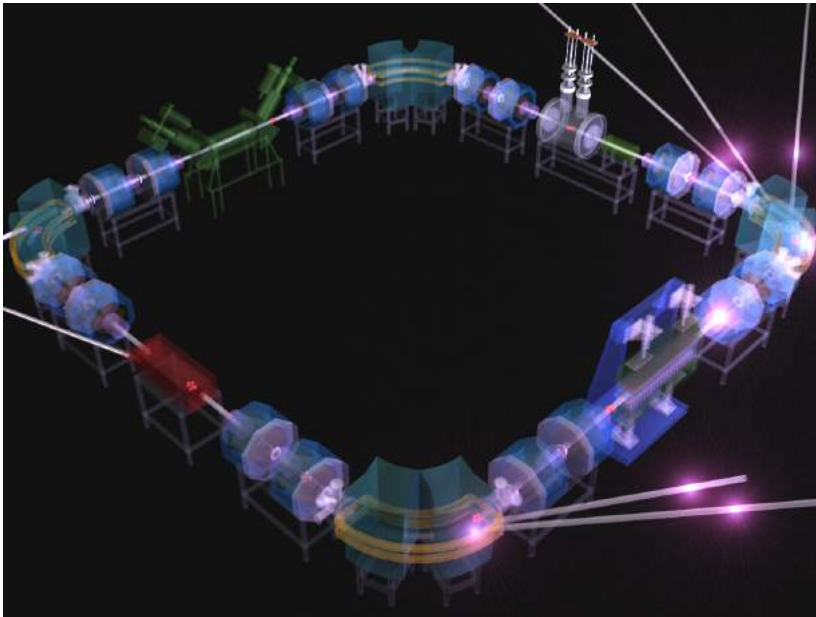
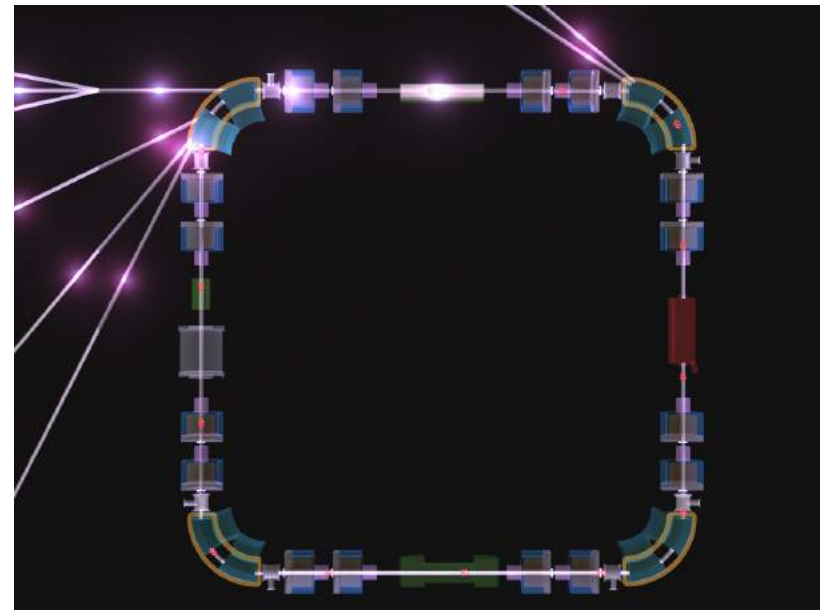
For a *relativistic effect*, when the speed of the emitting electrons increases to relativistic values ($v \approx c$) the radiation pattern is compressed into a *narrow cone in the direction of motion, resulting into an emission tangential to the particle orbit*.

Radiated power:

$$P_{\text{rad}} = \frac{2}{3} \frac{Q^2 c}{R^2} \left[\frac{E}{m_0 c^2} \right]^4$$

Q = particle charge, E = particle energy, m_0 = rest mass, R = radius of curvature

1949 Schwinger: classical theory of radiation from accelerated relativistic electrons



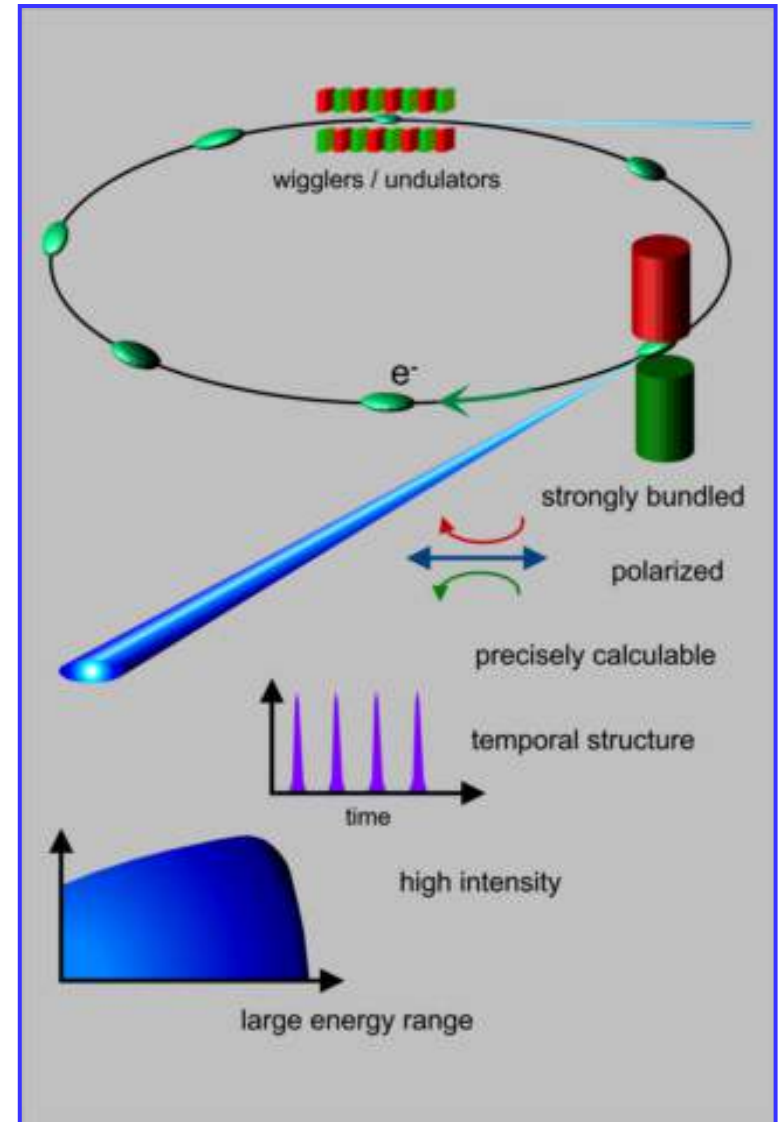
ASTRID (Aarhus - Denmark) <http://www.isa.au.dk/animations/pictures/pic-index.asp>

http://www.isa.au.dk/animations/Finalmovie/astrid_total_v2.mov

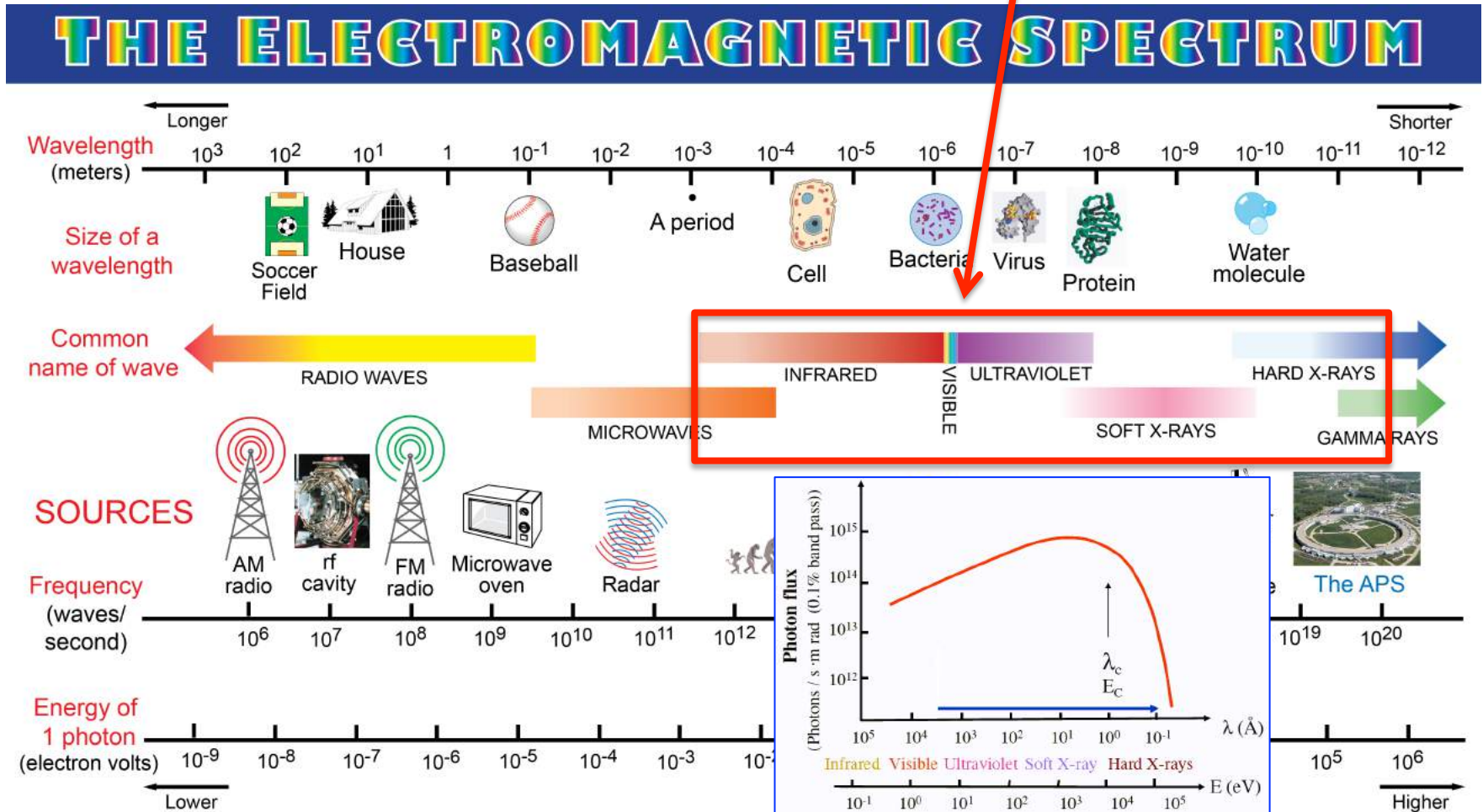
Synchrotron Radiation Properties

What makes synchrotron radiation interesting, powerful and unique?

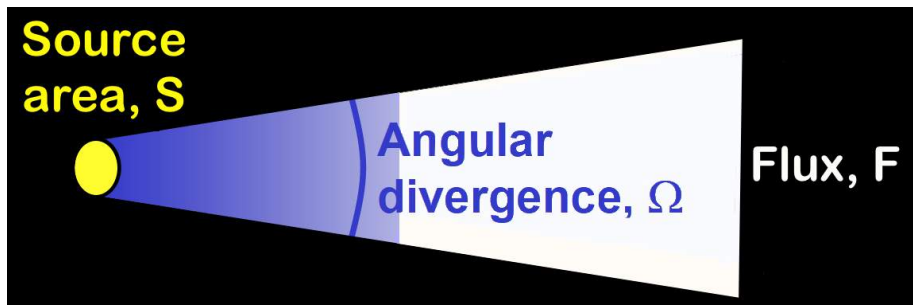
- **Continuum source from IR to X-rays (tunability)** which covers from **microwaves to hard X-rays**: the user can select the wavelength required for experiment-**continuous (Bending Magnet/Wiggler)** - **quasi-monochromatic (Undulator)**
- **Source in a clean UHV environment**
- **Very high flux and brightness (with undulators)** highly **collimated** photon beams generated by a **small divergence** and **small size sources**.
- **Highly Polarized**
- **Pulsed time structure** - pulsed length down to tens of picoseconds allows the resolution of processes on the same time scale
- **High stability** (submicron source stability)



Spectral range covered by Synchrotron Radiation!



Brightness



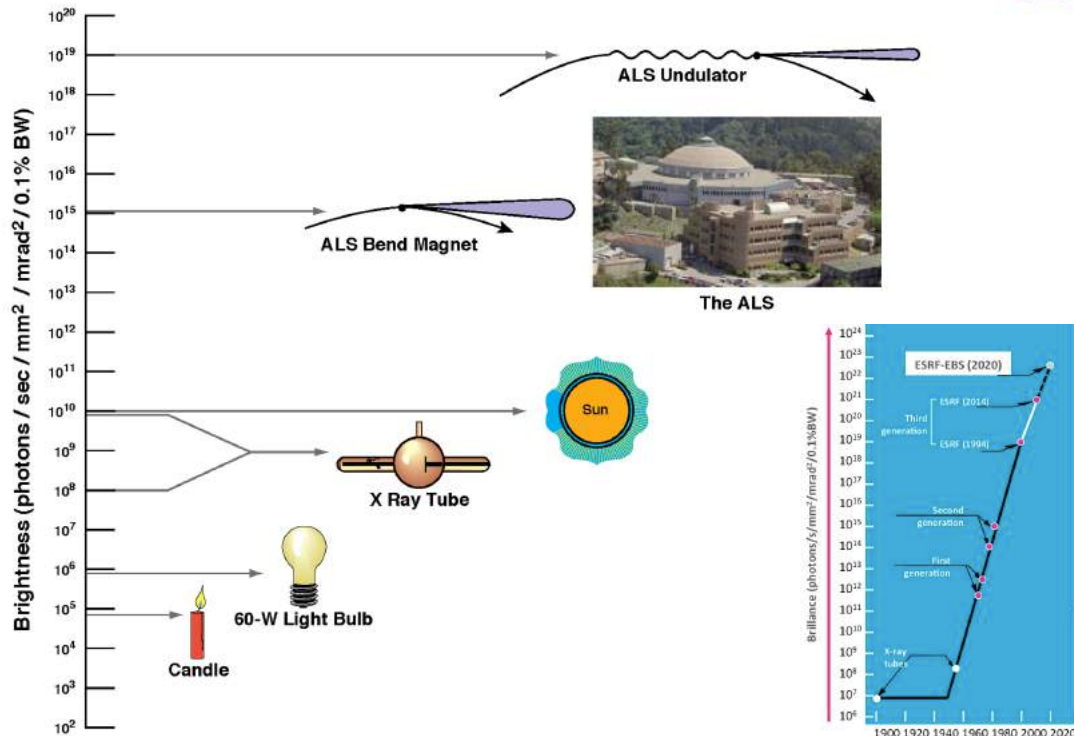
$$\text{Brightness} = \text{constant} \frac{F}{S \times \Omega}$$

Brightness

Synchrotron radiation sources have very high brightness.

How Bright Is the Advanced Light Source?

ALS

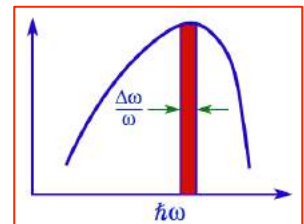


Brightness of third generation low emittance storage rings.

- *Electrons in vacuum can emit more power than electrons in a solid because the power does not damage their environment \Rightarrow **high flux**.*
- *The **source size** is not that of a single electron but the transverse cross section of the electron beam. The sophisticated trajectory control system makes it **very small**.*
- *Relativity induces **drastically reduced angular divergence** of the emission.*

Spectral brightness is that portion of the brightness lying within a *relative spectral bandwidth* $\Delta\omega/\omega$:

$$\text{Spectral Brightness} = \frac{\text{photons}}{\text{second} \cdot \text{mrad}^2 \cdot \text{mm}^2 \cdot 0.1\% \text{BW}}$$



3rd Generation Light Sources



ESRF - France



DIAMOND - UK



ALBA - Spain

Brightness is the main figure of merit of synchrotron radiation sources and its huge increase, was obtained designing low emittance machines, minimizing the source size and the beam divergence.

Ultimate SR facilities



Lund - Sweden



Sirius - Brazil

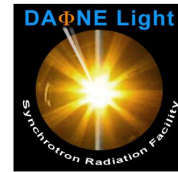


Shanghai -China

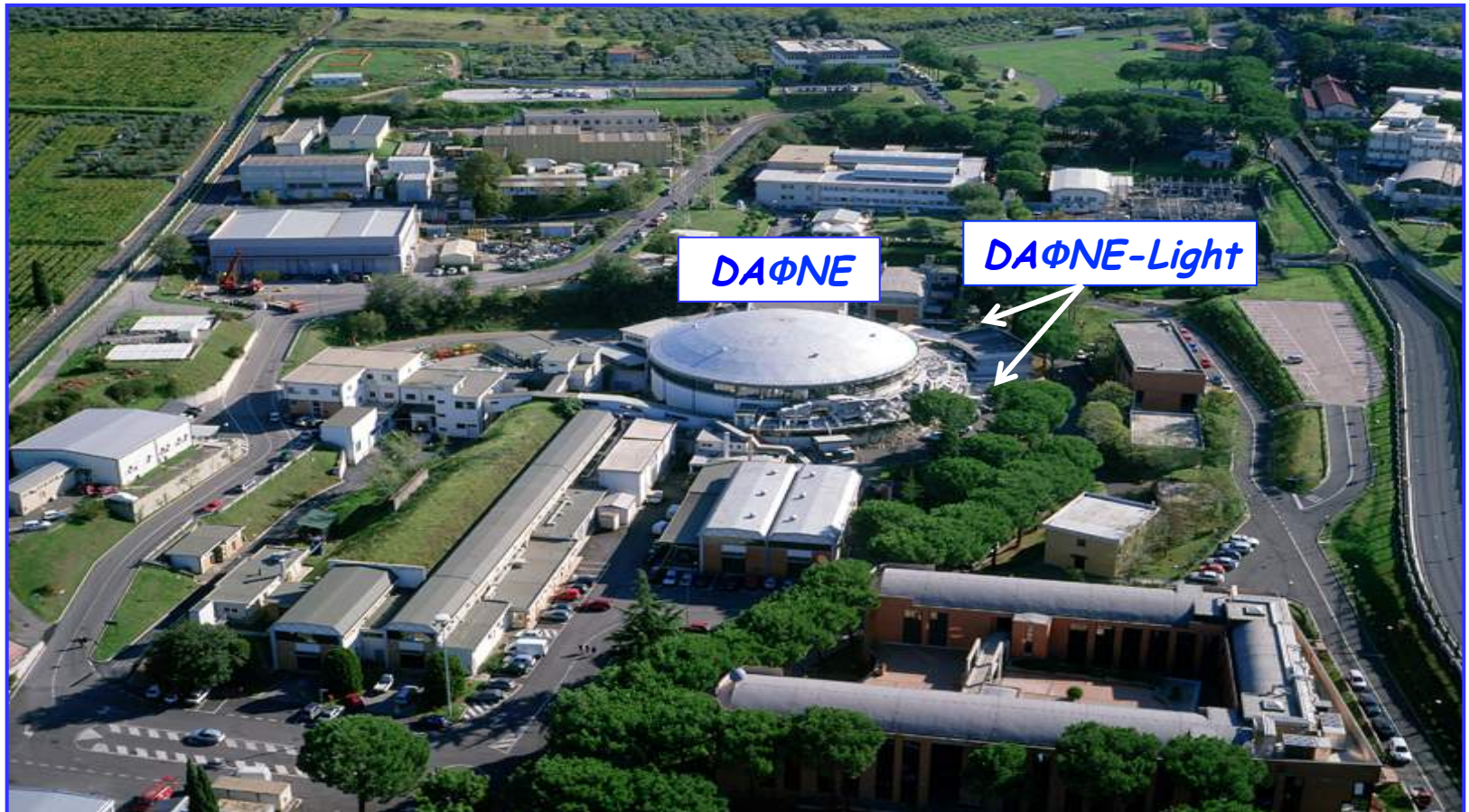
Synchrotron radiation @ INFN-Frascati National Laboratory



DAΦNE-Light



INFN-LNF Synchrotron Radiation Facility



Beamlines @ DAΦNE

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

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

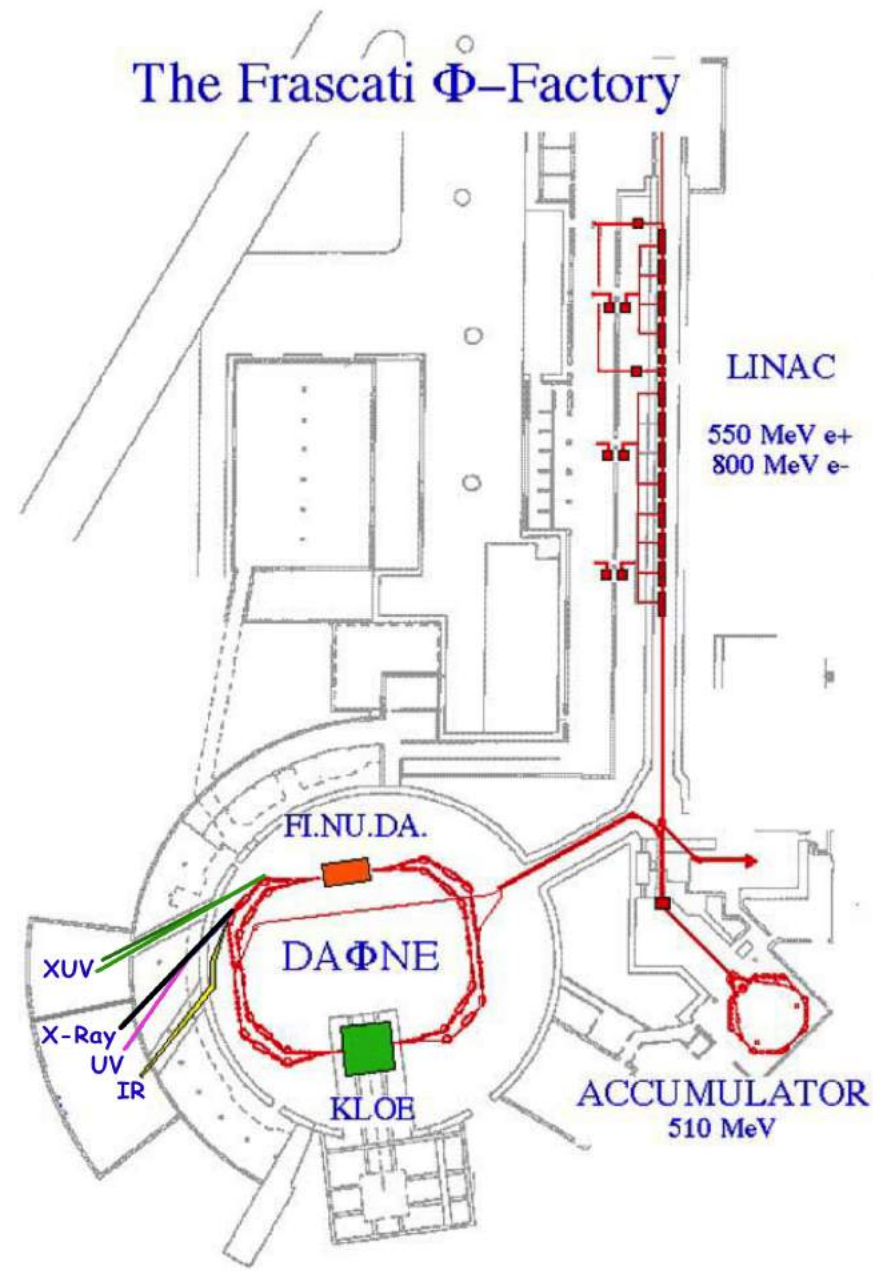
Open to Italian and EU users

3) **DXR2 - UV-VIS beamline** (2-10eV)
new setup.

XUV beamlines

4) **Low Energy Beamline** (35-200 eV)
ready for commissioning;

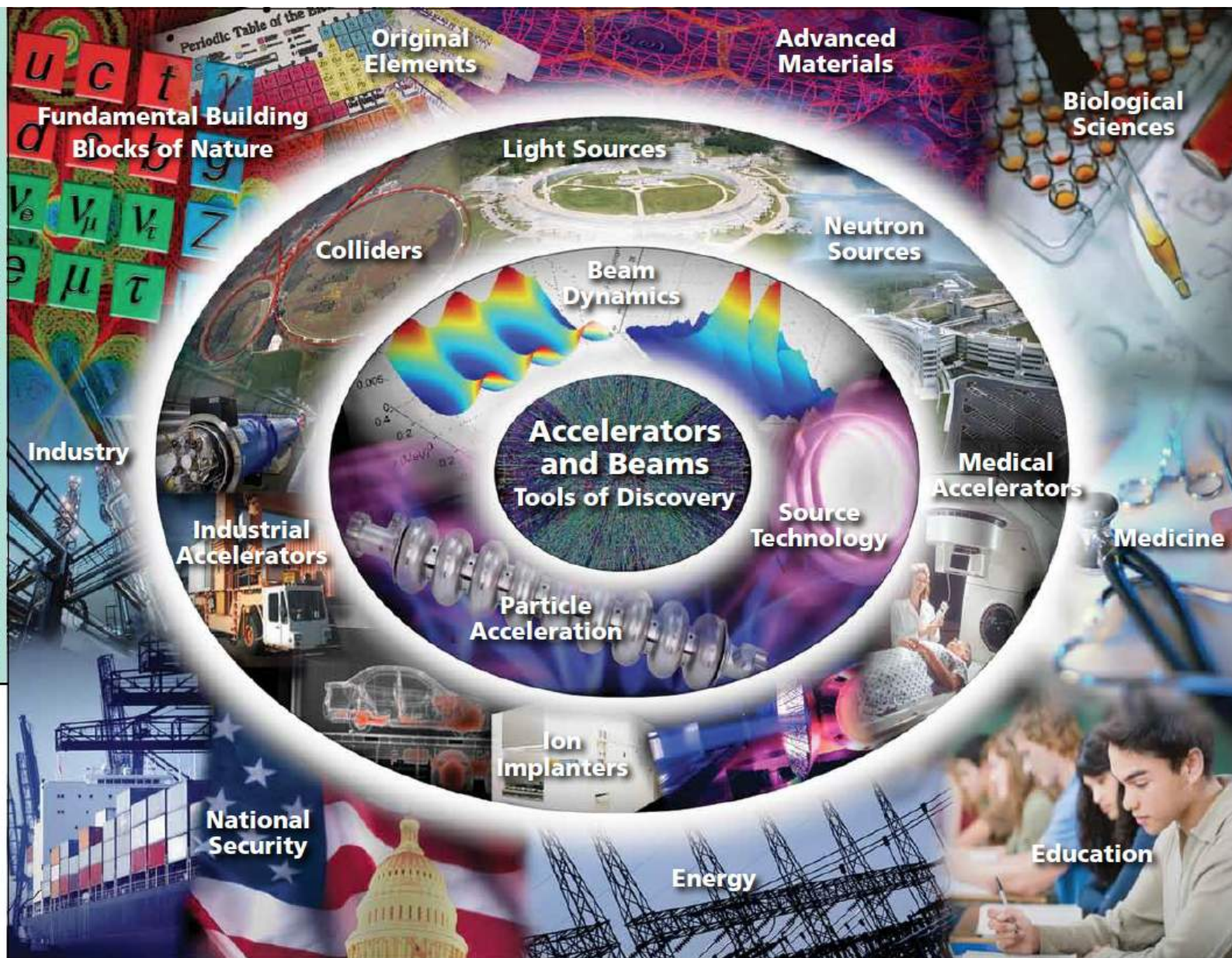
5) **High Energy Beamline** (60-1000eV)
ready for commissioning.



Available techniques

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

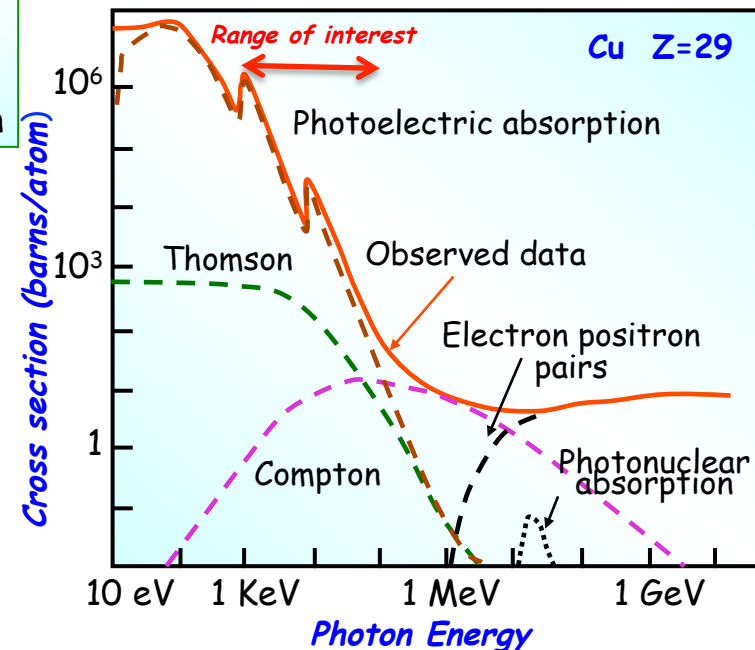
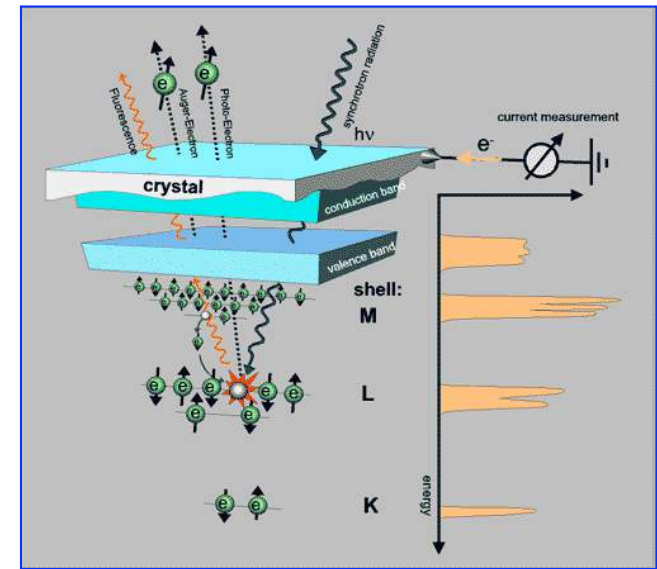
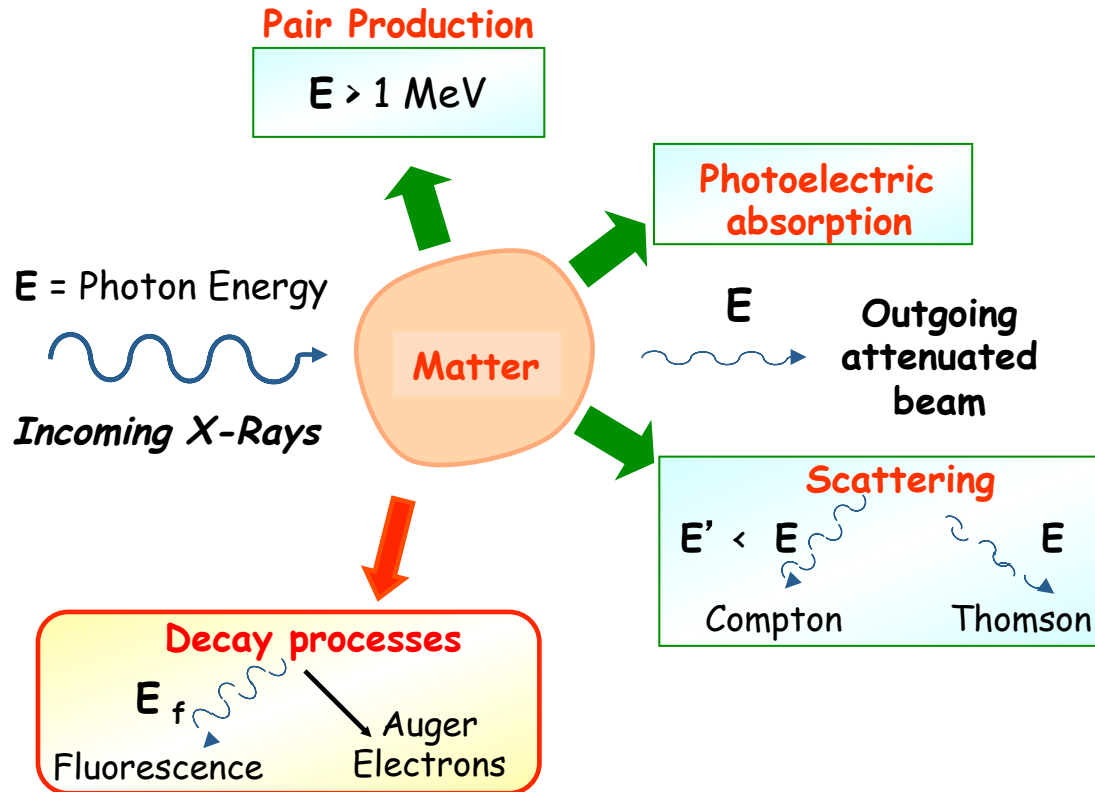
From accelerators to applications



Interactions of x-rays with matter

Interaction of X-rays with matter

Attenuation mechanisms for X-rays



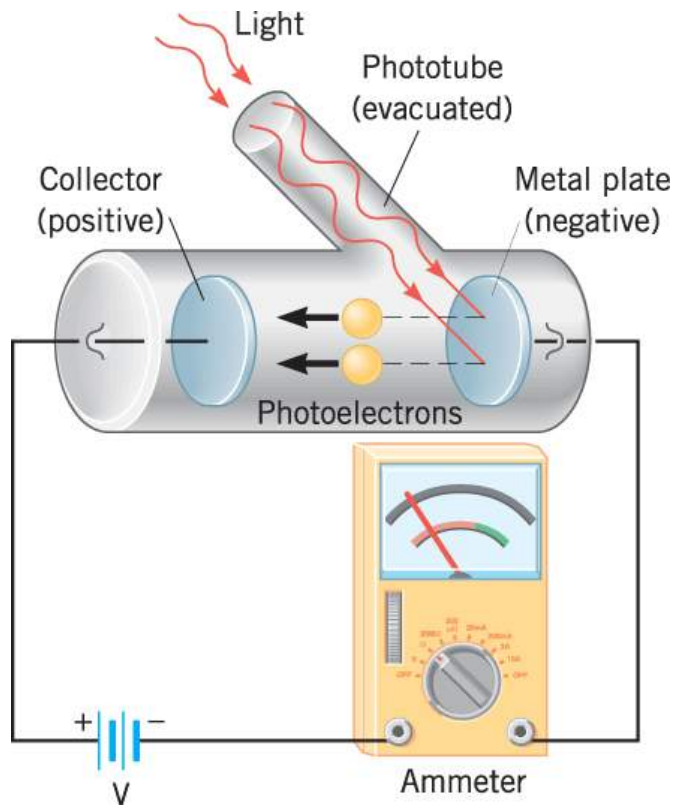
There are different types of interaction of X-rays with matter but taking into account the energy range of interest the one that will be taken into account is **x-ray absorption**.

X-ray Absorption Spectroscopy

*XAS local sensitive and chemical selective probe
that can provide structural, electronic and magnetic information.*

Photoelectric effect

Electromagnetic radiation can be used to push electrons, freeing them from the surface of a solid. This process is called the **photoelectric effect** (or **photoelectric emission** or **photoemission**), a material that can exhibit this phenomena is said to be **photoemissive**, and the ejected electrons are called **photoelectrons**; but there is nothing that would distinguish them from other electrons. All electrons are identical to one another in mass, charge, spin, and magnetic moment.



The photoelectric effect does not occur when the frequency of the incident light is less than the **threshold frequency**. **Different materials have different threshold frequencies.**

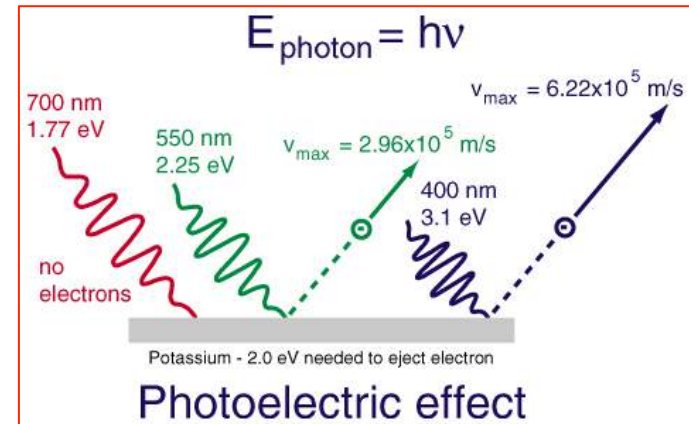
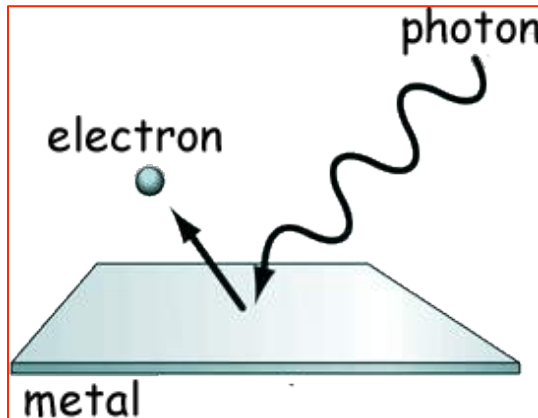
Photoelectric effect

Classical physics cannot explain why...

- no photoelectrons are emitted when the incident light has a frequency below the threshold,
- the maximum kinetic energy of the photoelectrons increases with the frequency of the incident light,
- the maximum kinetic energy of the photoelectrons is independent of the intensity of the incident light, and
- there is essentially no delay between absorption of the radiant energy and the emission of photoelectrons.

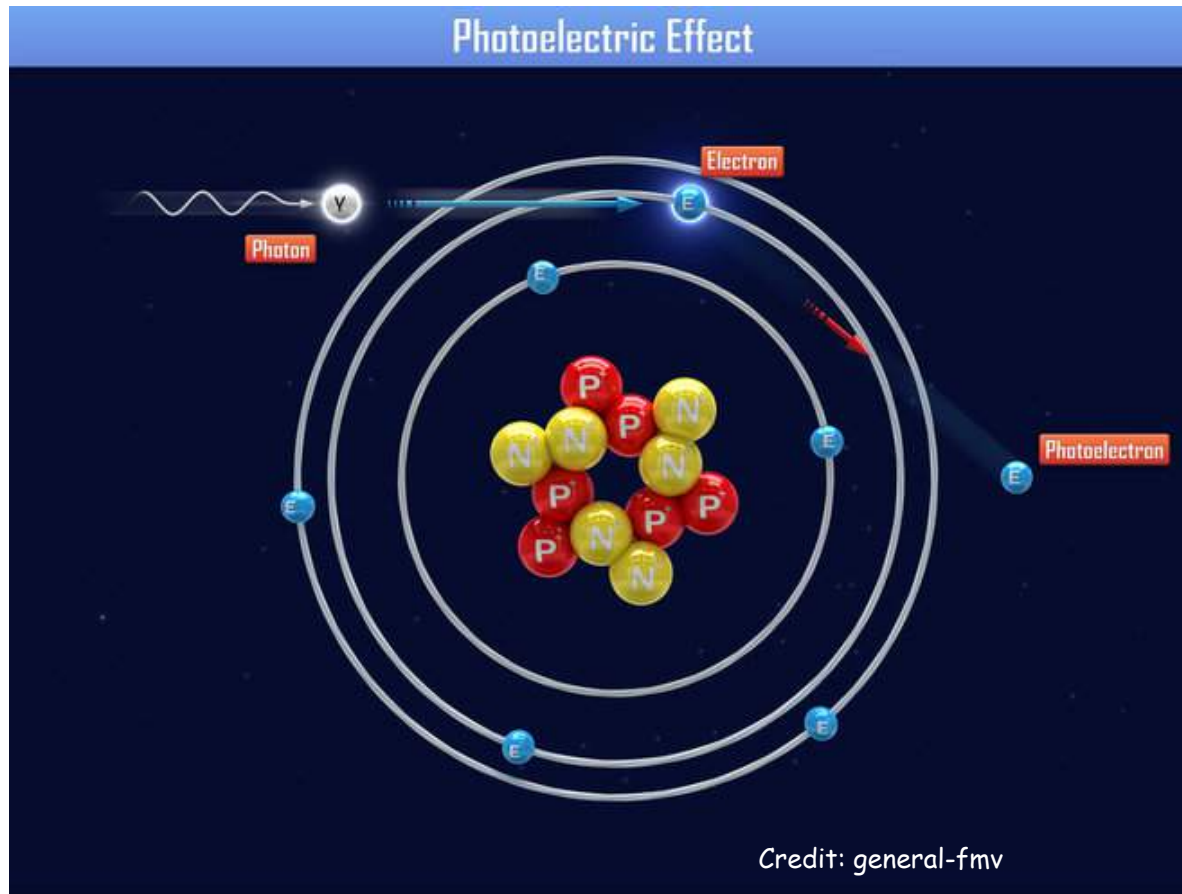
Modern physics states that...

- electromagnetic radiation is composed of discrete entities called photons
- the energy of a photon is proportional to its frequency
- the work function of a material is the energy needed per photon to extract an electron from its surface



In 1905, *Albert Einstein realized that light was behaving as if it was composed of tiny particles* (initially called quanta and later called photons) and that *the energy of each particle was proportional to the frequency of the electromagnetic radiation* (Nobel Prize in Physics in 1921).

Photoelectric effect



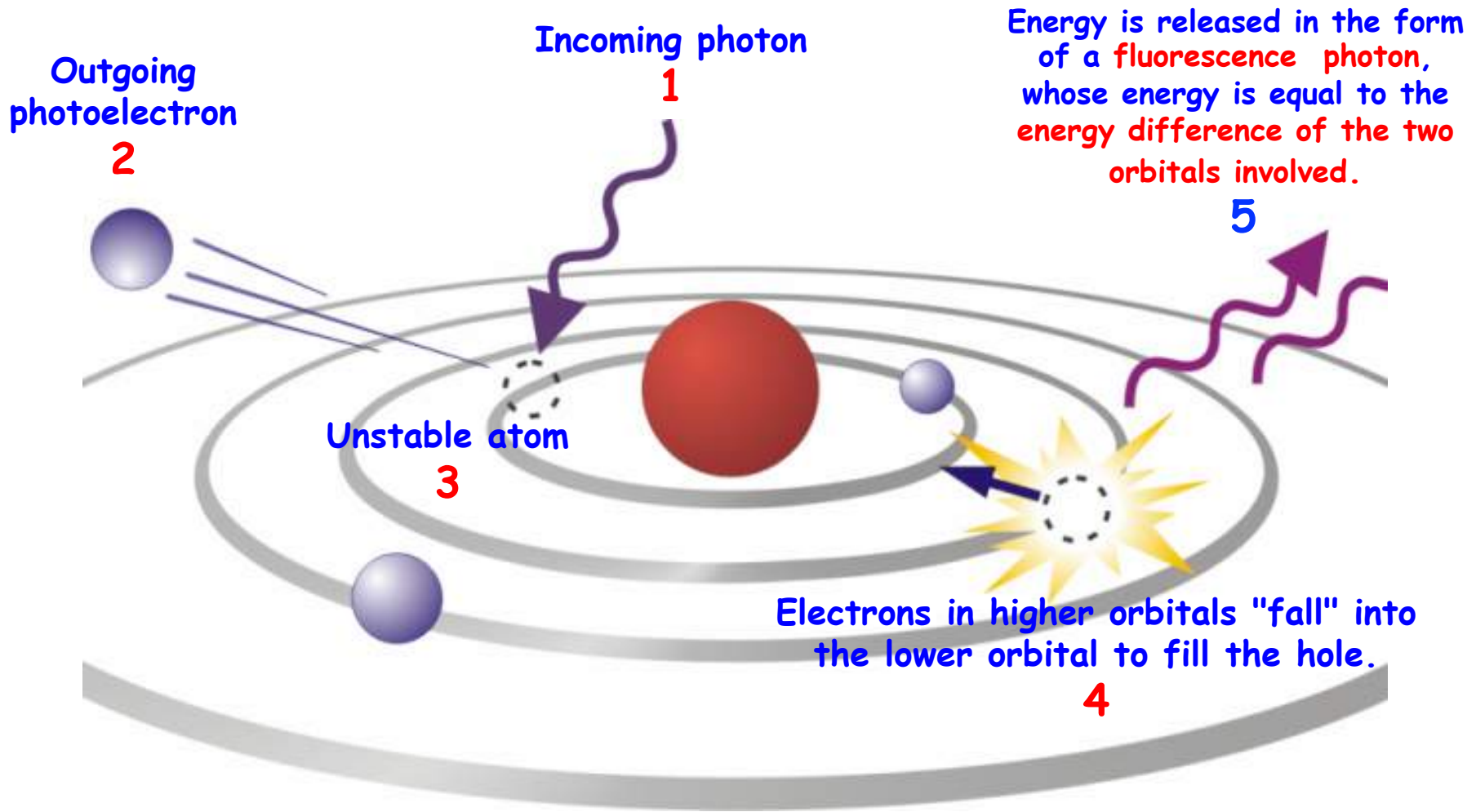
The **photoelectric effect** is the emission of electrons from matter upon the **absorption** of electromagnetic radiation, such as x-rays.

Some X-ray absorption energies

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

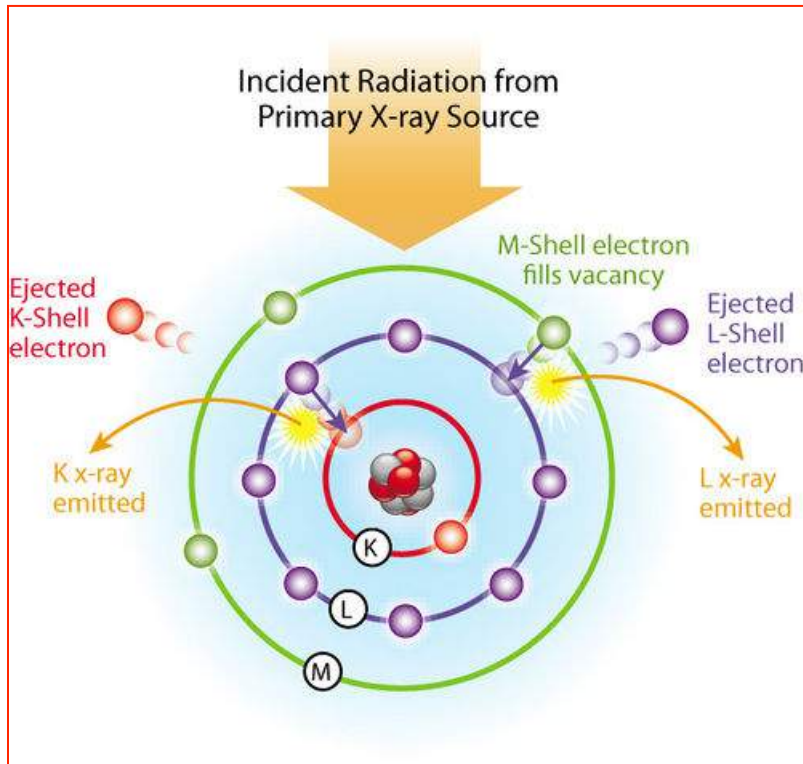
Element	Atomic Number	Atomic Weight	K Edge	L1 Edge	L2 Edge	L3 Edge	M1 Edge	M5 Edge
H	1	1.008	13.6	0.0	0.0	0.0	0.0	0.0
He	2	4.003	24.6	0.0	0.0	0.0	0.0	0.0
Li	3	6.941	54.8	0.0	0.0	0.0	0.0	0.0
Be	4	9.012	111.0	0.0	0.0	0.0	0.0	0.0
B	5	10.811	188.0	0.0	4.7	4.7	0.0	0.0
C	6	12.011	283.8	0.0	6.4	6.4	0.0	0.0
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

Absorption and fluorescence decay



The material emits radiation, which has energy characteristic of the atoms present.

Decay Process: X-ray Fluorescence (XRF)

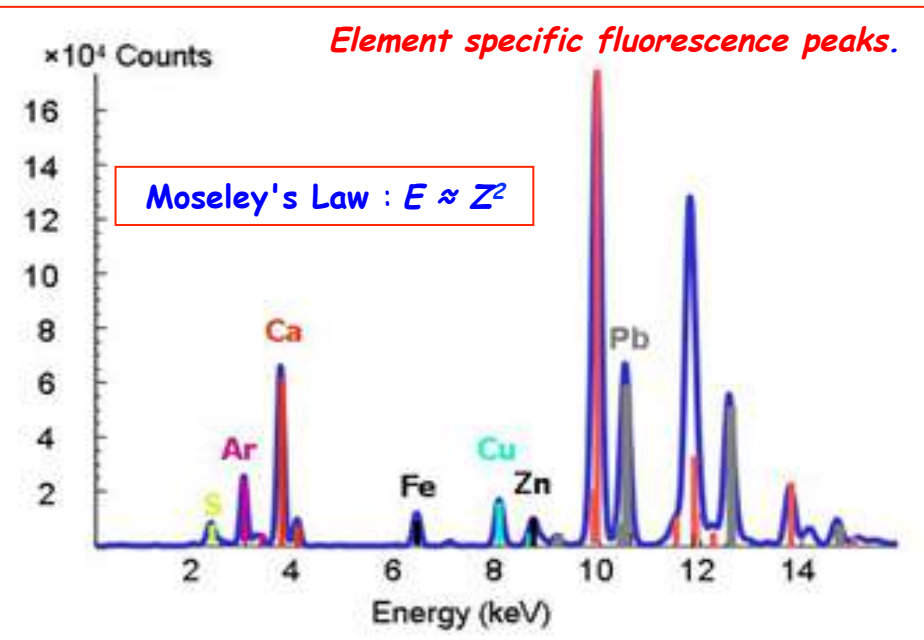


XRF produces a **non-destructive chemical analysis** of any kind of sample.

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.

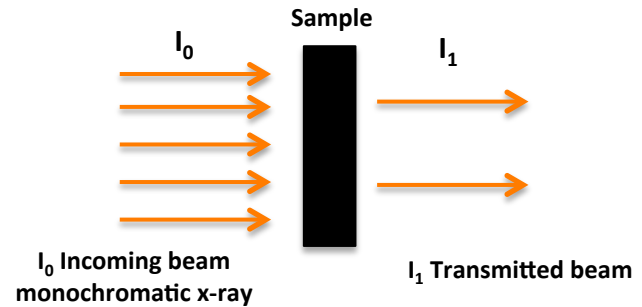
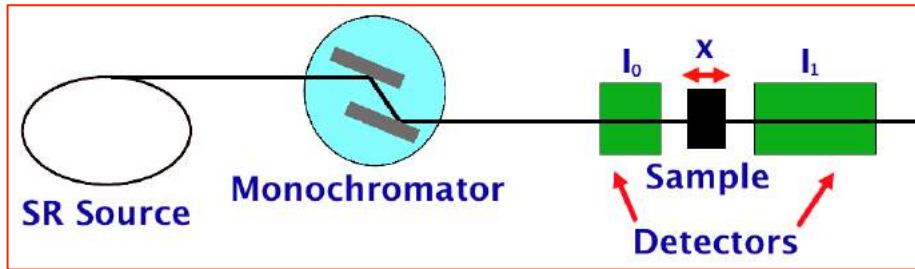


Some X-ray fluorescence energies

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



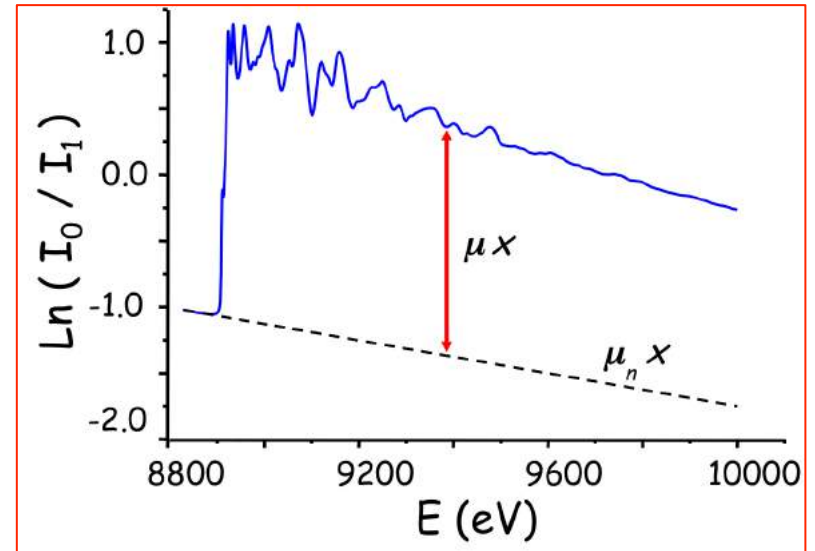
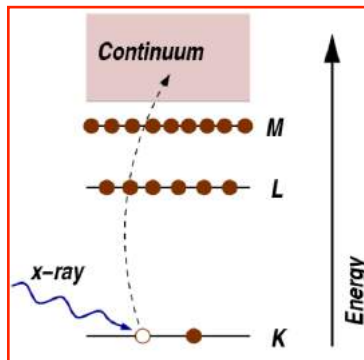
$$I_1 = I_0 \exp[-\mu(E) x]$$

Exponential attenuation or equation of Beer-Lambert

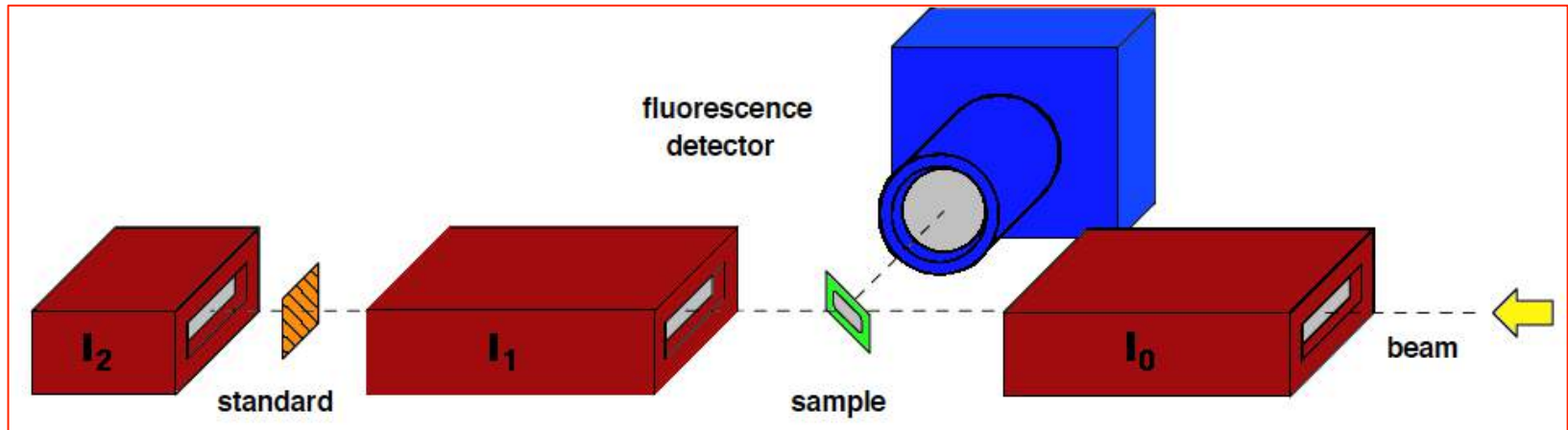
Absorption coefficient

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

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

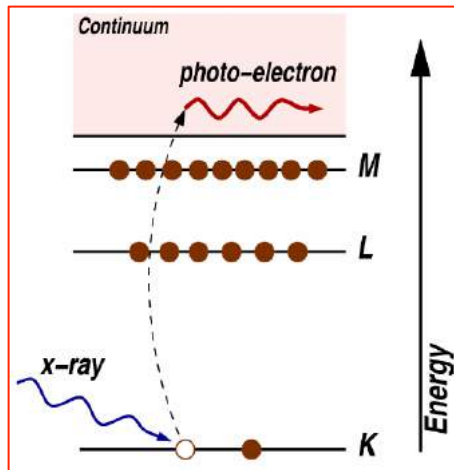


XAFS Spectroscopy modes

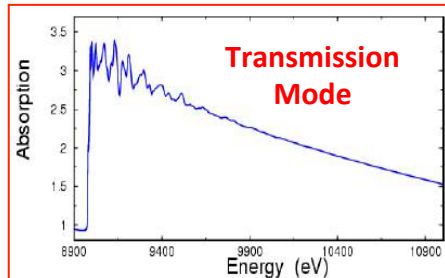


XAFS can be measured either in **Transmission** or **Fluorescence** mode.

X-ray absorption

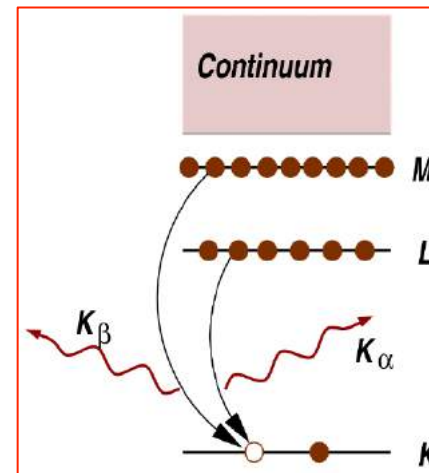


$$\mu(E)x = \ln\left(\frac{I_0}{I_1}\right)$$

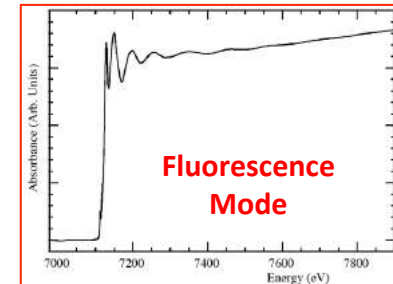


The probability of **Fluorescence** emission is directly proportional to the absorption probability.

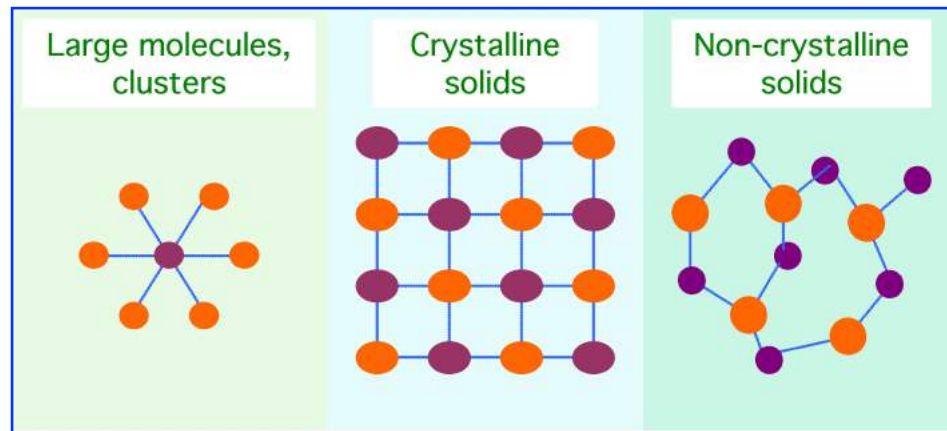
X-ray fluorescence



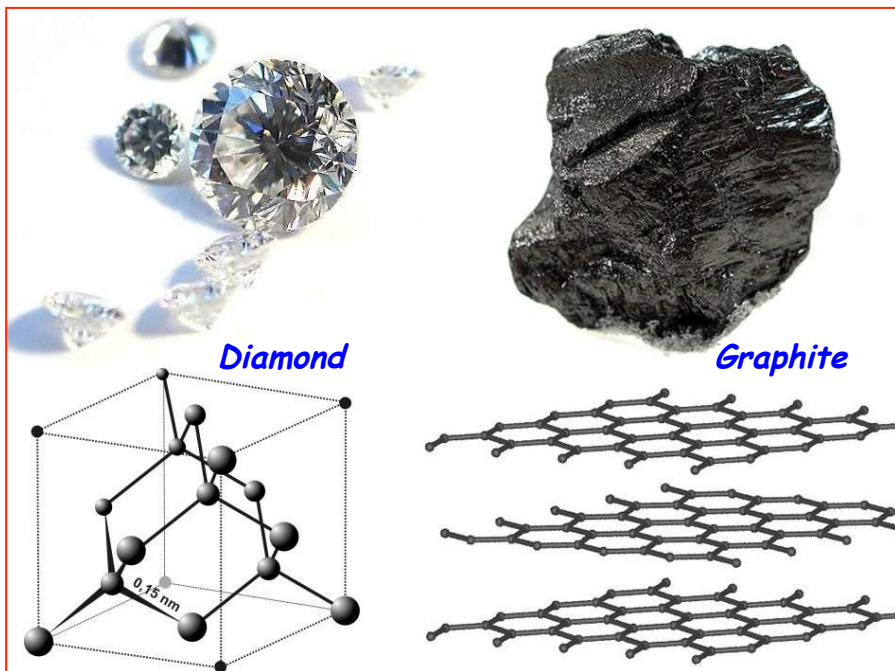
$$\mu(E)x = \left(\frac{I_F}{I_0}\right)$$



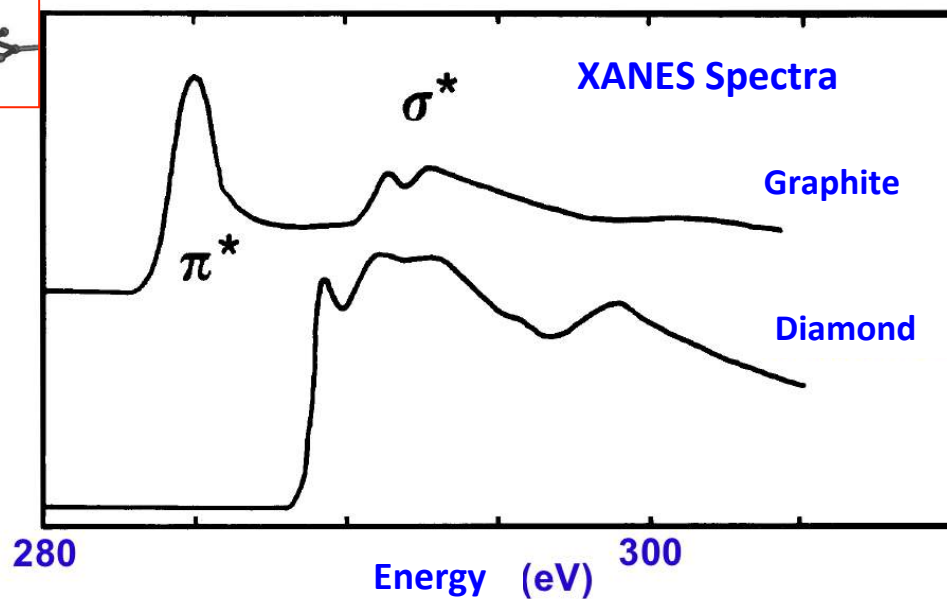
XAFS spectroscopy as a very powerful tool



XAFS - Carbon K edge



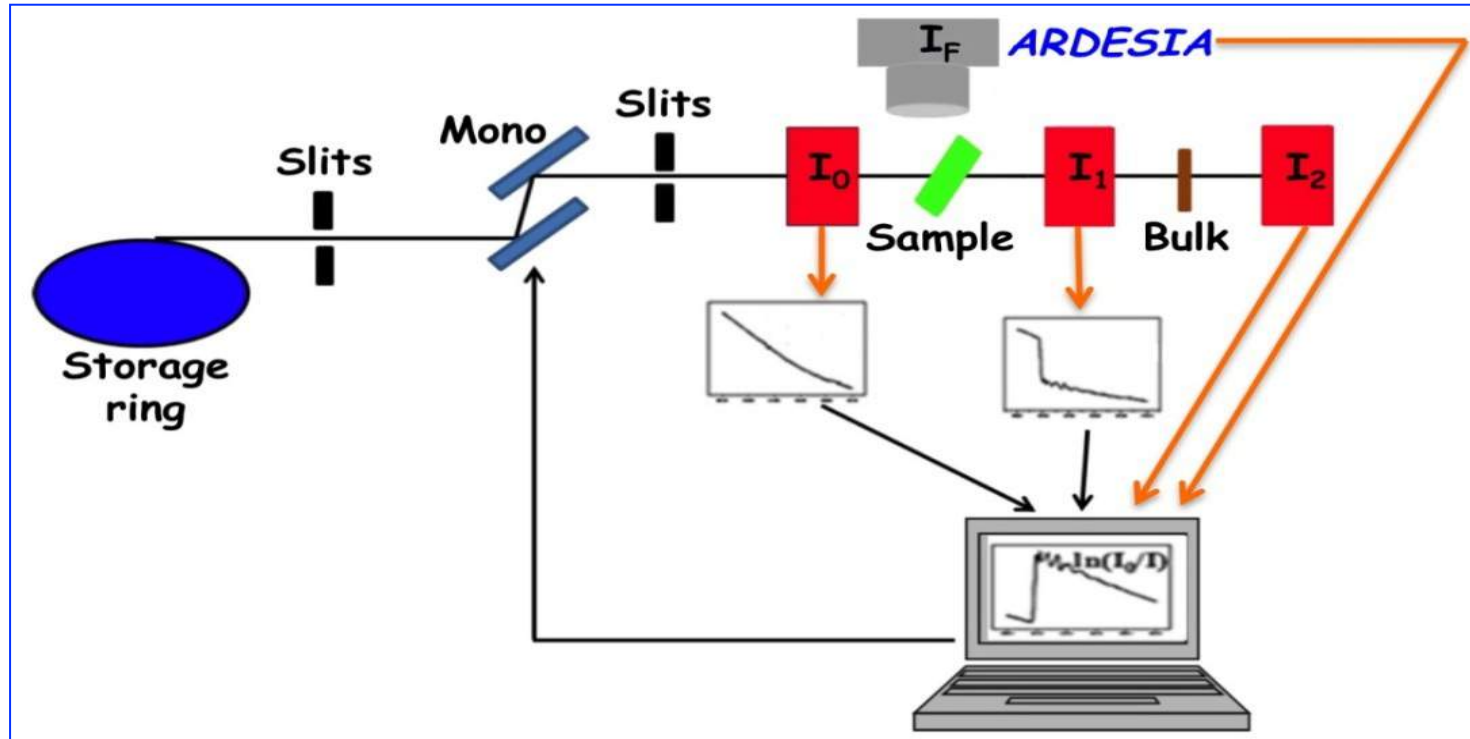
The quite different XANES spectra for Graphite and Diamond due to their different atomic structures.



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

XAFS beamline

DAFNE-L DXR1 beam line absorption spectroscopy



I_0, I_1, I_2 Gas ionization chambers - I_F SDD solid state detector

DAΦNE Ring

First Horizontal slits

Beam Stopper

DAΦNE shielding wall

Second slits

Mirror chamber

Fast Valves

Polymer Window

Precision slits

Double Crystal Monochromator

Resolution slits

Ionization Chamber

Experimental Chamber

Ionization Chamber

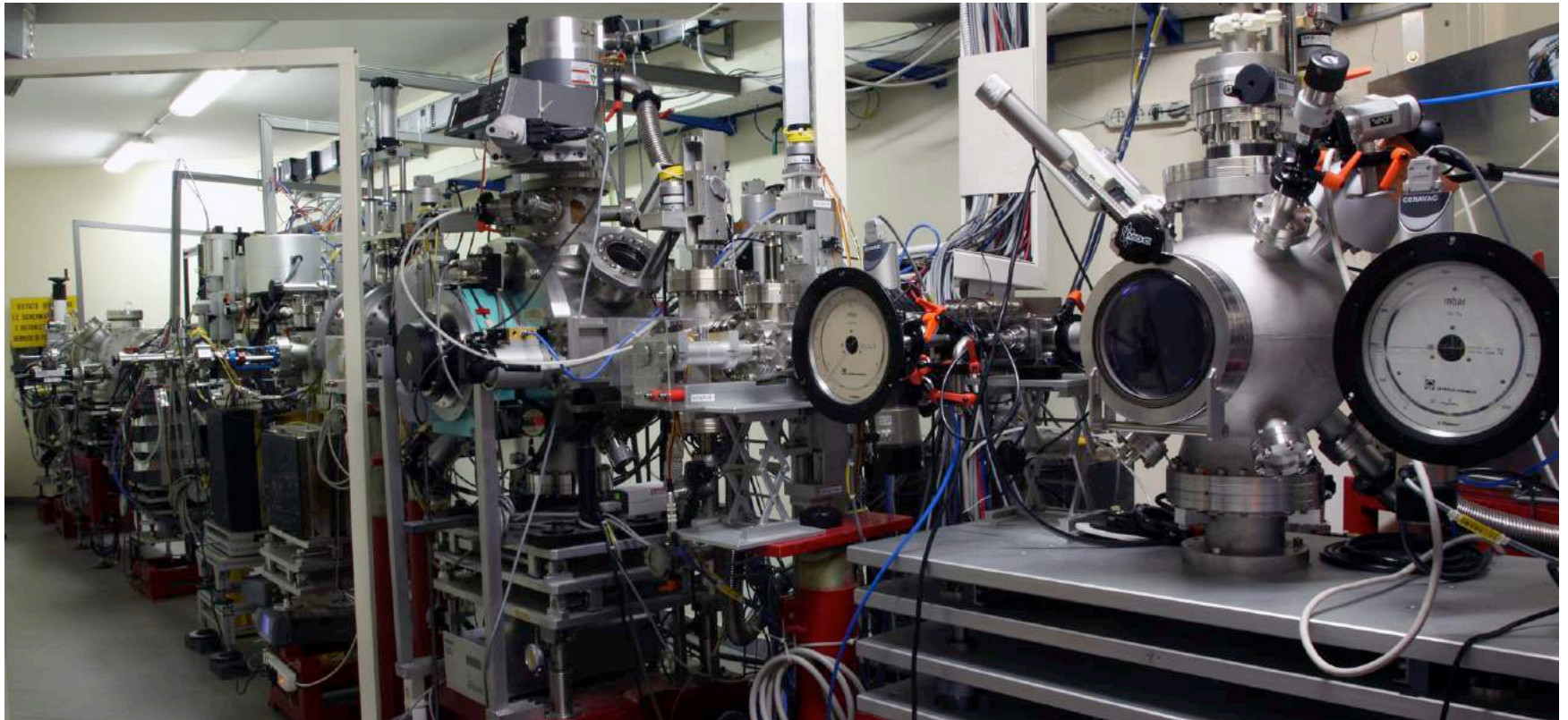
Lead Wall

X-ray
Exp. hutch

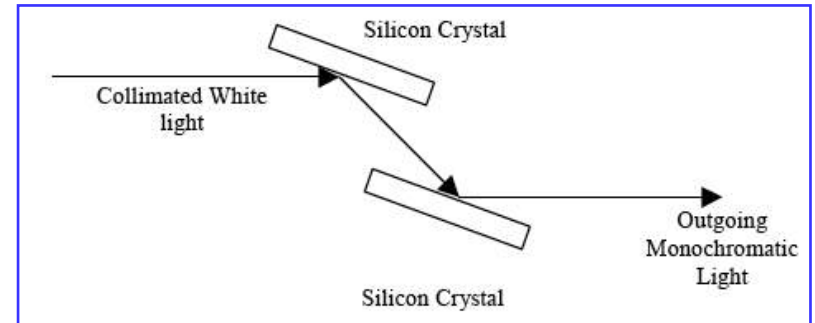
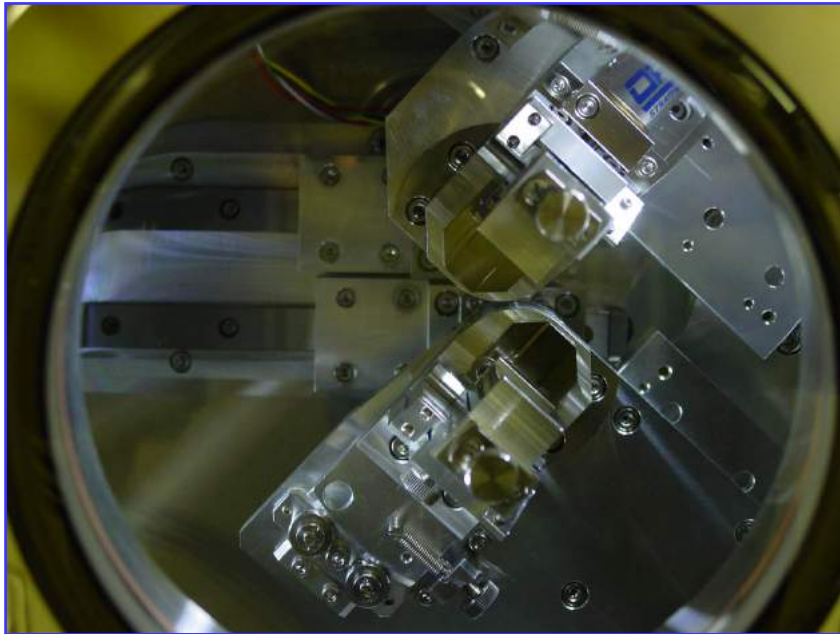
DAΦNE Soft X-ray DXR1 Beamline

- Wiggler 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
- Soft X-ray absorption spectroscopy and tests of Soft x-ray optics and detectors.

DXR1 Soft X-ray Beamline



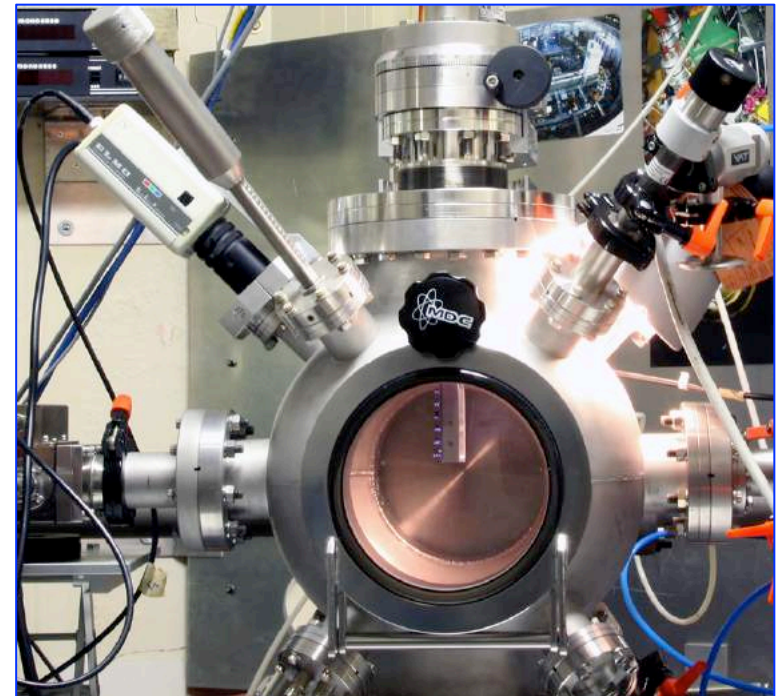
DXR1 Soft X-ray Beamline



$$n\lambda = 2d \sin \theta \quad \text{Bragg's Law}$$

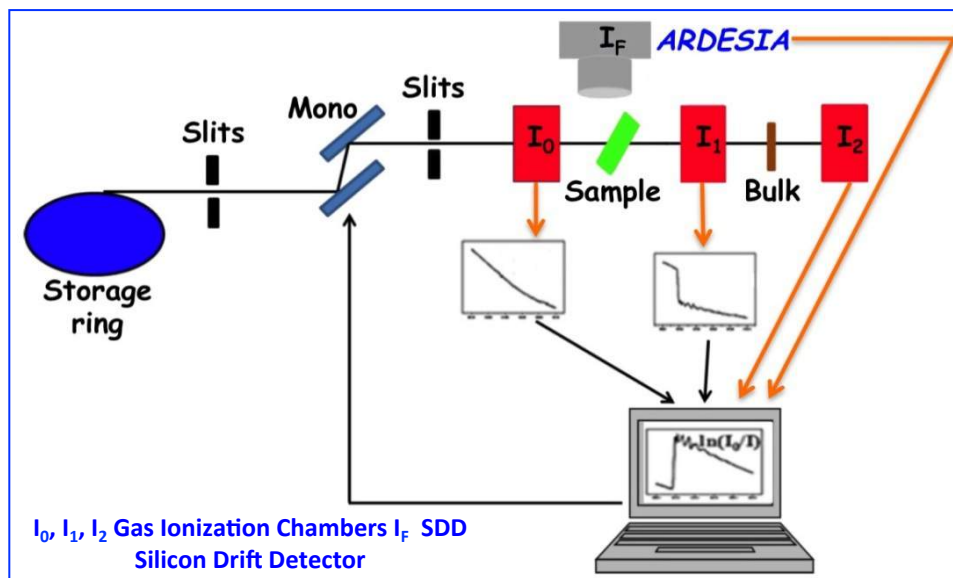
Double crystal monochromator with fixed exit boomerang mechanism.

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

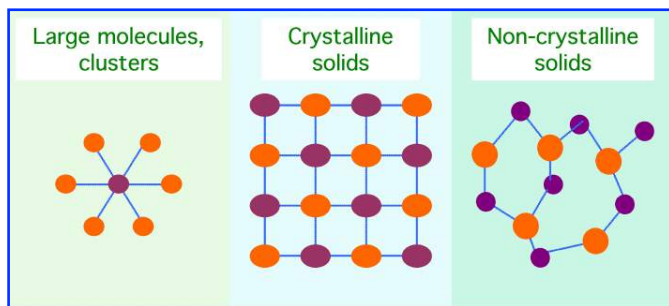
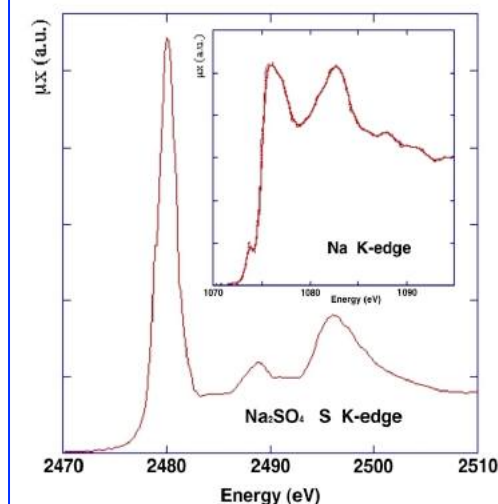
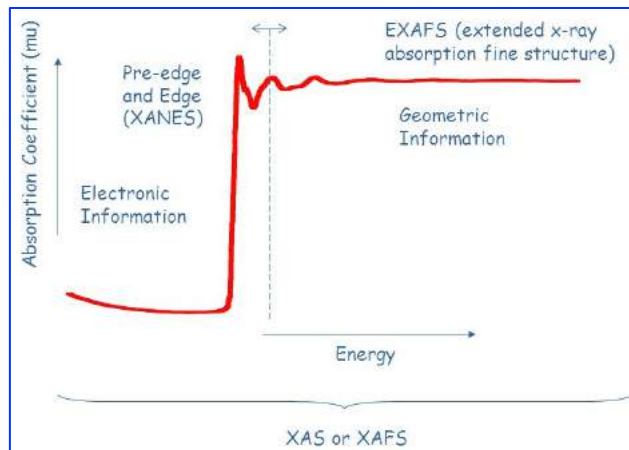
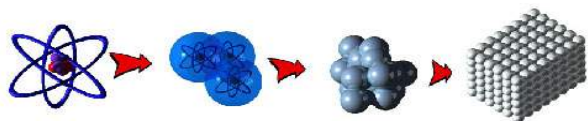
Soft X-ray spectroscopy -XAFS



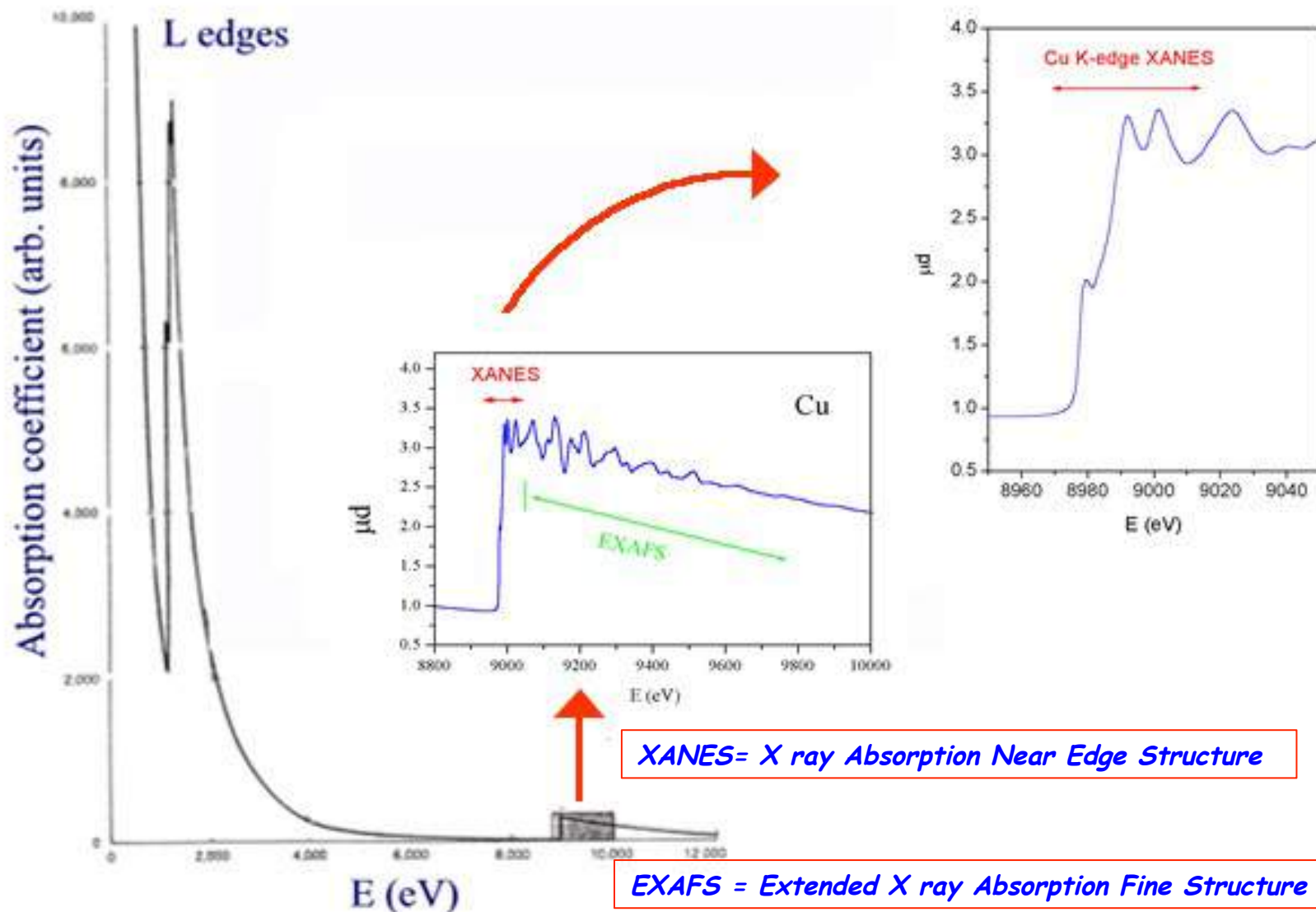
X-ray Absorption Fine Structure or XAFS spectroscopy

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.



XAFS = XANES + EXAFS

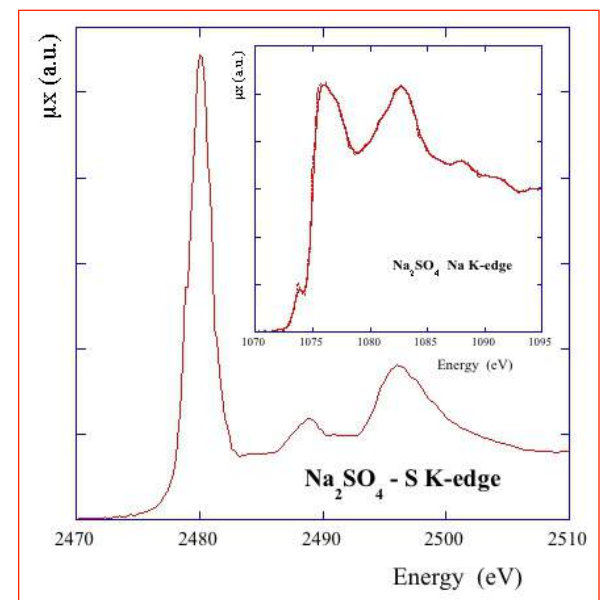
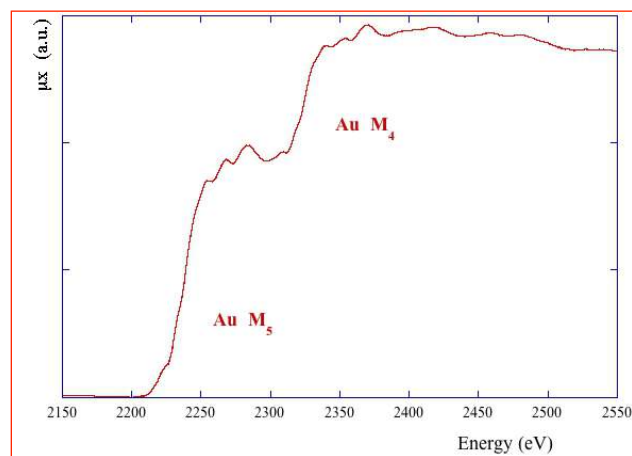
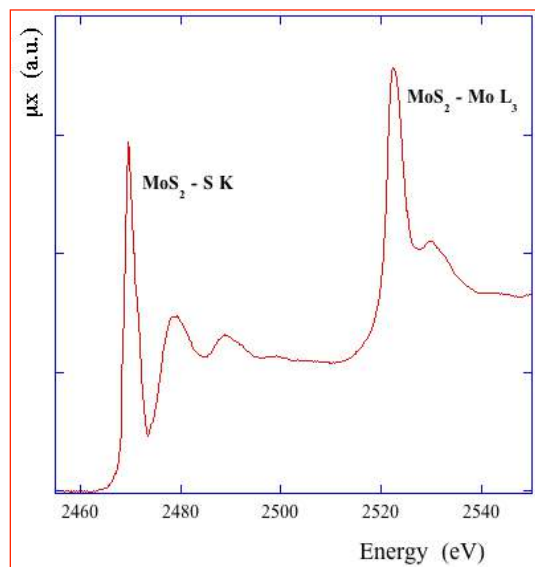
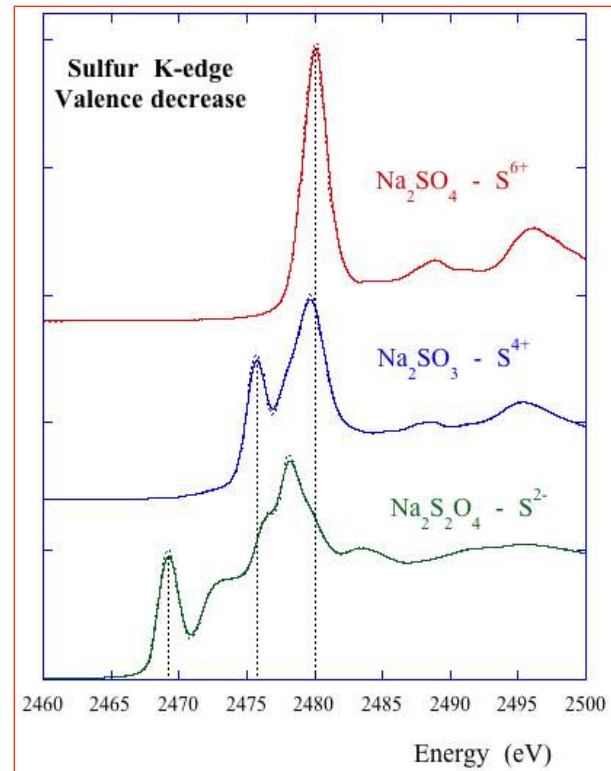
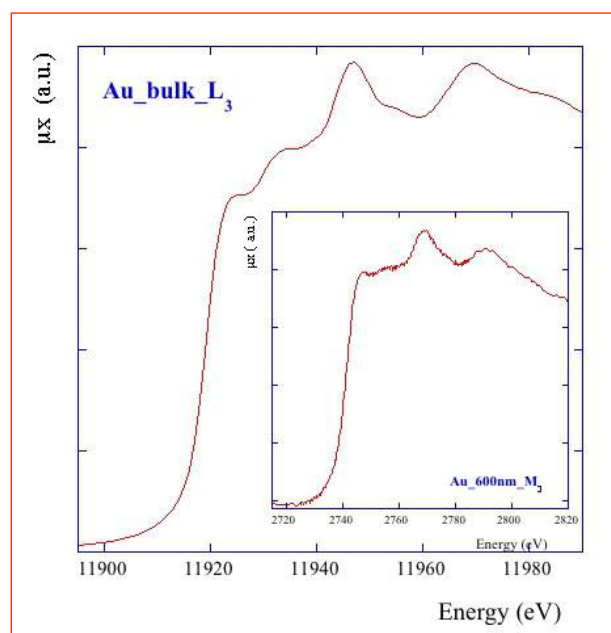
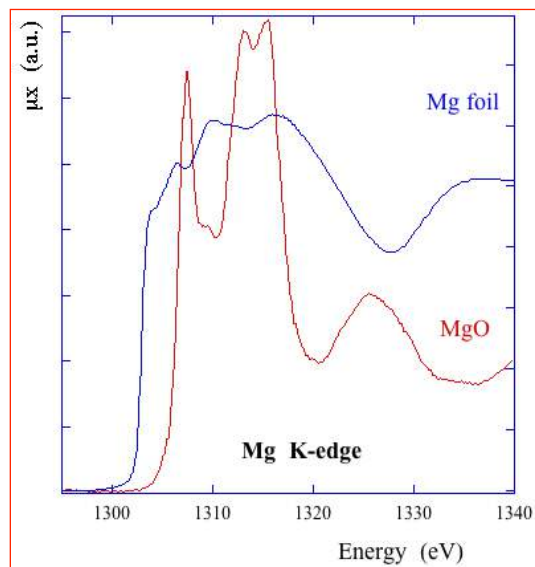


Elements that can be investigated

1 H 1.00794												5 B 10.811	6 C 12.0107	7 N 14.00674	8 O 15.9994	9 F 18.9984032	10 Ne 20.1797
3 Li 6.941	4 Be 9.012182											13 Al 26.981538	14 Si 28.0855	15 P 30.973761	16 S 32.066	17 Cl 35.4527	18 Ar 39.948
11 Na 22.989770	12 Mg 24.3050											31 Ga 69.723	32 Ge 72.61	33 As 74.92160	34 Se 78.96	35 Br 79.904	36 Kr 83.80
19 K 39.0983	20 Ca 40.078	21 Sc 44.955910	22 Ti 47.867	23 V 50.9415	24 Cr 51.9961	25 Mn 54.938049	26 Fe 55.845	27 Co 58.933200	28 Ni 58.6934	29 Cu 63.546	30 Zn 65.39	49 In 114.818	50 Sn 118.710	51 Sb 121.760	52 Te 127.60	53 I 126.90447	54 Xe 131.29
37 Rb 85.4678	38 Sr 87.62	39 Y 88.90585	40 Zr 91.224	41 Nb 92.90638	42 Mo 95.94	43 Tc (98)	44 Ru 101.07	45 Rh 102.90550	46 Pd 106.42	47 Ag 107.8682	48 Cd 112.411	81 Tl 204.383	82 Pb 207.2	83 Bi 208.98038	84 Po (209)	85 At (210)	86 Rn (222)
55 Cs 132.90545	56 Ba 137.327	57 La 138.9055	72 Hf 178.49	73 Ta 180.9479	74 W 183.84	75 Re 186.207	76 Os 190.23	77 Ir 192.217	78 Pt 195.078	79 Au 196.96655	80 Hg 200.59	82 Pb 207.2	83 Bi 208.98038	84 Po (209)	85 At (210)	86 Rn (222)	
87 Fr (223)	88 Ra (226)	89 Ac (227)	104 Rf (261)	105 Db (262)	106 Sg (263)	107 Bh (262)	108 Hs (265)	109 Mt (266)	110 (269)	111 (272)	112 (277)	114 (289) (287)		116 (289)			118 (293)

58 Ce 140.116	59 Pr 140.90765	60 Nd 144.24	61 Pm (145)	62 Sm 150.36	63 Eu 151.964	64 Gd 157.25	65 Tb 158.92534	66 Dy 162.50	67 Ho 164.93032	68 Er 167.26	69 Tm 168.93421	70 Yb 173.04	71 Lu 174.967
90 Th 232.0381	91 Pa 231.03588	92 U 238.0289	93 Np (237)	94 Pu (244)	95 Am (243)	96 Cm (247)	97 Bk (247)	98 Cf (251)	99 Es (252)	100 Fm (257)	101 Md (258)	102 No (259)	103 Lr (262)

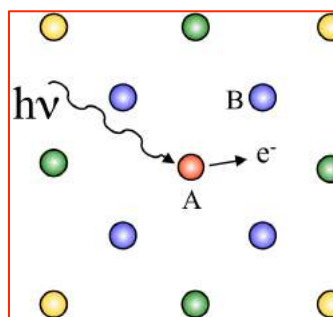
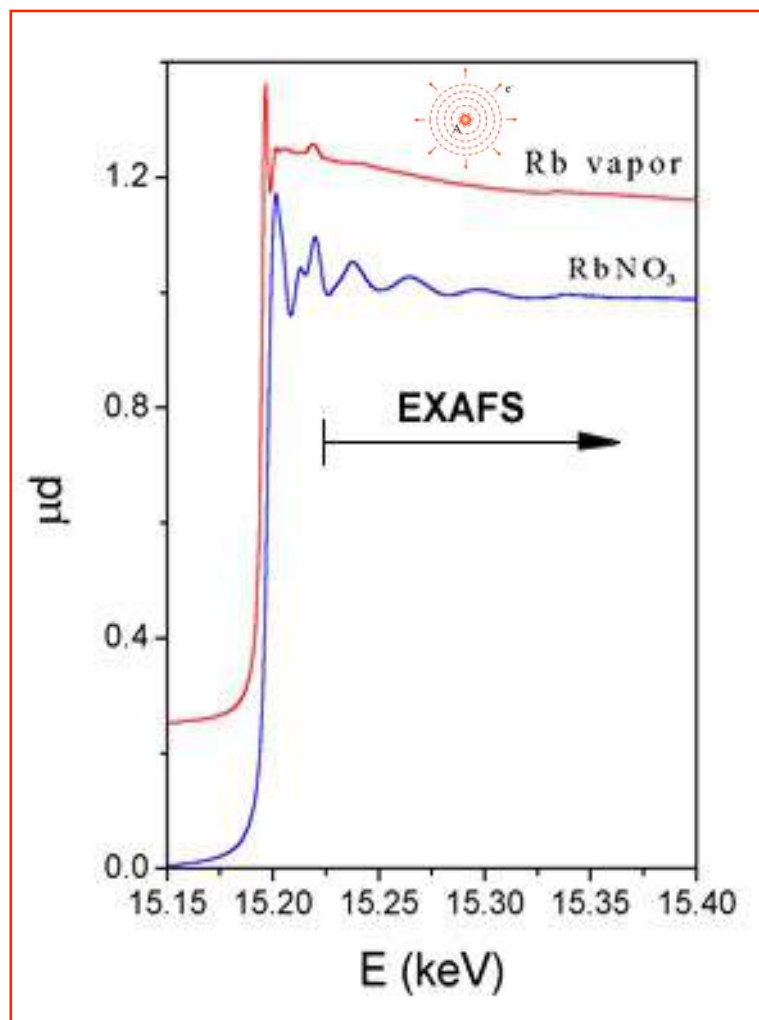
XANES examples



EXAFS

Extended X ray Absorption Fine Structure

EXAFS phenomenological interpretation



X-ray absorption

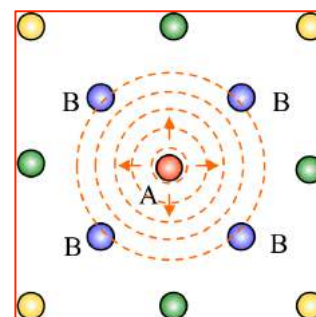
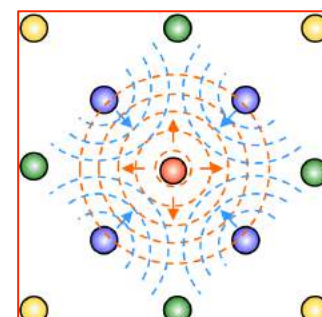
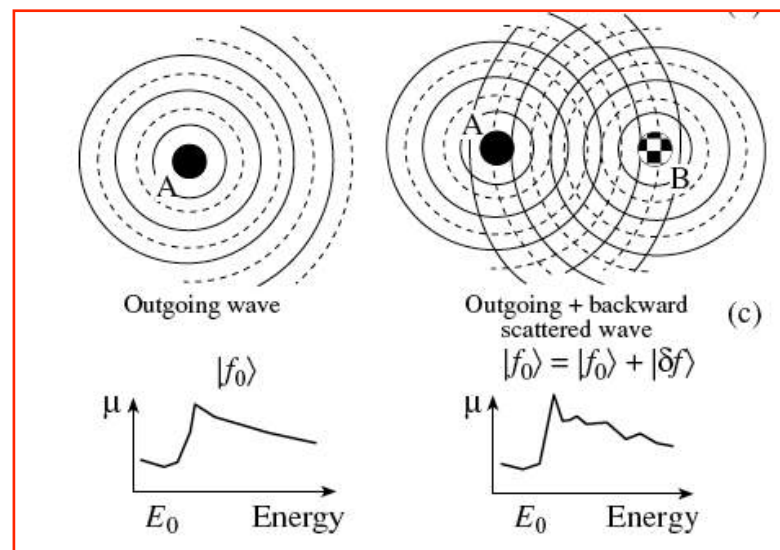


Photo-electron emission

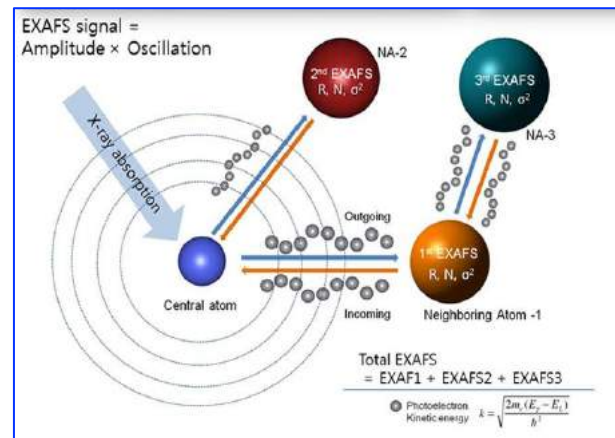
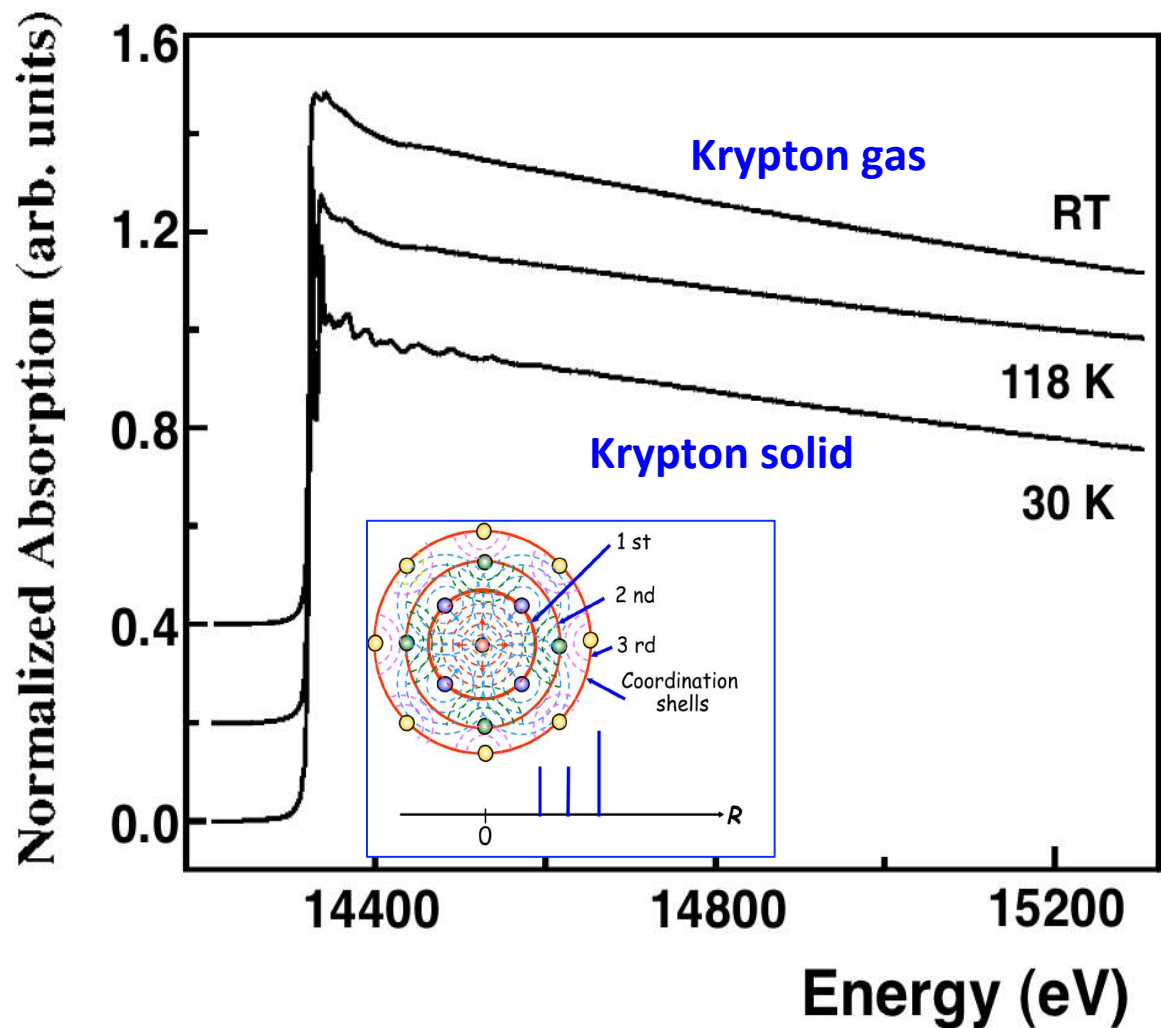


Interference



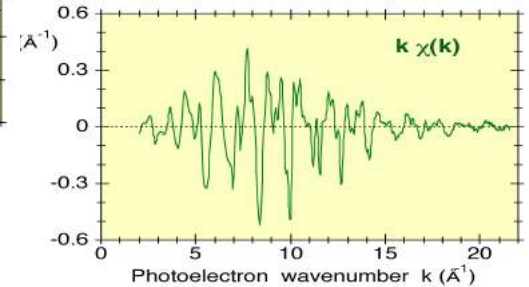
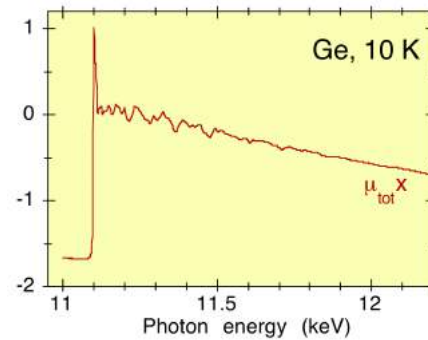
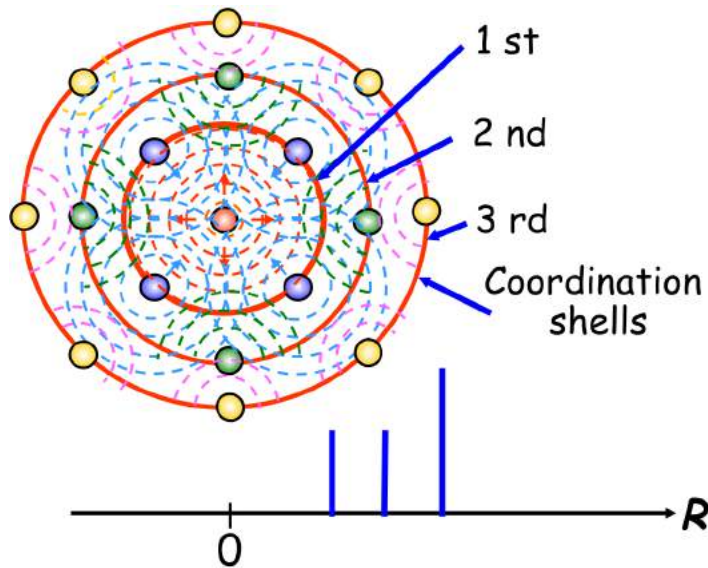
Auto -interference phenomenon of the outgoing photoelectron with its parts that are backscattered by the neighbouring atoms

X-ray Absorption



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

EXAFS formula



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

Coordination
number

Debye Waller
factor

Interatomic distance

$$k = \sqrt{\frac{2m}{\hbar^2} (E - E_0)}$$

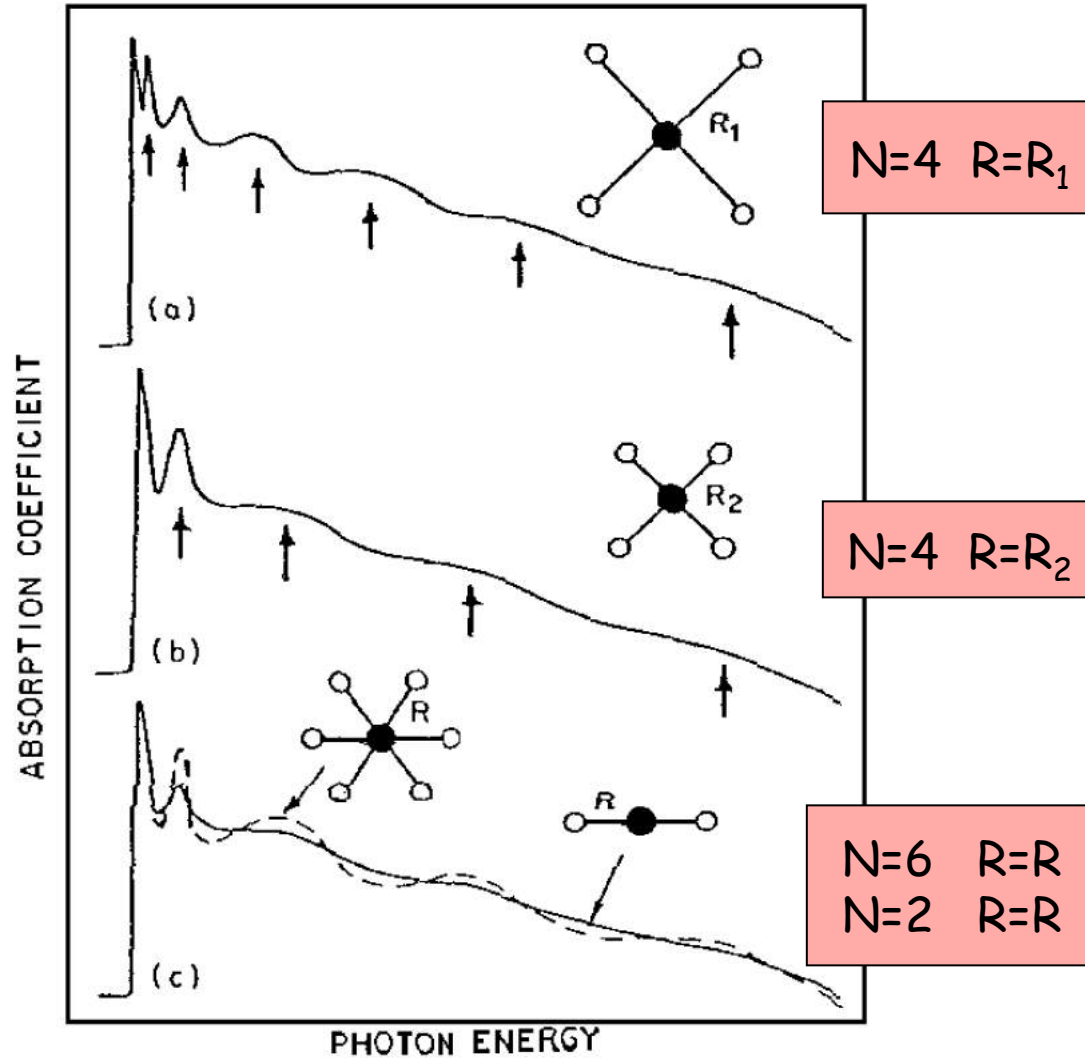
k = wavenumber

Thermal disorder: e^{-k^2/σ^2}

Electron mean free path: λ

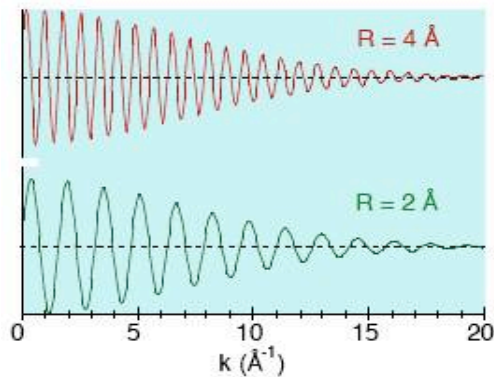
Inelastic scattering effect: S_0^2

EXAFS



EXAFS and structural information

$$\chi(k) = -\frac{S_0^2}{k} \sum_s \underbrace{N_s}_{\text{Coordination number}} \frac{|f_s(\pi, k)|}{R_s^2} e^{-k^2/\underbrace{\sigma_s^2}_{\text{Debye Waller factor}}} e^{-2R_s/\lambda_s} \sin\left(2k\underbrace{R_s}_{\text{Interatomic distance}} + \phi_s(k)\right)$$

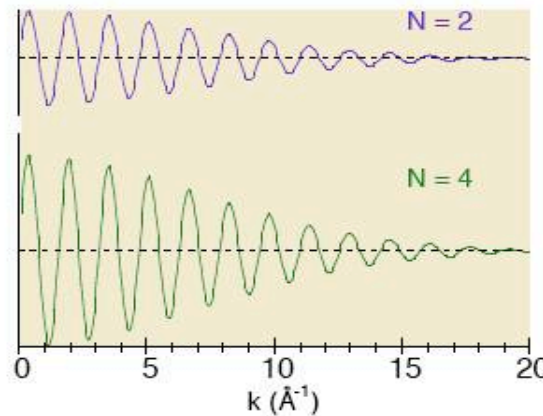


Frequency



Inter-atomic
distance

R

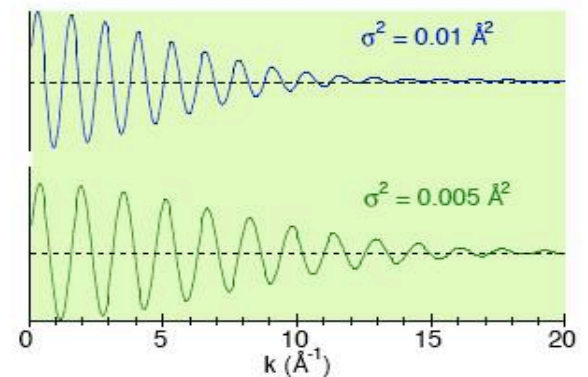


Amplitude



Coordination
number

N



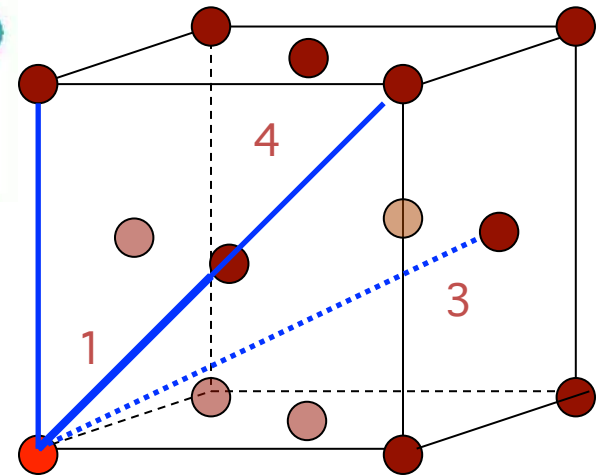
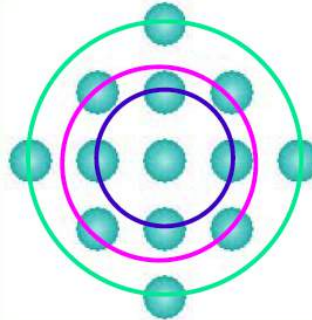
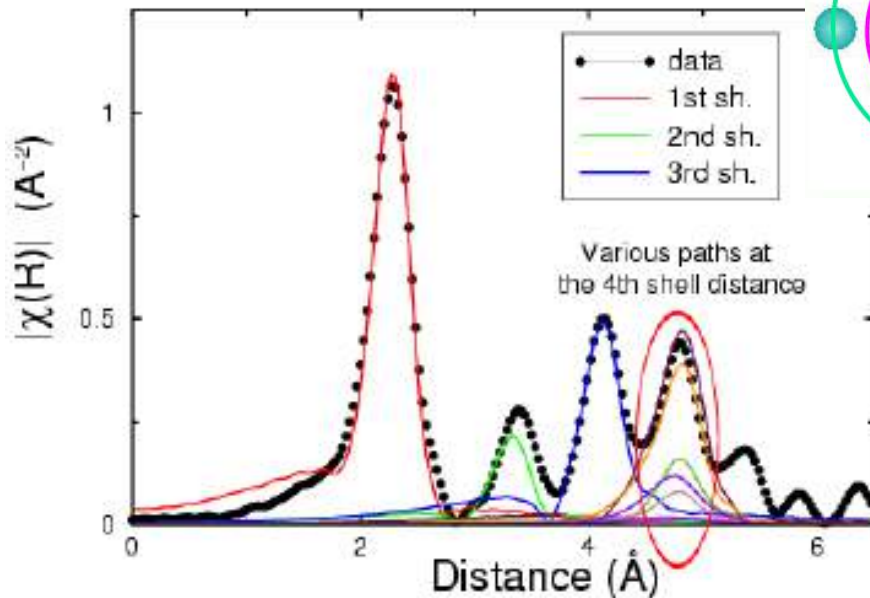
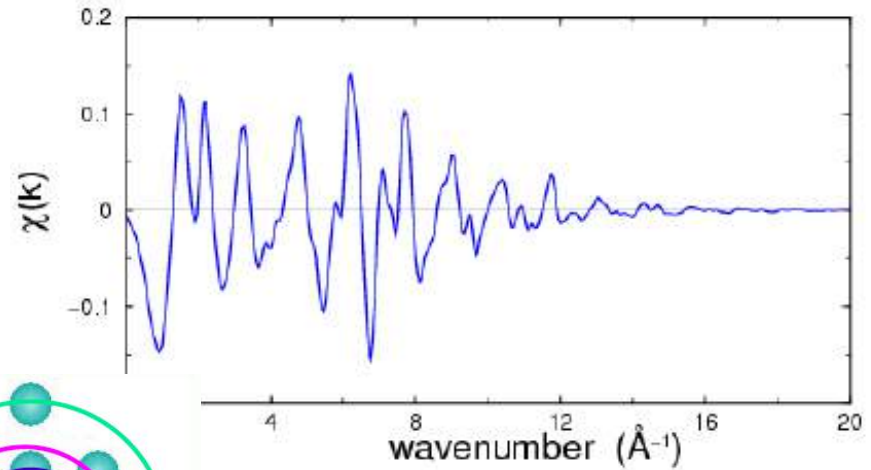
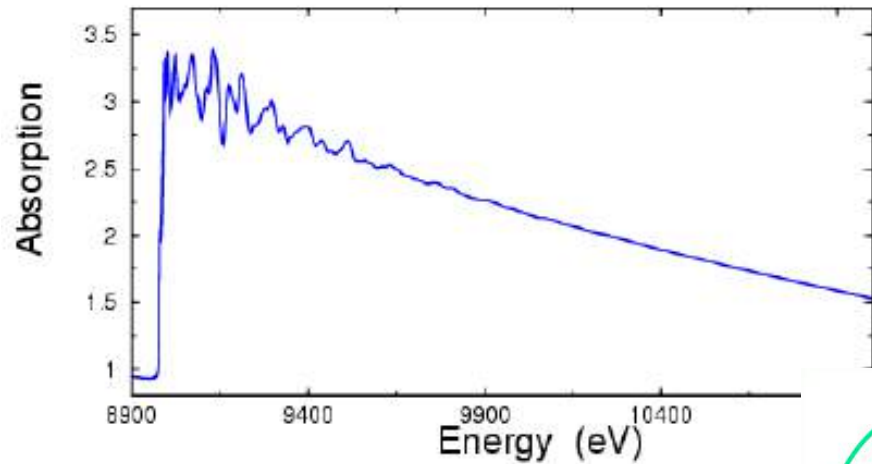
Damping



Disorder

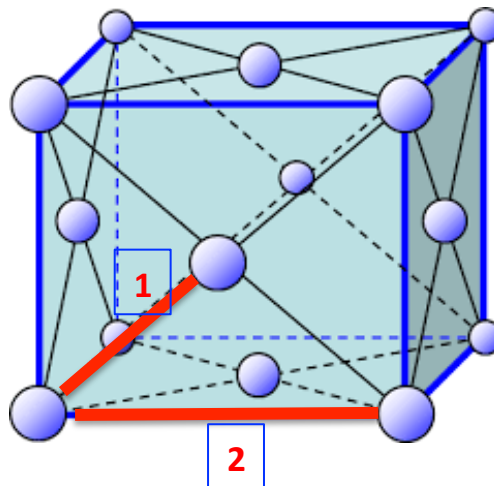
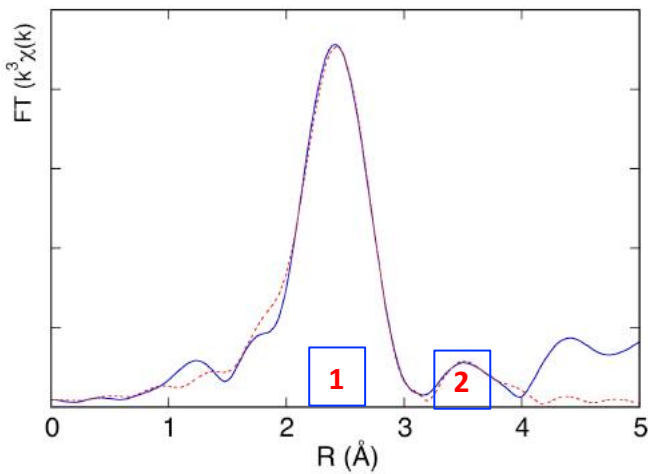
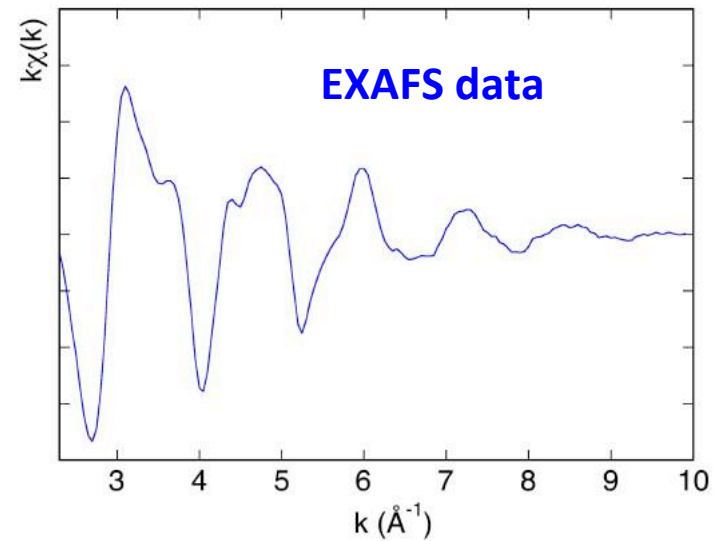
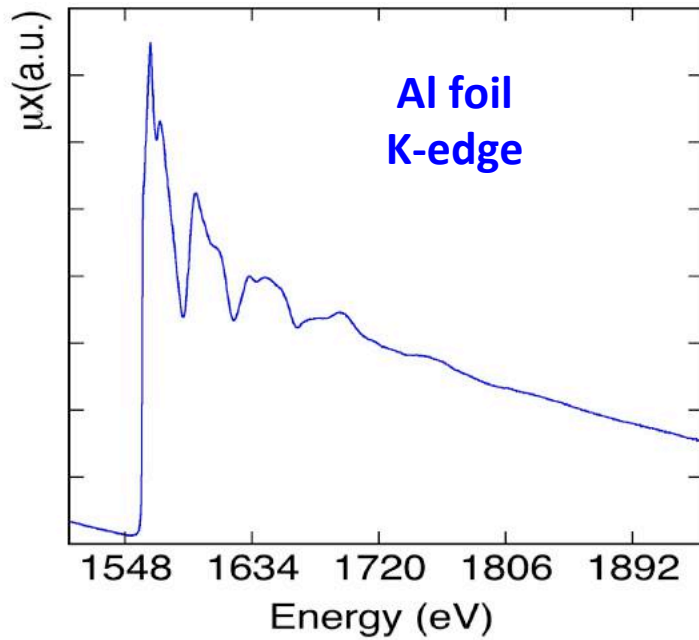
σ^2

EXAFS data analysis



Metallic Copper – FCC or Face Centered Cubic structure.

EXAFS DXR1



$$N_{1\text{Al}} = 12$$

$$R_{1\text{Al}} = 2.85 \text{ \AA}$$

$$\sigma_{1\text{Al}}^2 = 0.014 \text{ \AA}^2$$

First
shell

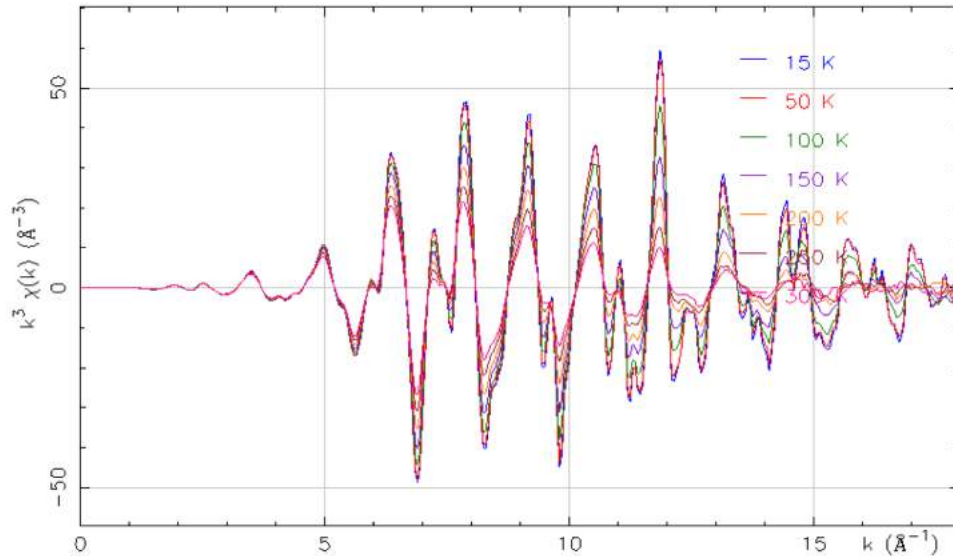
$$N_{2\text{Al}} = 6$$

$$R_{2\text{Al}} = 4.04 \text{ \AA}$$

$$\sigma_{2\text{Al}}^2 = 0.027 \text{ \AA}^2$$

Second
shell

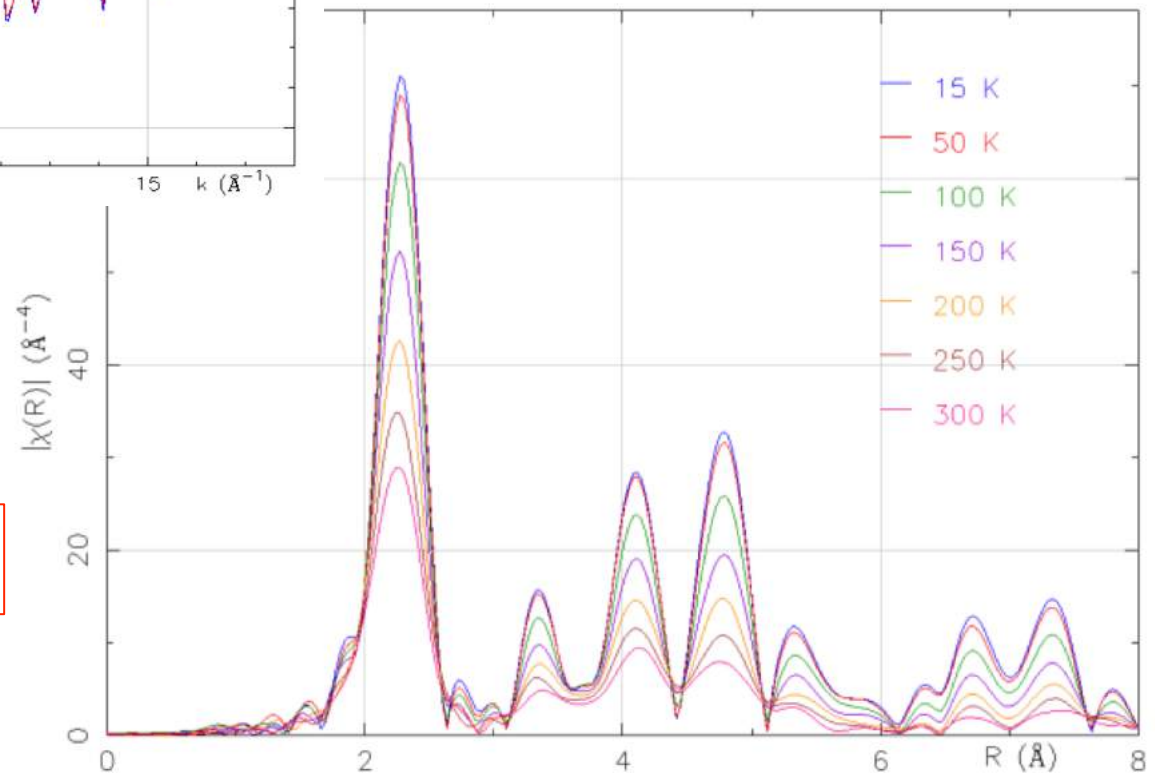
Cu foil and temperature effects



EXAFS spectra as a function of temperature.

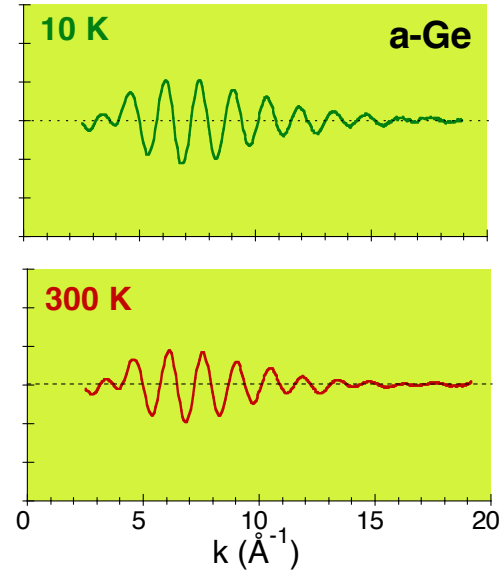
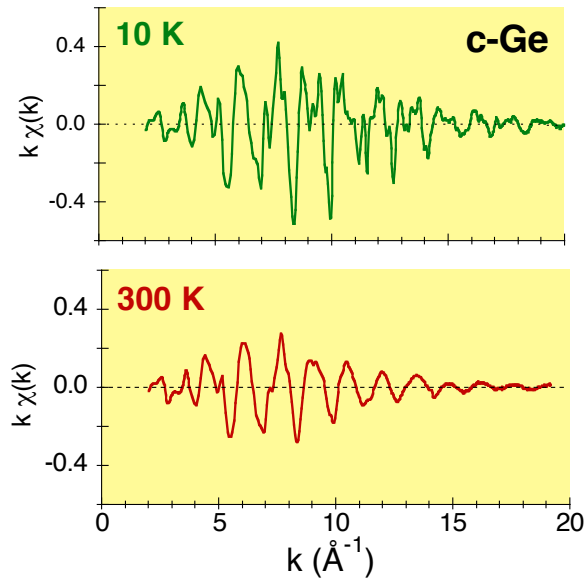
Thermal disorder: e^{-k^2/σ^2}

Fourier transforms of the EXAFS spectra.

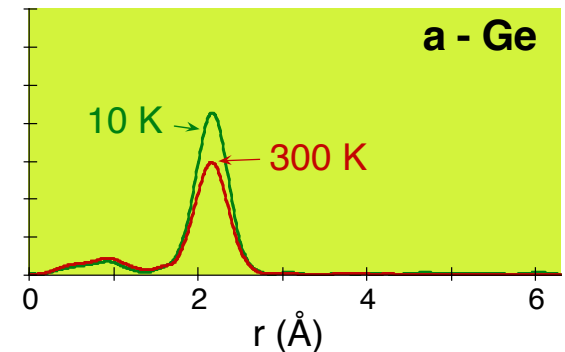
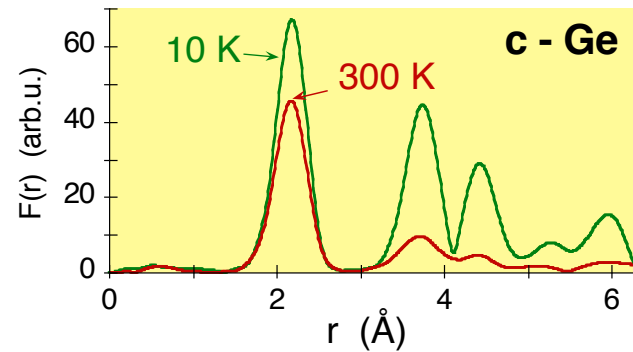


EXAFS data analysis

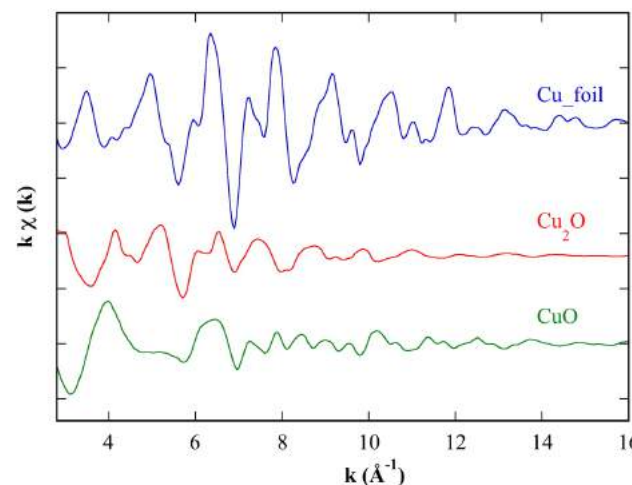
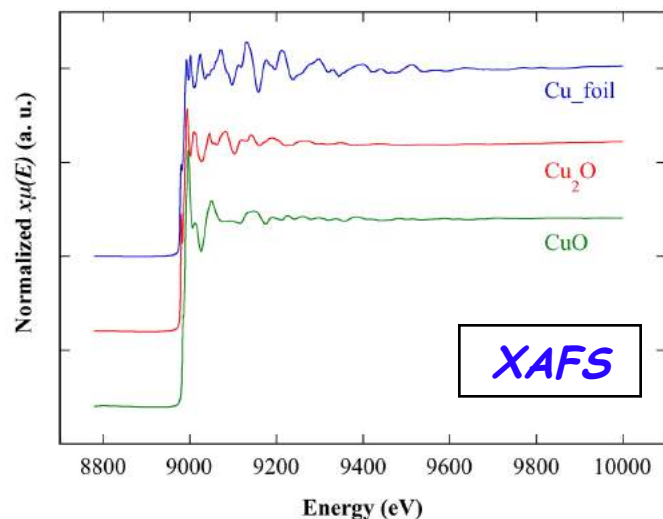
EXAFS signals



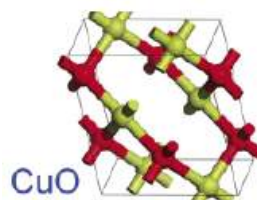
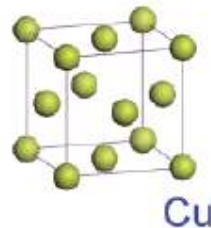
Fourier transforms



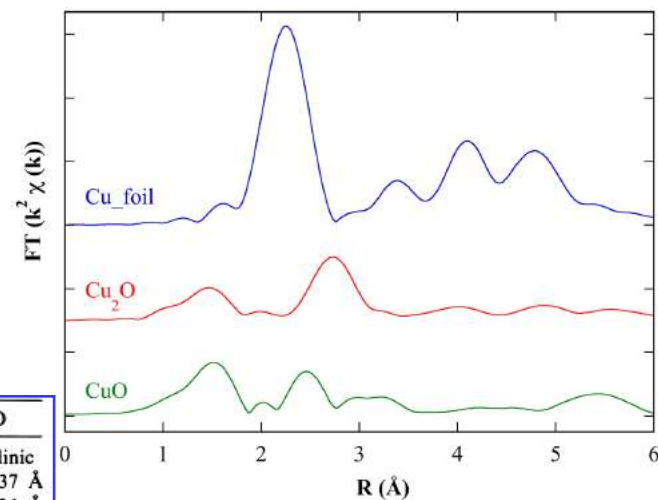
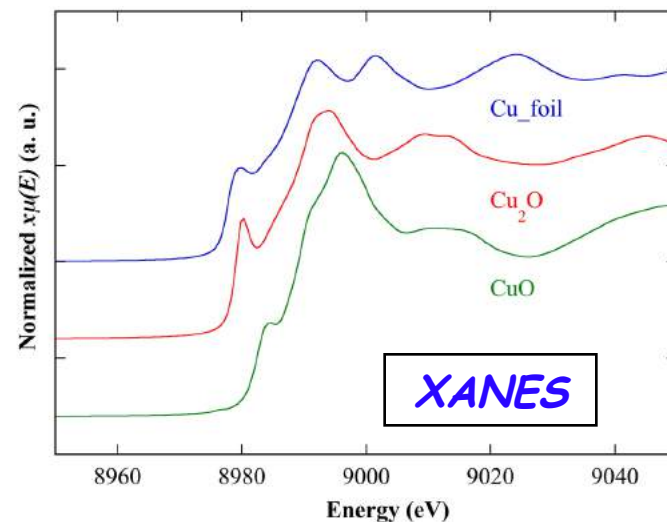
Cu Cu₂O CuO



Cu metal FCC $a = 3.61 \text{\AA}$



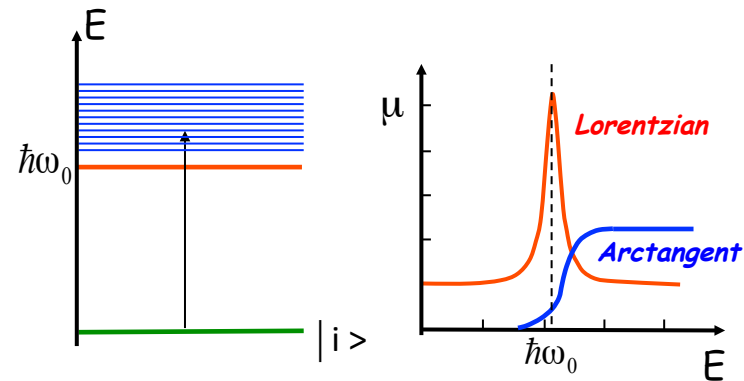
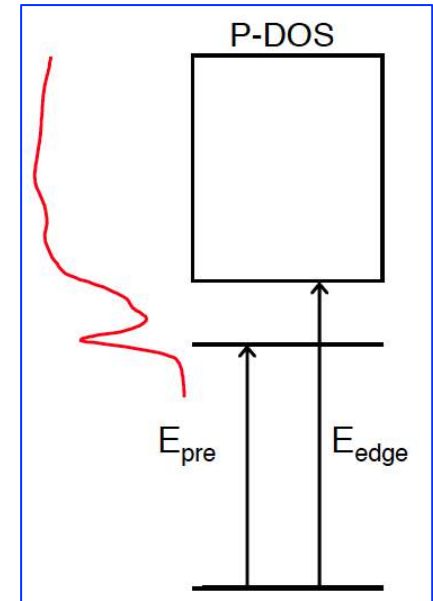
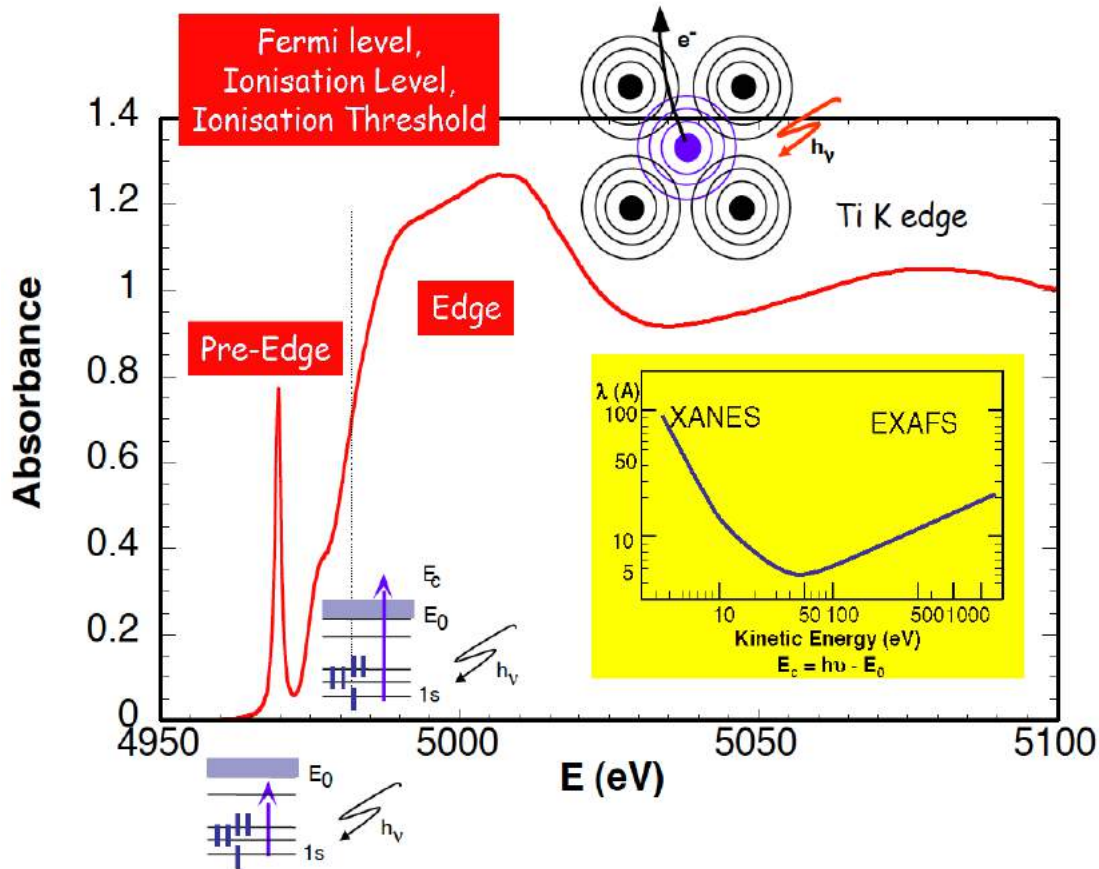
	Cu ₂ O	CuO
Lattice parameter	Cubic $a = 4.27 \text{\AA}$	Monoclinic $a = 4.6837 \text{\AA}$ $b = 3.4226 \text{\AA}$ $c = 5.1288 \text{\AA}$ $\beta = 99.54^\circ$
Shortest distances		
$d_{\text{Cu-O}}$	1.84 \AA	1.95 \AA
$d_{\text{O-O}}$	3.68 \AA	2.62 \AA
$d_{\text{Cu-Cu}}$	3.02 \AA	2.90 \AA



XANES

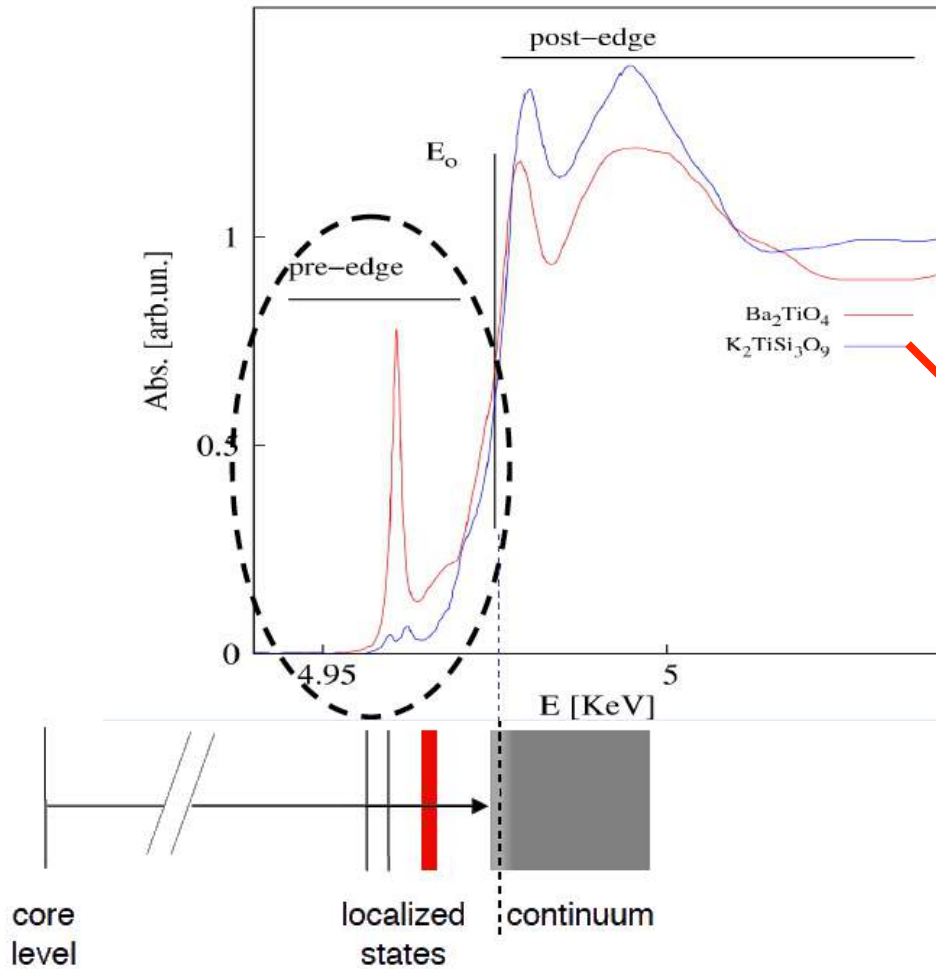
X ray Absorption Near Edge Structure

XANES

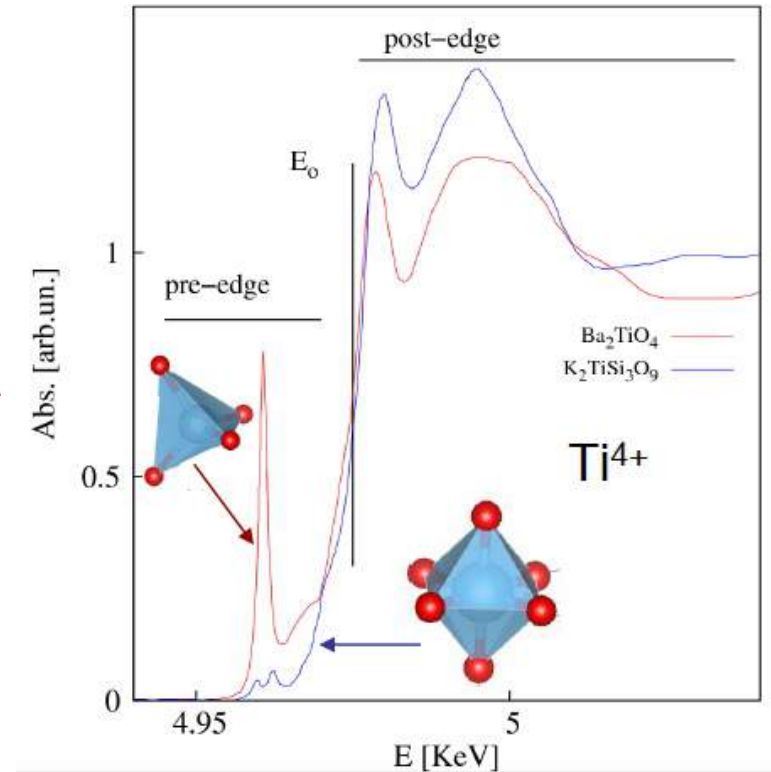


The local symmetry around the absorbing atom (symmetry, distance ligand-metal), the electronic structure of the absorbing atom (electronic filling of the valence state, oxidation state, spin state...)

Pre-edge region



Pre-edge peaks are due to electronic transitions (mainly dipole allowed) to empty bound states near the Fermi level.



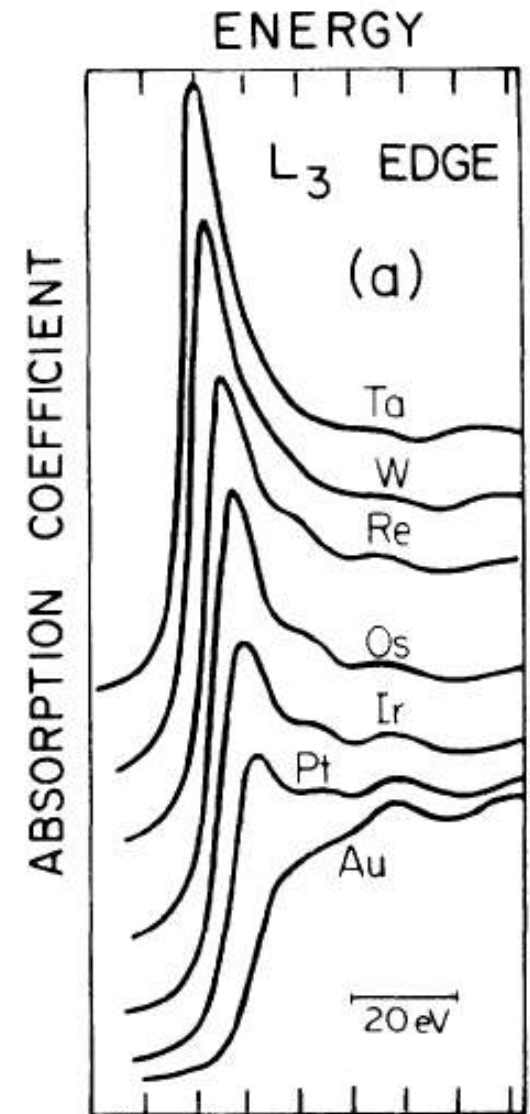
The peak due to $s \rightarrow p$ transitions (K edge) provides information on the absorber local geometry. In the Tetrahedral case (not centrosymmetric like Oh case) the $p-d$ mixing is allowed and this gives the largest pre-edge peak.

Shape of whitelines and L-edges

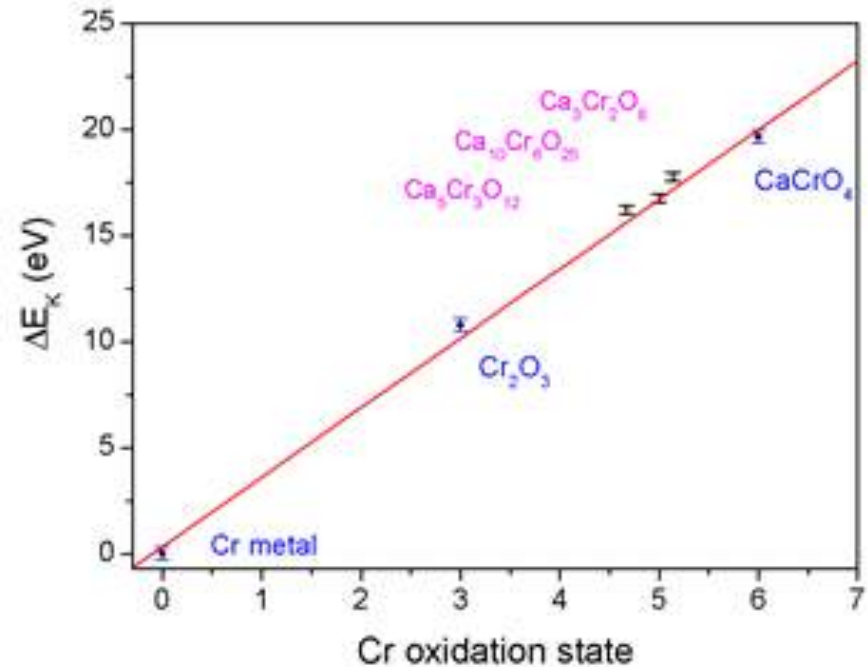
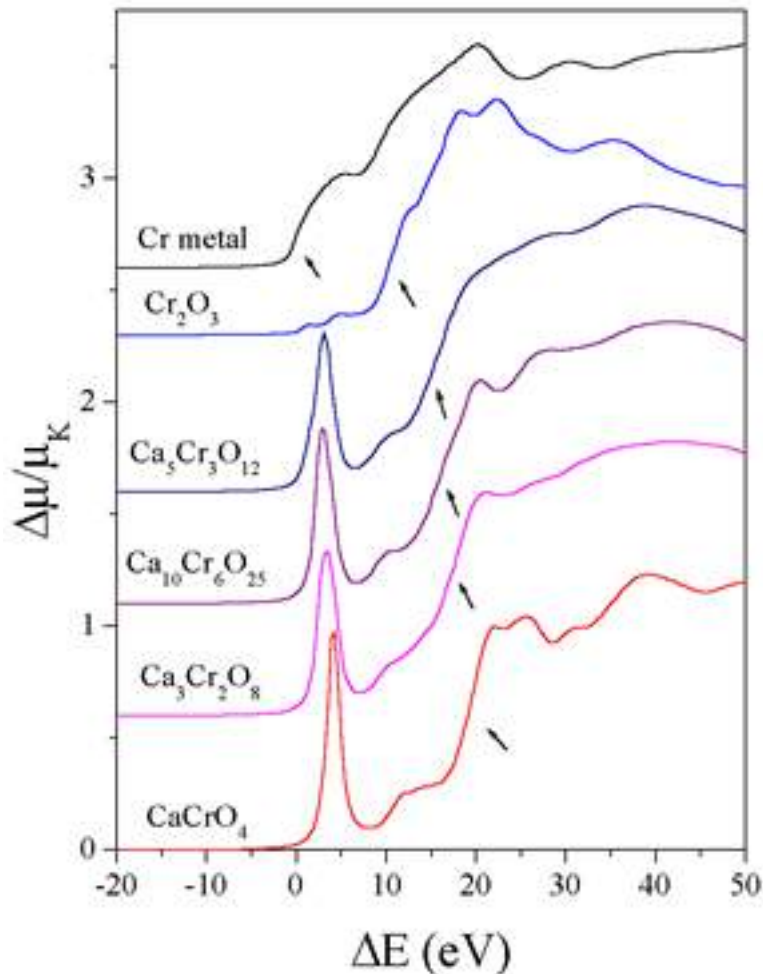
Whitelines present in the L-edges of atoms with 4d and 5d electrons, reflect holes in d-bands: the intensity decreases as a function of the increasing number of electrons in the d-band. In Au the 5d band is full.

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IA	IIA	IIIB	IVB	VB	VIB	VIB	VIII	VIII	VIII	IB	IIB	IIIA	IVA	VA	VIA	VIIA	VIIIA
1 H																	2 He
3 Li	4 Be											5 B	6 C	7 N	8 O	9 F	10 Ne
11 Na	12 Mg											13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
55 Cs	56 Ba	57 La	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
87 Fr	88 Ra	89 Ac	104 Rf	105 Db	106 Sg	107 Bh	108 Hs	109 Mt	110 Ds								
			58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu	
			90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr	
Groupe s			Groupe p					Groupe d					Groupe f				



XANES and oxidation state

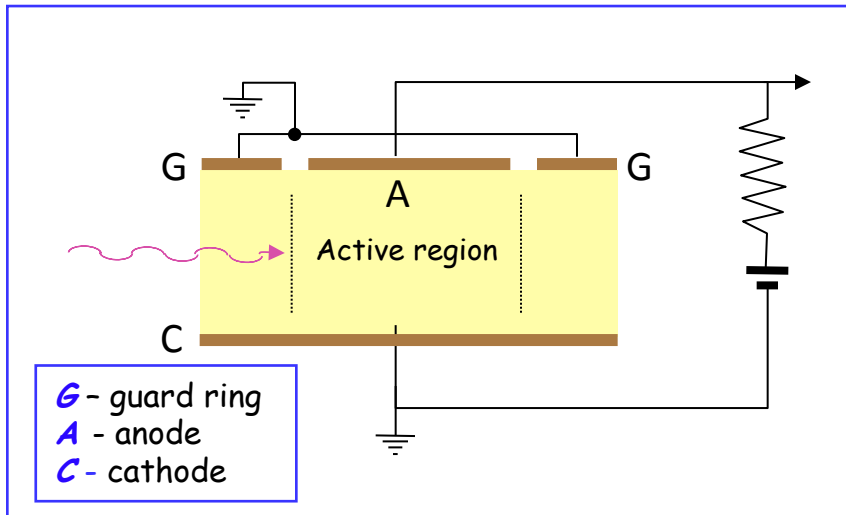


The edge, E_0 (arrow), defines the onset of continuous states (this is not the Fermi level).

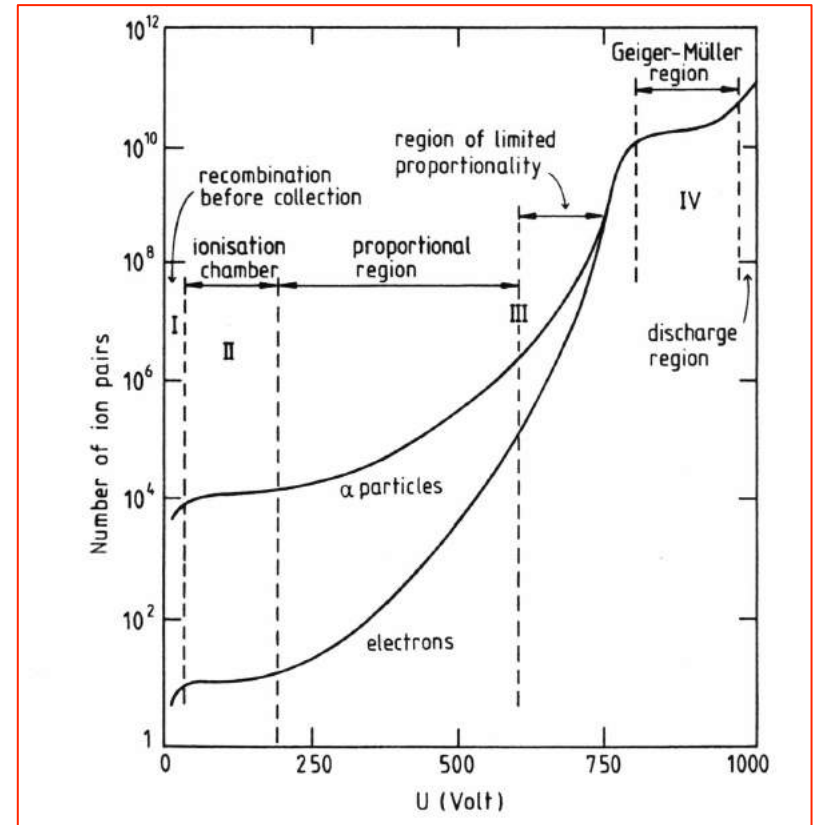
E_0 is a function of the absorber oxidation state and geometry. It may also increase by several eV due to oxidation.

Detectors and Gas ionization chambers

X-ray Ion Chamber

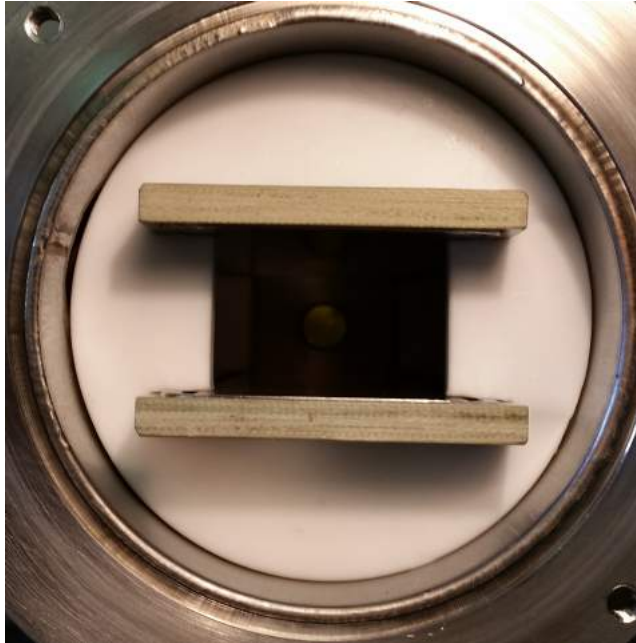


Gas ionization detectors are commonly used as integrating detectors to measure beam flux rather than individual photons. A typical detector consists of a rectangular gas cell with thin entrance and exit windows.



Inside the detector, an electric field is applied across two parallel plates. Some of the x-rays in the beam interact with the chamber gas to produce fast photoelectrons, Auger electrons, and/or fluorescence photons. The energetic electrons produce additional electron-ion pairs by inelastic collisions, and the photons either escape or are photo-electrically absorbed. The electrons and ions are collected at the plates, and the current is measured with a low-noise current amplifier. The efficiency of the detector can be calculated from the active length of the chamber, the properties of the gas, and the x-ray absorption cross section at the appropriate photon energy.

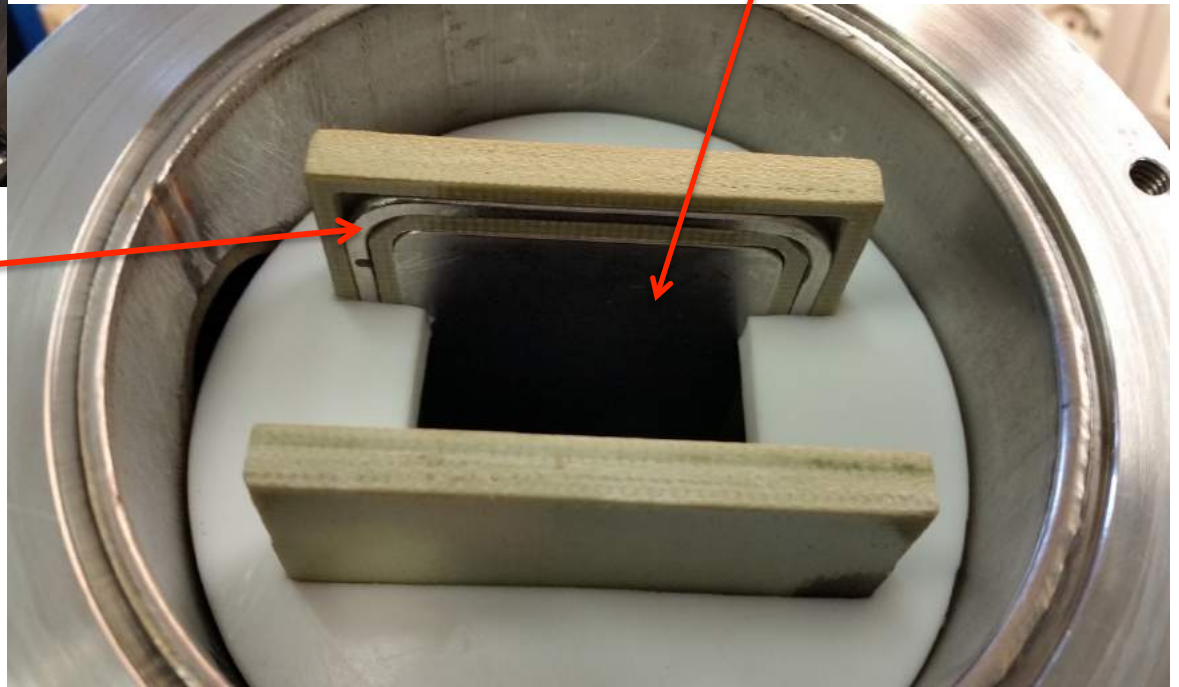
X-ray Ion chamber



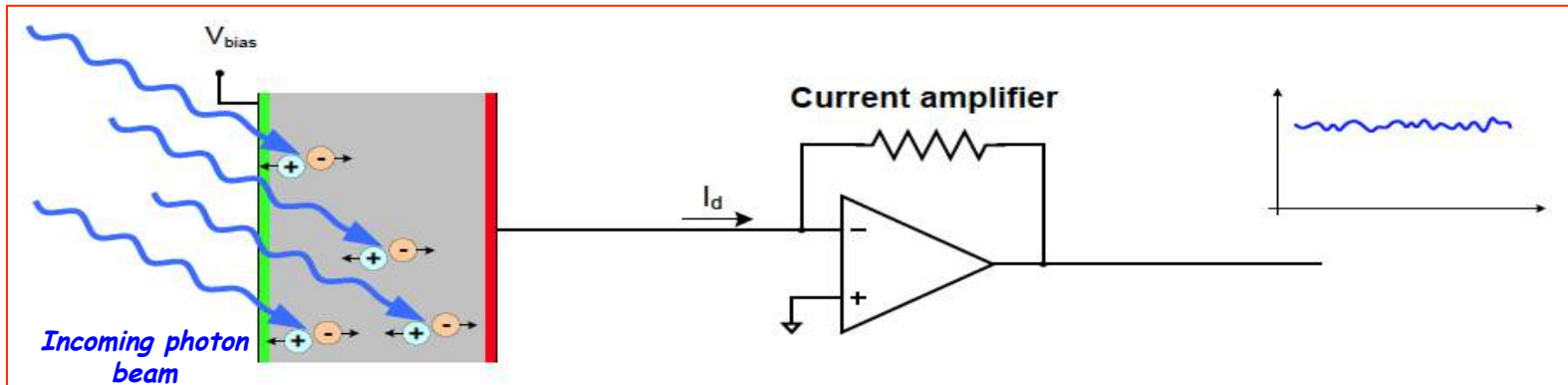
Ion chamber with parallel plates.

Guard ring

In the parallel plate chamber the charge-collecting electrode is surrounded by an annular ring. The annular ring represents the guard ring (or guard electrode) and is separated from the collecting electrode by a narrow insulating gap, and the applied voltage to the guard ring is the same as that to the collecting electrode.



Direct detection: charge conversion scheme and intensity measurement



The measured intensity is usually integrated during a well defined time interval and is proportional to the number of incident X-ray photons (N_{ph}).

Intrinsic statistical noise (Poisson statistics):

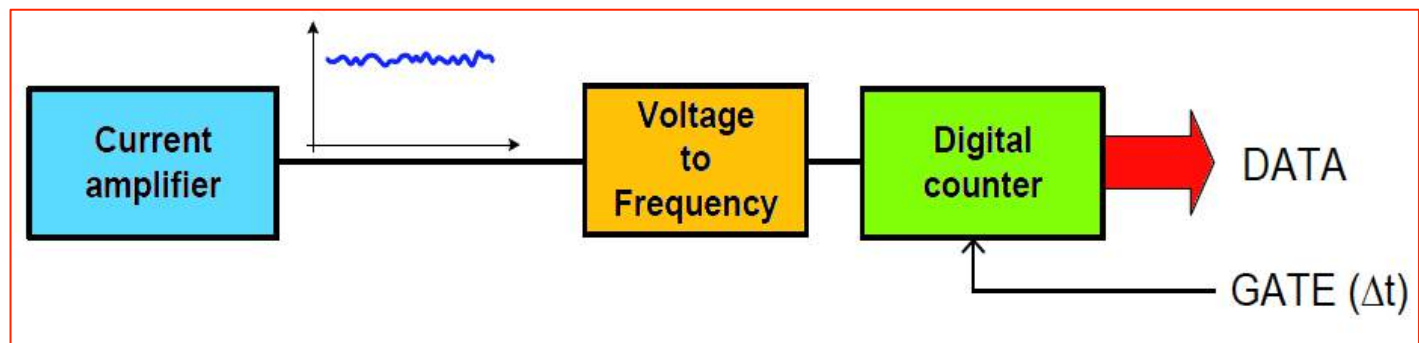
$$\sigma_{N_{ph}} = \sqrt{N_{ph}}$$

Effective:

$$\sigma_{N_{ph}} = \sqrt{F N_{ph}}$$

Fano factor F accounts empirically for deviation from Poisson statistics $F \approx 0.2$ for gasses, ≈ 0.1 for semiconductors

The measured intensity is integrated during the exposure time Δt .

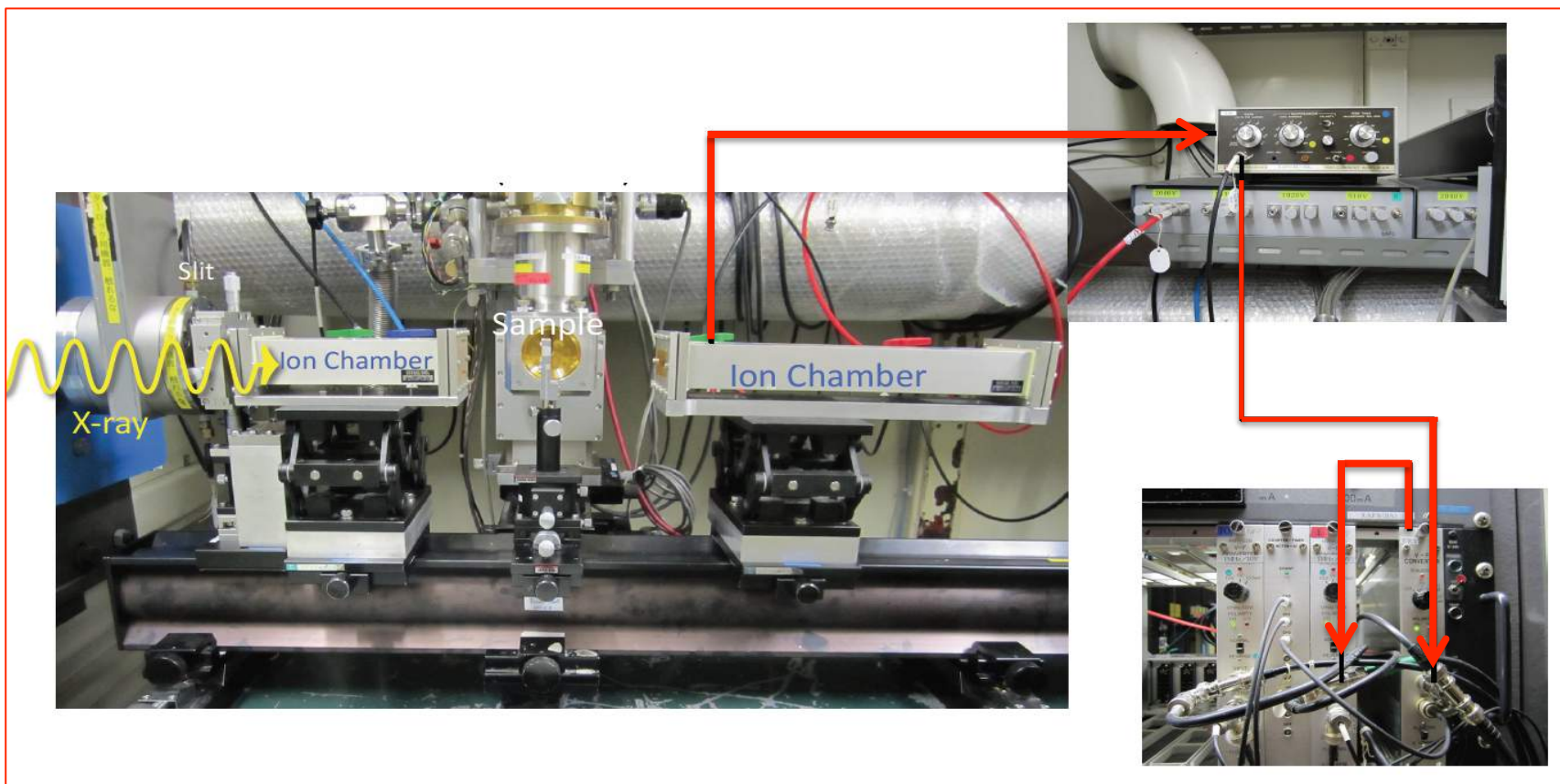


Setup: XAFS in transmission mode

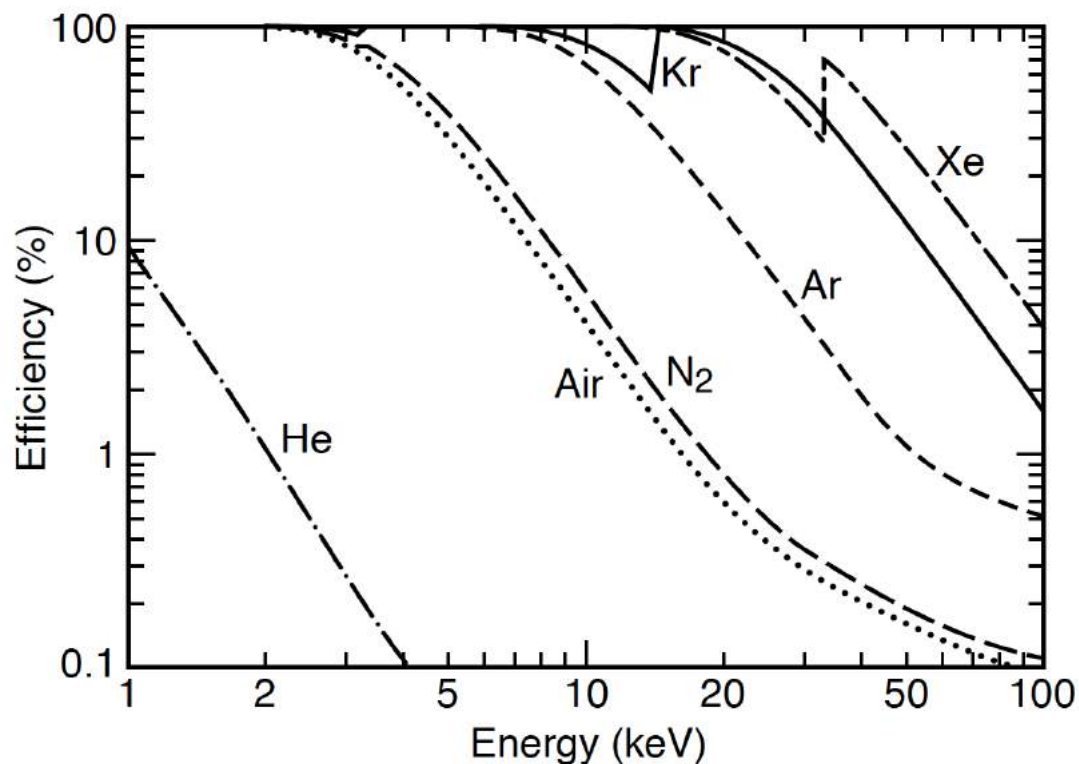
$$\mu(E) = \frac{1}{x} \ln \left(\frac{I_0}{I_1} \right)$$

Current proportional to the x-ray intensity

Current amplifier and
converter of I to V



Ion chamber characteristics



Efficiency of a 10-cm-long gas ionization chamber as a function of energy, for different gases at normal pressure.

The efficiency of the detector can be calculated from the active length of the chamber, the properties of the chamber gas, and the x-ray absorption cross section at the appropriate photon energy

Once the efficiency is known, the photon flux can be estimated from chamber current and the average energy required to produce an electron-ion pair

Element	Energy (eV)
Helium	41
Nitrogen	36
Air	34.4
Neon	36.3
Argon	26
Krypton	24
Xenon	22

Photon flux evaluation

$$I = Ne = I_0 T \gamma e$$

I_0 = Incoming photon flux (ph/s)

T = Ion chamber window transmission

γ = gas efficiency (electrons/ph)

L = length of the ion chamber plate

$$N \cong \frac{E}{\langle V_i \rangle}$$

N = Number of electron-ion pairs produced

E = X ray energy

$\langle V_i \rangle$ = Average energy required to produce an electron-ion pair

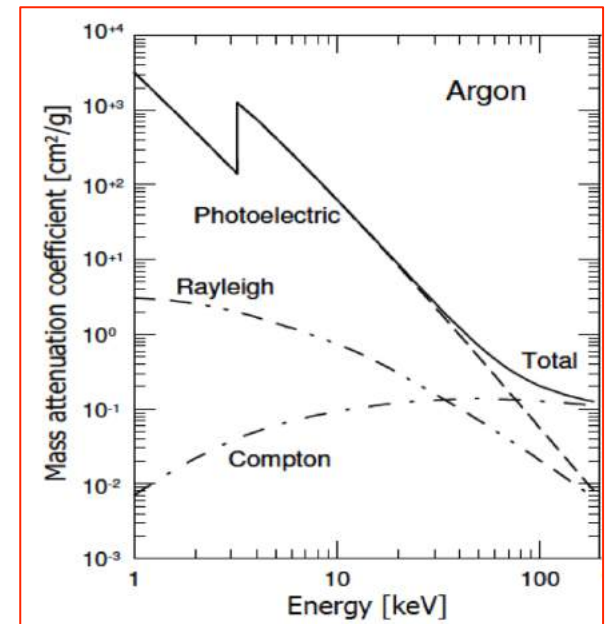
$$I_0 (\text{ph} / \text{s}) = \frac{I(A)}{\gamma e(C)} \frac{\langle V_i \rangle (eV)}{E(eV)} \frac{1}{1 - e^{-\mu L (cm)}}$$

$$\mu (cm^{-1}) = \left[\frac{\mu}{\rho} (E) \right] \rho$$

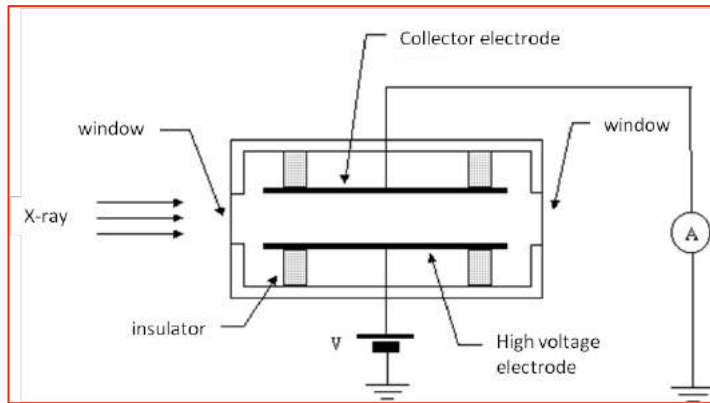
$\left[\frac{\mu}{\rho} \right]$ = gas mass attenuation coefficient

ρ = gas density function of pressure (γ)

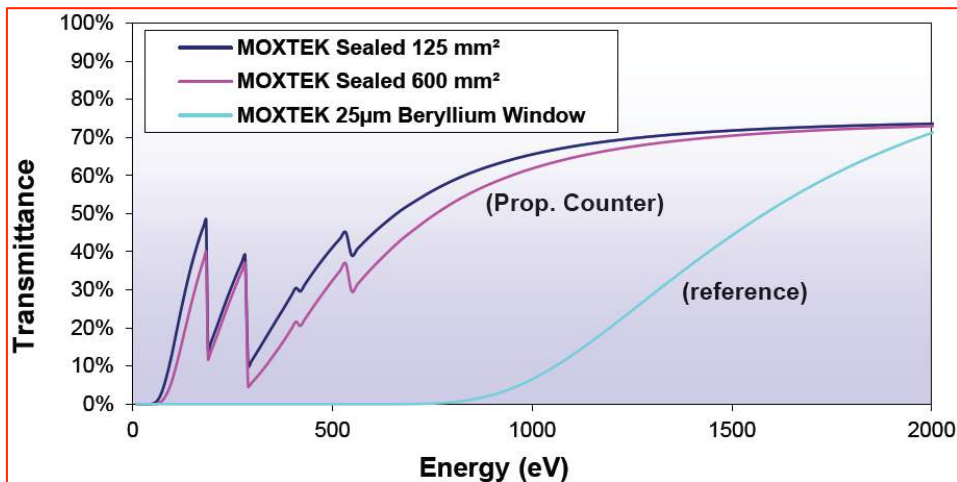
$\gamma = I_0$ 10%; I_1 90%



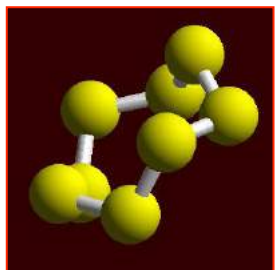
X-ray ion chambers and windows



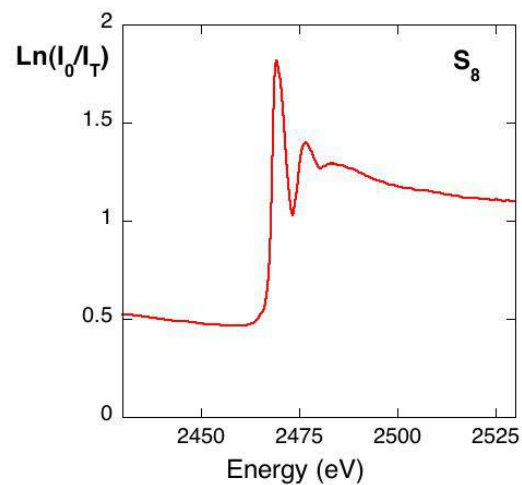
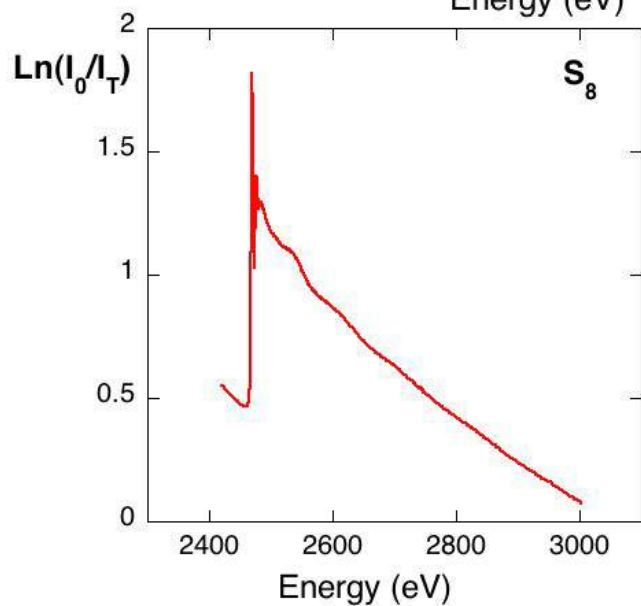
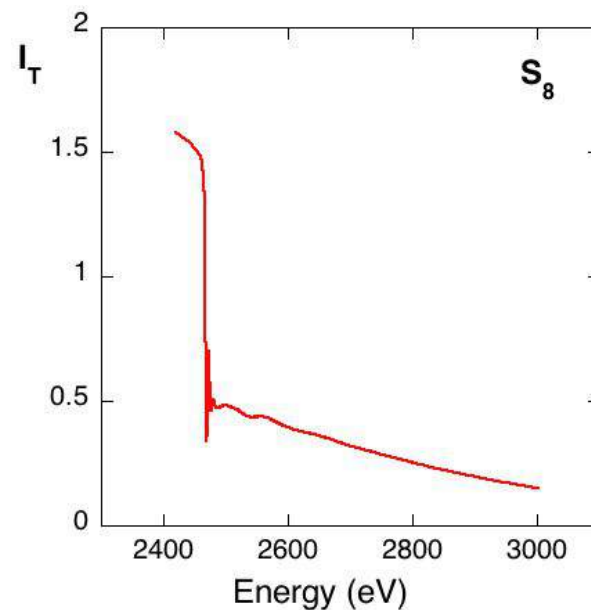
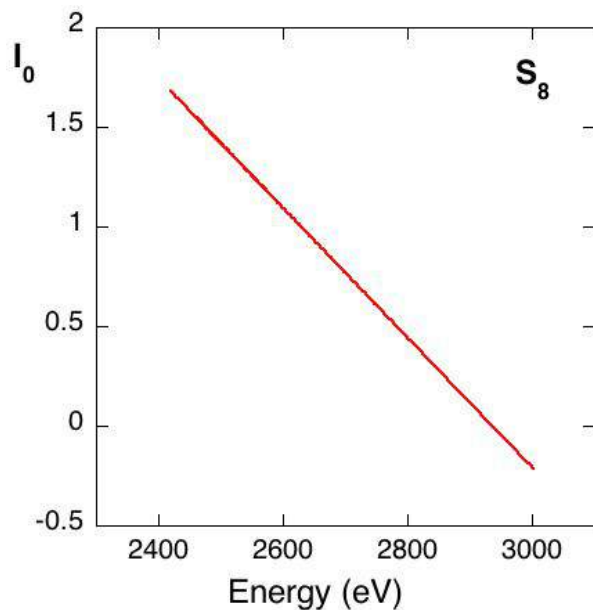
Unmounted and mounted MOXTEK ultrathin windows



XAFS S_8



S_8



Thank you for your attention



Supplementary material

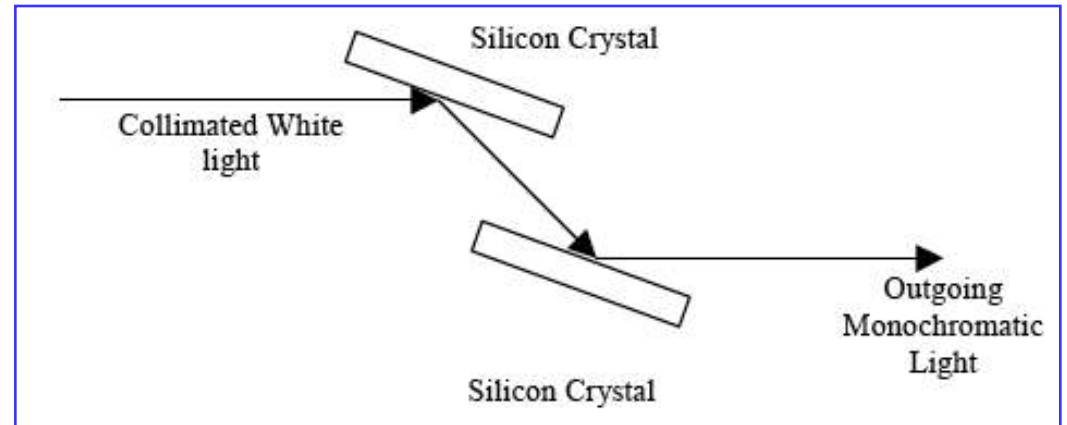
Reminder

- *Speed of light* $c = 2.99792458 \times 10^8 \text{ m/s}$
 - *Electron charge* $e = 1.6021 \times 10^{-19} \text{ Coulombs}$
 - *Electron volts* $1 \text{ eV} = 1.6021 \times 10^{-19} \text{ Joule}$
 - *Energy and rest mass* $1 \text{ eV}/c^2 = 1.78 \times 10^{-36} \text{ kg}$
- | | |
|-----------------|--|
| <i>Electron</i> | $m_0 = 511.0 \text{ keV}/c^2 = 9.109 \times 10^{-31} \text{ kg}$ |
| <i>Proton</i> | $m_0 = 938.3 \text{ MeV}/c^2 = 1.673 \times 10^{-27} \text{ kg}$ |
- *Relativistic energy, E* $E = mc^2 = m_0 \gamma c^2$
 - *Lorentz factor, γ* $\gamma = 1/[(1-v^2/c^2)^{1/2}] = 1/[(1-\beta^2)^{1/2}]$
 $\beta = v/c$
 - *Relativistic momentum, p* $p = mv = m_0 \gamma \beta c$
 - *E-p relationship* $E^2/c^2 = p^2 + m_0^2 c^2$
for ultra-relativistic particles $\beta \approx 1, E = pc$
 - *Kinetic energy* $T = E - m_0 c^2 = m_0 c^2 (\gamma - 1)$

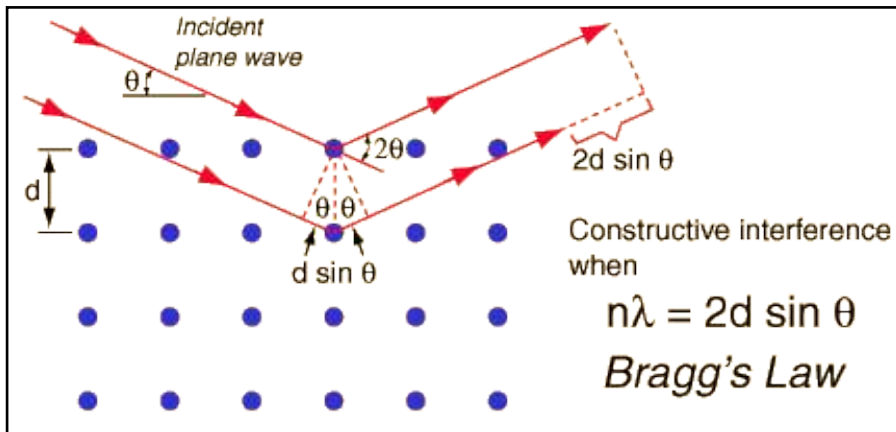
Monochromator



Prism and visible light



X-rays and crystals



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

$$n\lambda = 2d \sin \theta \quad \text{Bragg's Law}$$

Anti-matter positron production

