Chapter 10 - Nuclear Based Analytical Methods

Radiography

NDT	X-ray radiography
NDT	Neutron radiography

X-Ray Methods

XRD	X-ray diffraction
XRC	X-ray crystallography
XRF	X-ray fluorescence
EXAFS	Extended X-ray Absorption Fine Structure
XANES	X-ray Absorption Near-Edge Structure

Electron Methods

AES	Auger Electron Spectroscopy
ESCA	Electron Spectroscopy for Chemical Analysis
LEED	Low Energy Electron Diffraction
XPS	X-ray Photoemission Spectroscopy

Neutron Methods

NAA	Neutron Activation Analysis (INAA, PGNAA, RNAA, FNAA)
NS	Neutron Scattering

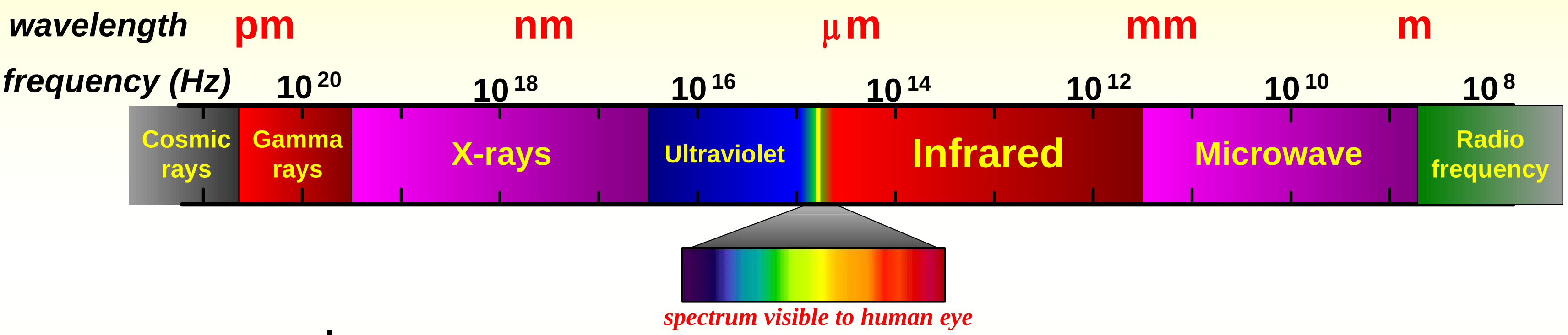
Mass Spectral Methods

AMS	Accelerator Mass Spectroscopy
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
FTICR-MS	Fourier Transform Ion Cyclotron Resonance Mass Spectrometry
SIMS	Secondary Ion Mass Spectrometry

Charged Particle Methods

APXS	Alpha Particle X-ray Spectrometry
PIXE	Proton Induced X-ray Emission
RBS	Rutherford Back Scattering

The Electromagnetic Spectrum



nuclear proton - neutron energy levels	atomic electron energy levels	molecular electron energy levels	molecular vibration energy levels	molecular rotation energy levels	nuclear spin energy levels
Gamma spectroscopy	X-ray spectroscopy	UV-Vis spectroscopy	Infrared spectroscopy	Microwave spectroscopy	NMR spectroscopy

Industrial Radionuclide Uses

Liquid level measurements

Thickness measurements

Corrosion measurements

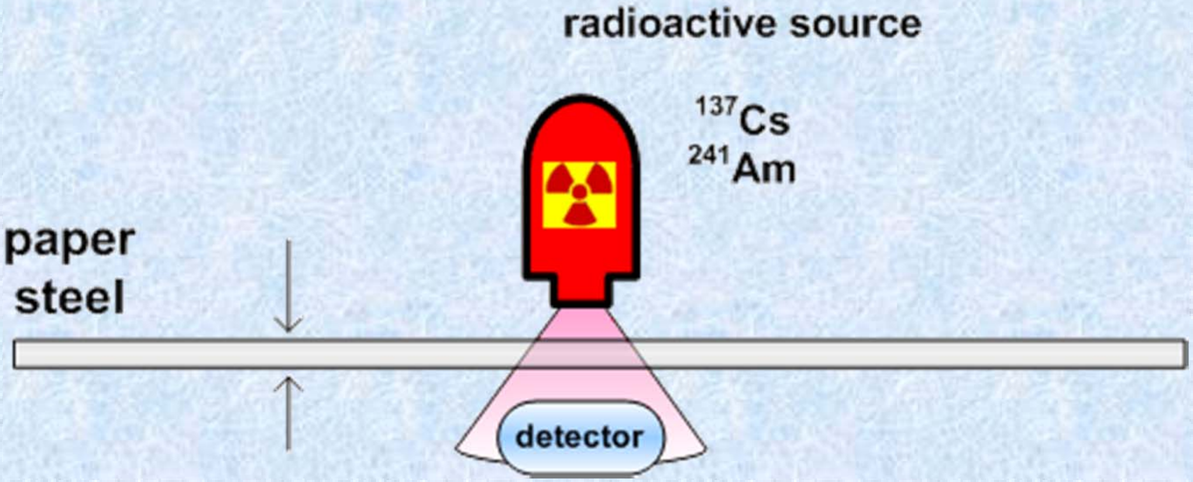
Mass flow measurements

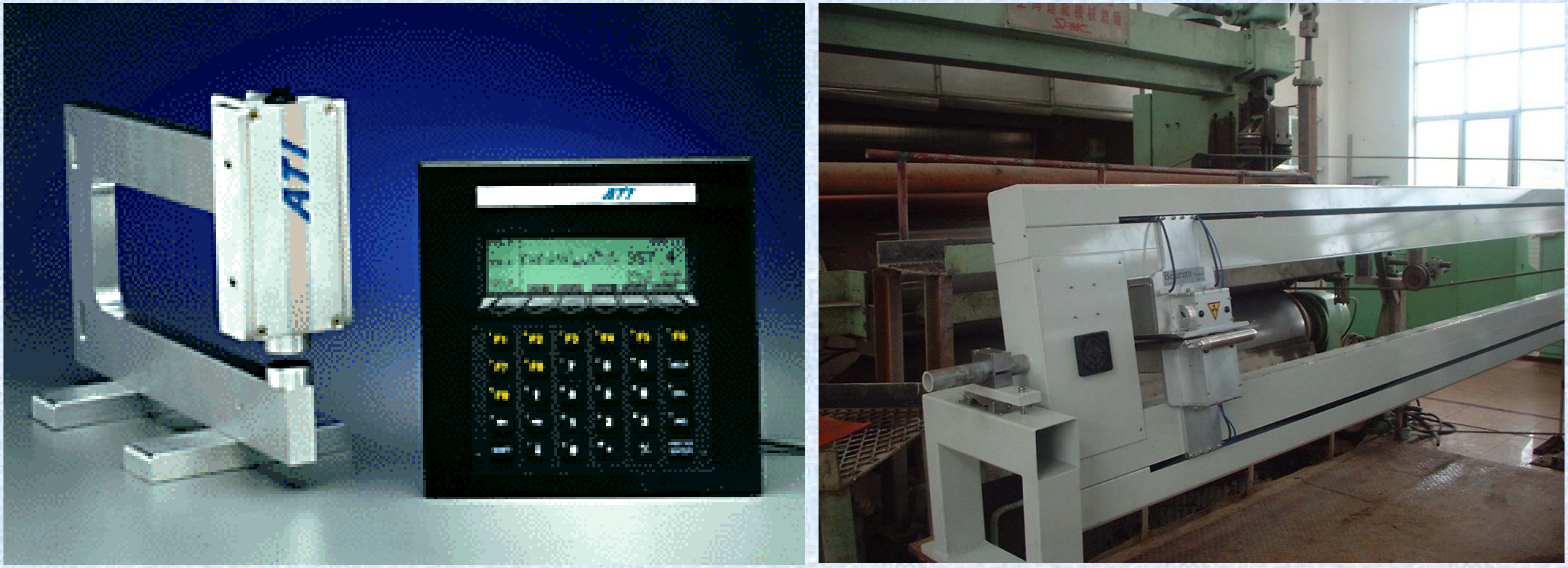
Leak detection

Chemical process measurements

- distillation efficiency
- solvent entrainment

Nuclear Thickness Gauges

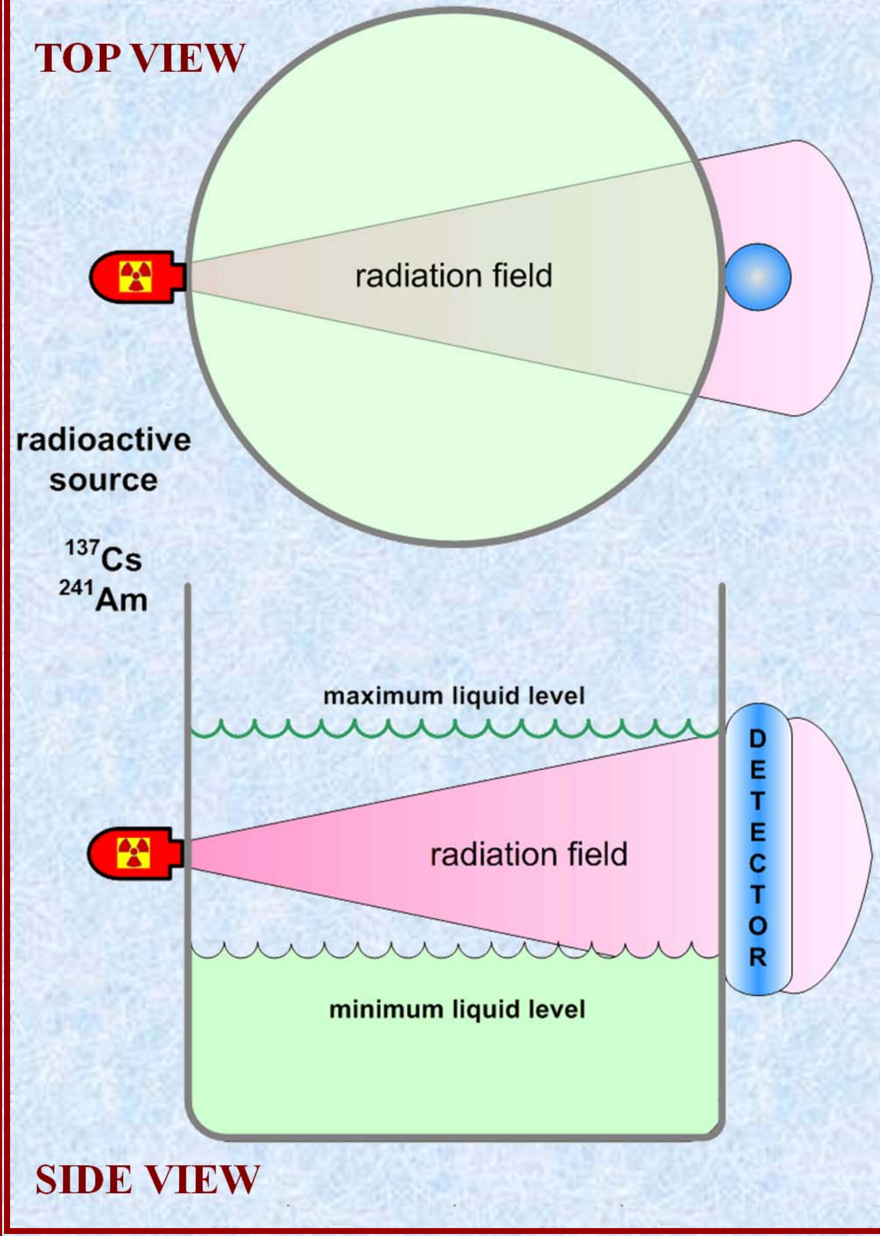




Nuclear Thickness Gauges

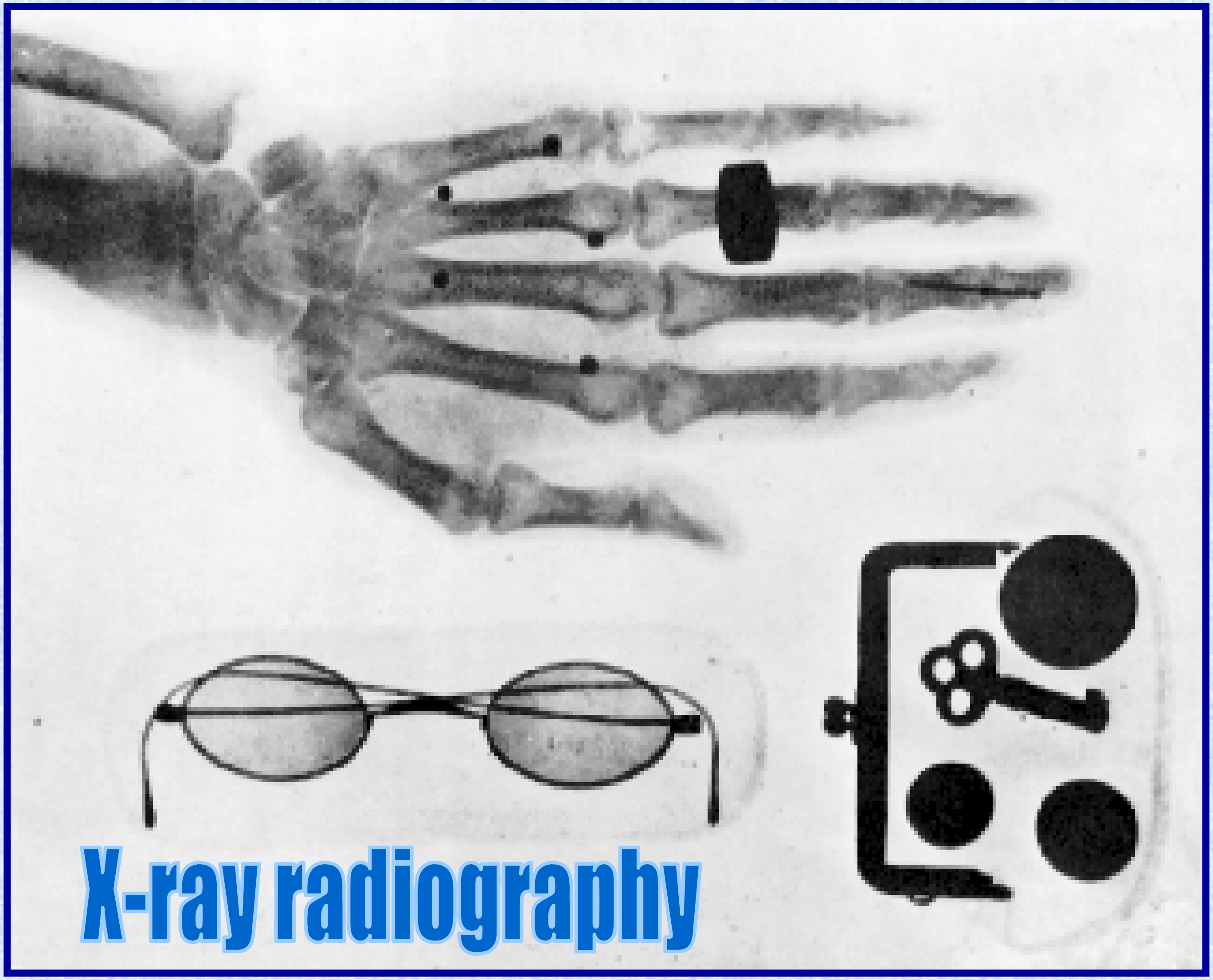
Radiation	Source	$t_{1/2}$	Application
α	U or Ra	Long	Thickness control in manufacturing paper, aluminum; $\leq$ 60 g/m^2
Soft β	^{147}Pm (0.2 MeV)	2.6 y	Thickness control; $\leq$ 400 g/m^2
β, soft γ	^{204}Tl (0.8 MeV)	3.8 y	Thickness: 1-10 mm steel, 3-50 mm glass; 8-100 kg/m^2
Hard β	^{144}Ce (3 MeV)	0.78 y	Thickness $\leq$1 mm steel; $\leq$ 10 kg/m^2
X	^{109}Cd (88 keV)	1.24 y	Detection of S-content in hydrocarbons
n, γ	RaBe, ^{137}Cs	30 y	Moisture-density meter for civil engineering and agriculture
γ	^{60}Co (1.3 MeV)	5.3 y	4 MBq source for backscatter on $\leq$ 20 mm steel, 0.4 - 40 GBq for remote level indication
Soft γ	^{192}Ir (0.3 MeV)	74 d	400 GBq, 26 kg: $\leq$ 40 mm steel radiography
Medium γ	^{137}Cs (0.7 MeV)	30 y	400GBq, 45kg: $\leq$ 70 mm steel pipeline inspection
Hard γ	^{60}Co (1.3 MeV)	5.3 y	10 TBq, 900 kg: $\leq$ 180 mm steel radiography

Nuclear Liquid Levels Gauges

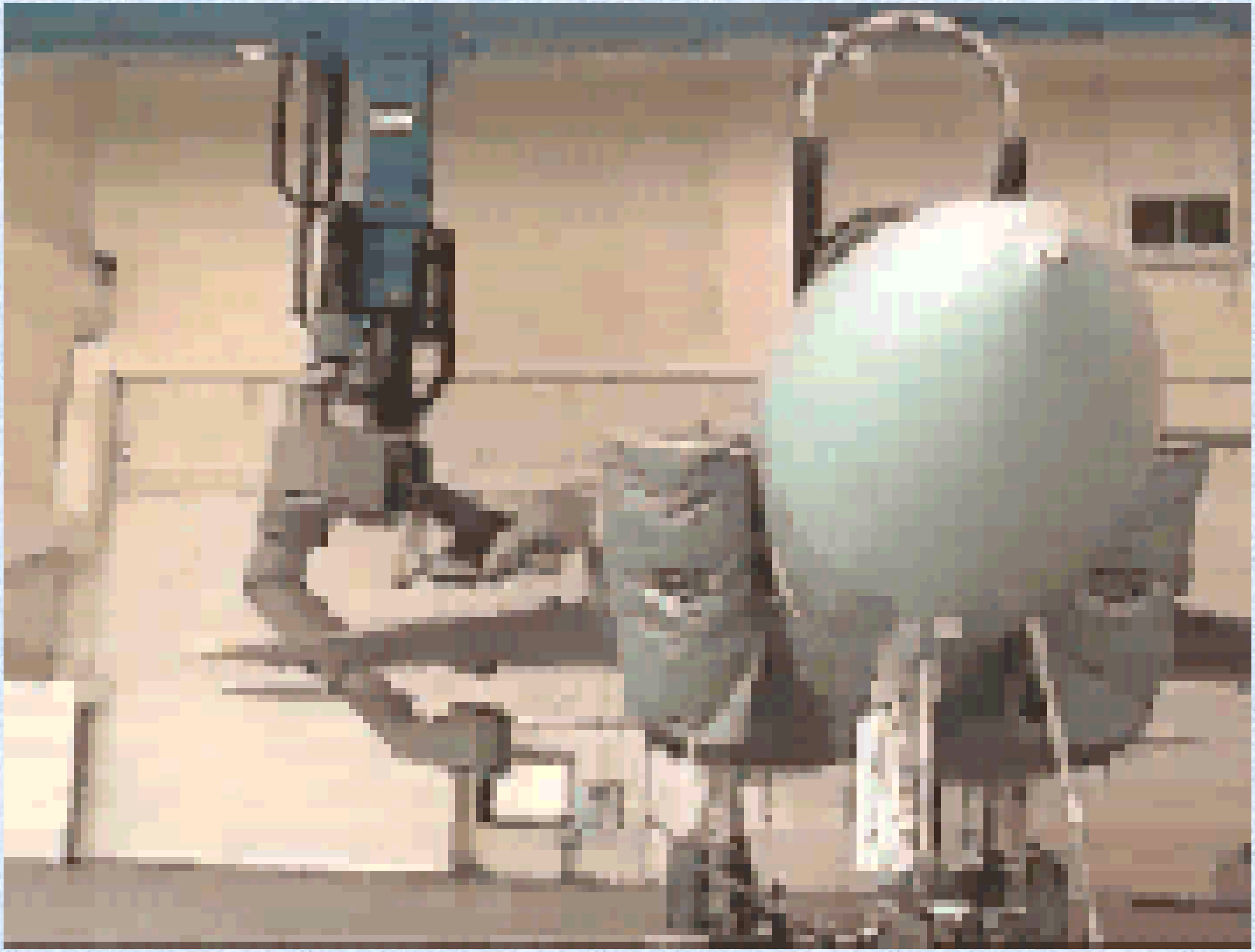


Radiography - NDT

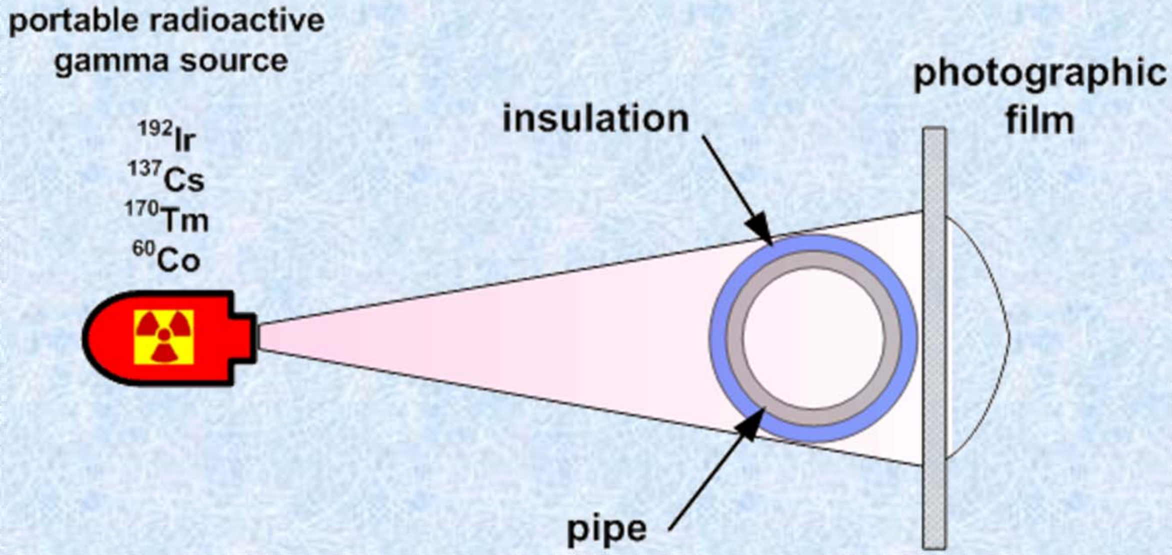
Non-destructive Testing



Industrial Radiography



Industrial Radiography



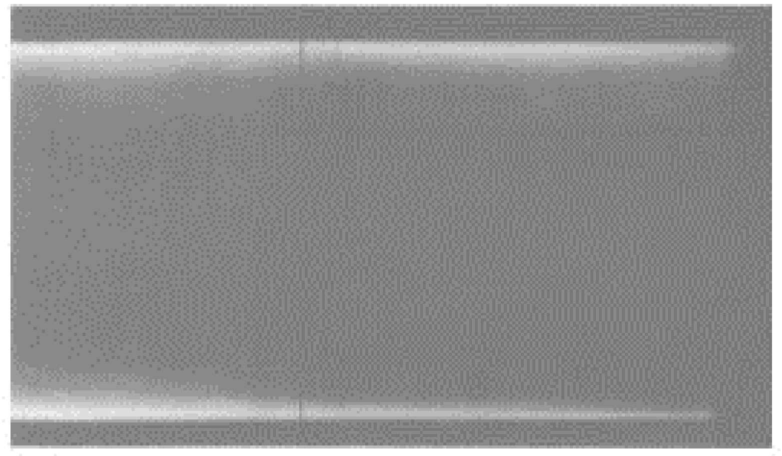
portable radioactive
gamma source

^{192}Ir
^{137}Cs
^{170}Tm
^{60}Co

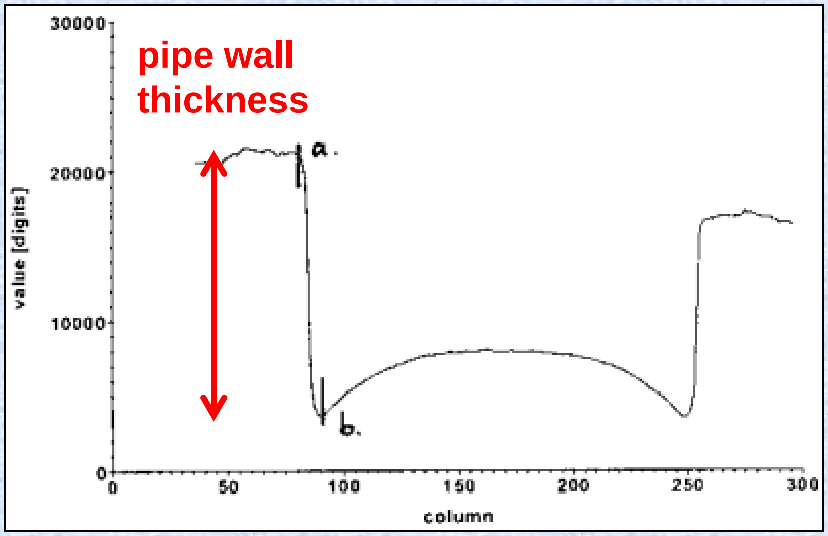
insulation

pipe

photographic
film

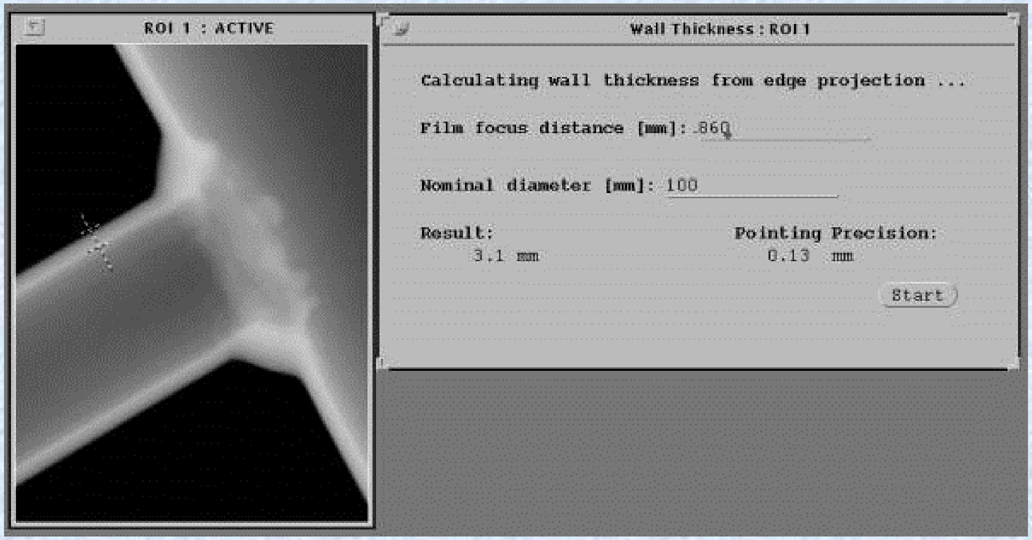


pipe radiograph



densitometer trace

Industrial Radiography



Calculating wall thickness from edge projection ...

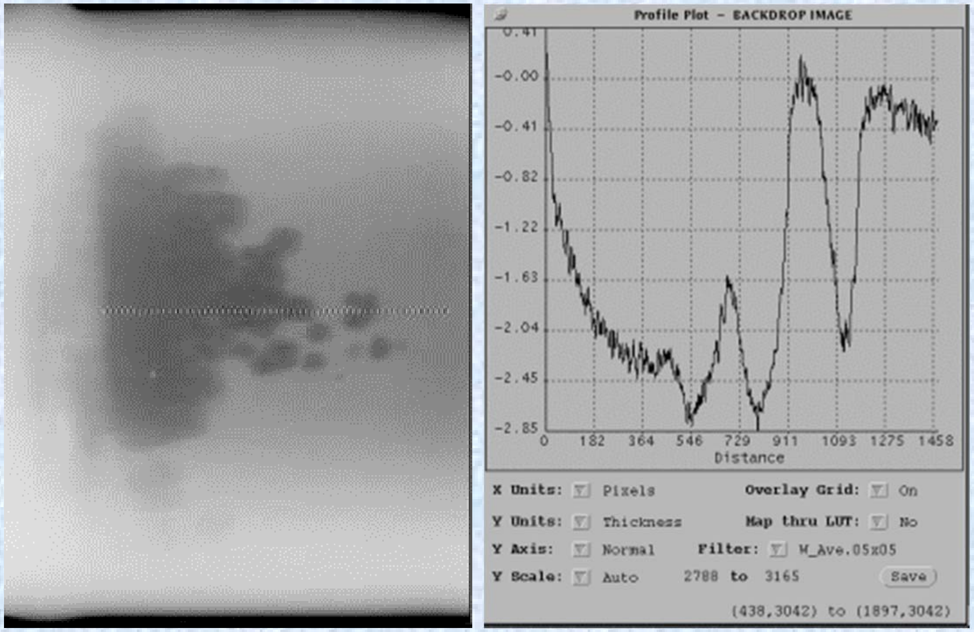
Film focus distance [mm]: 860

Nominal diameter [mm]: 100

Result: 3.1 mm

Pointing Precision: 0.13 mm

weld integrity
measurements
(in the field)



X Units: Pixels

Overlay Grid: On

Y Units: Thickness

Map thru LUT: No

Y Axis: Normal

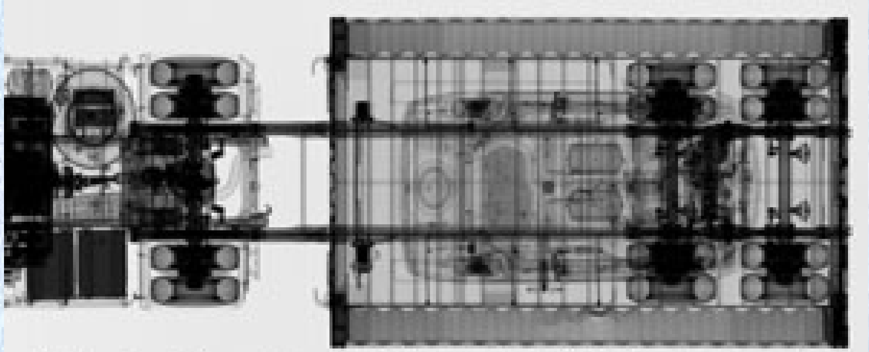
Filter: W_Ave.05x05

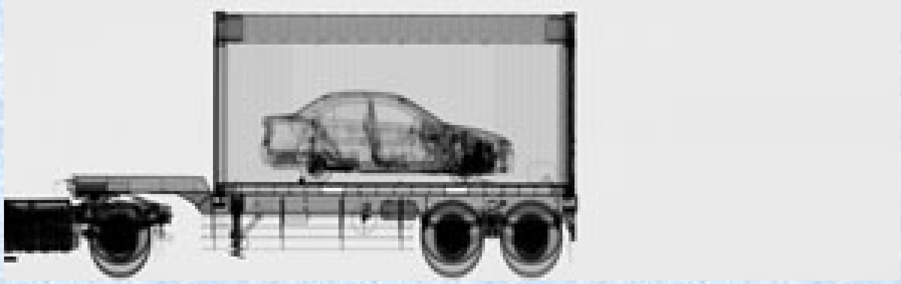
Y Scale: Auto 2788 to 3165

(438,3042) to (1897,3042)

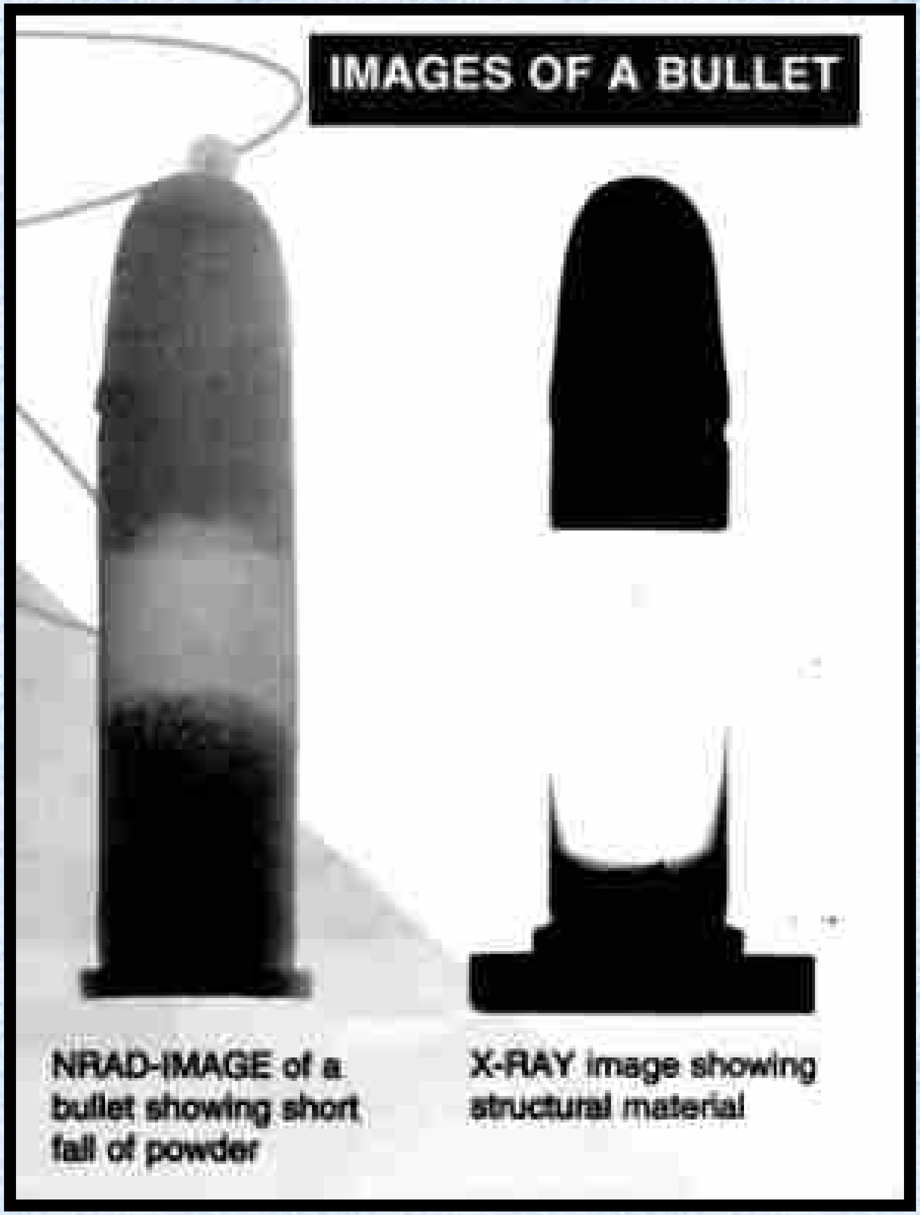
corrosion
measurements
(in the field)

Homeland Security Radiography





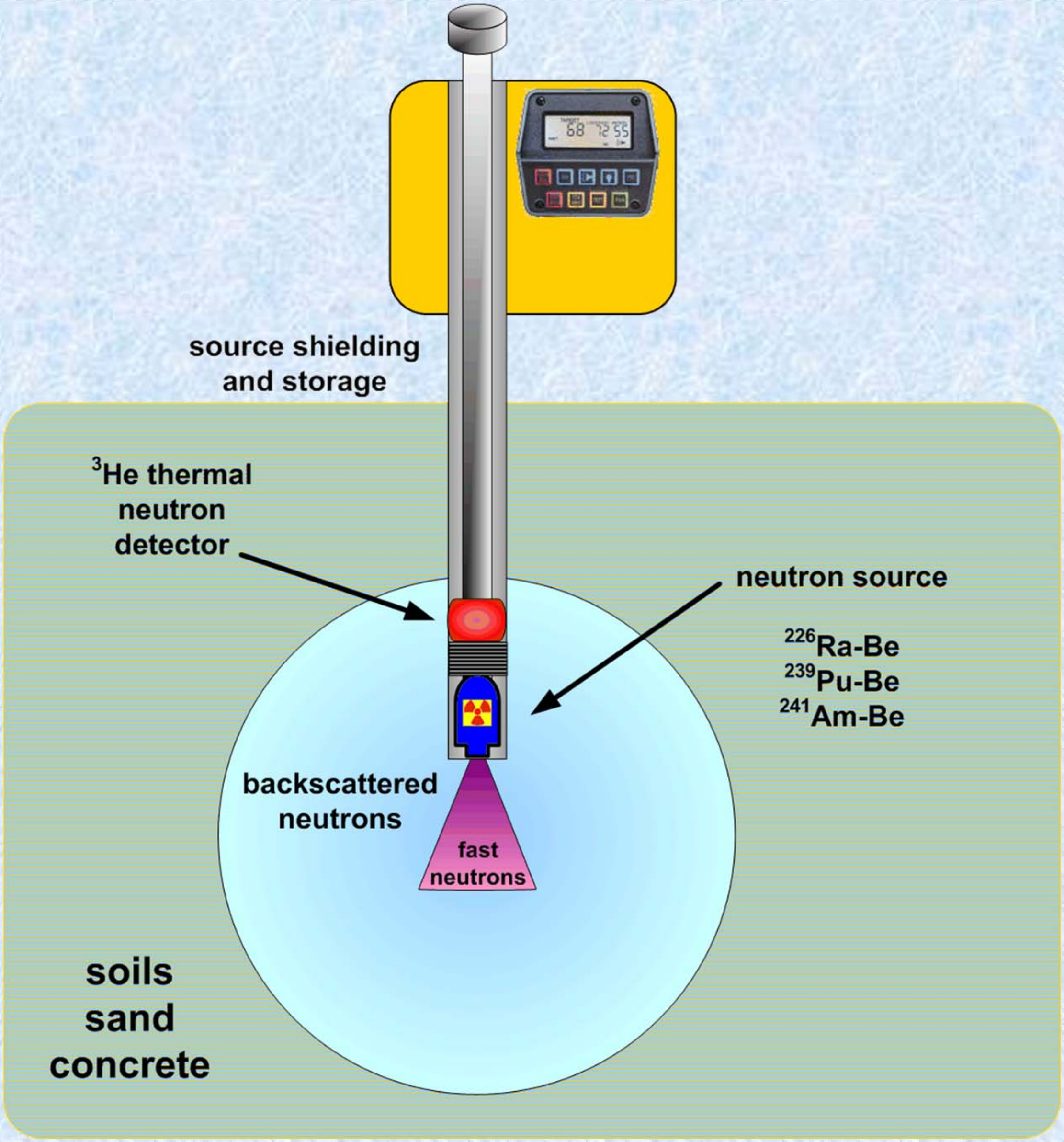
Neutron Radiography



NRAD-IMAGE of a bullet showing short fall of powder

X-RAY image showing structural material

Neutron Moisture Gauges



source shielding and storage

^{3}He thermal neutron detector

neutron source

^{226}Ra-Be
^{239}Pu-Be
^{241}Am-Be

backscattered neutrons

fast neutrons

soils
sand
concrete

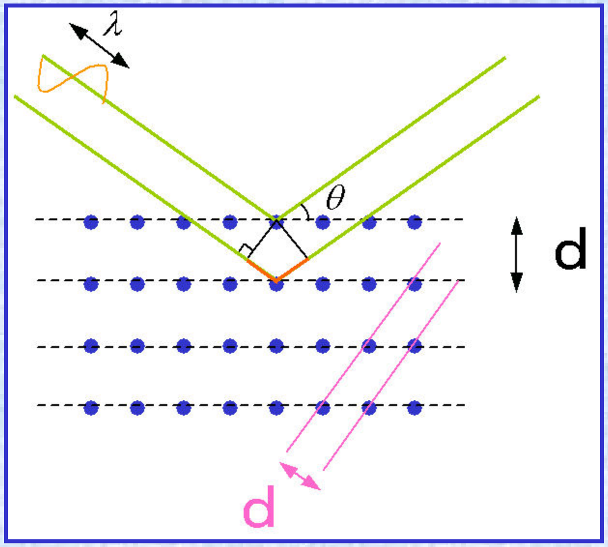
X-Ray Diffraction

Moon rock

micrograph of polished thin section
showing many mineral constituents

Need to more than just gross chemical composition.

What are the constituents of the rock?
(gives information about how rock was formed and what its geochemical history has been.)



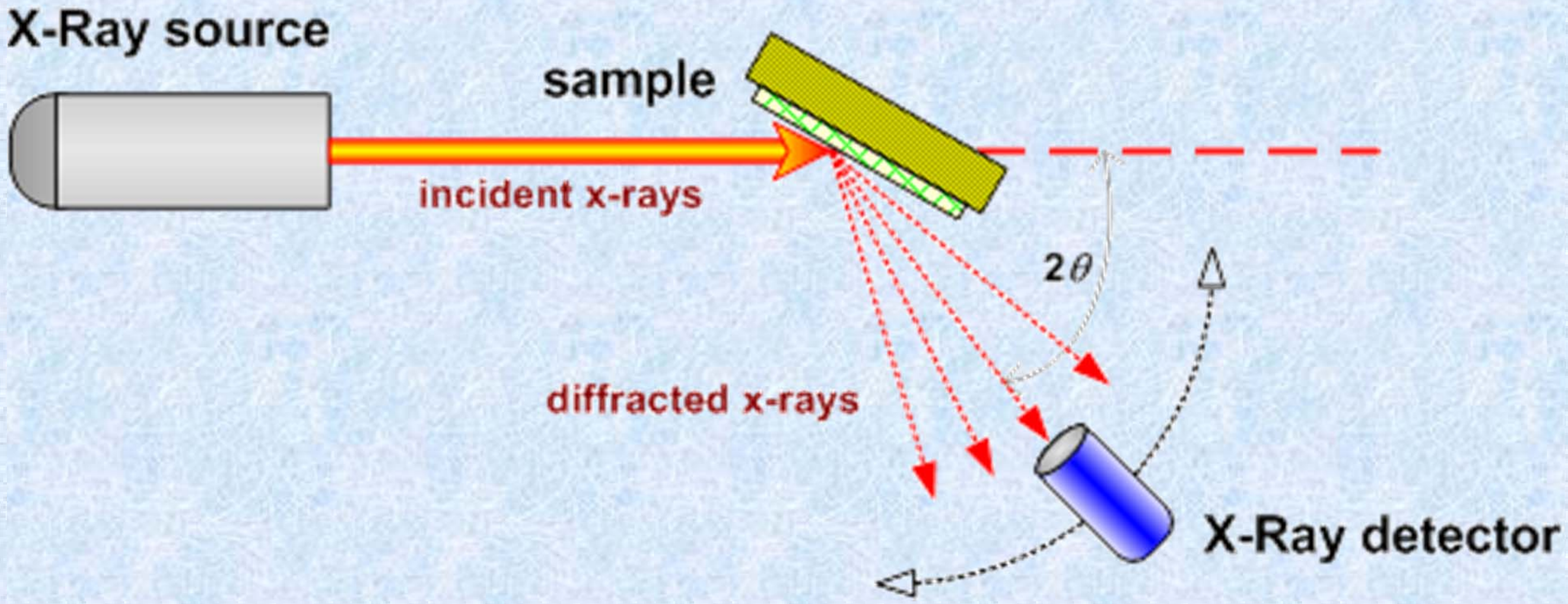
Analyze minerals by using Bragg's Law of Diffraction

$$n\lambda = 2d \times \sin(\Theta)$$

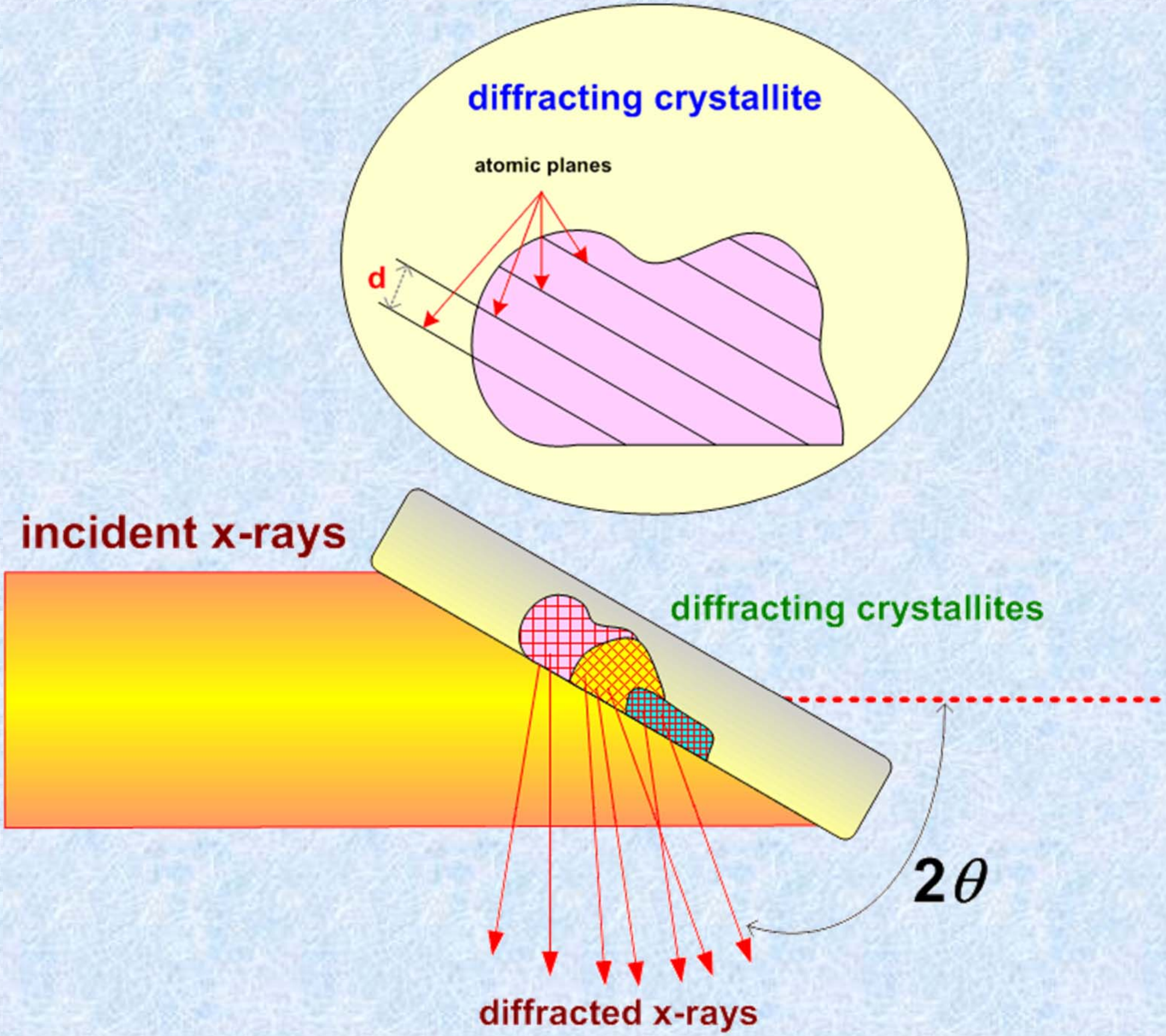
X-Ray Diffraction

Bragg's Law of Diffraction

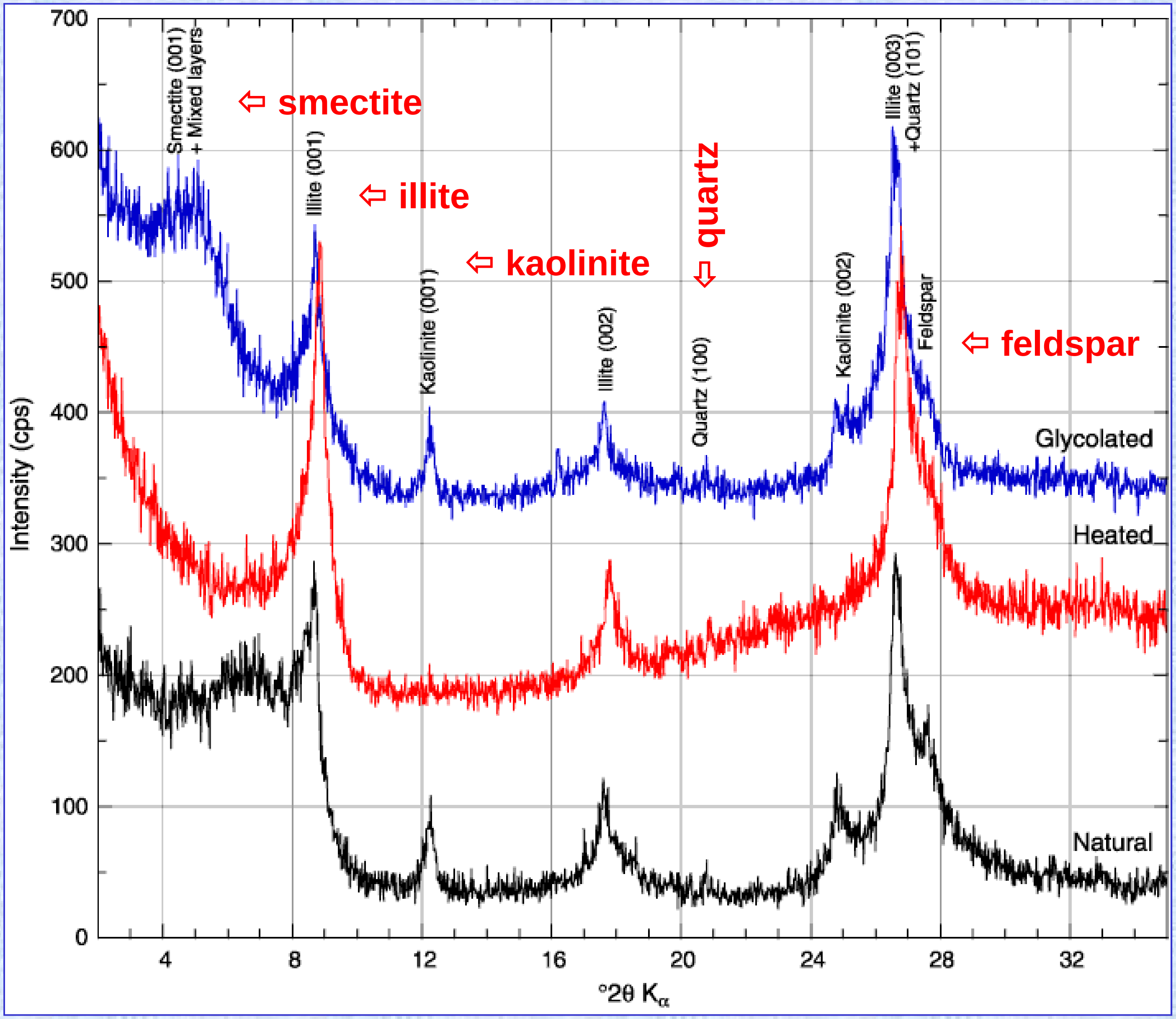
$n\lambda = 2d \times \sin(\Theta)$



X-Ray Diffraction



Pottery XRD



⇦ smectite

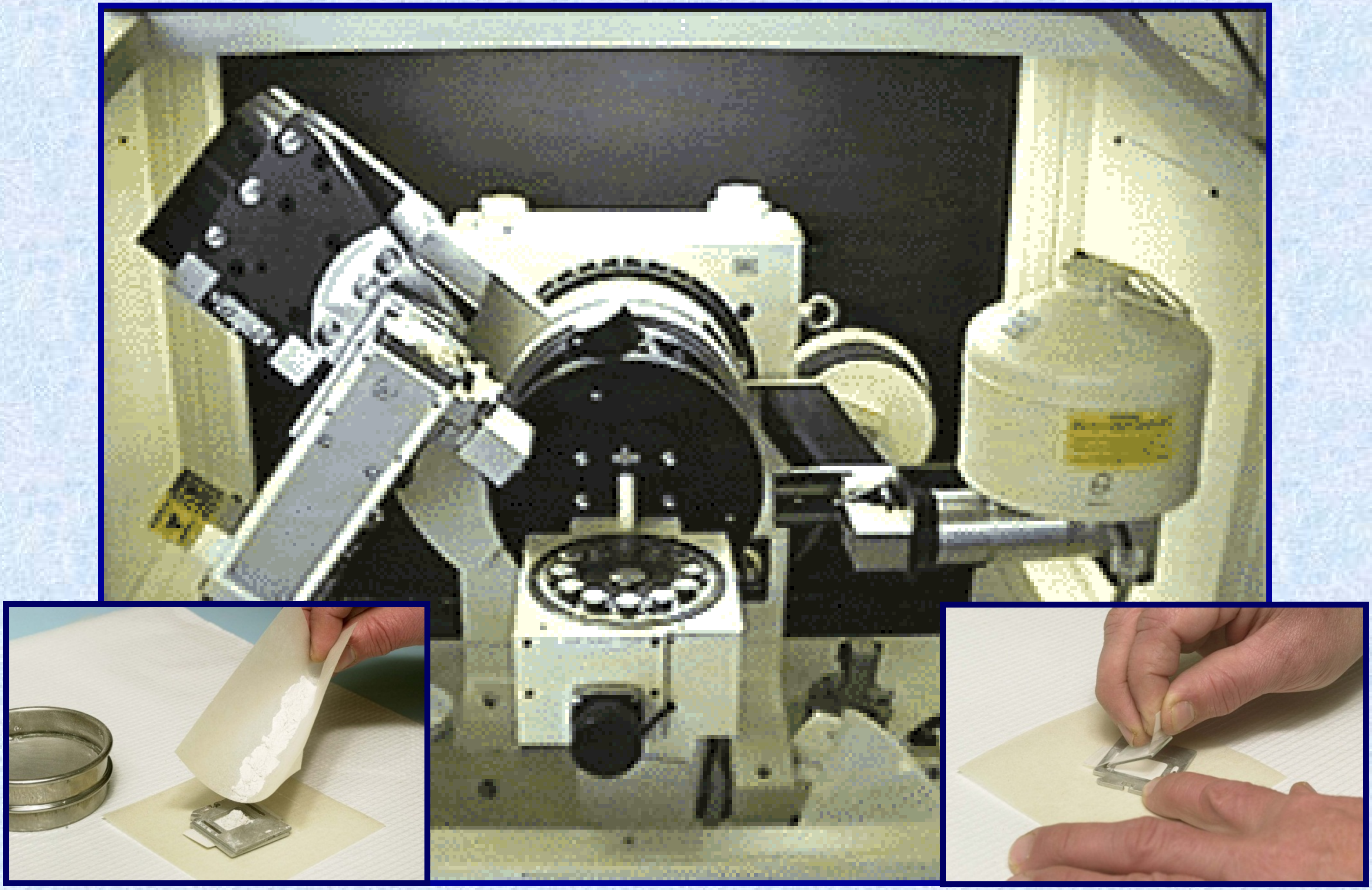
⇦ illite

⇦ kaolinite

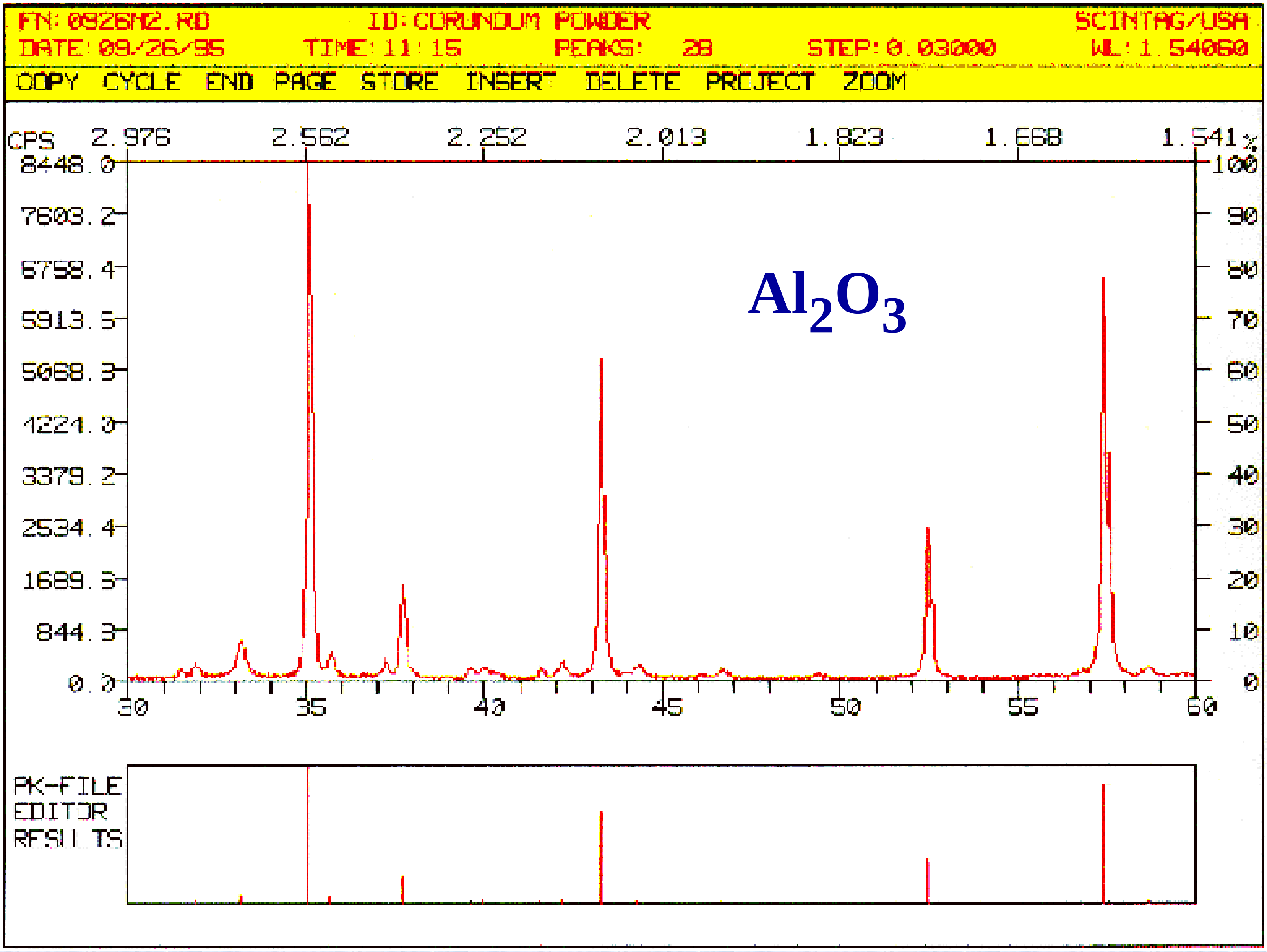
quartz ⇩

⇦ feldspar

X-Ray Diffractometer



X-Ray Powder Diffraction

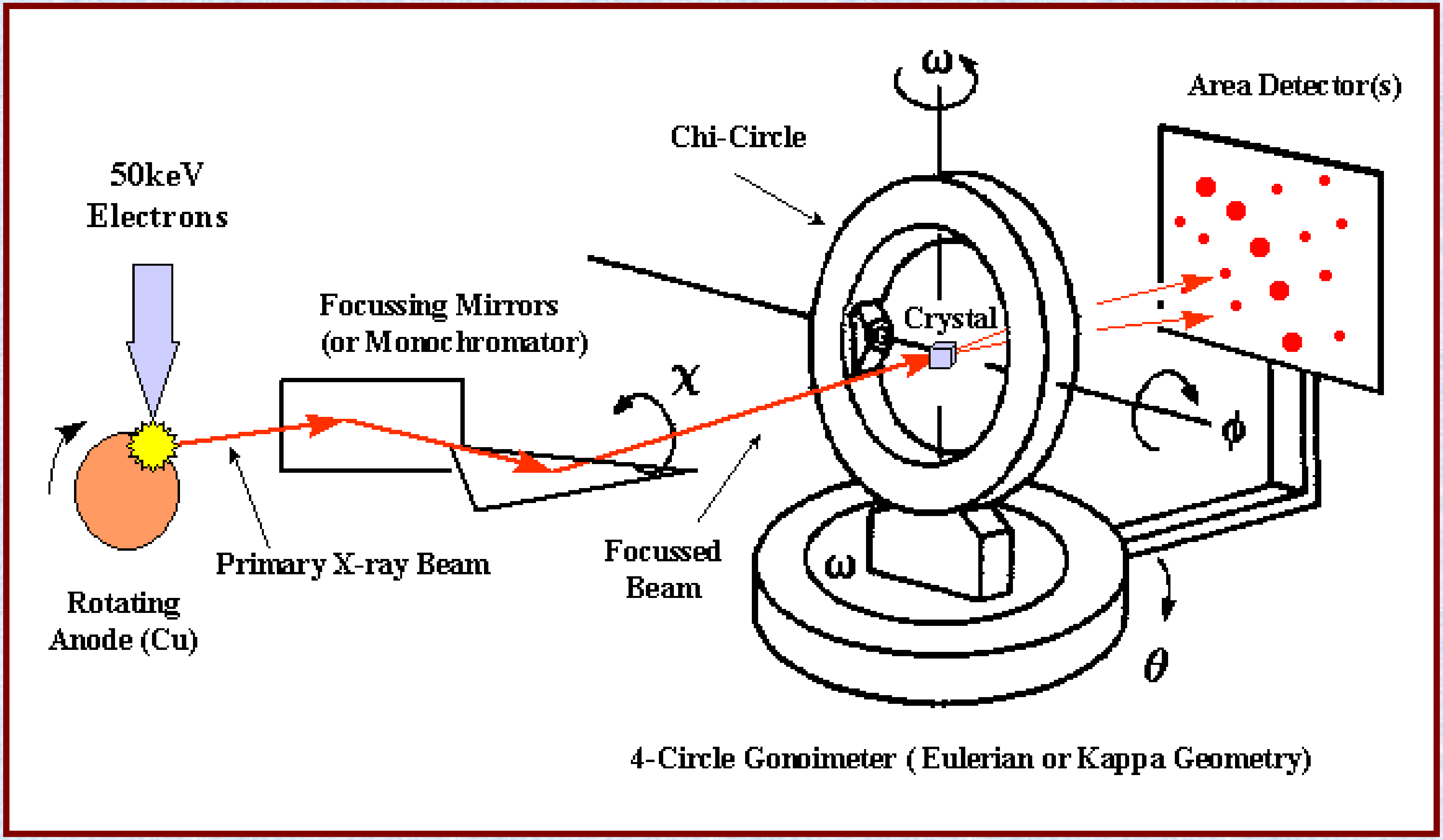


FN: 0926M2.RD ID: CORUNDUM POWDER SCINTAG/USA
DATE: 09/26/95 TIME: 11:15 PEAKS: 28 STEP: 0.03000 WL: 1.54060

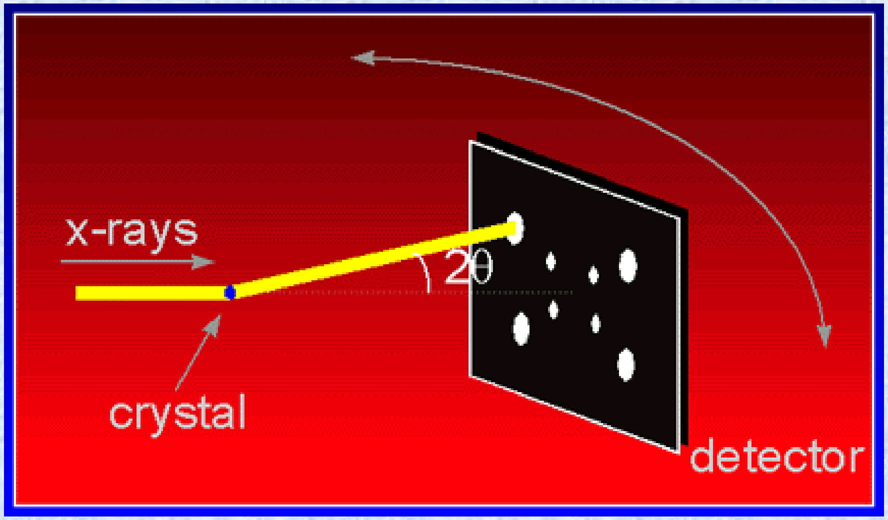
Al_2O_3

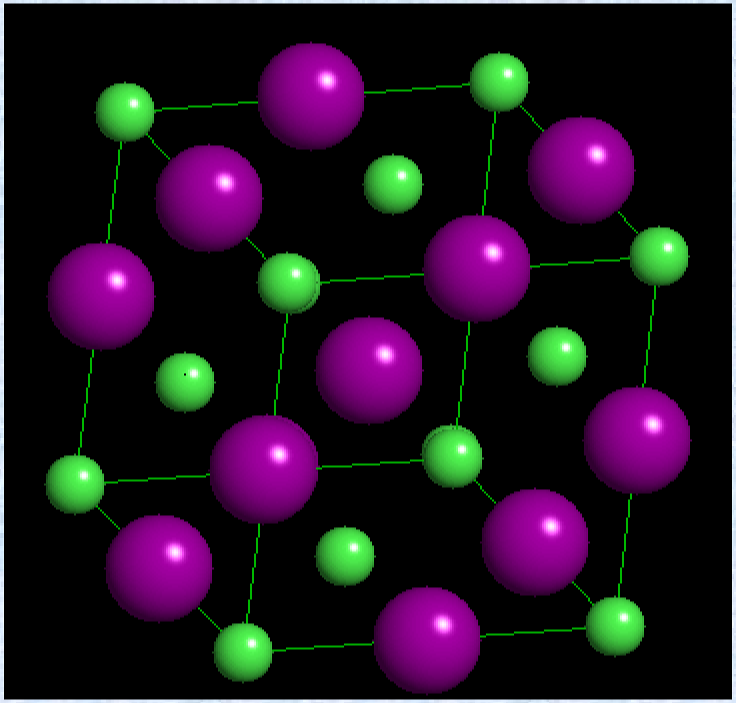
X-Ray Crystallography

www-structure.llnl.gov/Xray/101index.html

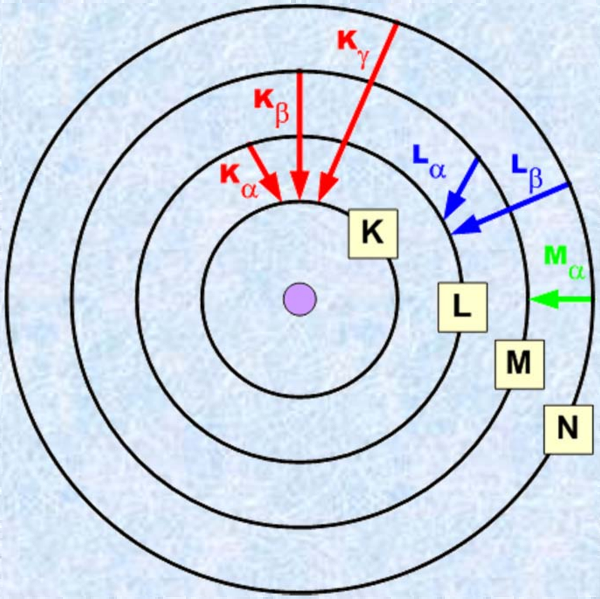


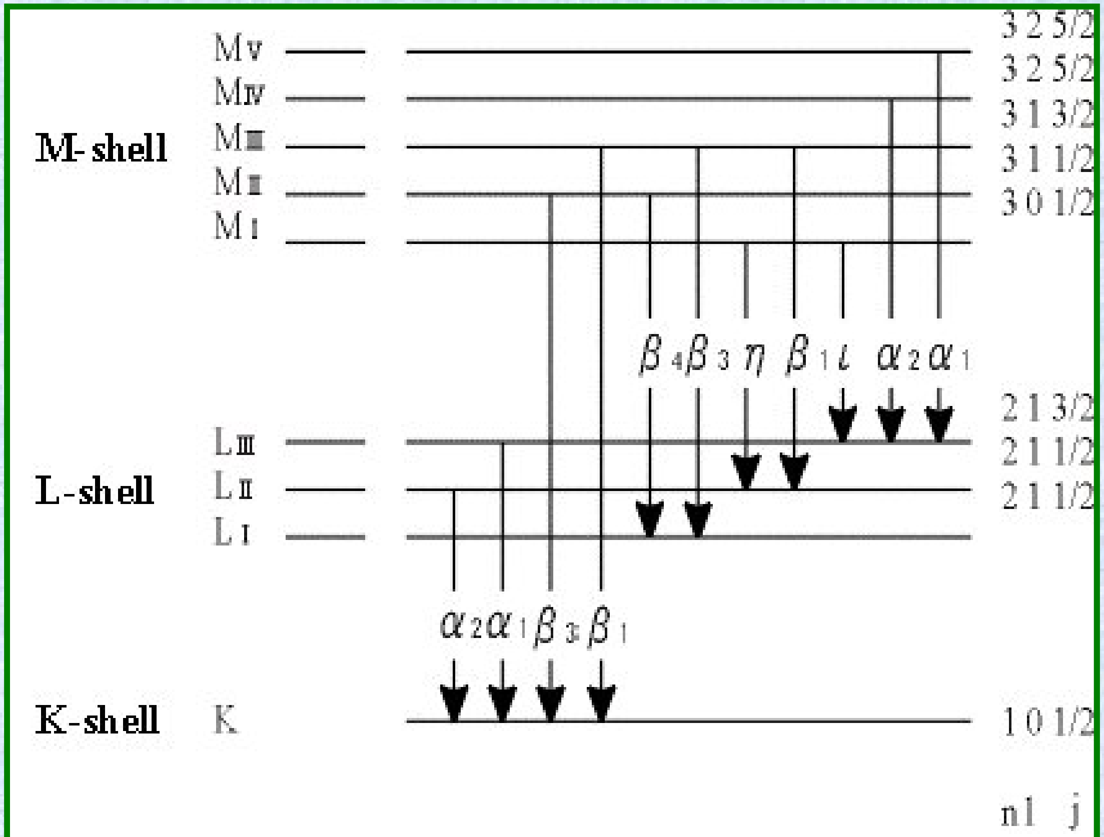
X-Ray Crystallography


x-rays
crystal
2θ
detector

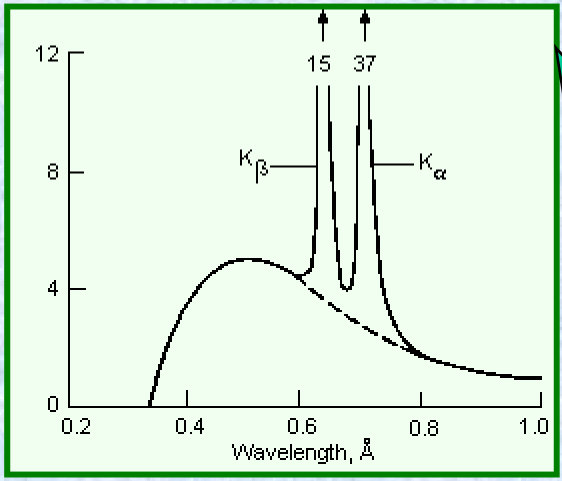


X-Ray Line Spectra





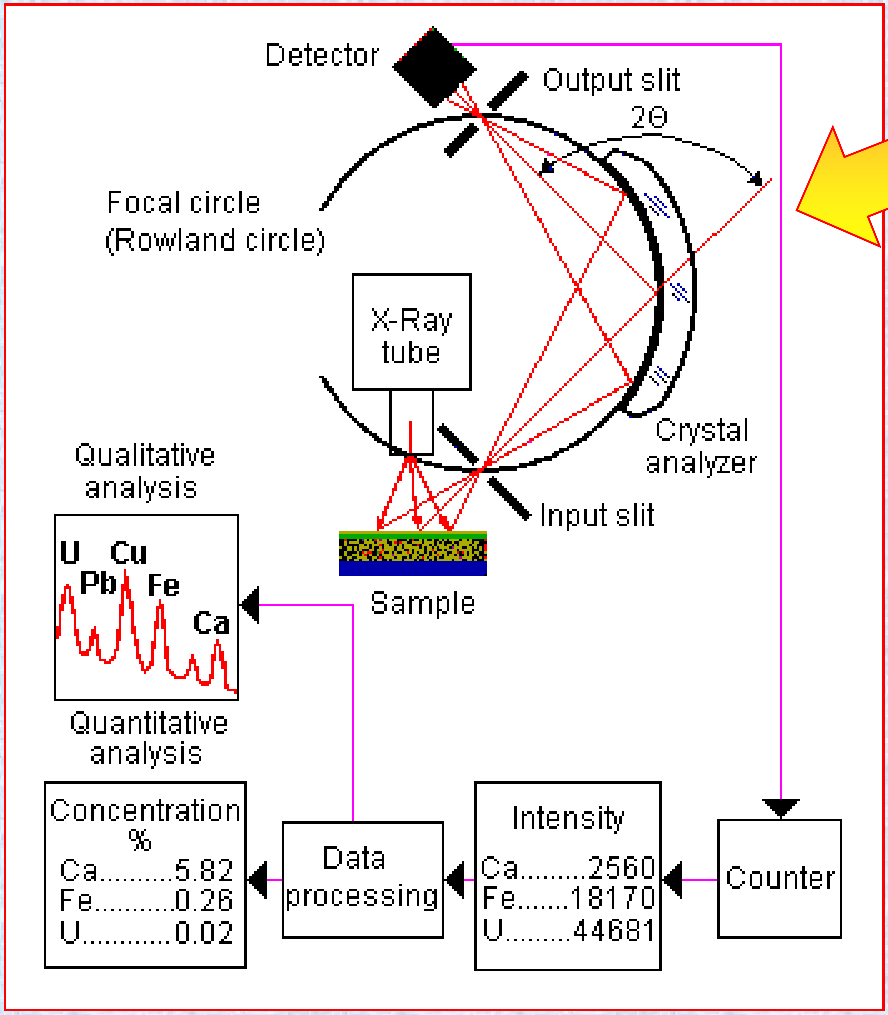
Energy level and characteristic X-ray



“line” X-ray spectrum

X-Ray Fluorescence Spectrometry

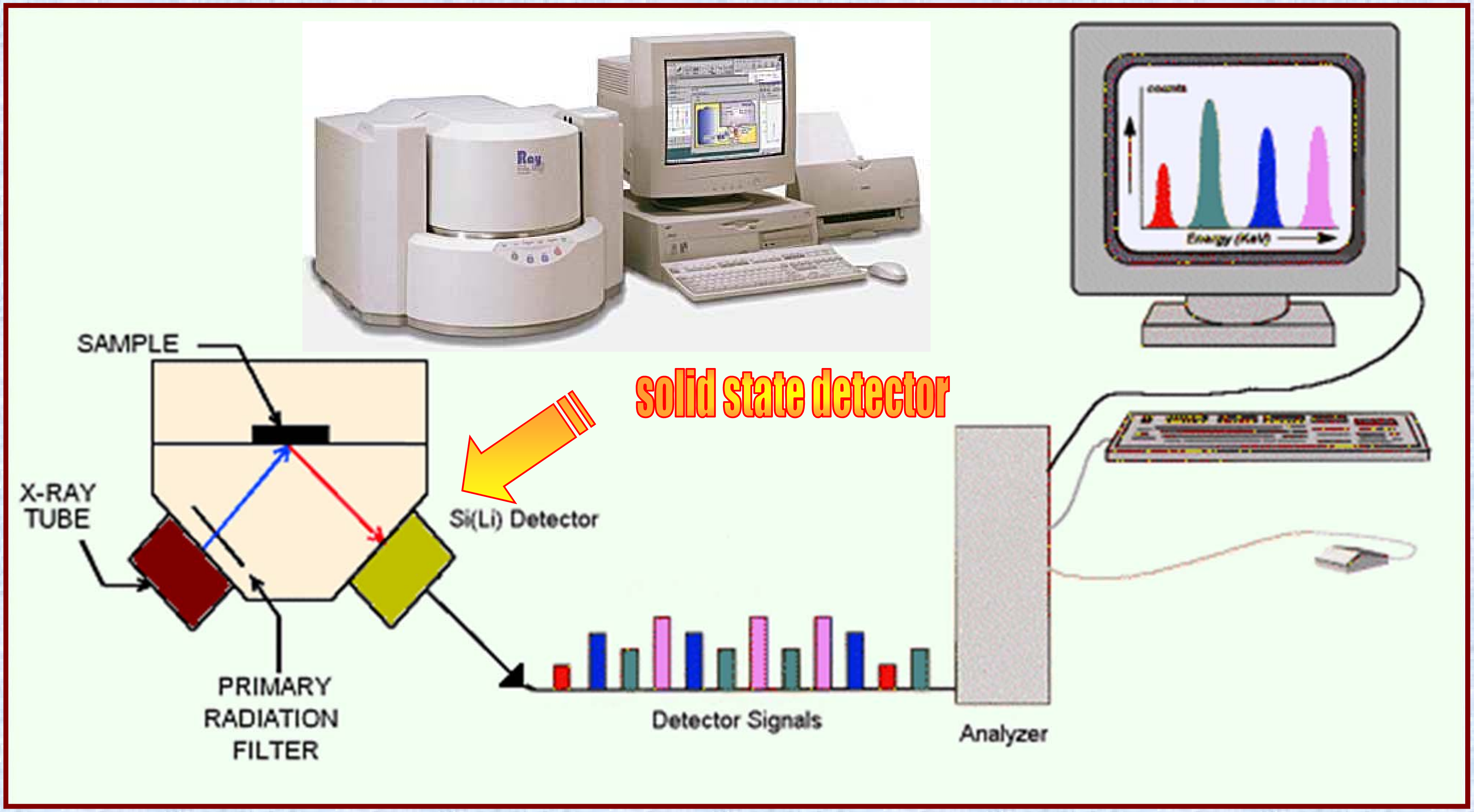
Wavelength Dispersive XRF



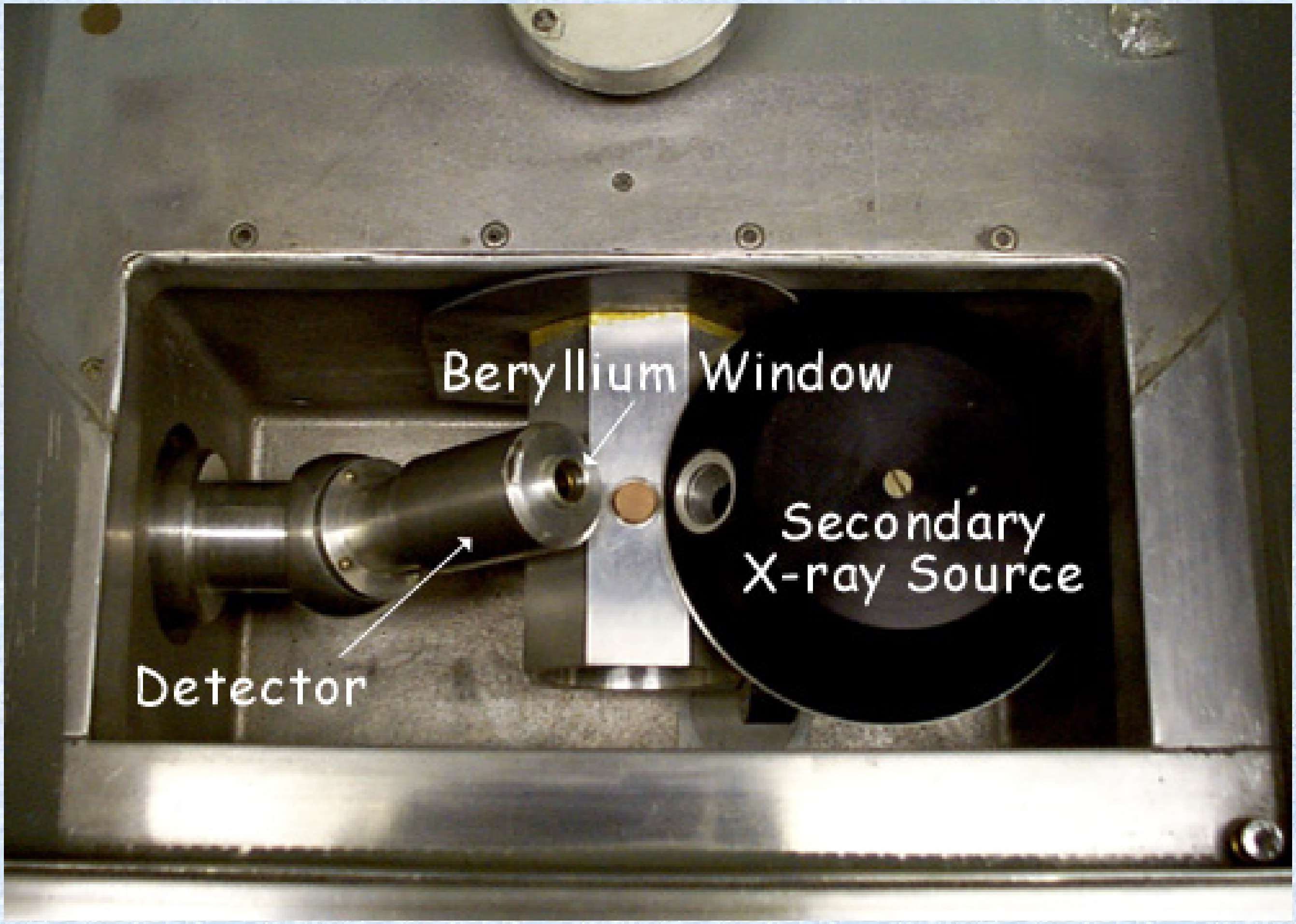
X-ray monochromator

X-Ray Fluorescence Spectrometry

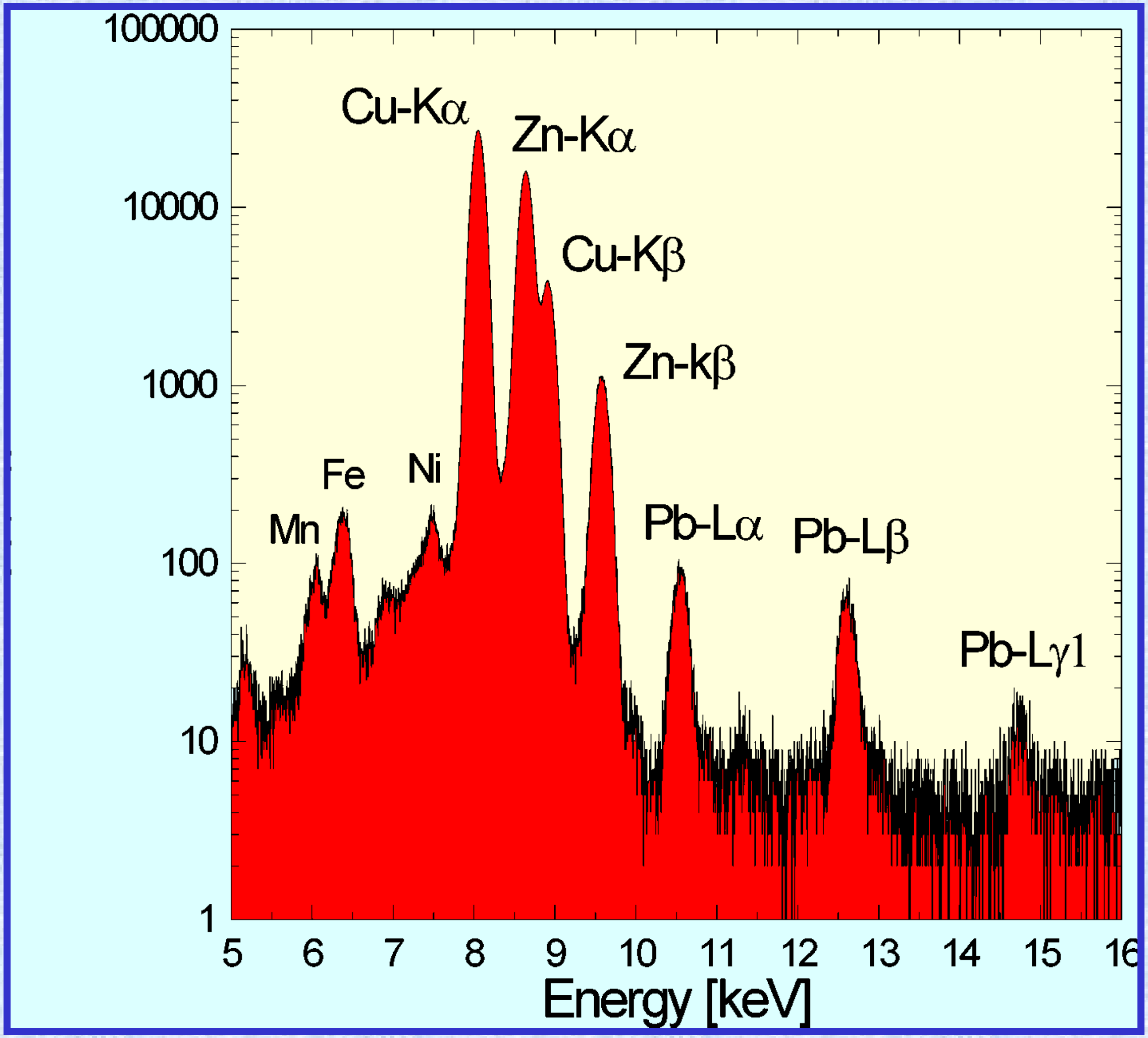
Energy Dispersive XRF



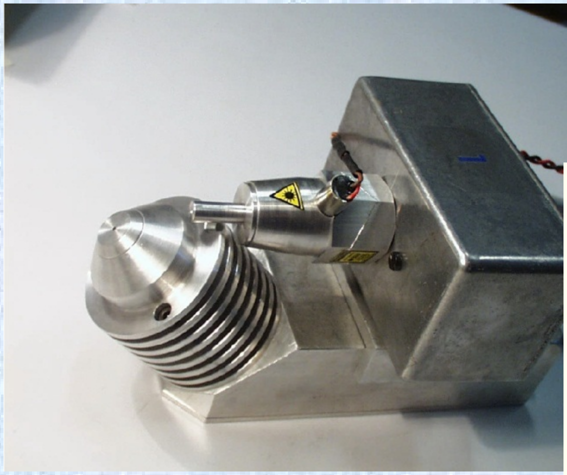
X-Ray Fluorescence Spectrometry

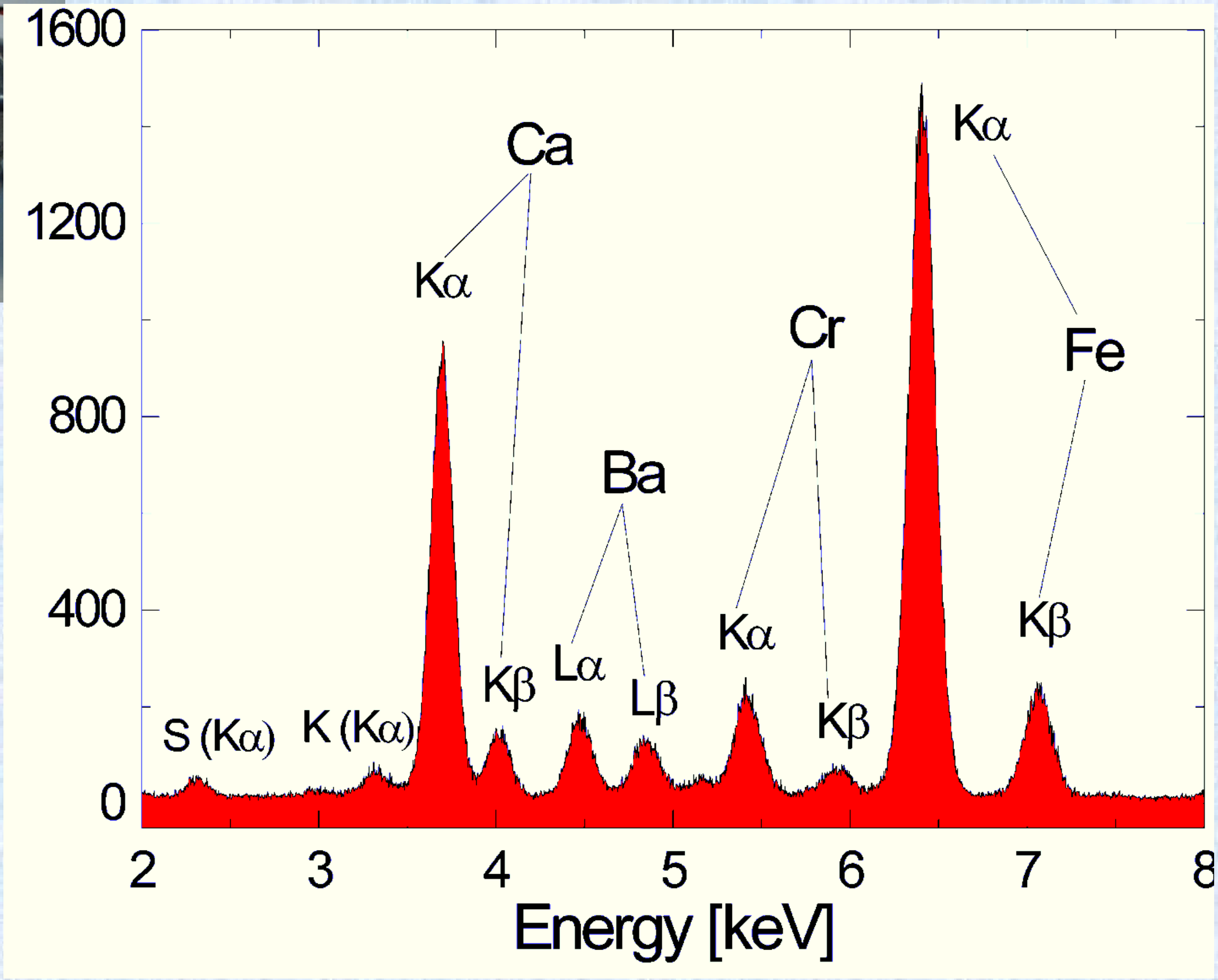


X-Ray Fluorescence Spectrum



PORTABLE XRF ANALYZERS





Fresco painting

Green vegetation

Original attribution: XVI century

Pigments:

Fe - ochre, always used

Cr - chromium green, introduced in 1860

Ba - barium white, introduced at the end of 1700

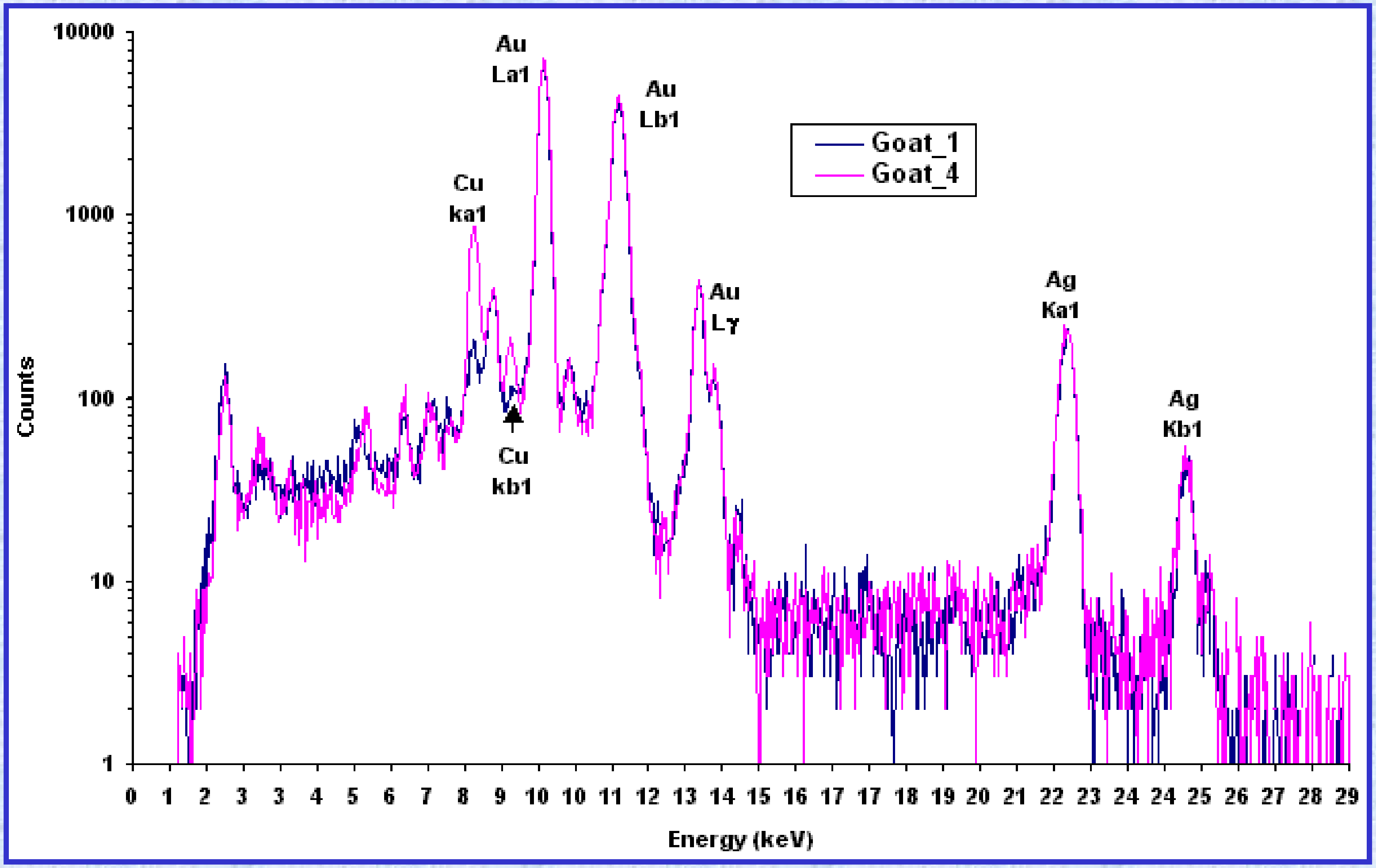
EDXRF and the Gold Ibex from Akrotiri, Greece

A gold ibex that was recently found at Akrotiri was analyzed using x-ray fluorescence techniques (XRF). Other artifacts were also analyzed, ranging from pottery to wall paintings and daggers.

The gold ibex was perhaps the most unique object analyzed in that it is the only artifact made of precious metals found at Akrotiri. All other precious objects were removed from the site by Thera inhabitants before the eruption that devastated the island and buried the settlement beneath meters of ash. It is also of note because it comes from an important public building which was in close proximity to the famous Xeste 3 building where the rituals for the young boys and girls of the local elite class took place. The fact that the ibex was found *in-situ* makes its importance even greater since this object is stylistically unique and it could be considered even as fake in the international art market.

Unique of its kind, it was discovered in mint condition, inside a wooden box inside a clay chest (larnax), next to a large pile of horns, mainly of goats. Excavation of the find-spot is still in progress and it is therefore too early to draw conclusions about the figurine's significance (I would say that it was offered as a gift from someone coming from the East or that it is a sacrificial object related to worship or other rituals). The figure is hollow and was cast by the "lost wax" method. The legs, neck, and tail of the animal were soldered on after the removal of the inside core. In the finishing process the figure was hammered, as deduced from the tool-marks."

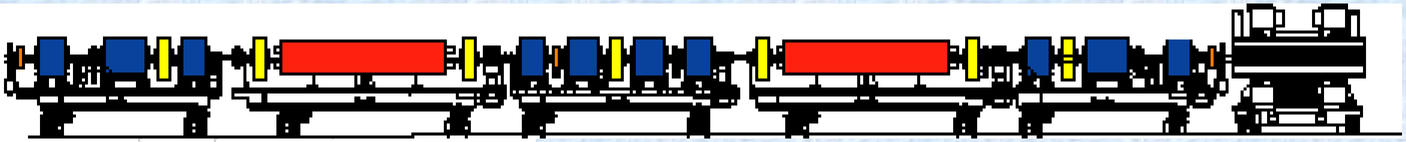
EDXRF and the Gold Ibex from Akrotiri, Greece

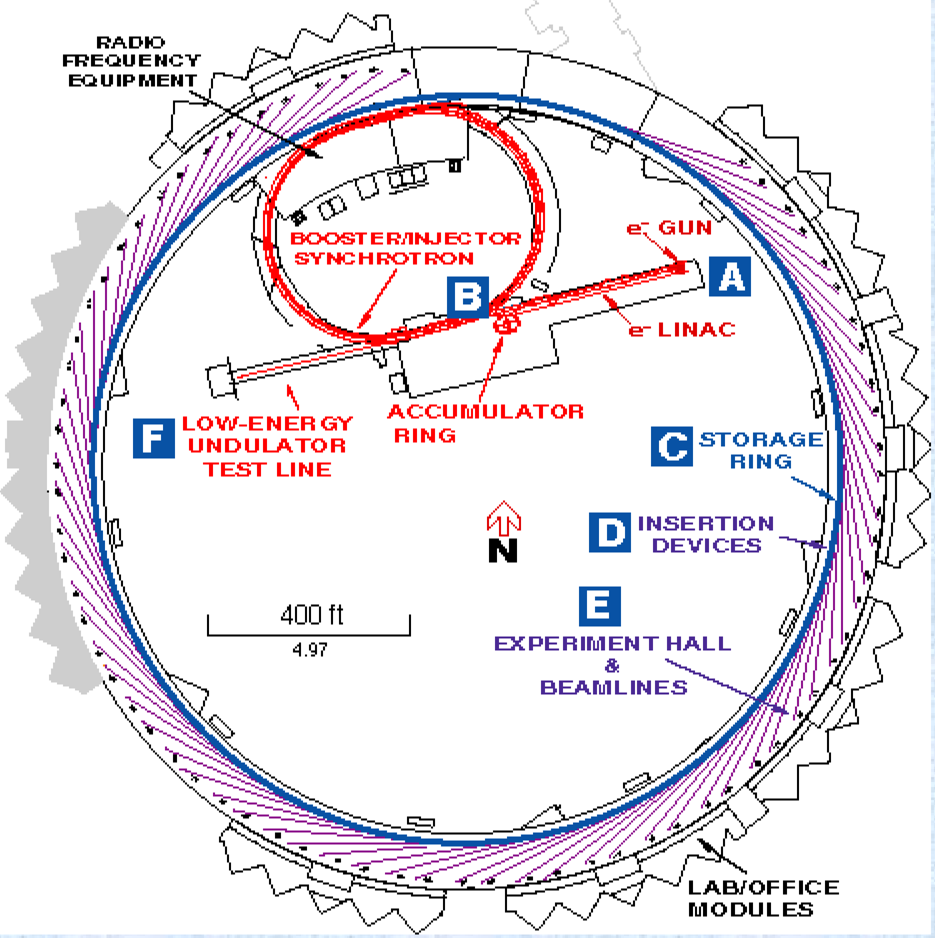


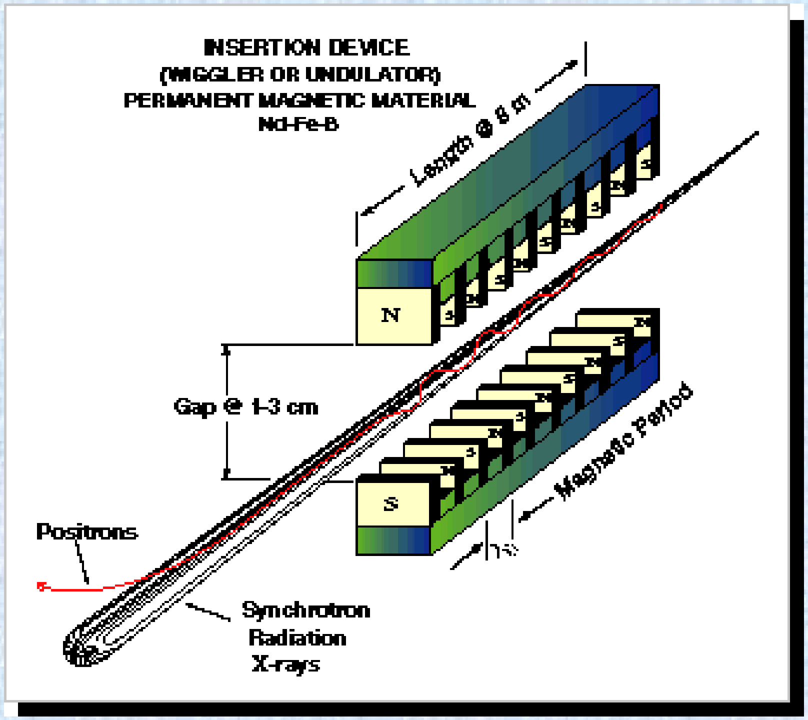
Two spectra taken from the Gold Ibex are shown above. The Goat_1 spectrum was taken from just above the front left leg. This position is free of visual contaminants and considered to be the base spectrum. The Goat_4 spectrum is from the braze/weld of the tail. It shows an increase in the amount of copper.

ADVANCED LIGHT SOURCES

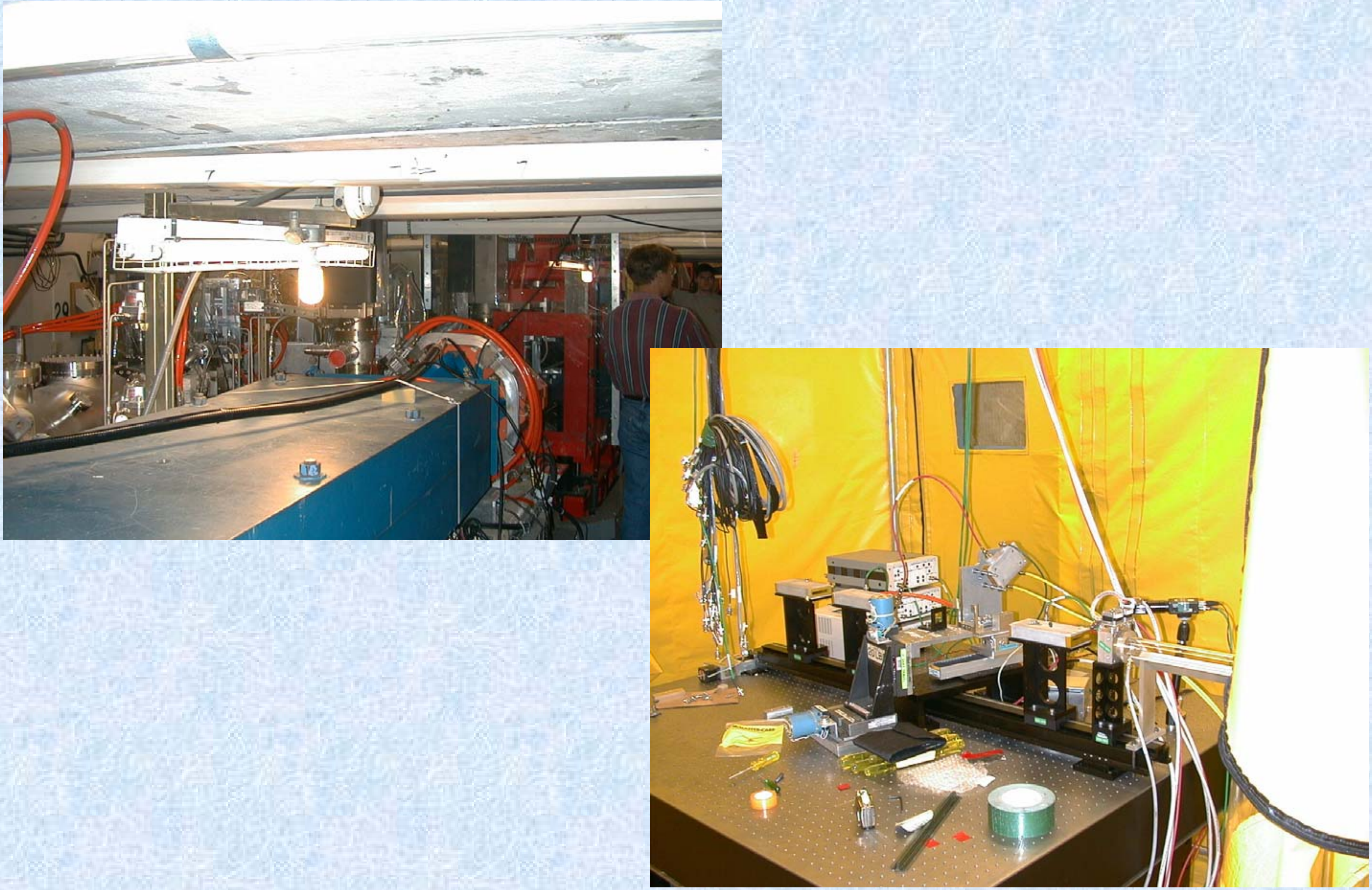
ADVANCED LIGHT SOURCES



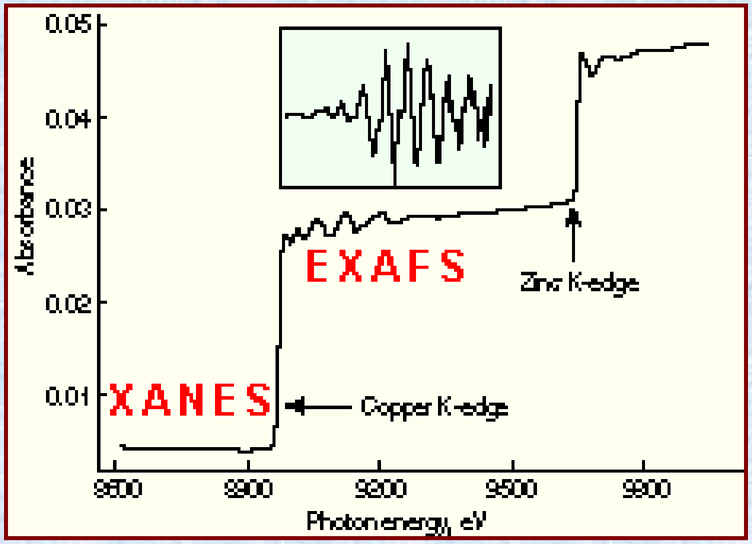




ADVANCED LIGHT SOURCES



ADVANCED LIGHT SOURCES



XANES

- peaks and shoulders in the rising edge: about electronic configuration, bonds, and symmetry
- position of the edge: oxidation state of the absorber

EXAFS

- type of back-scattering atoms surrounding the central atom
- number of back-scattering atoms
- distance of back-scattering atoms to the central atom
- disorder of back-scattering atoms

X-RAY ABSORPTION NEAR EDGE STRUCTURE (XANES)

XANES features are intense, concentrated in a small energy region and exhibit weak temperature sensitivity.

The XANES region gives extensive chemical information in the form of

valence and charge transfer,

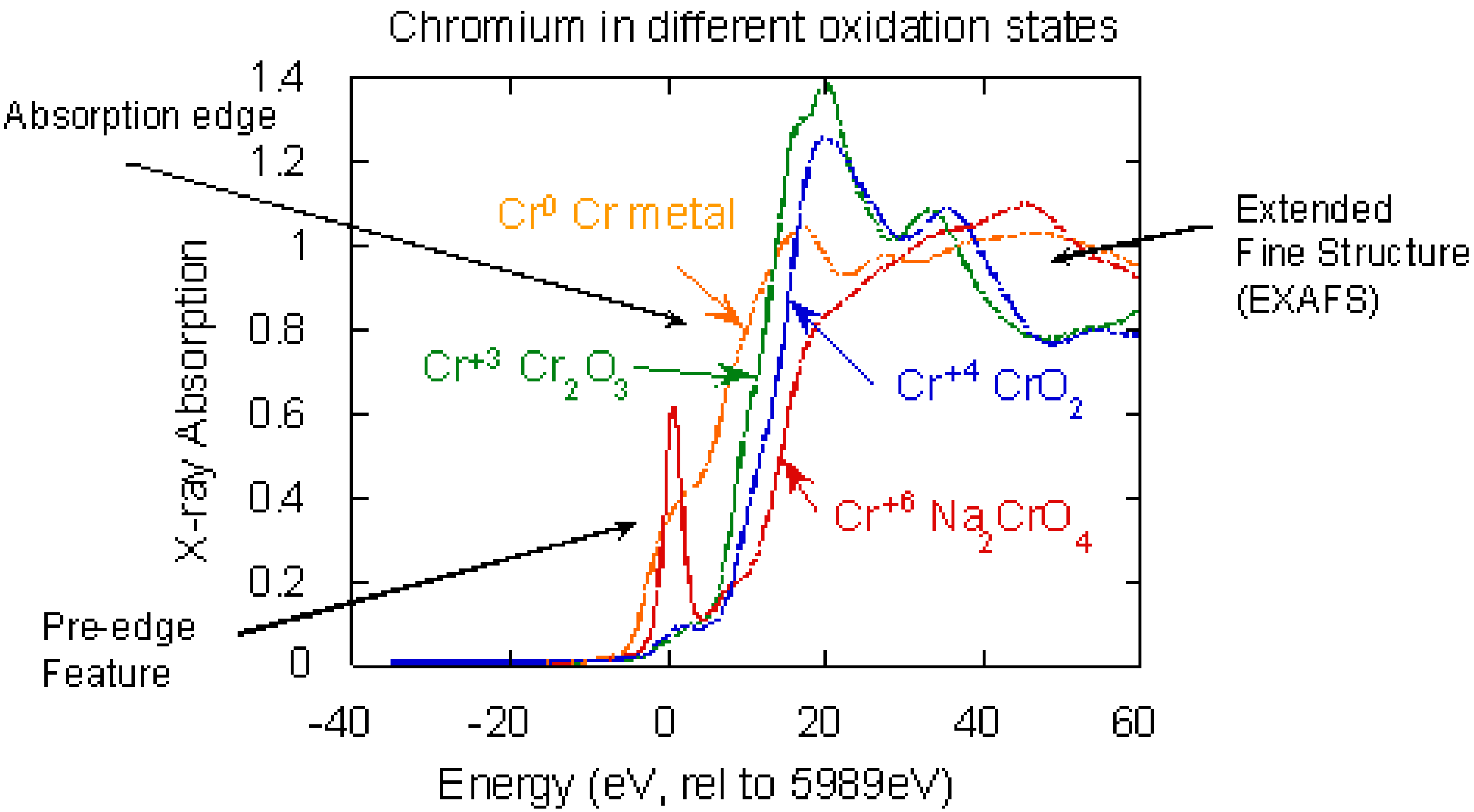
bond lengths,

number and type of nearest neighbors.

Most of the XANES work published is qualitative in nature. Use is made, for example, of the relative height of a pre-edge peak, the position of the inflection point, the intensity of the white line, or the presence or absence of some feature. A quantitative description of XANES is desperately needed.

X-RAY ABSORPTION NEAR EDGE STRUCTURE (XANES)

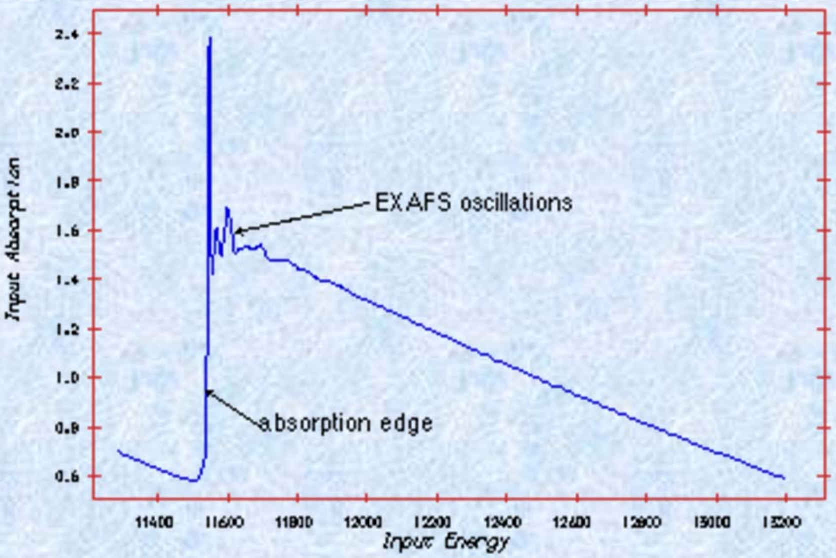
XANES Spectra



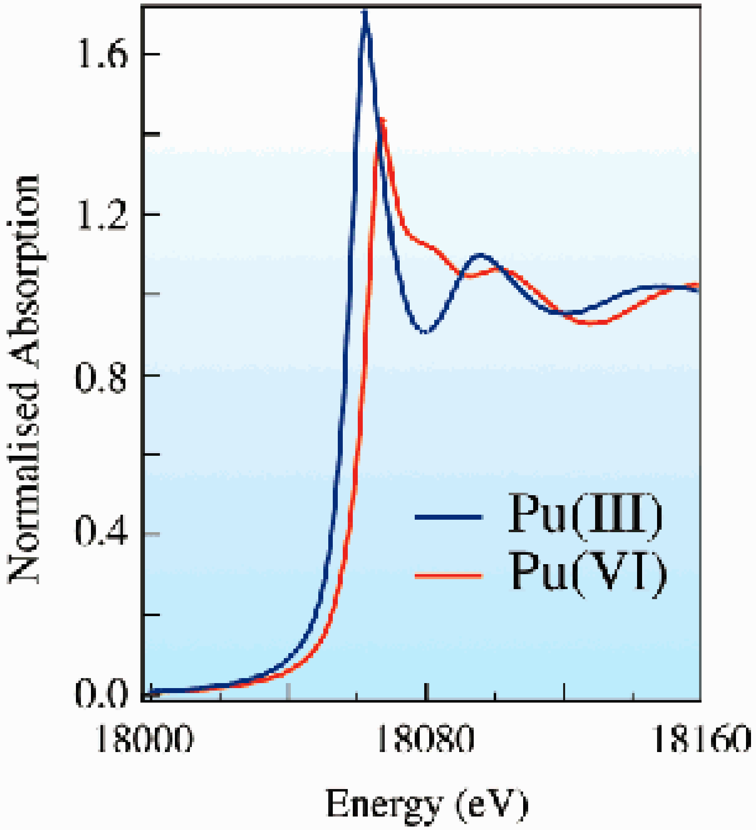
EXTENDED X-RAY ABSORPTION FINE STRUCTURE (EXAFS)

EXAFS spectra are a plot of the value of the absorption coefficient of a material against energy over a 500 - 1000 eV range (including an absorption edge near the start of the spectrum).

Through careful analysis of the oscillating part of the spectrum after the edge, information relating to the coordination environment of a central excited atom can be obtained.

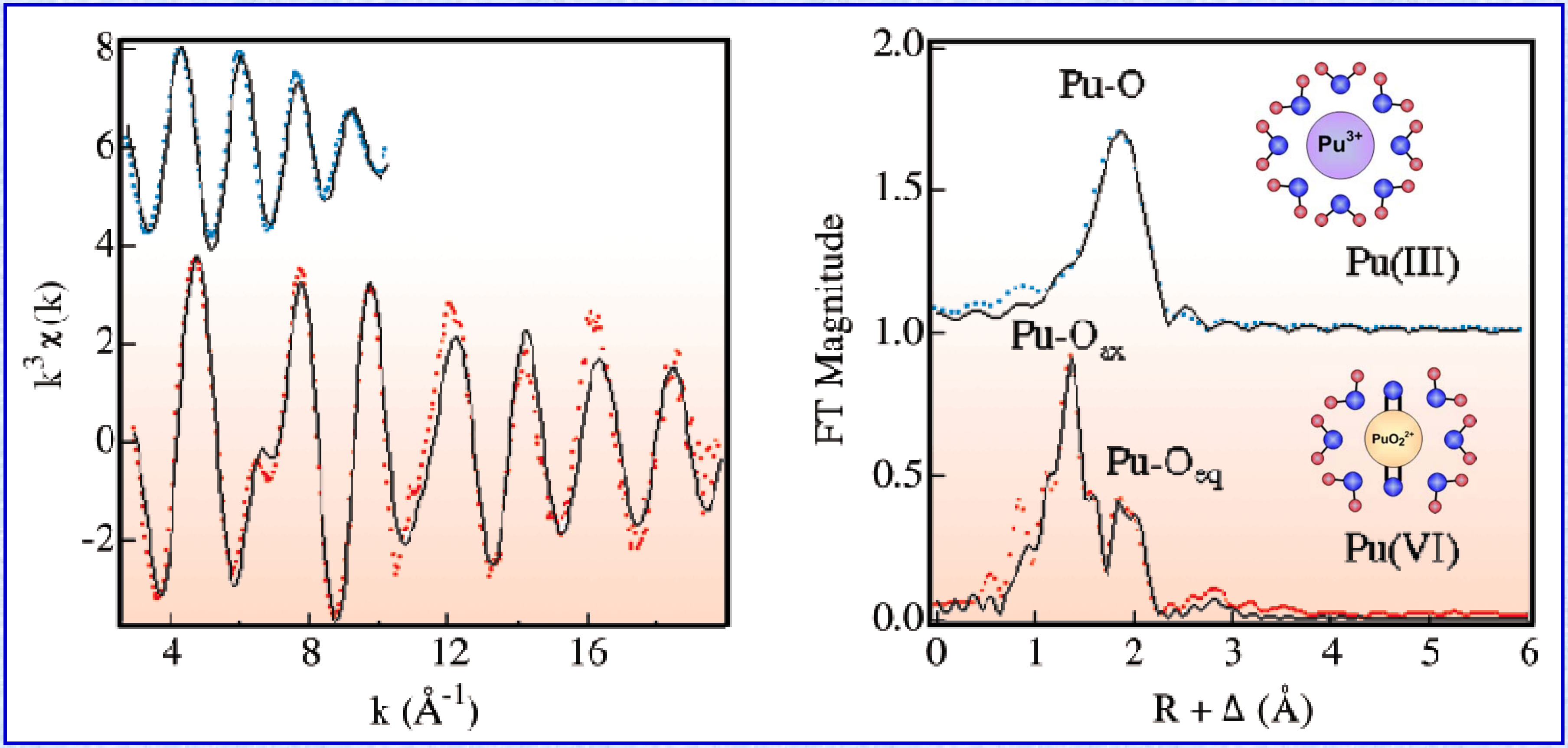


EXTENDED X-RAY ABSORPTION FINE STRUCTURE (EXAFS)

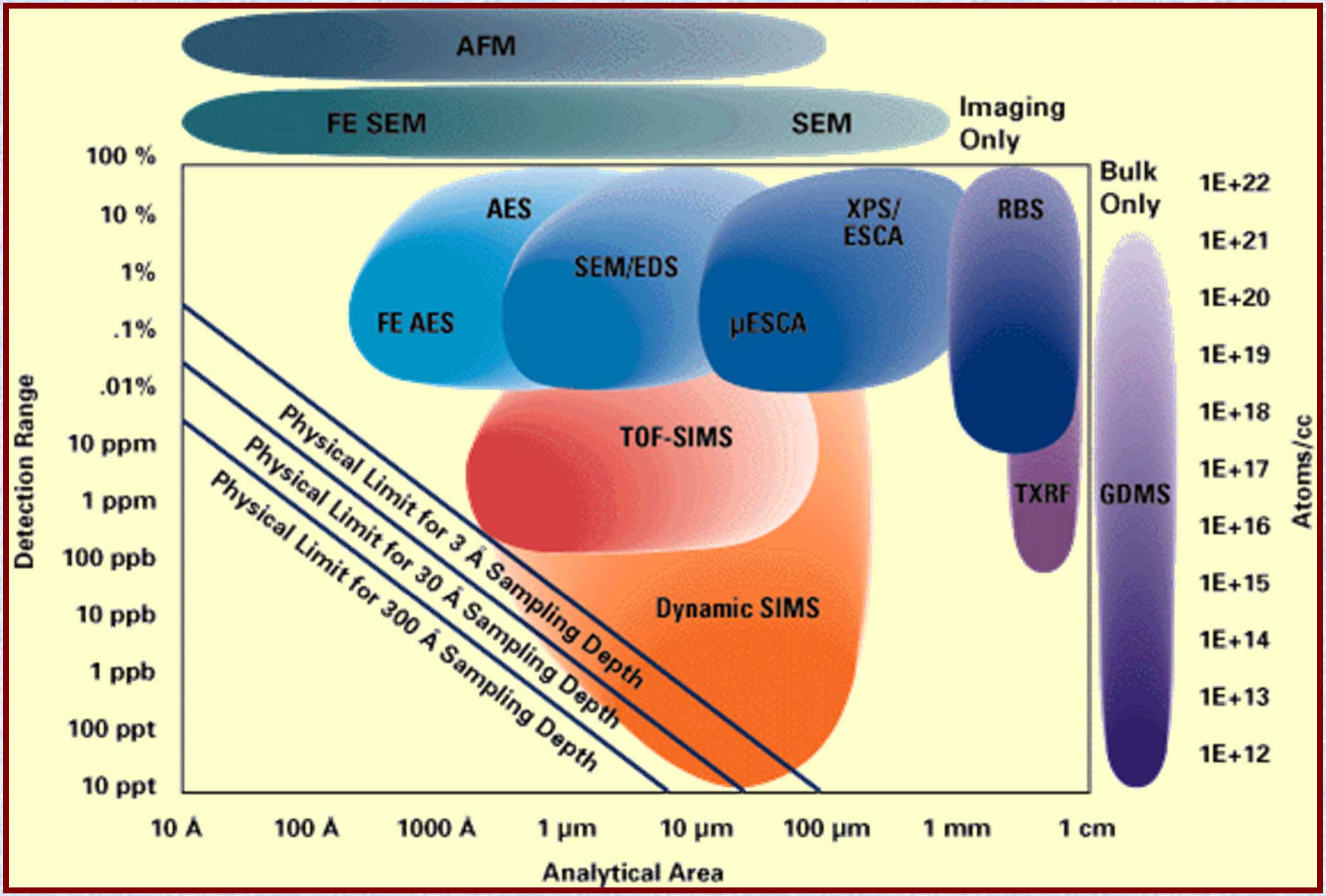


EXTENDED X-RAY ABSORPTION FINE STRUCTURE (EXAFS)

EXAFS of Hydrated Plutonium



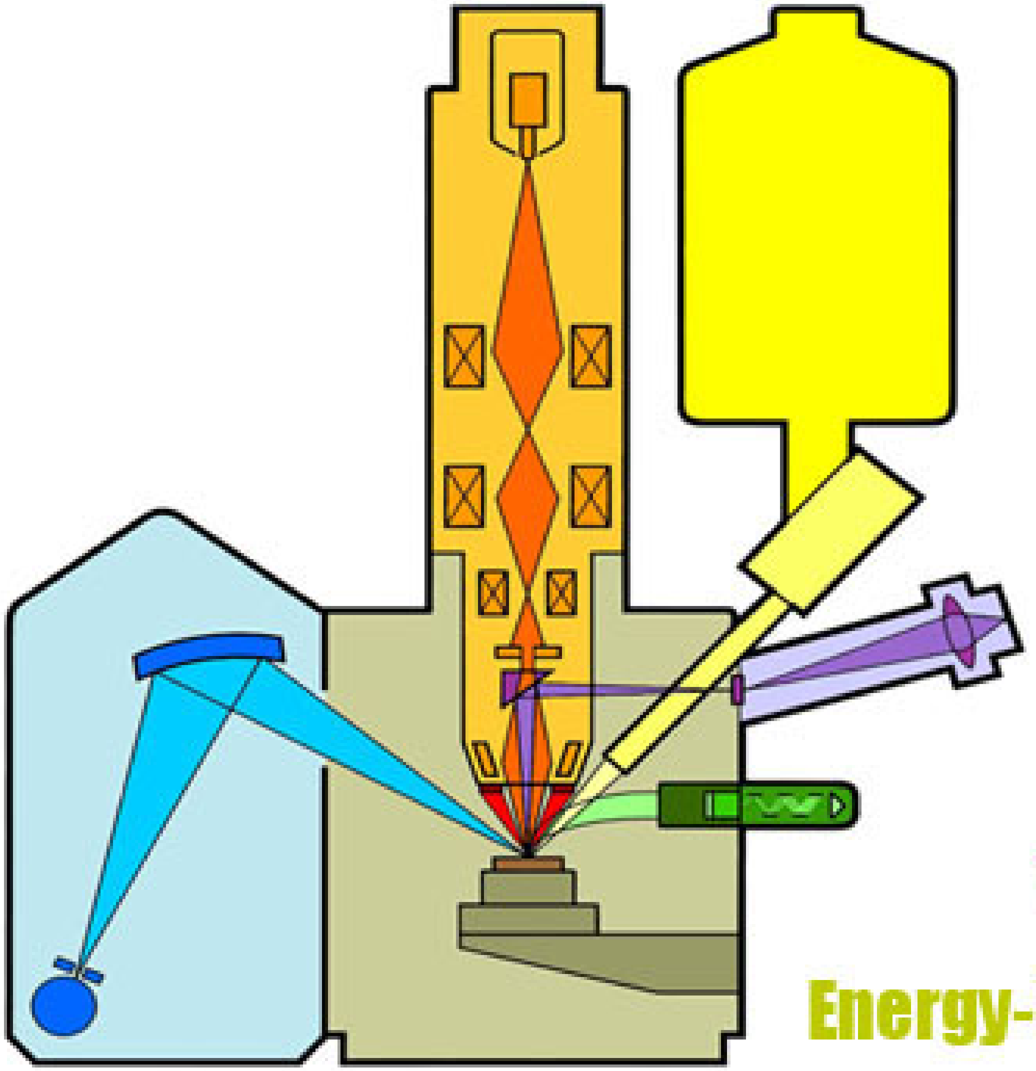
Surface Analysis Techniques



Surface Analysis Techniques

AES	**Auger Electron Spectroscopy**
EDX	**Energy Dispersive X-ray Analysis**
ESCA	**Electron Spectroscopy for Chemical Analysis**
EXAFS	**Extended X-ray Absorption Fine Structure**
LEED	**Low Energy Electron Diffraction**
PIXE	**Proton Induced X-Ray Emission**
RBS	**Rutherford Back Scattering**
SIMS	**Secondary Ion Mass Spectrometry**
XANES	**X-ray Absorption Near-Edge Structure**
XPS	**X-ray Photoemission Spectroscopy**

Electron Microprobe



Electron Column

Sample Chamber

Secondary Electron Detector

Backscattered Electron Detector

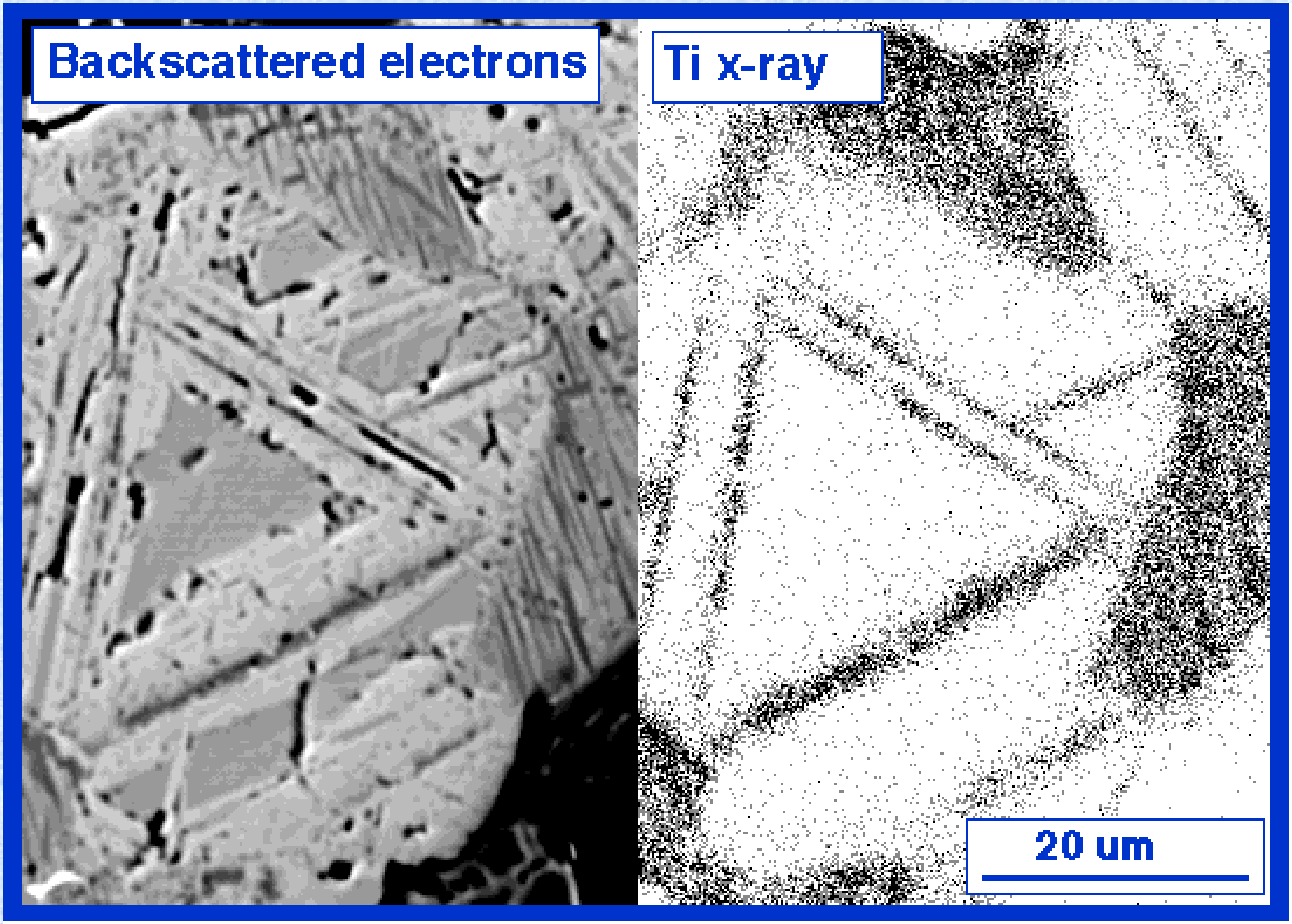
Wavelength-Dispersive Spectrometers

Visible-Light Microscope

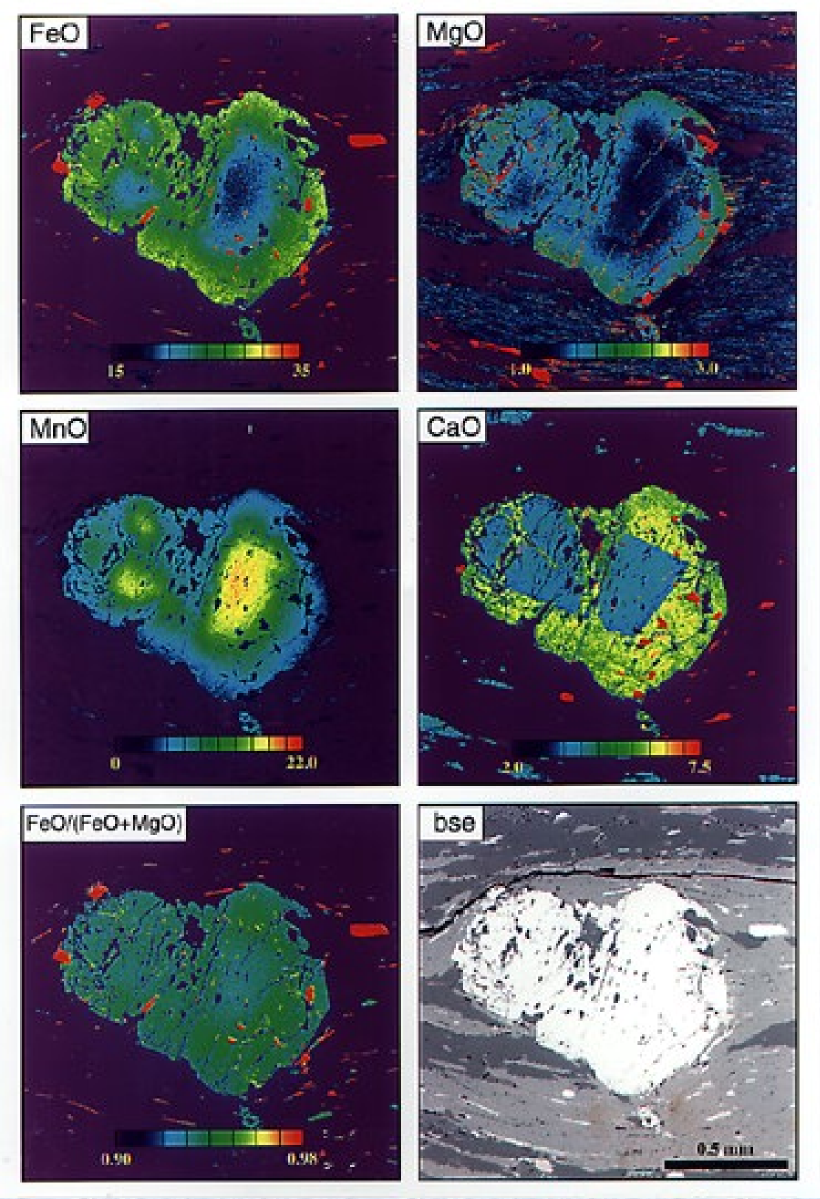
Energy-Dispersive Spectrometer

Electron Microprobe

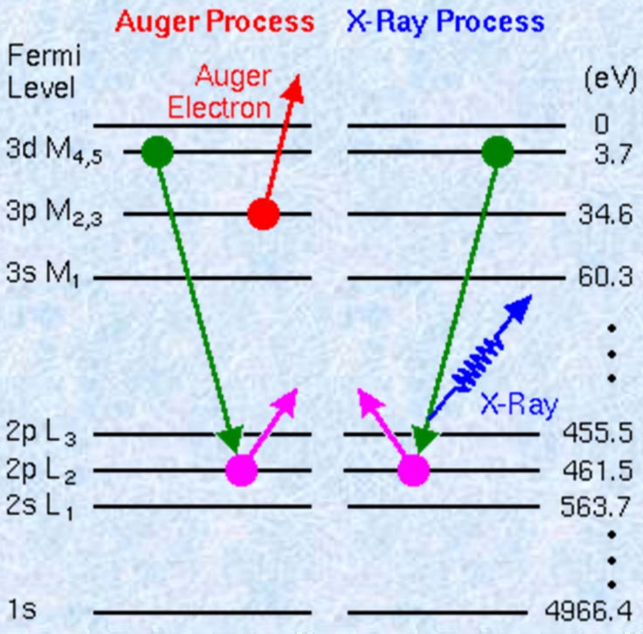
Electron Microprobe



Electron Microprobe



Auger Electron Spectroscopy



Competing paths for energy dissipation in titanium as an example.

LMM Auger electron energy is ~423.2 eV

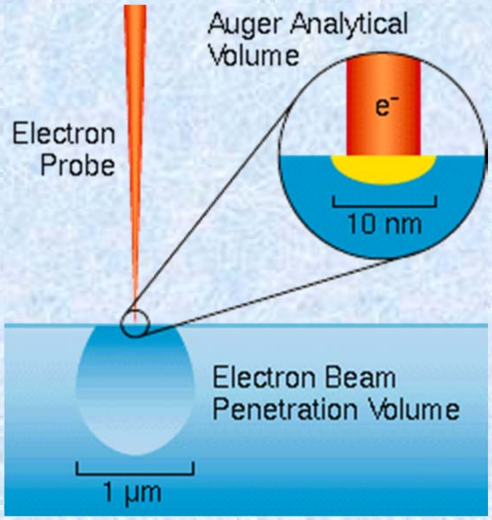
$$E_{Auger} = E_{L2} - E_{M4} - E_{M3}$$

$$461.5 - 3.7 - 34.6 = 423.2$$

X-ray photon energy is *~457.8 eV*

$$E_{h\nu} = E_{L2} - E_{M4}$$

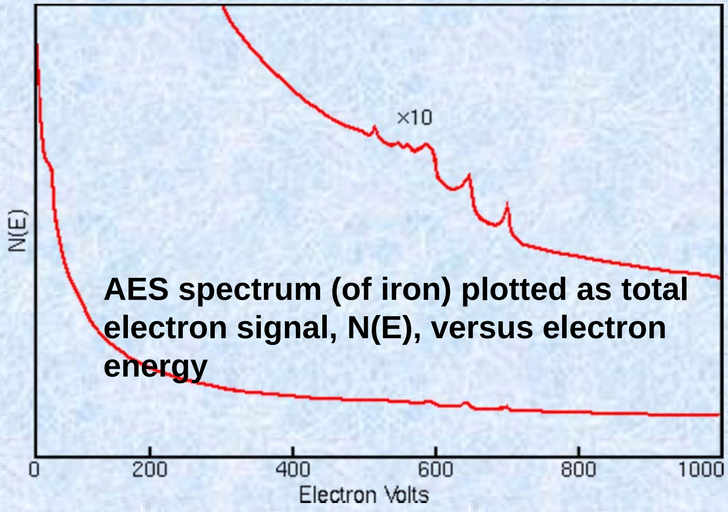
$$461.5 - 3.7 = 457.8$$



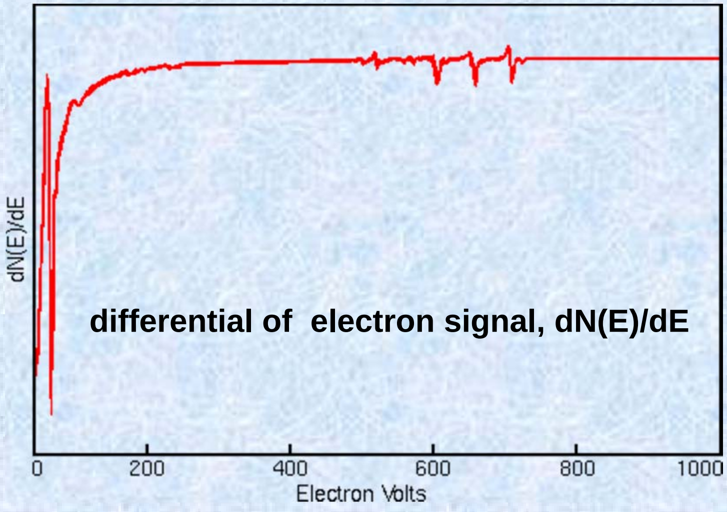
X-ray analytical volume increases with electron beam energy and decreases for materials with higher atomic numbers.

Auger Electron Spectroscopy

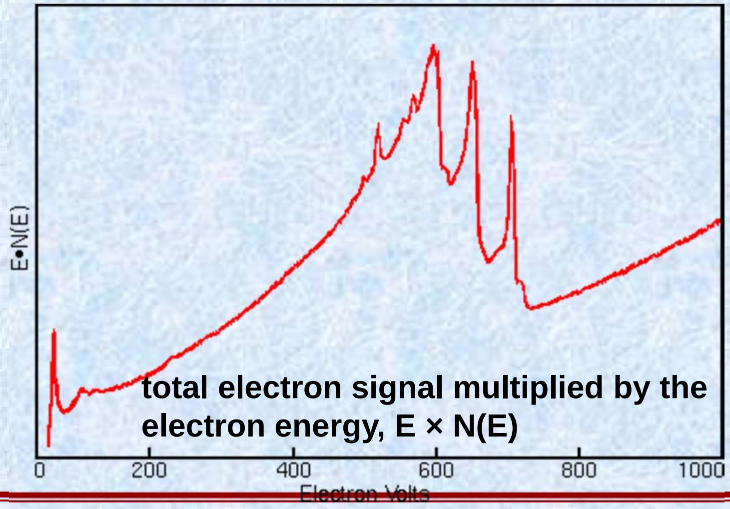
Data manipulation and display algorithms



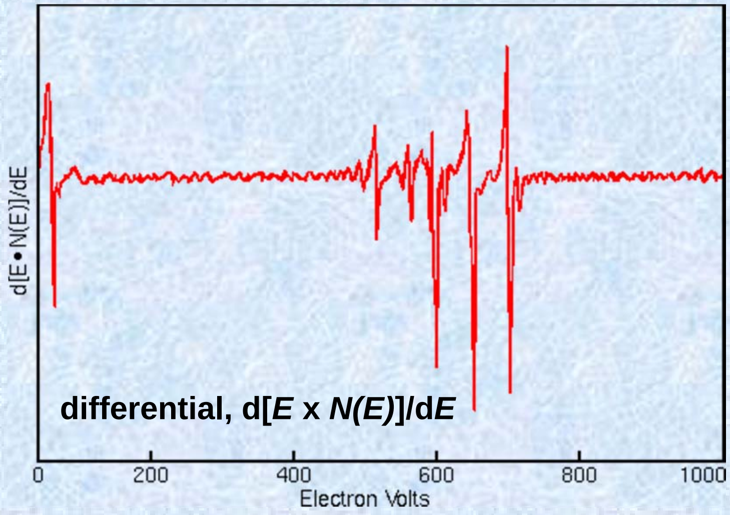
AES spectrum (of iron) plotted as total electron signal, N(E), versus electron energy



differential of electron signal, dN(E)/dE



total electron signal multiplied by the electron energy, E × N(E)



differential, d[*E* x *N(E)*]/d*E*

Auger Electron Spectroscopy

Easily characterized

Metals, semiconductors, thin films

Difficult to impossible

Glasses/ceramics, polymers

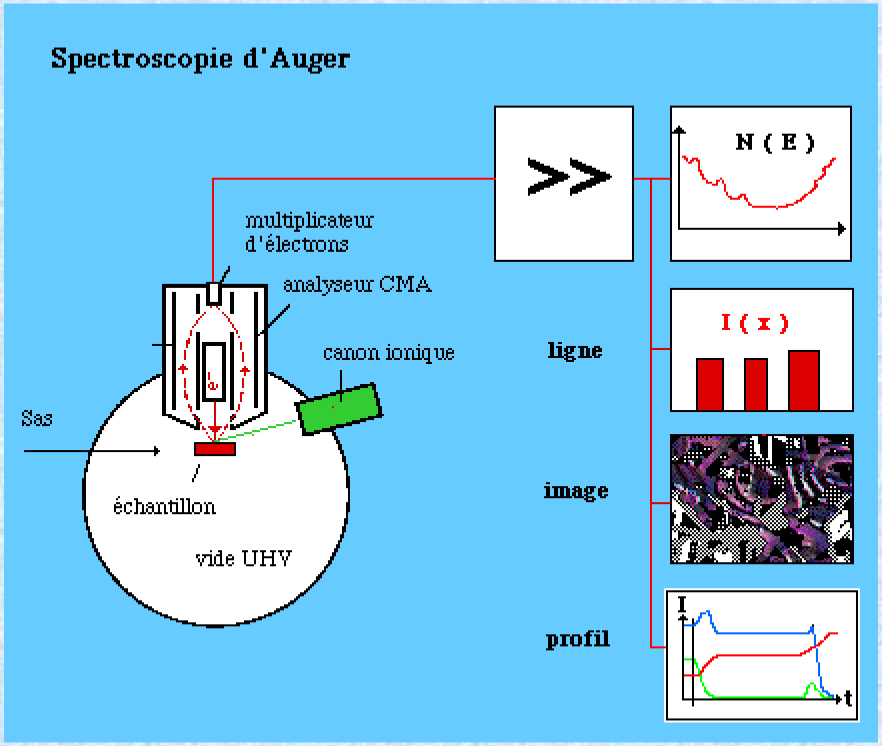
Some composites

Biological materials (bone, hair, etc.)

Requirements

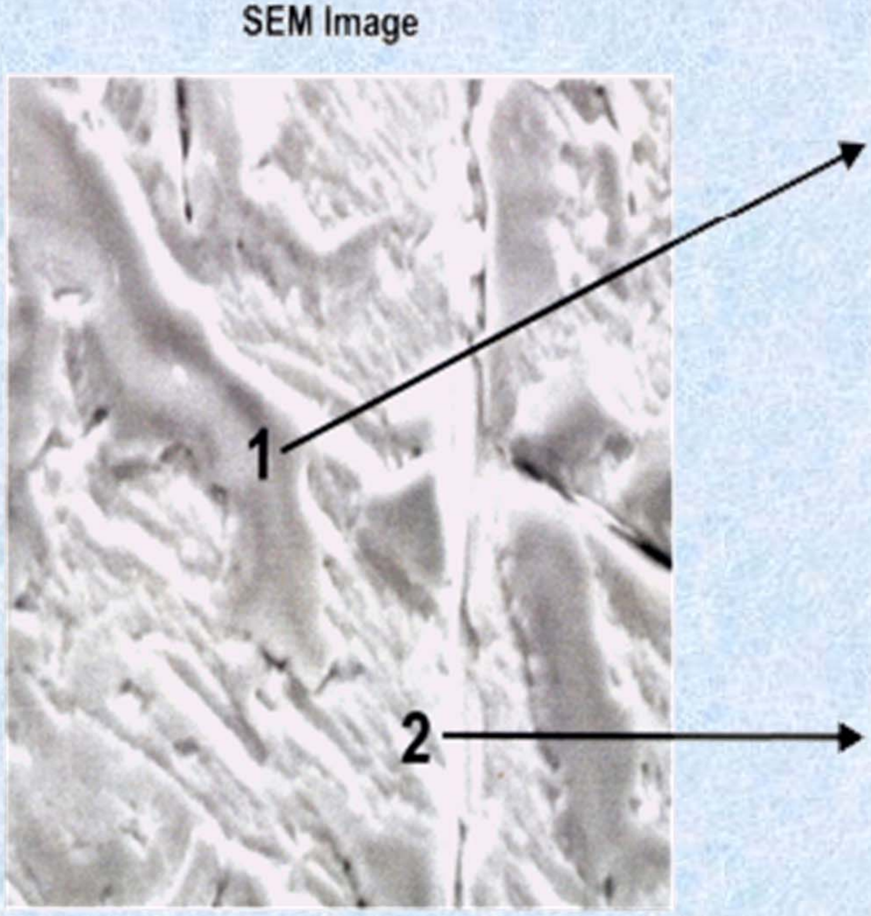
Vacuum-compatible, conducting, semiconductor, or thin film on conducting substrate

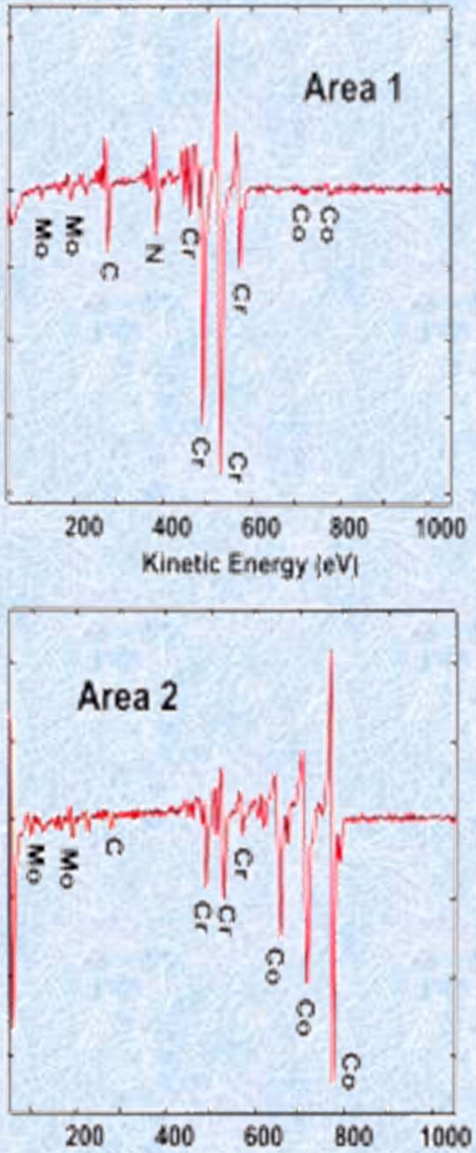
Sputter depth profiling routinely used



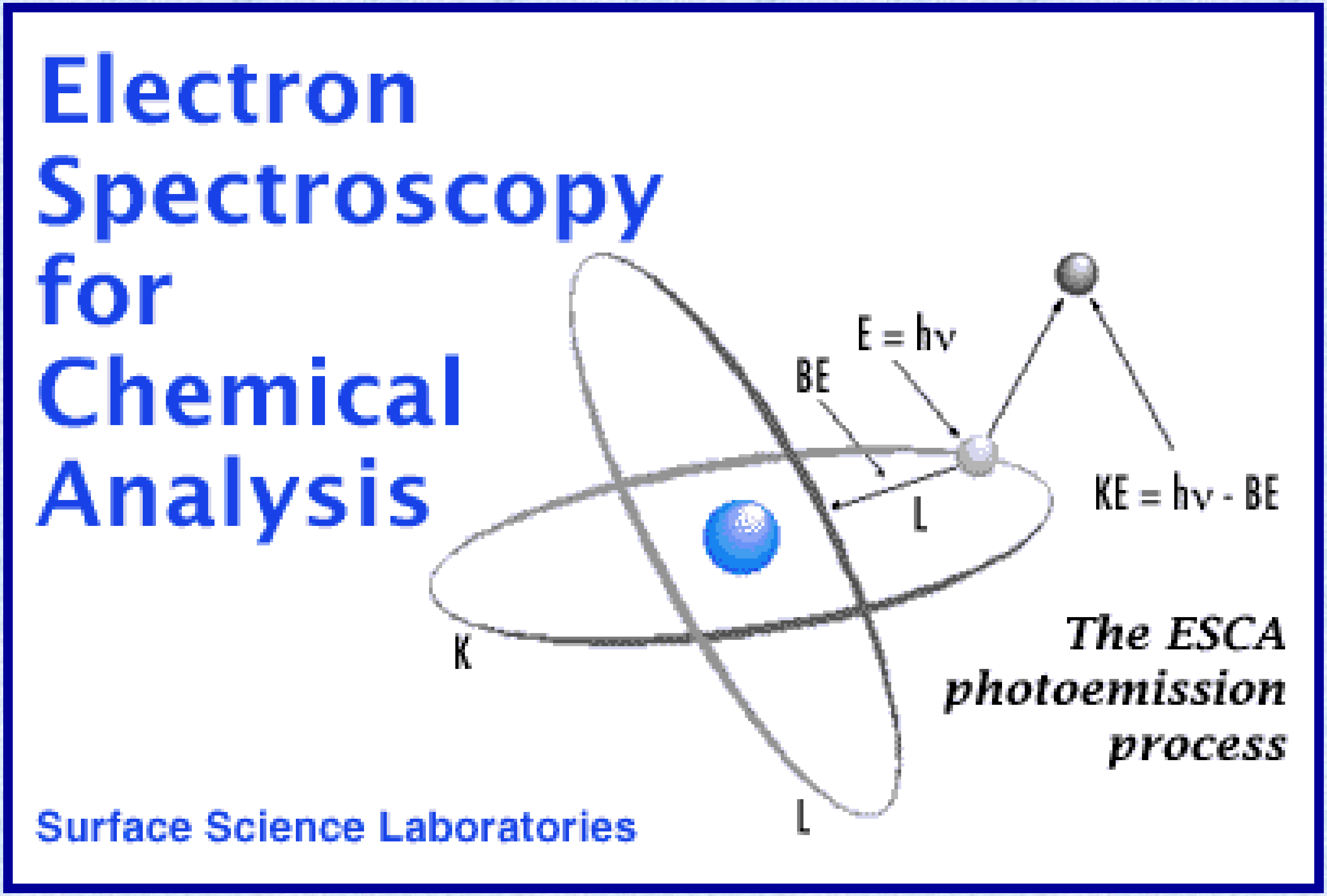
Auger Electron Spectroscopy

Note chromium enrichment in area 1





ESCA - X-Ray Photoelectron Spectroscopy



Electron
Spectroscopy
for
Chemical
Analysis

The ESCA photoemission process

Surface Science Laboratories

ESCA - X-Ray Photoelectron Spectroscopy

- ESCA analysis involves only the top 20-50Å of the sample, making it an extremely surface sensitive technique

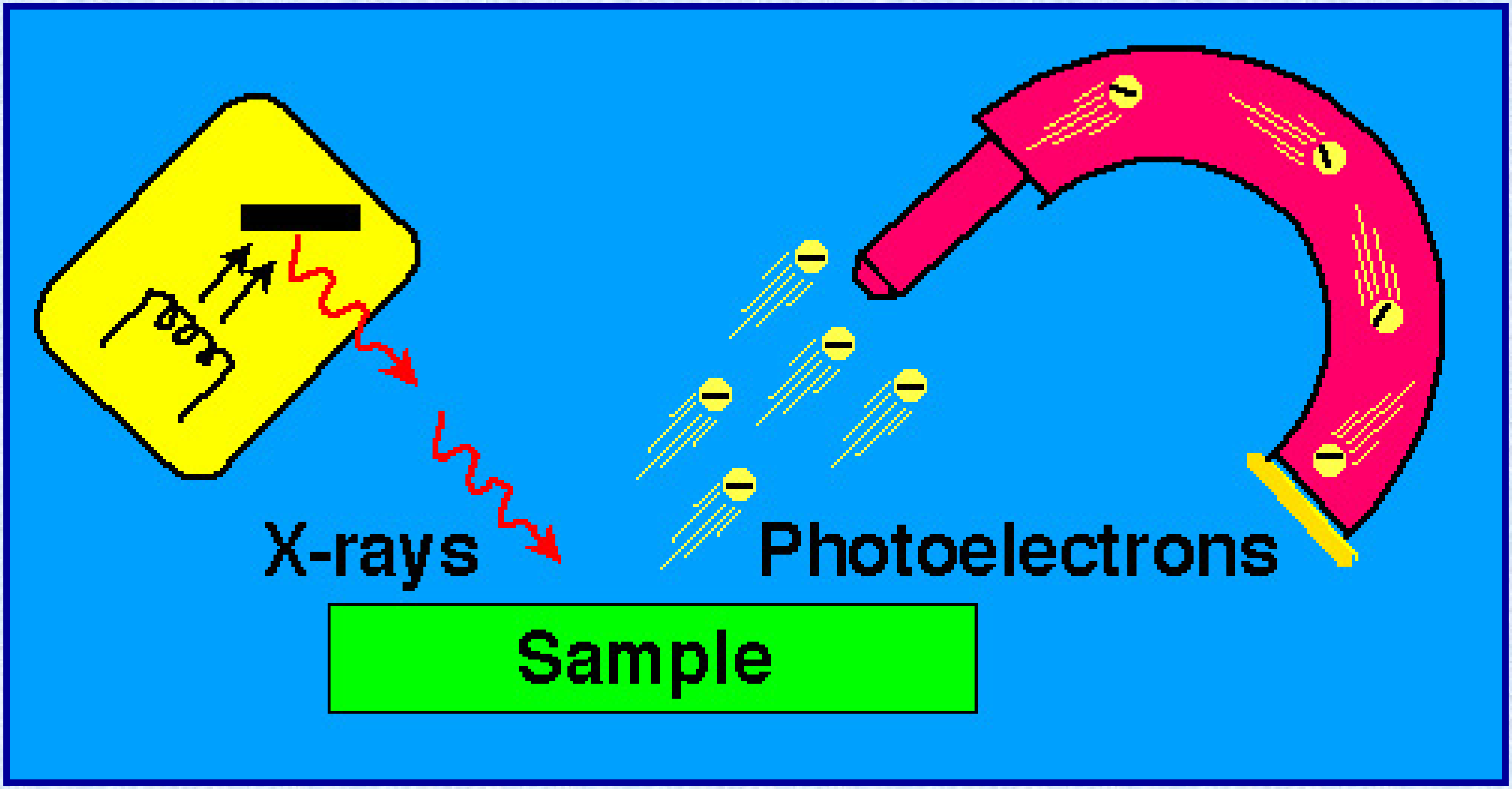
- ESCA spectra can also provide information about an element's chemical environment or oxidation state. The chemical environment of an atom affects the strength with which electrons are bound to it. Atoms associated with different chemical environments produce peaks with slightly different binding energies which is referred to as chemical shift

- Distinct chemical states which are close in energy can be deconvoluted using peak fitting programs to give percent composition of each state.

ESCA - X-Ray Photoelectron Spectroscopy

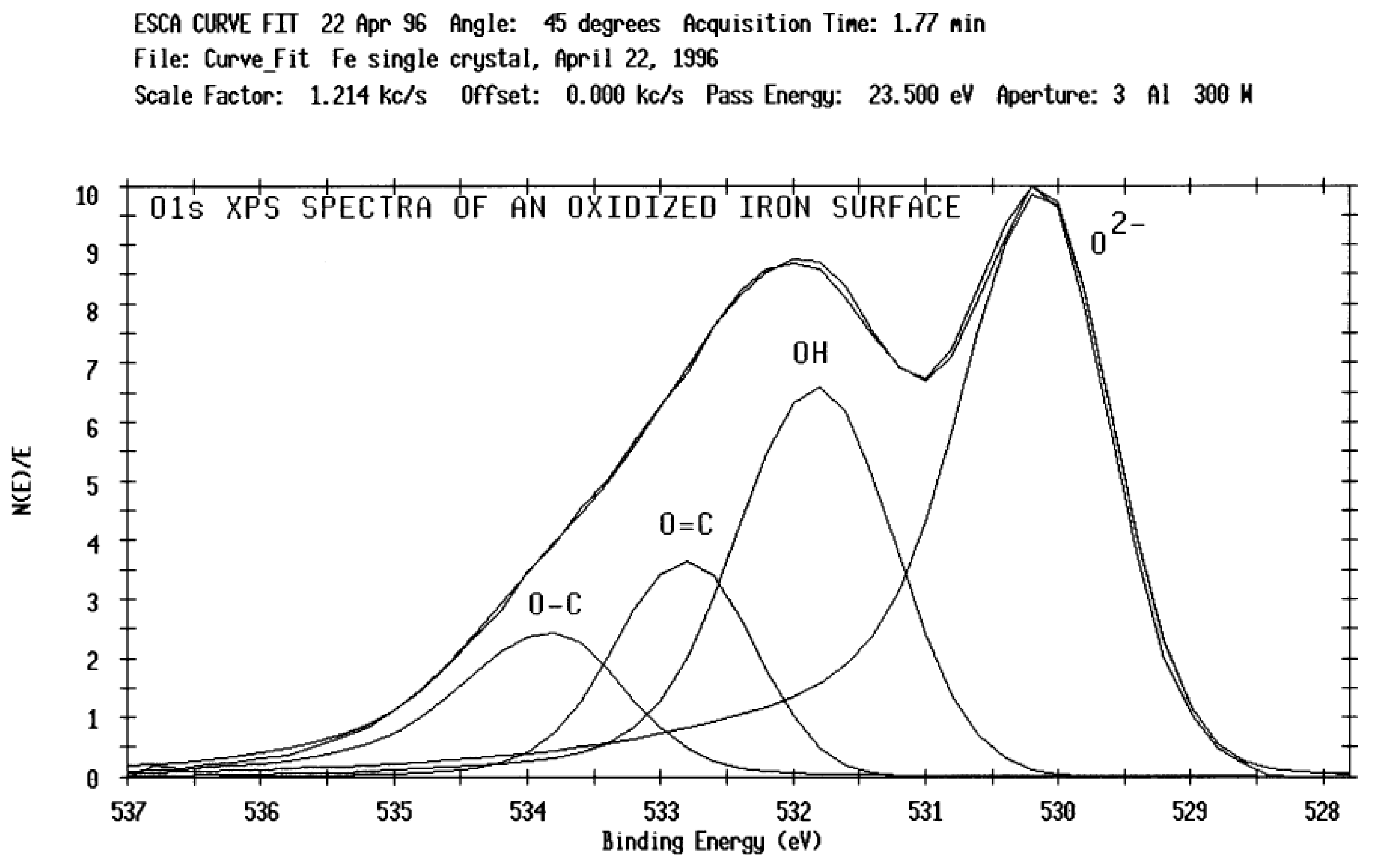
- incident radiation is a monochromatic X-ray beam
- core electrons are emitted by the sample according to the **photoelectric effect**
- emitted electrons have a kinetic energy equal to the X-ray energy less the **binding energy of the electron** and the work function of the instrument
- detected kinetic energies of the electrons are converted to binding energies, thereby enabling element identification
- energy spectra are reported as binding energy vs. intensity
- using sensitivity factors, peak intensities can provide quantitative elemental surface compositions

ESCA - X-Ray Photoelectron Spectroscopy



ESCA - X-Ray Photoelectron Spectroscopy

Deconvoluted ESCA Spectrum

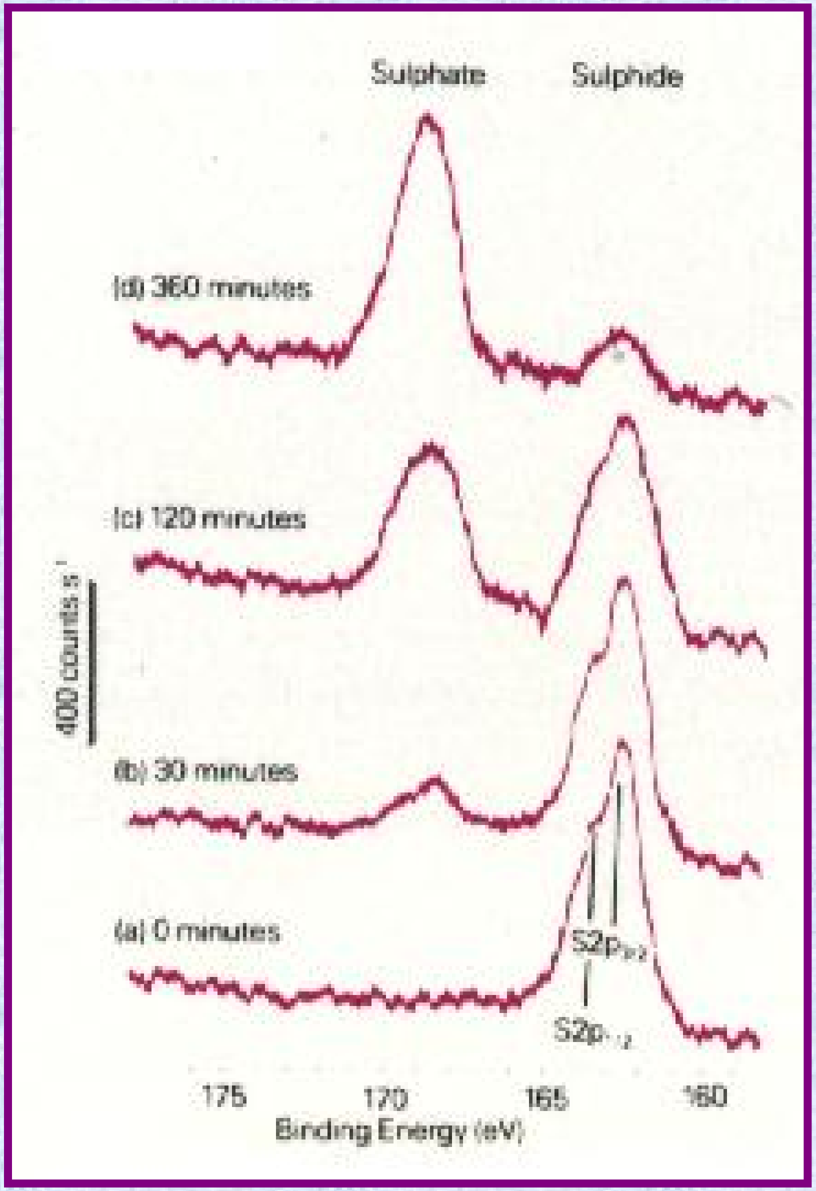


ESCA CURVE FIT 22 Apr 96 Angle: 45 degrees Acquisition Time: 1.77 min

File: Curve_Fit Fe single crystal, April 22, 1996

Scale Factor: 1.214 kc/s Offset: 0.000 kc/s Pass Energy: 23.500 eV Aperture: 3 Al 300 W

XPS Spectrum



oxidation of pyrite, FeS_2, surface

Neutron Activation Analysis

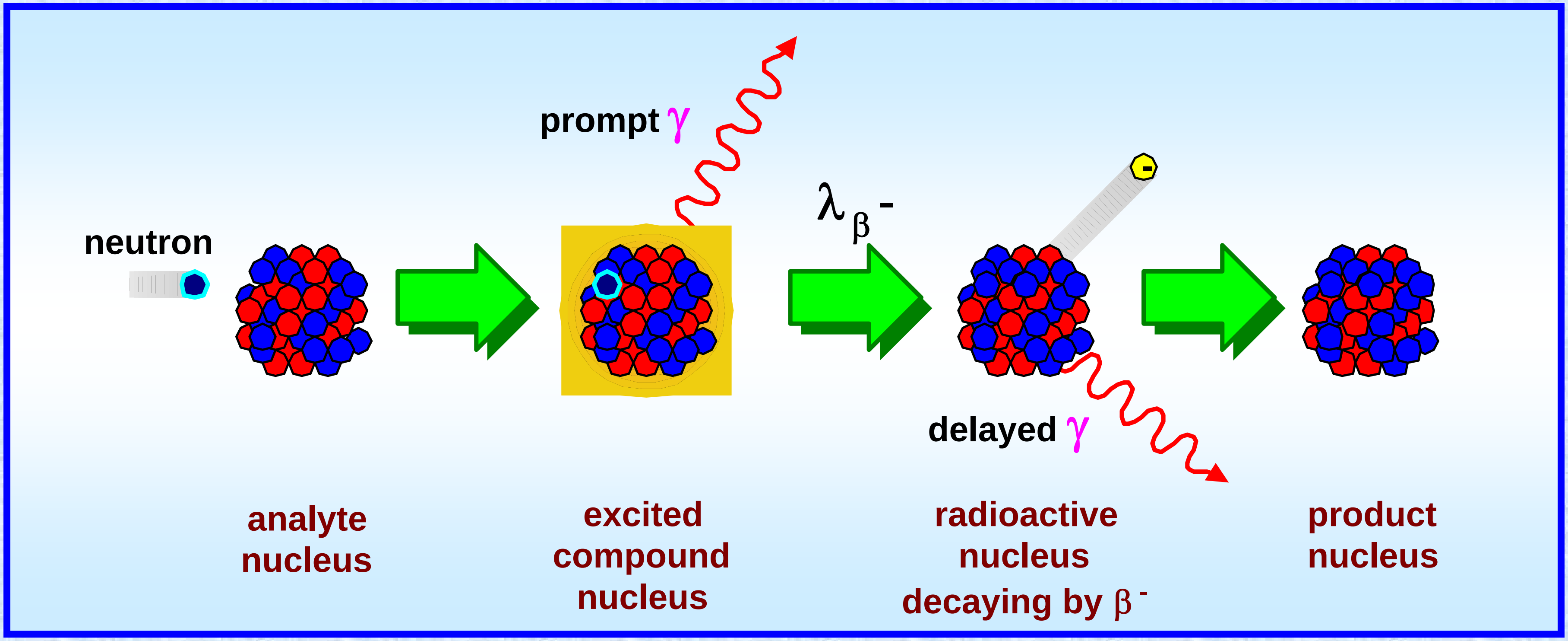
Instrumental Neutron Activation Analysis (INAA) is a method of identifying and measuring trace quantities of elements in many types of materials.

Sixty-seven common elements become radioactive when exposed to the neutron flux in the reactor, and subsequently emit radiation that is characteristic for each element and permits identification. More than fifty of the sixty-seven elements can be identified and measured quite readily.

The technique is particularly useful for analyzing geological and environmental samples and for analyzing industrial samples to maintain quality control.

The sensitivity and accuracy of the technique, the variety of materials that can be analyzed, the large number of elements that can be detected, and its essentially non-destructive nature make neutron activation analysis an outstanding analytical tool.

Neutron Activation Analysis



INAA — instrumental neutron activation analysis
PGNAA — prompt gamma neutron activation analysis
RNAA — radiochemical neutron activation analysis
FNAA — fast neutron activation analysis

Neutron Activation Analysis

The counts recorded during a count time t_c, following an irradiation time t_{irr}, and wait time t_w, is:

$$C = \frac{mEN_A\sigma_n\phi\Psi}{1.128A\lambda f}\left[1-e^{-\lambda t_{irr}}\right]\left[e^{-\lambda t_w}\right]\left[1-e^{-\lambda t_c}\right]$$

where:

Ψ = the overall efficiency of the detector set-up, including effects of geometry, detector response, and self-shielding.

m = mass of the irradiated element, g

E = abundance of the isotope of interest

N_A = Avogadro's number, atoms/g atom

σ_n = neutron absorption cross-section of the irradiated species, cm^2

A = Atomic mass of the irradiated element, AMU

ϕ = thermal neutron flux, neutrons/cm^2/s

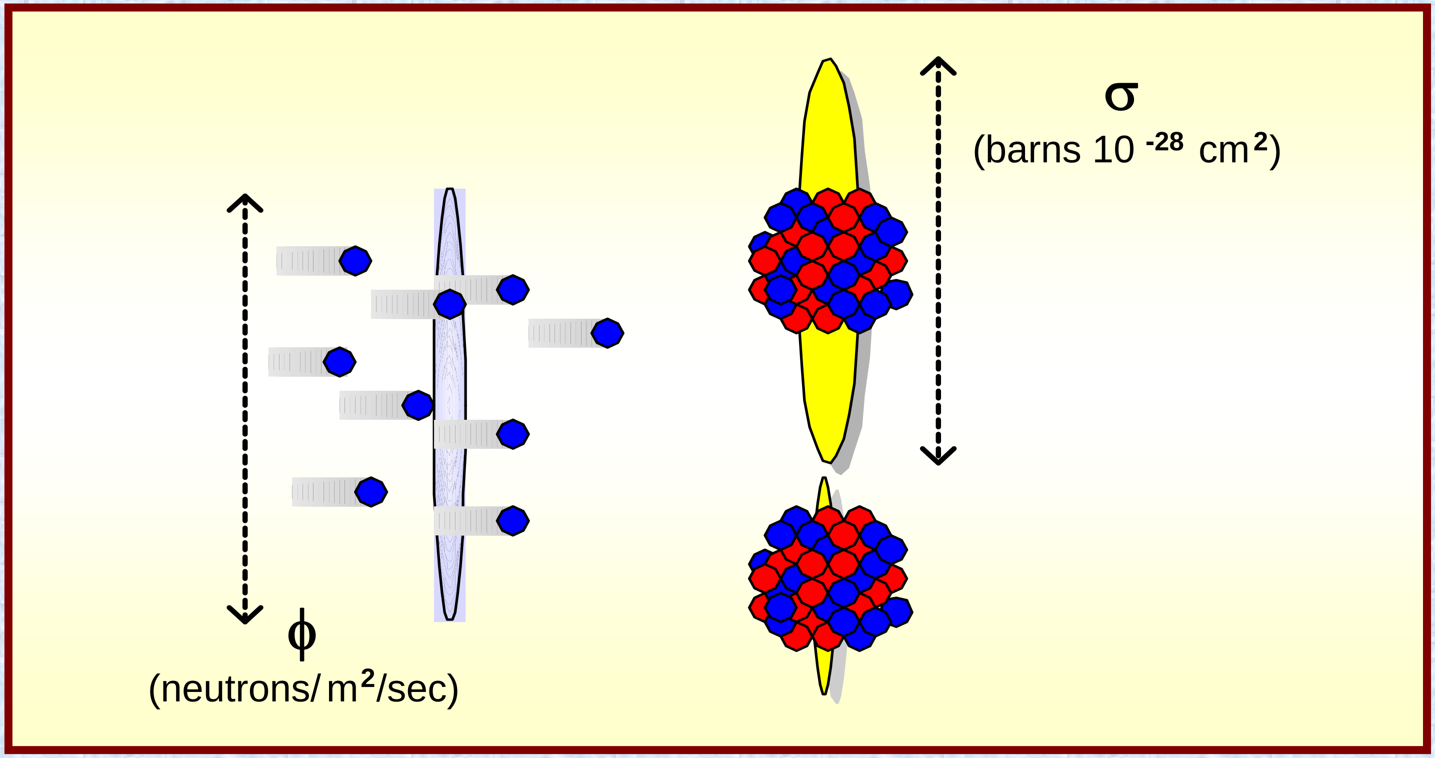
f = branching ratio for the gamma-ray of interest

λ = decay constant of the radioisotope produced, sec

1.128 = correction for a Maxwellian distribution of neutrons

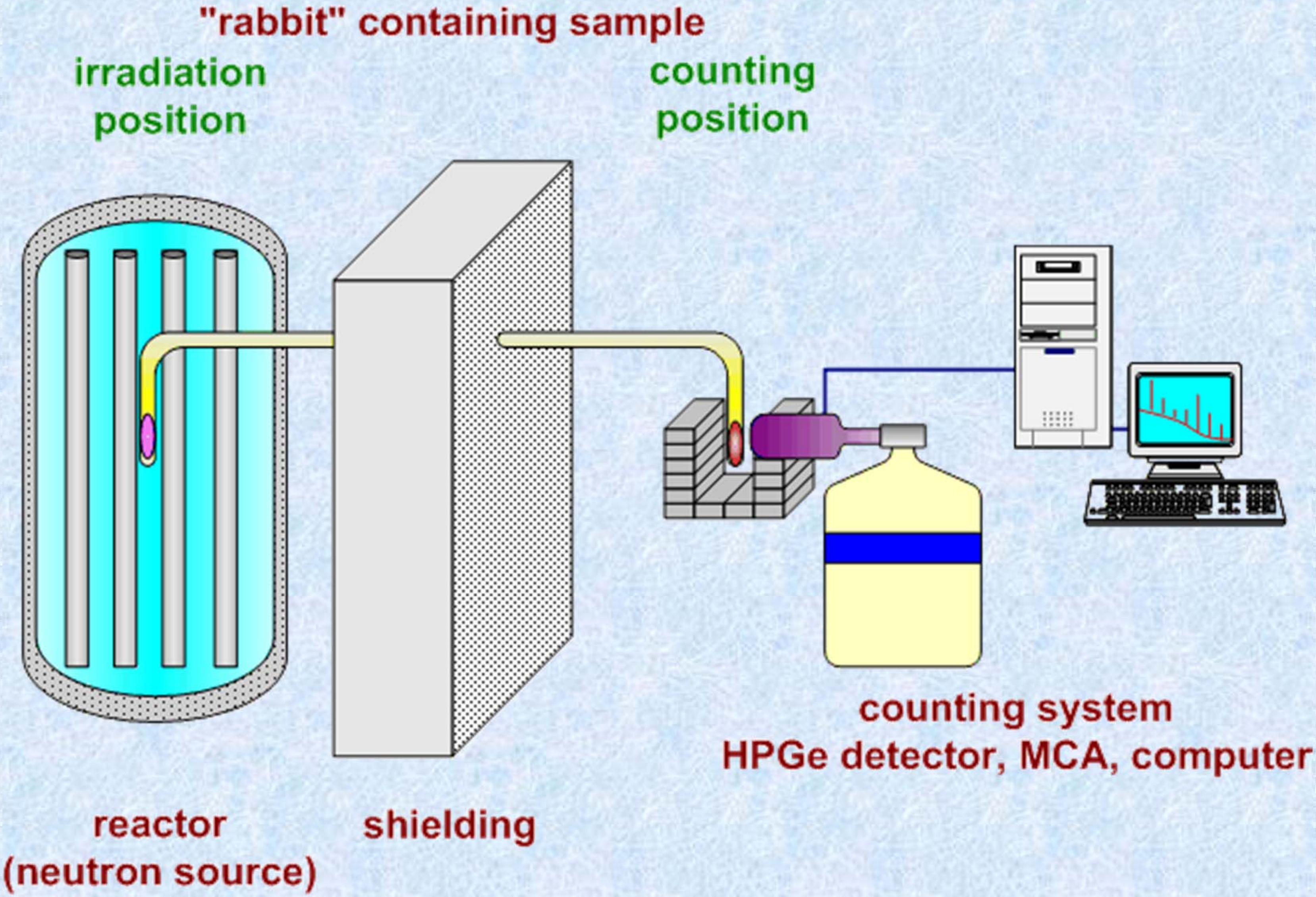
The expression clearly indicates the activity of the irradiated material generated by the thermal neutron flux at the location of the irradiation, corrected for decay before and during counting.

Neutron Activation Analysis



$$C = \frac{mEN_A\sigma_n\phi\Psi}{1.128A\lambda f}\left[1-e^{-\lambda t_{irr}}\right]\left[e^{-\lambda t_w}\right]\left[1-e^{-\lambda t_c}\right]$$

Neutron Activation Analysis



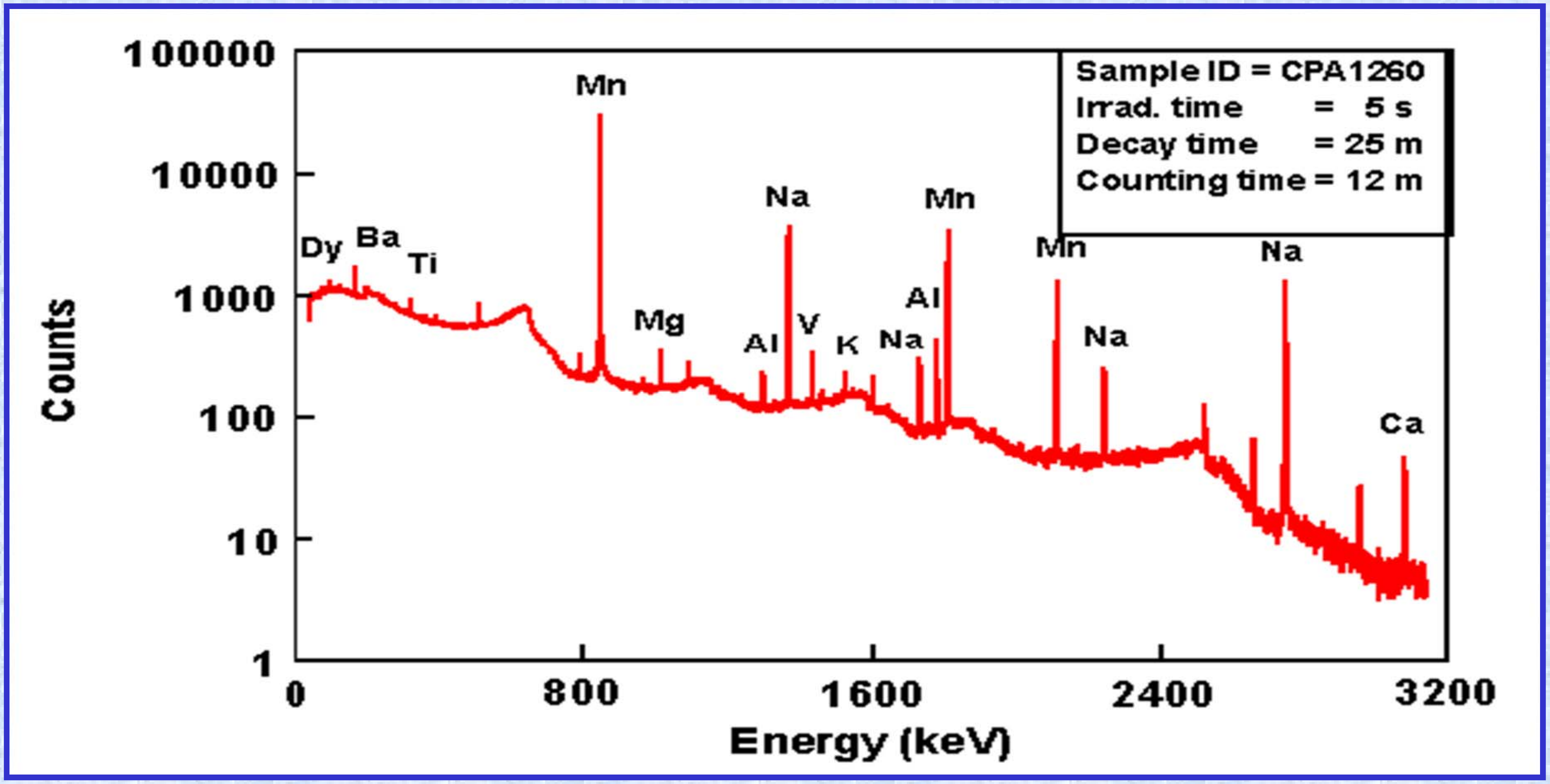
Neutron Activation Analysis

Sensitivity (nanograms)	Elements
0.001	Dy, Eu
0.001 – 0.010	In, Lu, Mn
0.010 – 0.100	Au, Ho, Ir, Re, Sm, W
0.100 – 1	Ag, Ar, As, Br, Cl, Co, Cs, Cu, Er, Ga, Hf, I, La, Sb, Sc, Se, Ta, Tb, Th, Tm, U, V, Yb
1 – 10	Al, Ba, Cd, Ce, Cr, Hg, Kr, Gd, Ge, Mo, Na, Nd, Ni, Os, Pd, Rb, Rh, Ru, Sr, Te, Zn, Zr
10 – 100	Bi, Ca, K, Mg, P, Pt, Si, Sn, Ti, Tl, Xe, Y
100 – 1000	F, Fe, Nb, Ne
10000	Pb, S

Applications of Neutron Activation Analysis

- **Anthropology**
  - New Mexican Pottery
  - Bronze Age pottery in Hungary
  - Aztec Pottery
- **Archeology**
  - Texcoco Fabric marked pottery
  - Persian Coins
  - Abila pottery
  - trade patterns
- **Chemical Engineering**
  - fuel cells
  - automotive catalysts
- **Chemistry**
  - Silicate Glass studies
- **Geology**
  - Gold Followers
  - Palaeo-oceanic Environments
  - Magma Systems
- **Graduate Research**
  - Deep Crystal Growth
  - Ocean Sediments
  - Discrimination Diagrams
  - K-bentonites
- **Other Graduate Research**
  - relationship between porphyry and epithermal deposits
  - Looking at two mass extinctions
  - origin of volcanic and plutonic rocks
  - Tourmaline
- **Nuclear Engineering**
- **Material studies**
  - high accuracy calibration of germanium detectors
- **Environmental Research**
  - Florida Everglades
- **Industrial Applications**
  - elements in glass and base materials
  - composition of polyethylene plastics
  - analyzing metals and halogens
  - analysis services

Neutron Activation Analysis of Pottery



Archaeometry
University of Missouri - Columbia

Neutron Activation Analysis of Olmec Pottery

Research Articles

Olmec Pottery Production and Export in Ancient Mexico Determined Through Elemental Analysis

Jeffrey P. Blomster,[1*] Hector Neff,[2] Michael D. Glascock[3]

The first Mesoamerican civilization, the Gulf Coast Olmec, is associated with hierarchical society, monumental art, and an internally consistent ideology, expressed in a distinct style and salient iconography. Whether the Olmec style arose in just one area or emerged from interactions among scattered contemporaneous societies remains controversial. Using elemental analysis, we determined the regional clay sources of 725 archaeological ceramic samples from across Mesoamerica. Exported Olmec-style ceramics originated from the San Lorenzo region of the Gulf Coast, supporting Olmec priority in the creation and spread of the first unified style and iconographic system in Mesoamerica.

[1] Department of Anthropology, George Washington University, Washington, DC 20052, USA.
[2] Department of Anthropology, California State University, Long Beach, CA 90840, USA.
[3] Research Reactor Center, University of Missouri, Columbia, MO 65211, USA.

Neutron Activation Analysis of Olmec Pottery

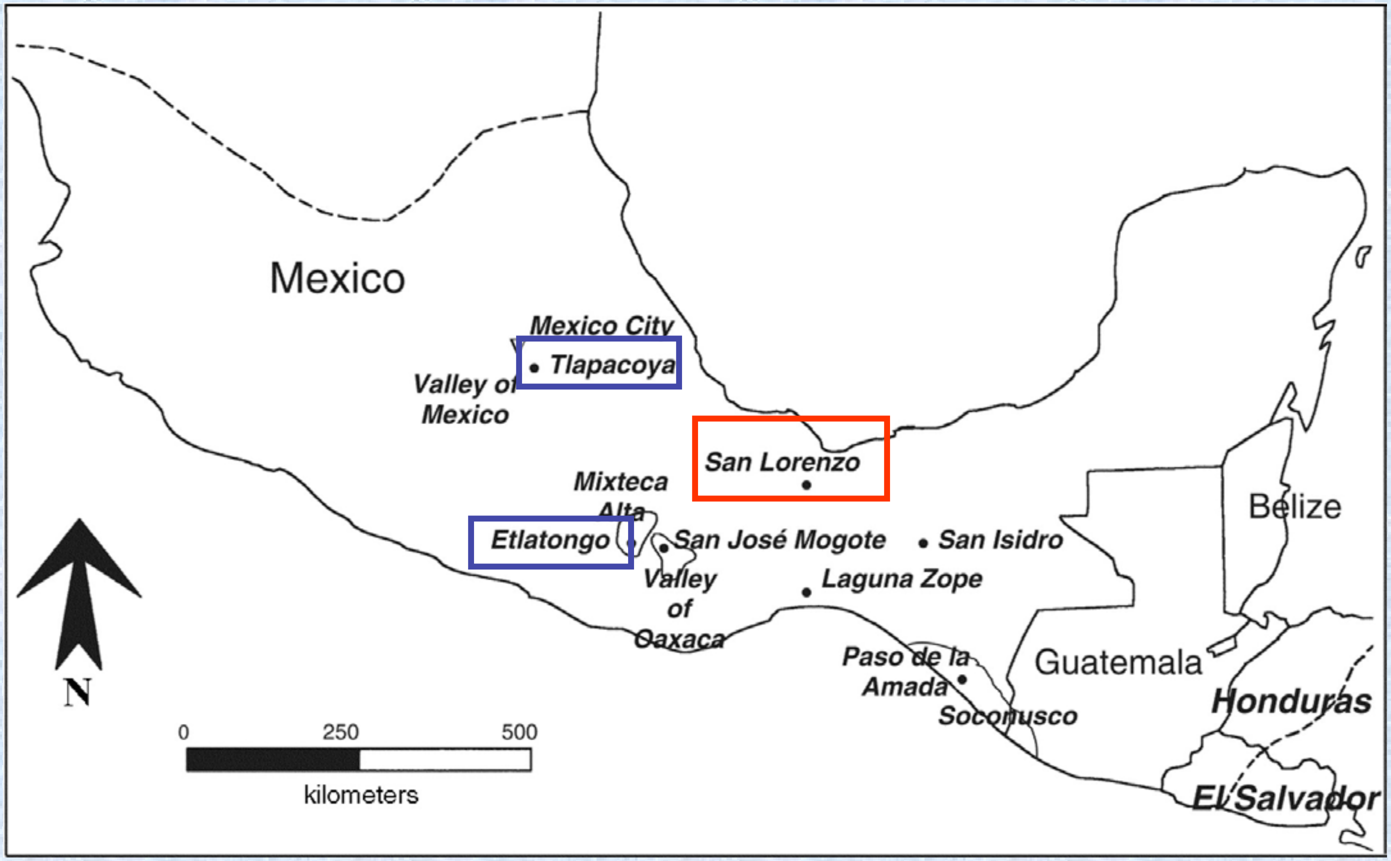


Fig. 1. Map of Early Formative regional centers, showing modern national borders and Mexico City for reference

J. P. Blomster et al., Science 307, 1068 -1072 (2005)

Neutron Activation Analysis of Olmec Pottery

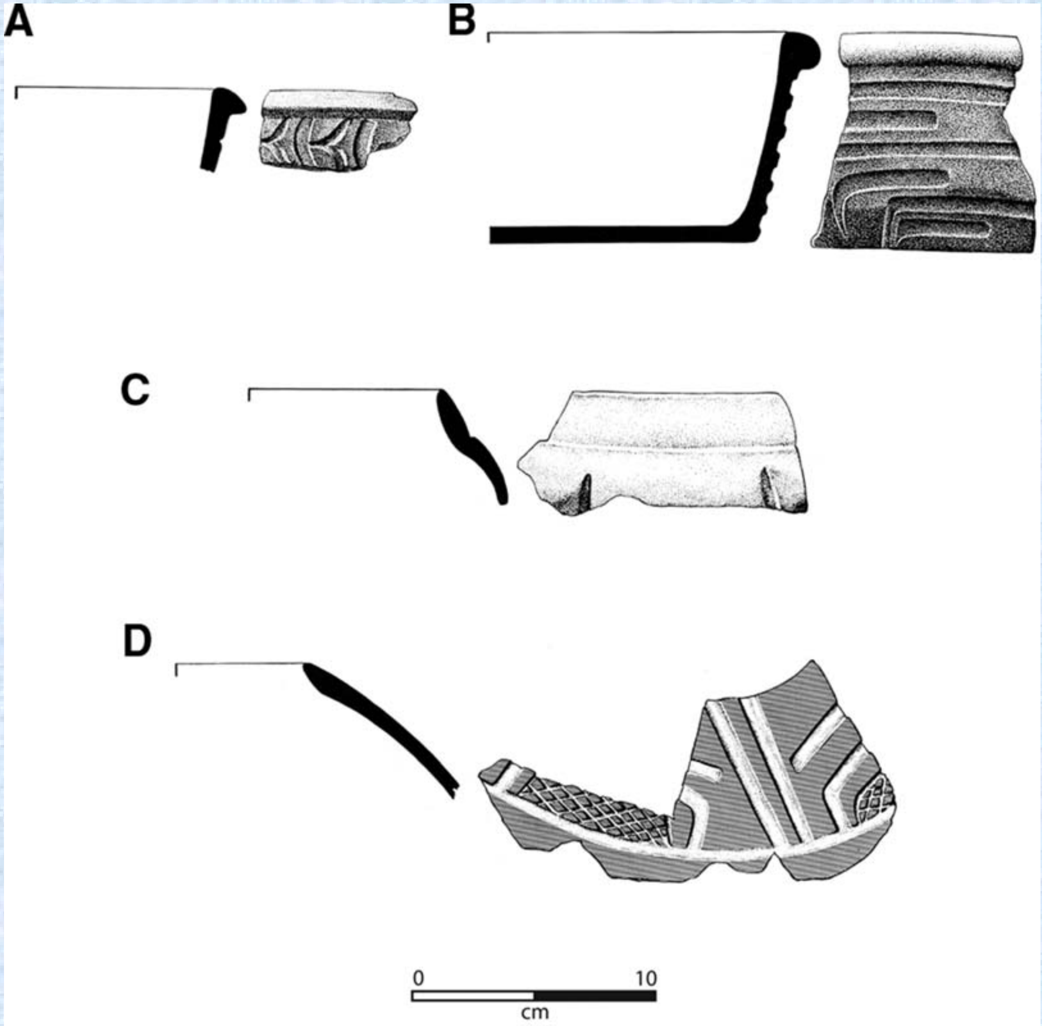


Fig. 2. Profile and exterior views of Olmec pottery types produced at San Lorenzo

J. P. Blomster et al., Science 307, 1068 -1072 (2005)

Neutron Activation Analysis of Olmec Pottery

Science

AAAS

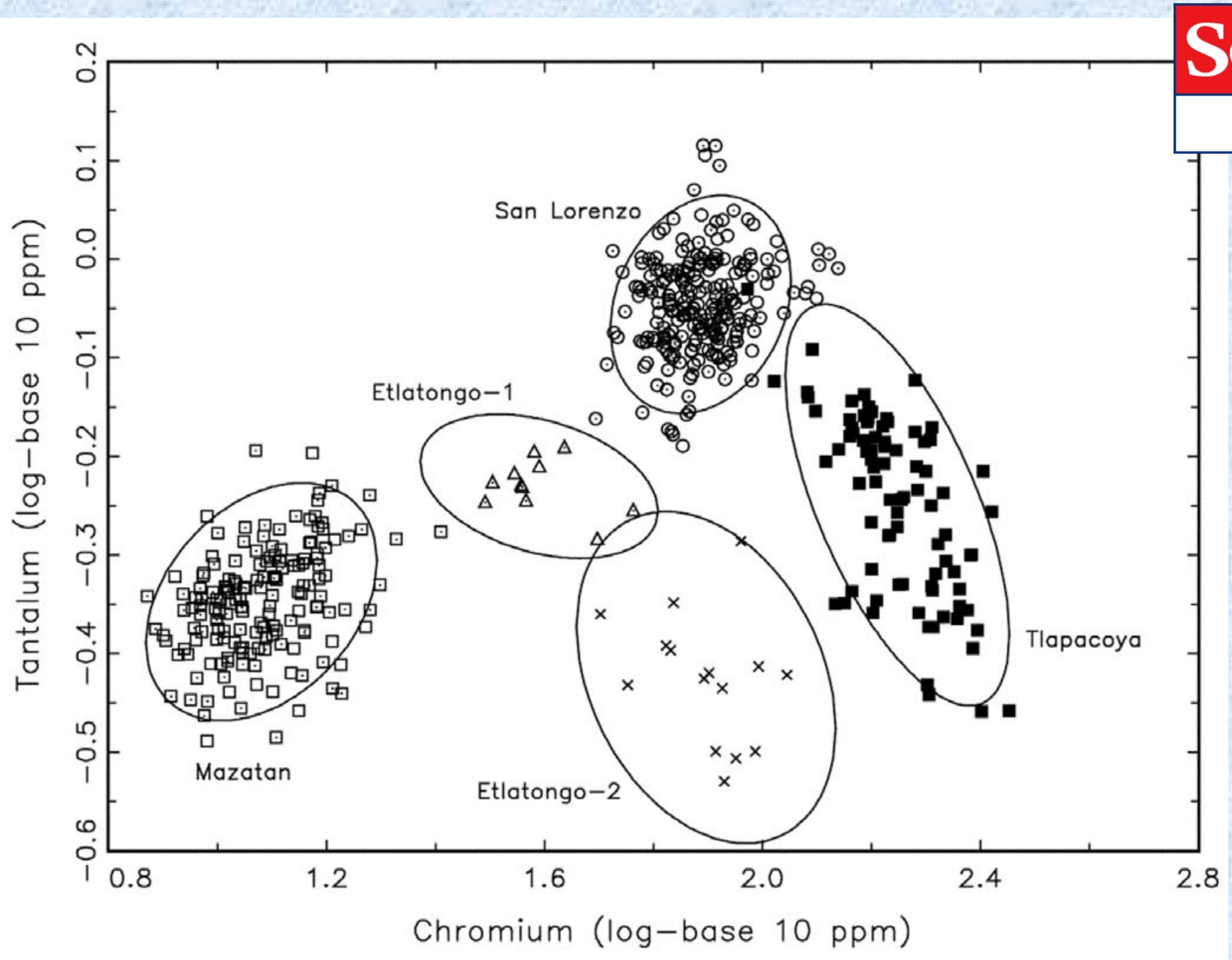
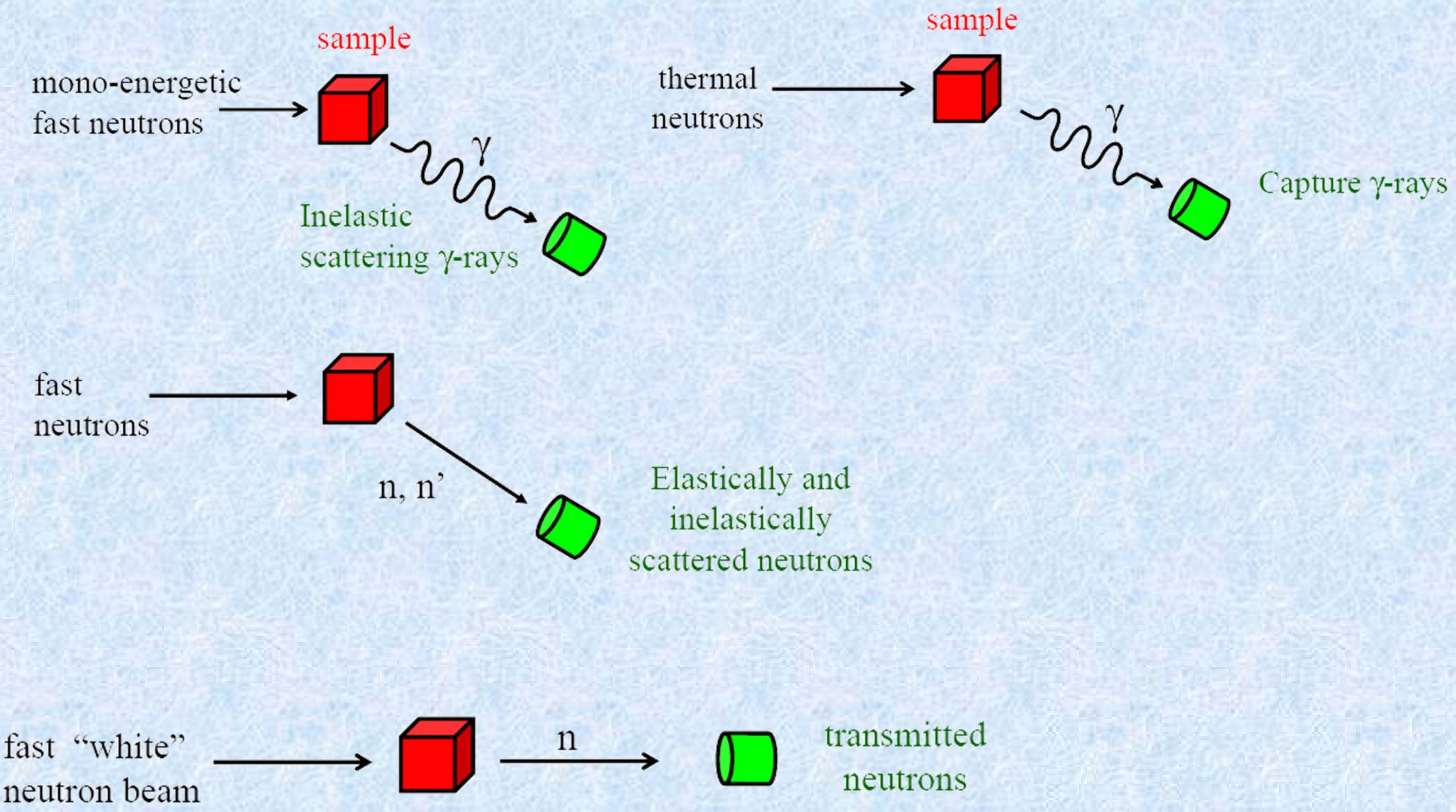


Fig. 4. Bivariate plot of chromium and tantalum concentrations in pottery samples assigned to five well-defined reference groups in the Early Formative pottery database

J. P. Blomster et al., Science 307, 1068 -1072 (2005)

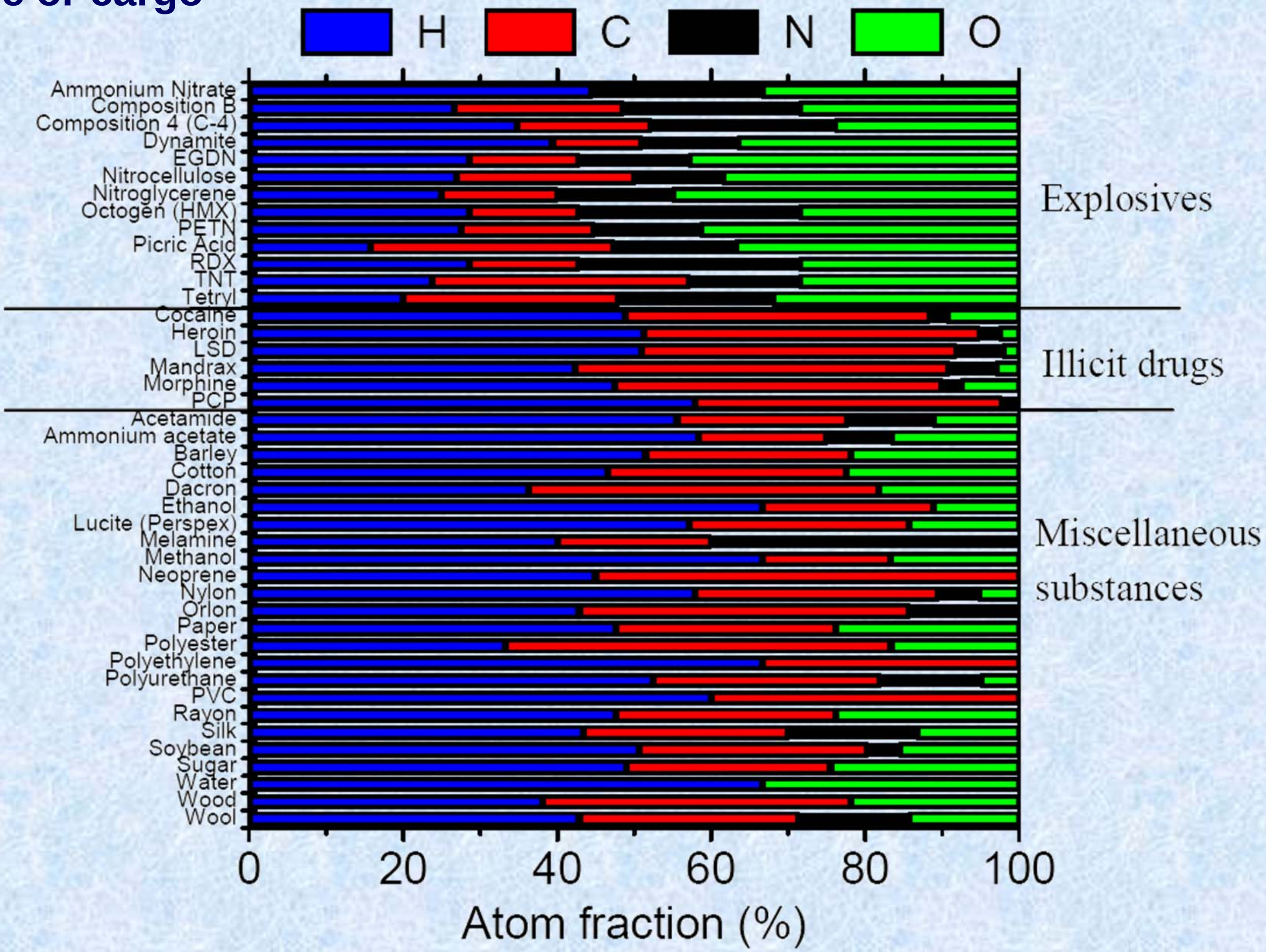
Neutron Scattering

Possible neutron interactions with matter



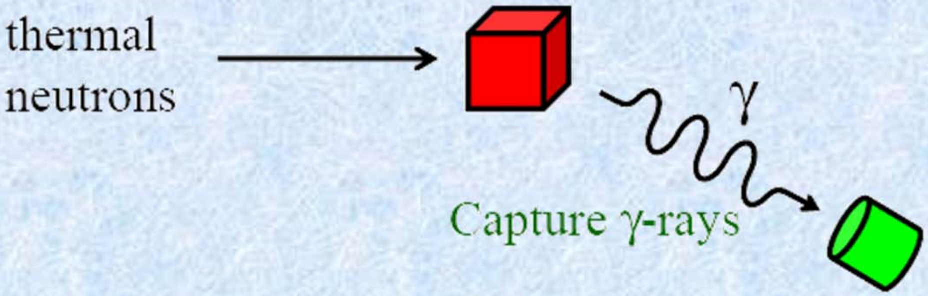
Neutron Scattering

Chemical composition of contraband and harmless materials which could be found in luggage or cargo



Neutron Scattering

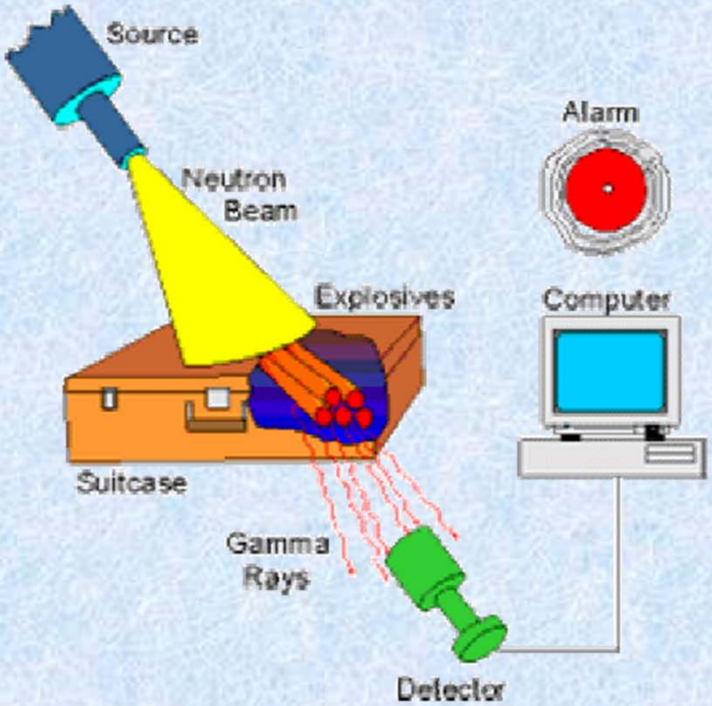
Neutron interrogation of luggage



thermal neutrons

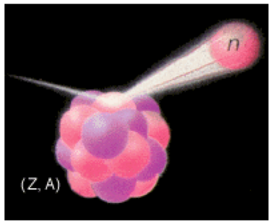
γ

Capture γ-rays

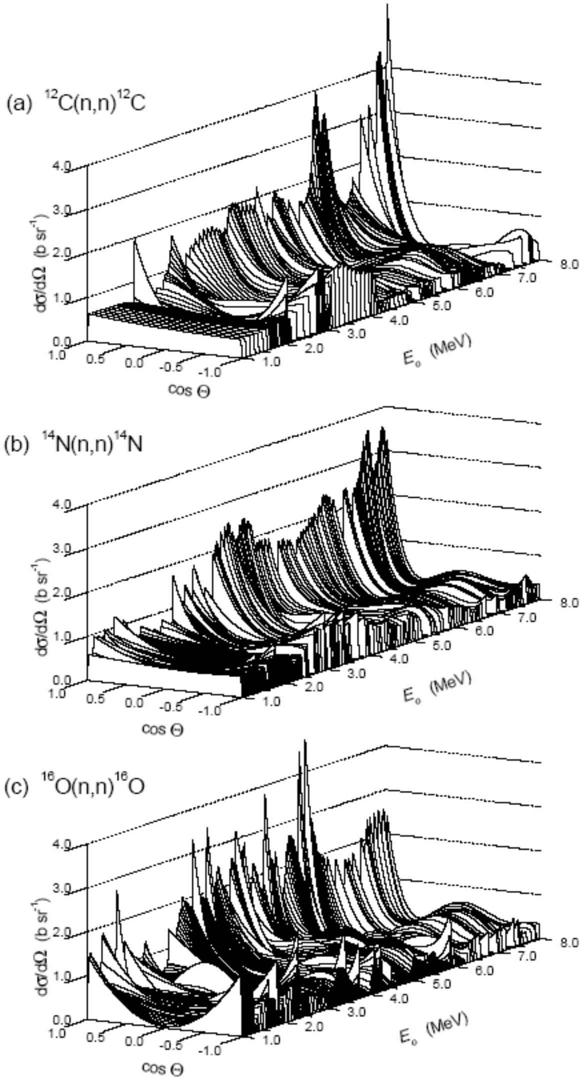


Neutron Scattering

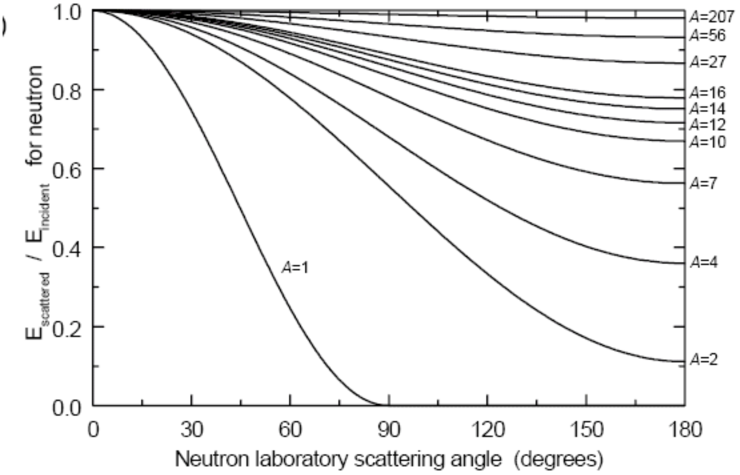
Neutron elastic scattering



Differential cross section



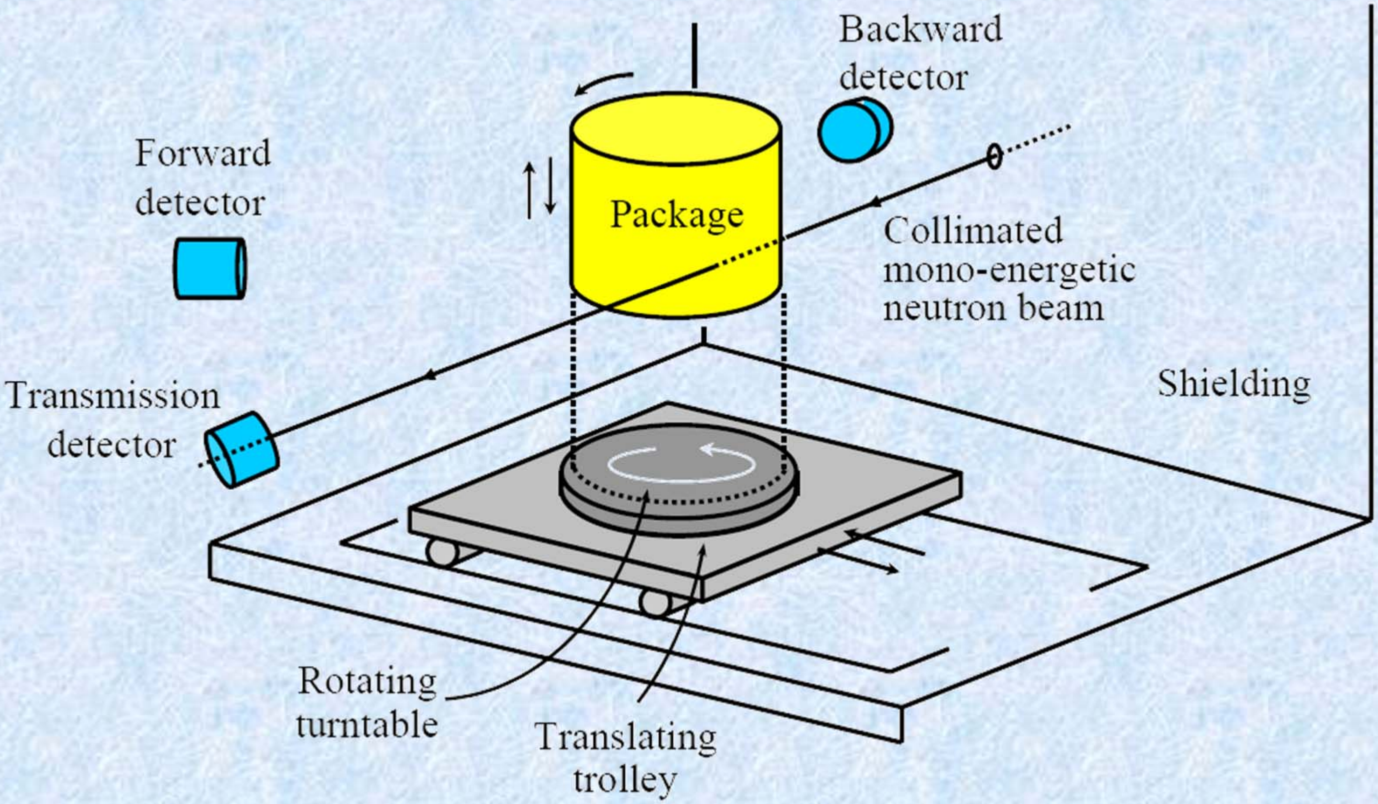
Kinematics



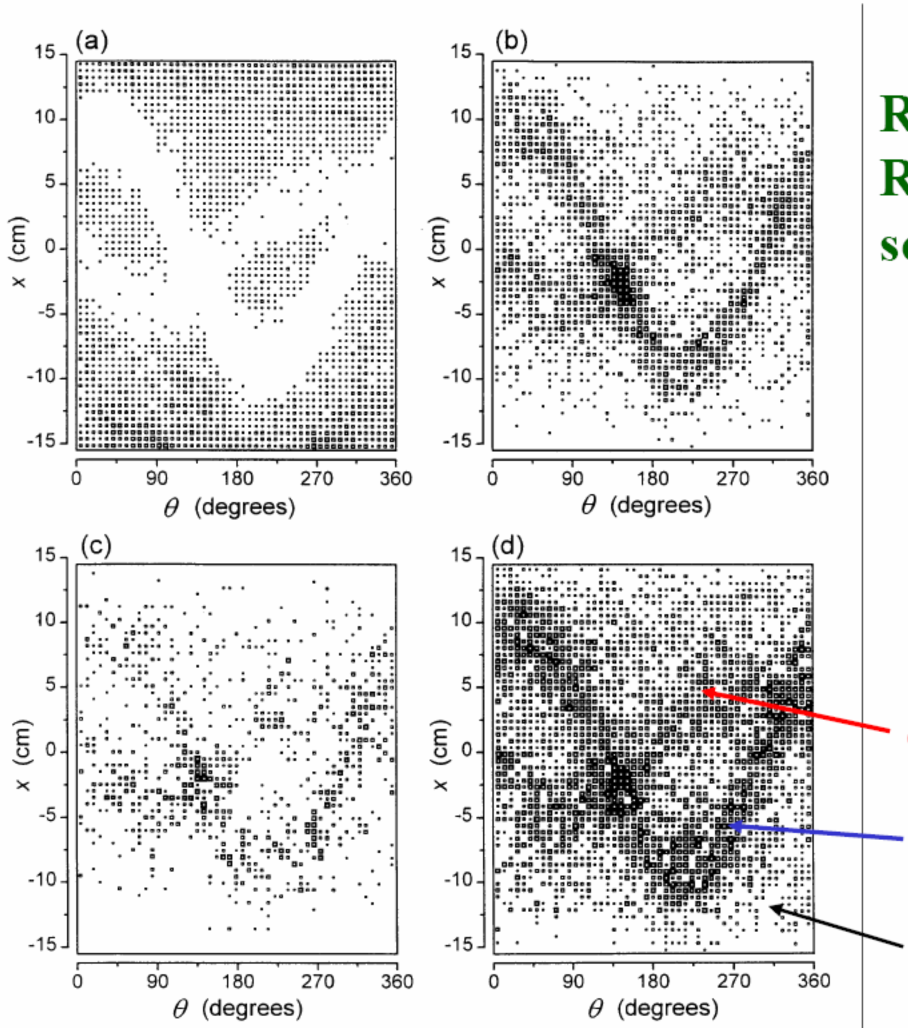
Neutron Scattering

Location of contraband in FNSA measurements

Rotate and translate package across collimated beam.



Neutron Scattering



Results from a Rotational-Translational scan

(a) transmission detector
(b) 45° detector
(c) 150° detector (H-suppressed)
(d) 150° detector (H-enhanced)

explosive

illicit drug

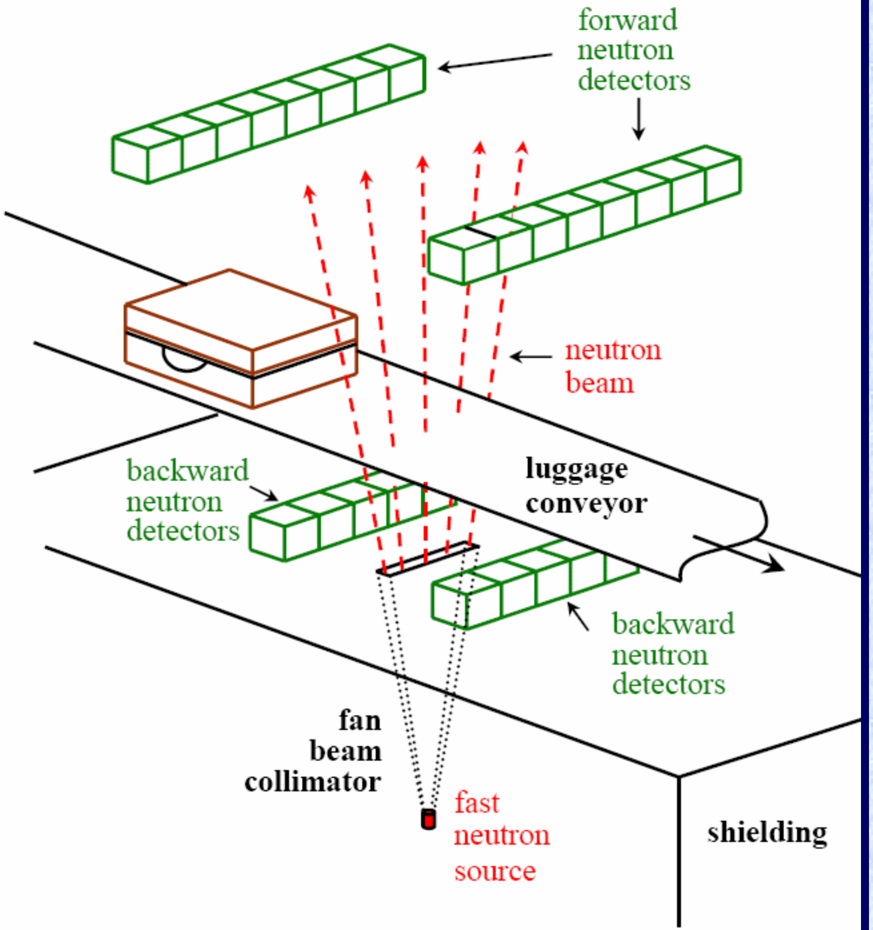
dense cotton clothing

Neutron Scattering

Proposed system for the detection of illicit drugs in **incoming airline luggage.**

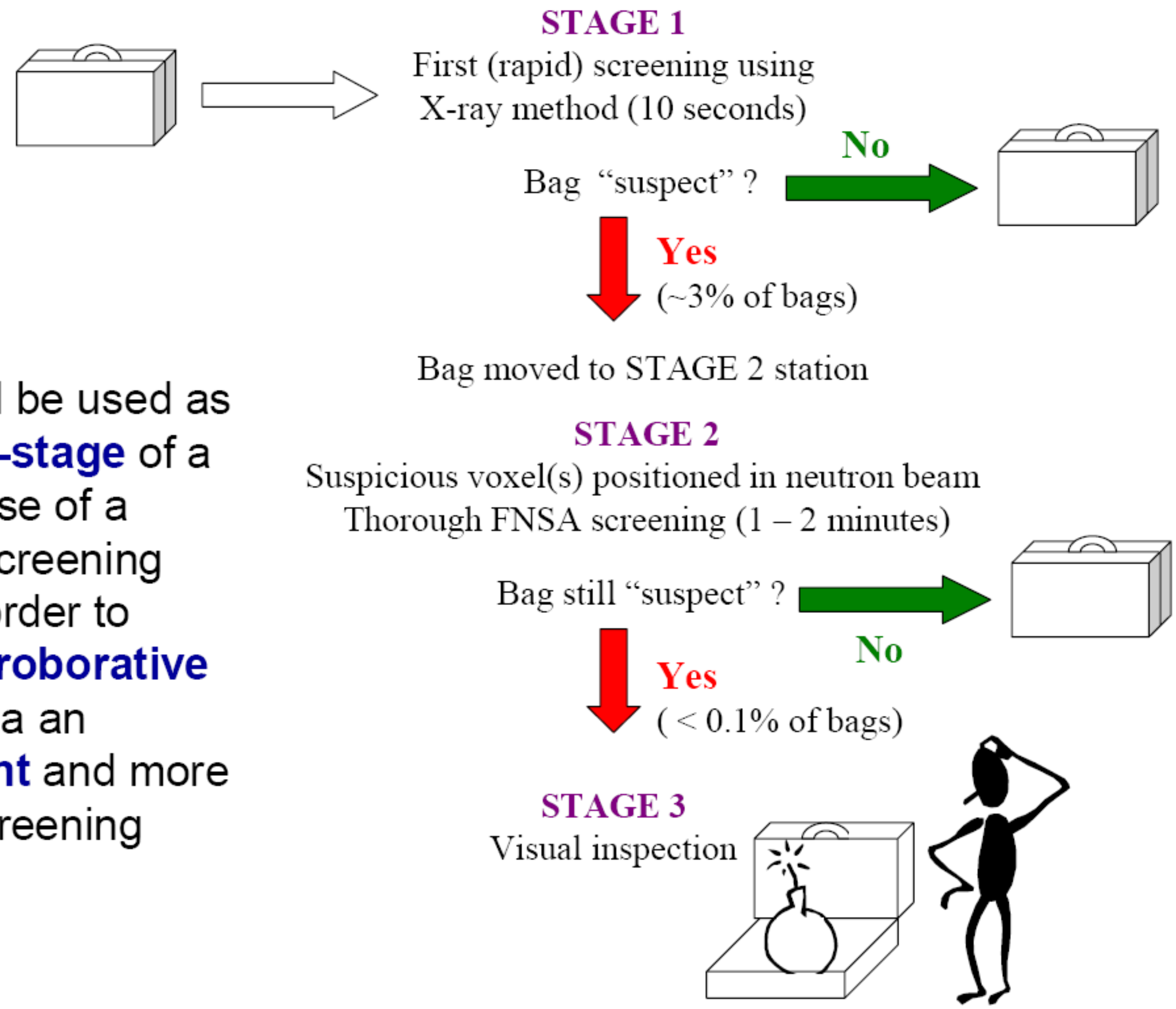
FNSA could be used as the **first stage** of a two-stage screening system. The second stage is manual inspection by customs officials.

Neutrons scattered out of the beam by a passing suitcase are detected in arrays of neutron detectors placed at forward and backward angles.



Neutron Scattering

Multi-stage interrogation protocol for explosives in outgoing luggage



STAGE 1
First (rapid) screening using
X-ray method (10 seconds)

Bag "suspect" ?

No

Yes
(~3% of bags)

Bag moved to STAGE 2 station

STAGE 2
Suspicious voxel(s) positioned in neutron beam
Thorough FNSA screening (1 – 2 minutes)

Bag still "suspect" ?

No

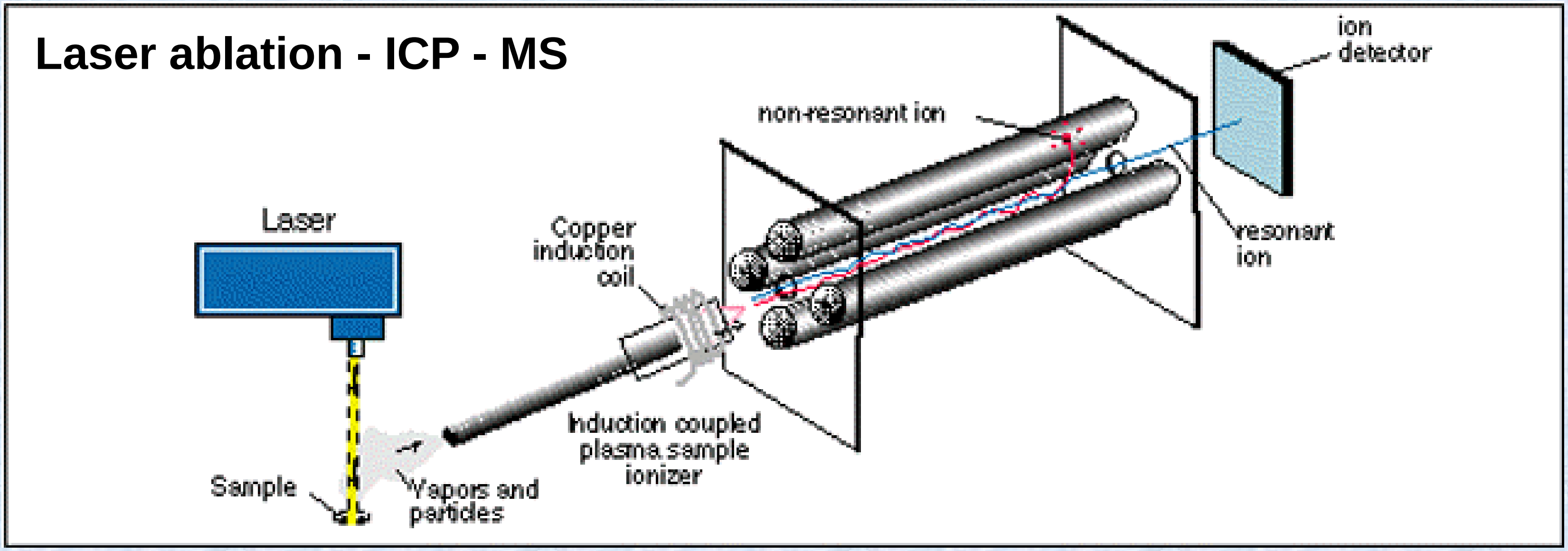
Yes
(< 0.1% of bags)

STAGE 3
Visual inspection

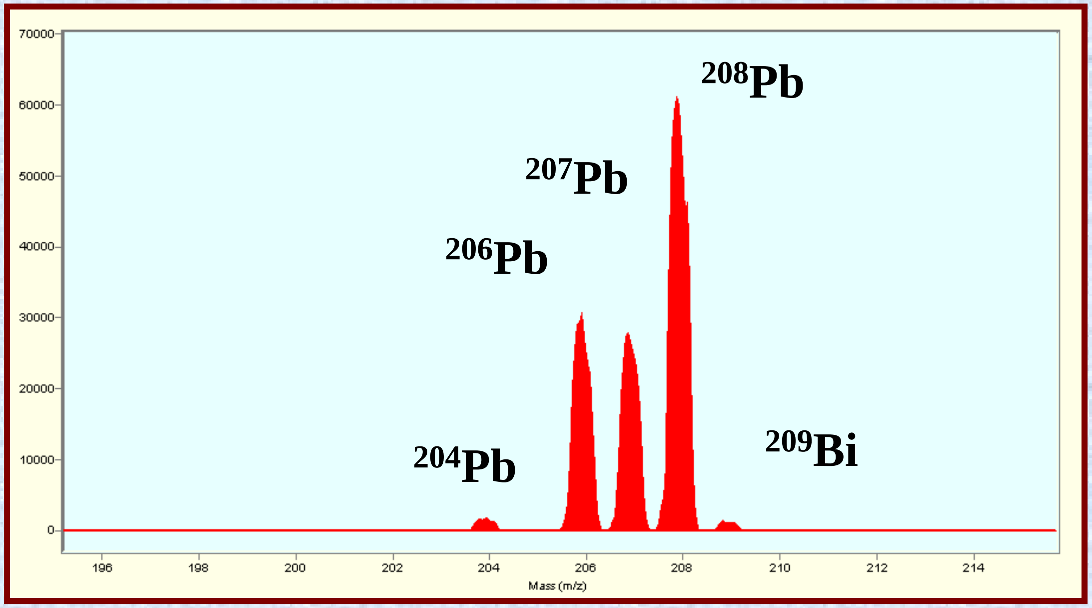
FNSA could be used as the **second-stage** of a second phase of a two-stage screening system, in order to provide **corroborative evidence** via an **independent** and more sensitive screening

http://www.phy.uct.ac.za/people/buffler/FNSA.pdf

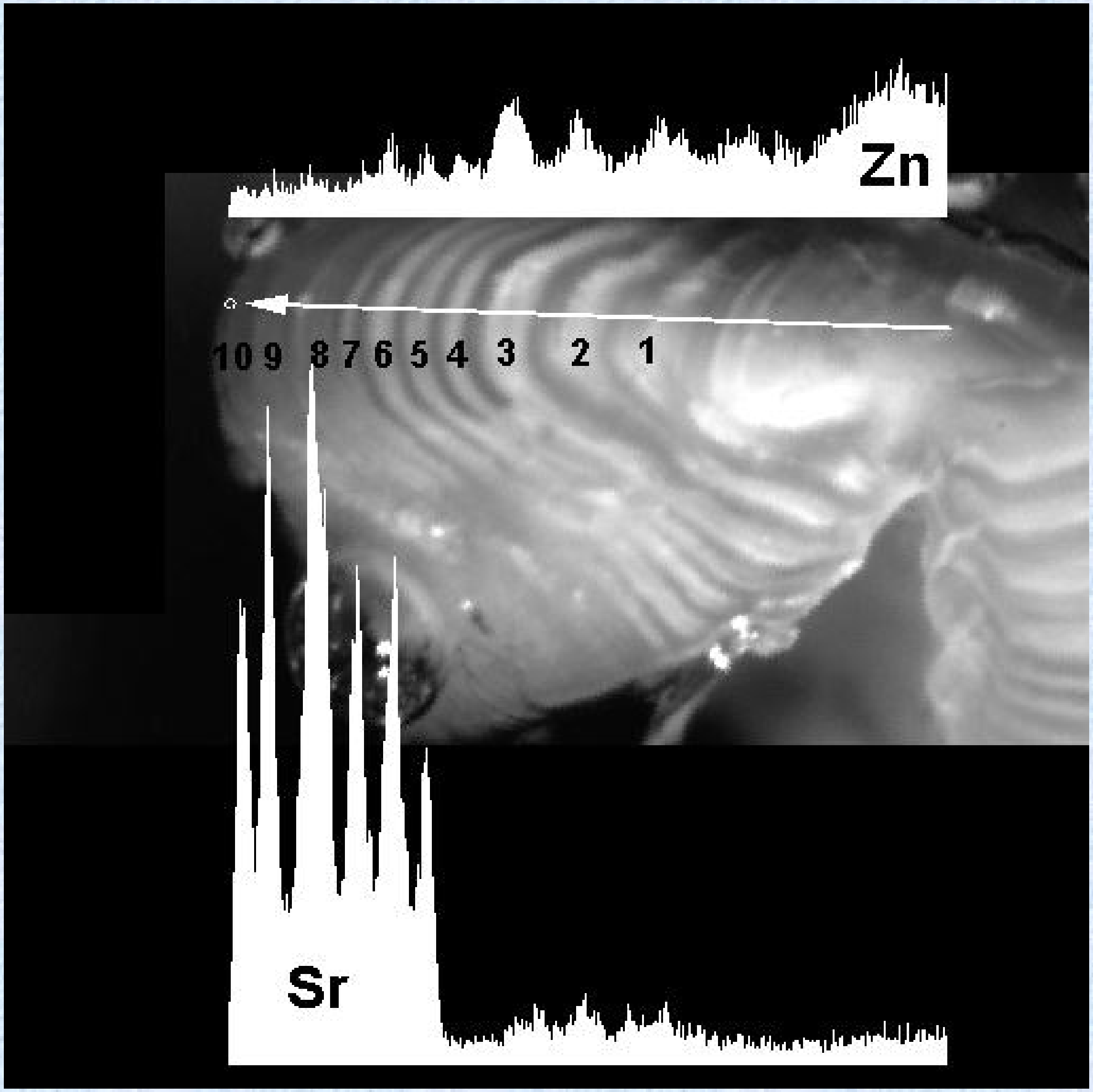
Laser Ablation - ICP - MS



laser ablation pits in zircon microcrystal



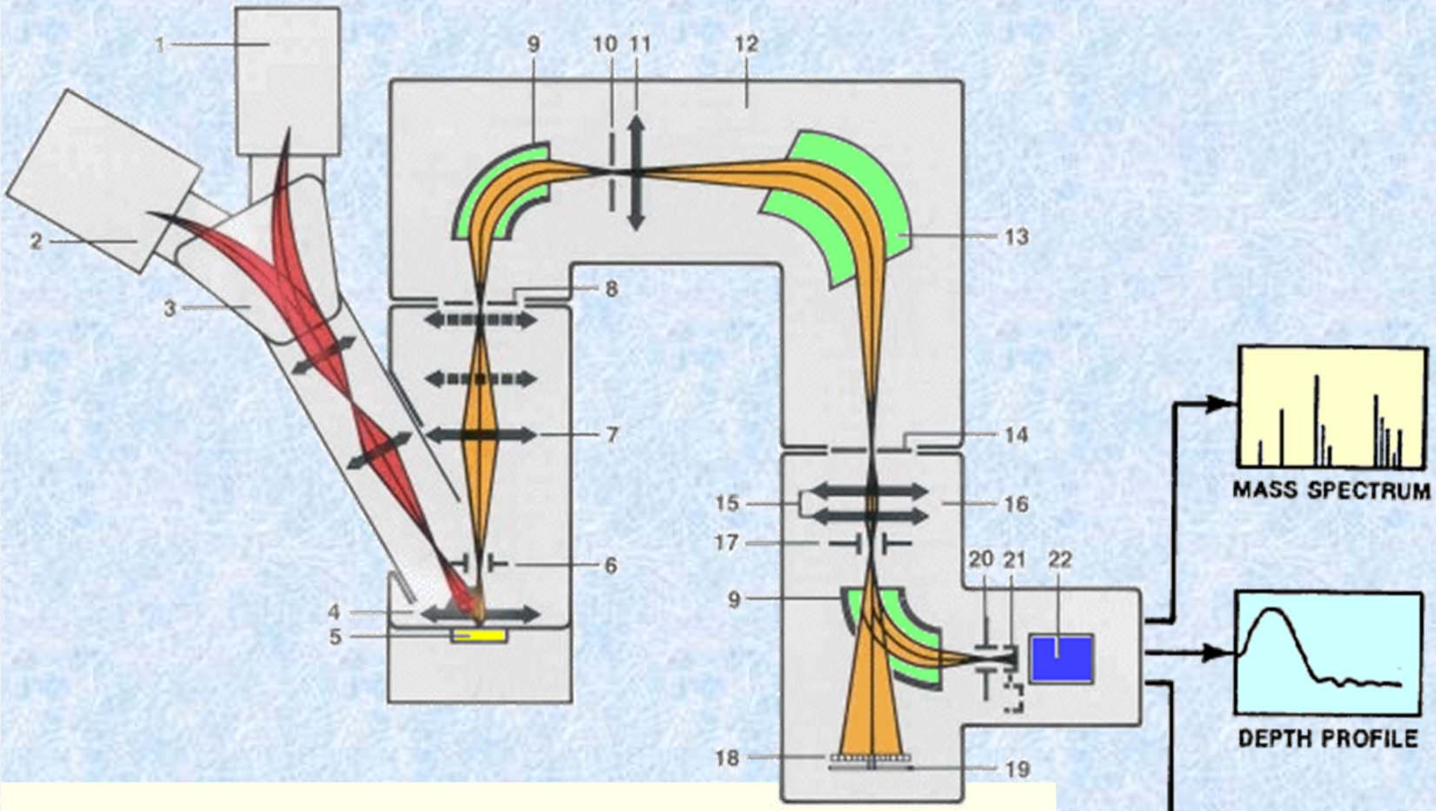
Laser Ablation - ICP - MS



http://www.umanitoba.ca/geoscience/faculty/halden/enviro.html

otolith from an anadroumous charr — the high Sr in year 5 probably marks the first marine excursion

SIMS - Secondary Ion Mass Spectroscopy

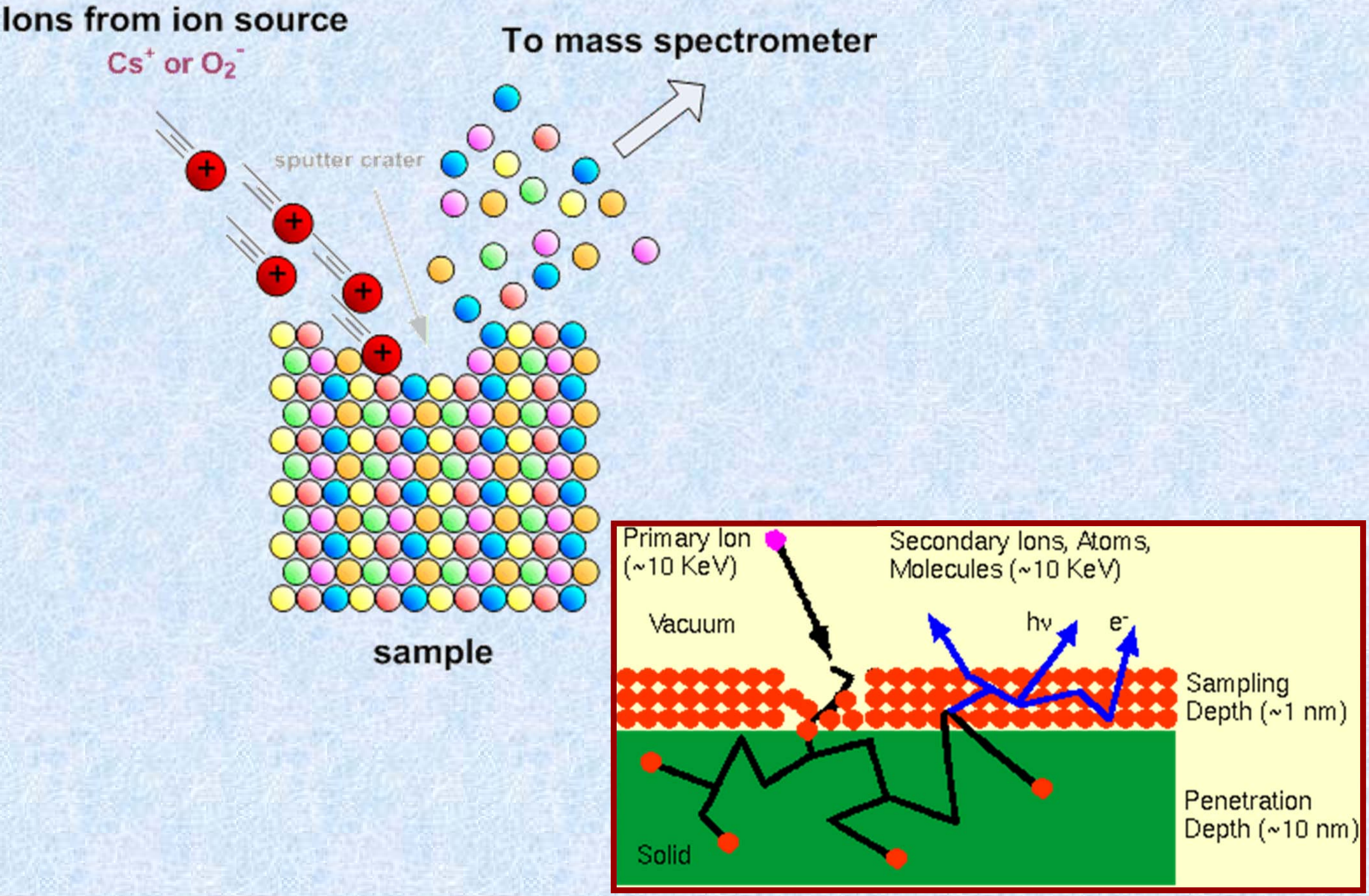


1 Cesium ion source
2 Duoplasmatron ion source
3 Primary beam mass filter
4 Immersion lens
5 Specimen
6 Dynamic transfer system
7 Transfer optical system
8 Entrance slit
9 Electrostatic sector
10 Energy slit
11 Spectrometer lens

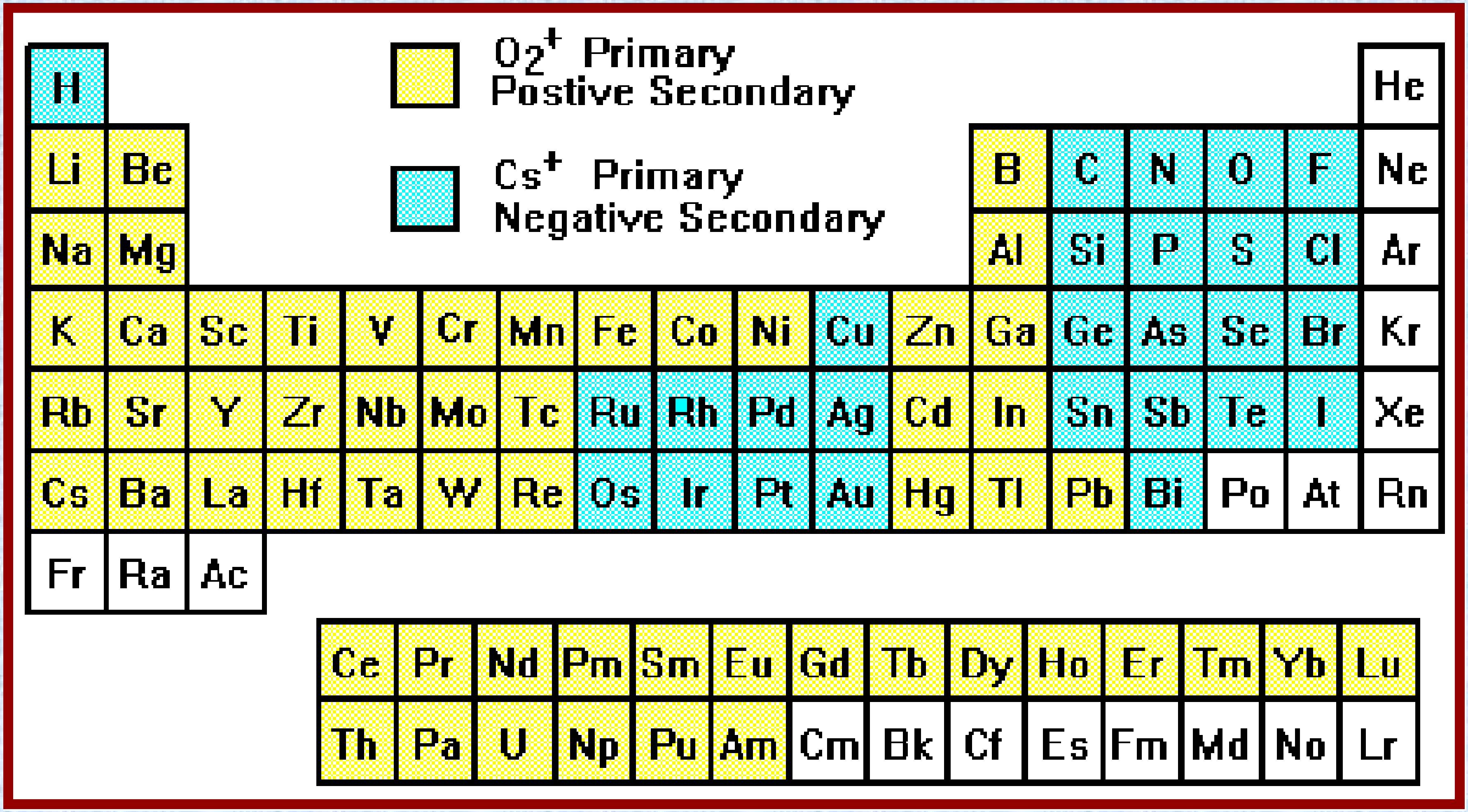
12 Spectrometer
13 Electromagnet
14 Exit slit
15 Projection lenses
16 Projection display and detection system
17 Deflector
18 Channel-plate
19 Fluorescent screen
20 Deflector
21 Remote controlled Faraday cup
22 Electron multiplier

SIMS Instrument

SIMS - Sampling Depth



SIMS - Elements



🟨 O_2^+ Primary
Postive Secondary

🟦 Cs^+ Primary
Negative Secondary

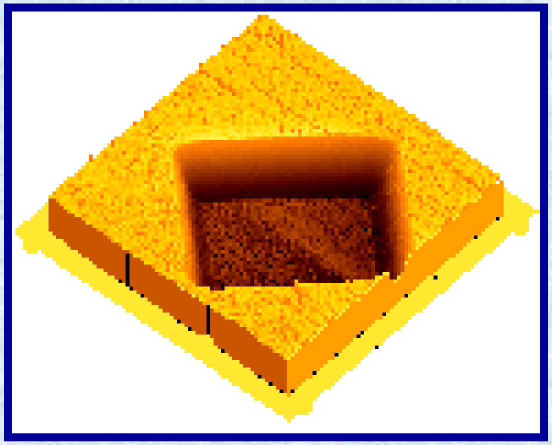
🟦 H																	He
🟨 Li	🟨 Be											🟨 B	🟦 C	🟦 N	🟦 O	🟦 F	Ne
🟨 Na	🟨 Mg											🟨 Al	🟦 Si	🟦 P	🟦 S	🟦 Cl	Ar
🟨 K	🟨 Ca	🟨 Sc	🟨 Ti	🟨 V	🟨 Cr	🟨 Mn	🟨 Fe	🟨 Co	🟨 Ni	🟦 Cu	🟨 Zn	🟨 Ga	🟦 Ge	🟦 As	🟦 Se	🟦 Br	Kr
🟨 Rb	🟨 Sr	🟨 Y	🟨 Zr	🟨 Nb	🟨 Mo	🟨 Tc	🟦 Ru	🟦 Rh	🟦 Pd	🟦 Ag	🟨 Cd	🟨 In	🟦 Sn	🟦 Sb	🟦 Te	🟦 I	Xe
🟨 Cs	🟨 Ba	🟨 La	🟨 Hf	🟨 Ta	🟨 W	🟨 Re	🟦 Os	🟦 Ir	🟦 Pt	🟦 Au	🟨 Hg	🟨 Tl	🟨 Pb	🟦 Bi	Po	At	Rn
Fr	Ra	Ac															
			🟨 Ce	🟨 Pr	🟨 Nd	🟨 Pm	🟨 Sm	🟨 Eu	🟨 Gd	🟨 Tb	🟨 Dy	🟨 Ho	🟨 Er	🟨 Tm	🟨 Yb	🟨 Lu	
			🟨 Th	🟨 Pa	🟨 U	🟨 Np	🟨 Pu	🟨 Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr	

SIMS - Advantages

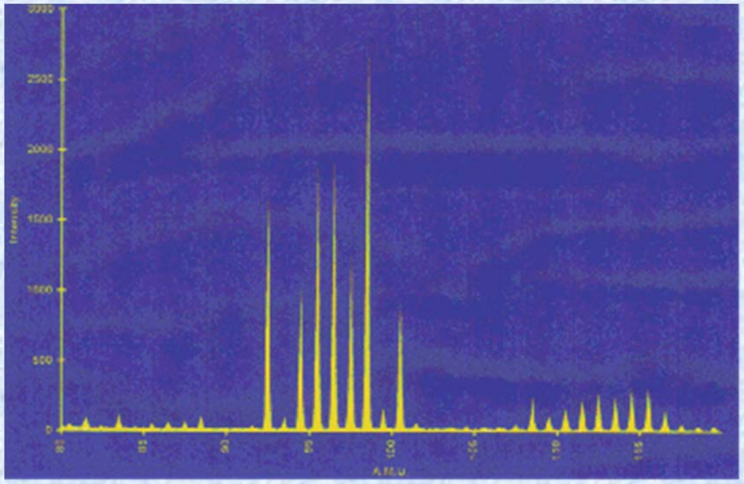
The advantages Time-of-Flight SIMS analysis are:

- Surface sensitivity
- Chemical compound identification
- Elemental and chemical mapping
- Trace element sensitivity (ppm or ppb, in some cases)
- Retrospective analysis
- Analysis of insulating and conducting samples
- Depth profiling
- Nominal sample damage
- Color output

SIMS - Sampling



SIMS sputtering crater



SIMS atomic mass spectrum

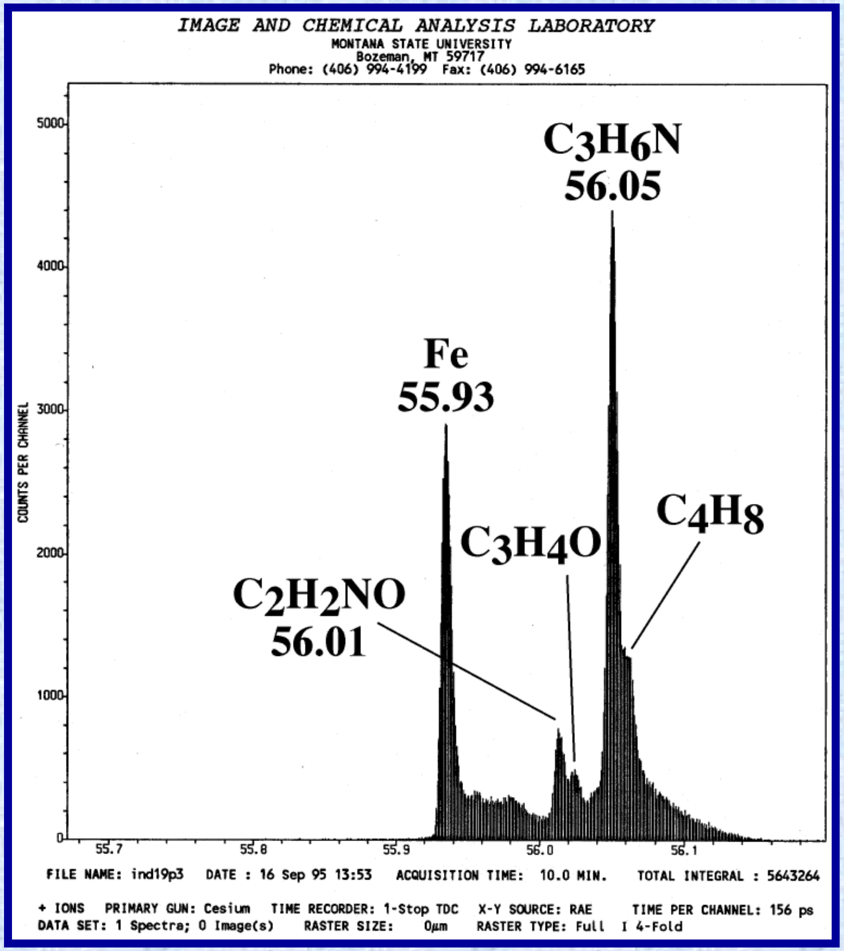


IMAGE AND CHEMICAL ANALYSIS LABORATORY
MONTANA STATE UNIVERSITY
Bozeman, MT 59717
Phone: (406) 994-4199 Fax: (406) 994-6165

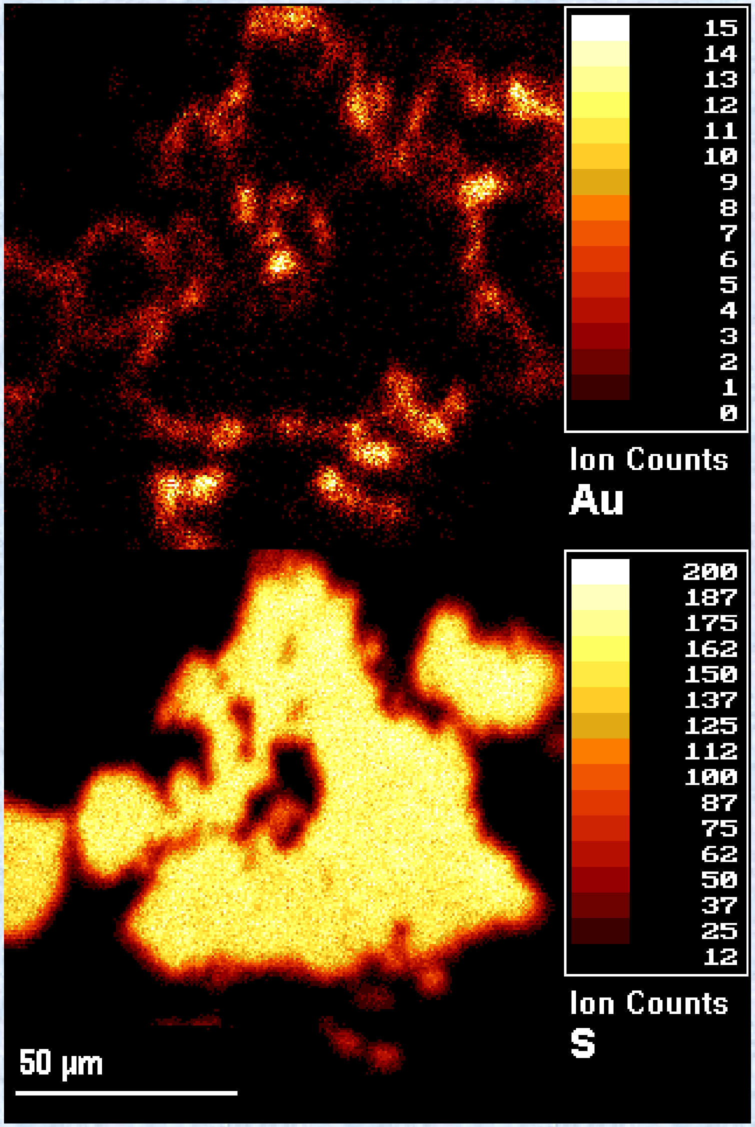
FILE NAME: ind19p3 DATE : 16 Sep 95 13:53 ACQUISITION TIME: 10.0 MIN. TOTAL INTEGRAL : 5643264

+ IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: RAE TIME PER CHANNEL: 156 ps
DATA SET: 1 Spectra; 0 Image(s) RASTER SIZE: 0µm RASTER TYPE: Full I 4-Fold

SIMS molecular spectrum

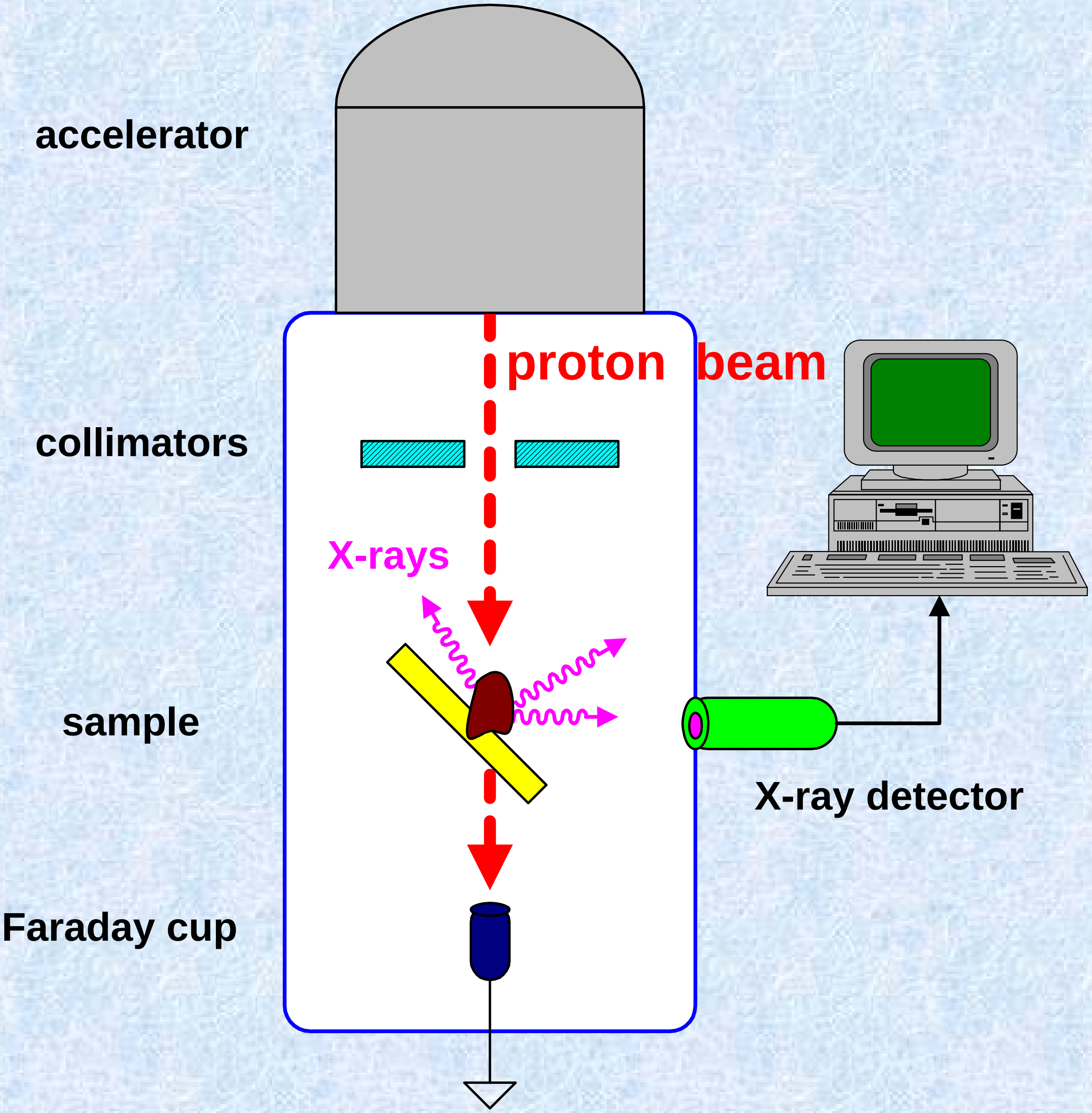
SIMS - Imaging

rock containing pyrite, FeS_2; gold, Au; and bornite, Cu_5FeS_4

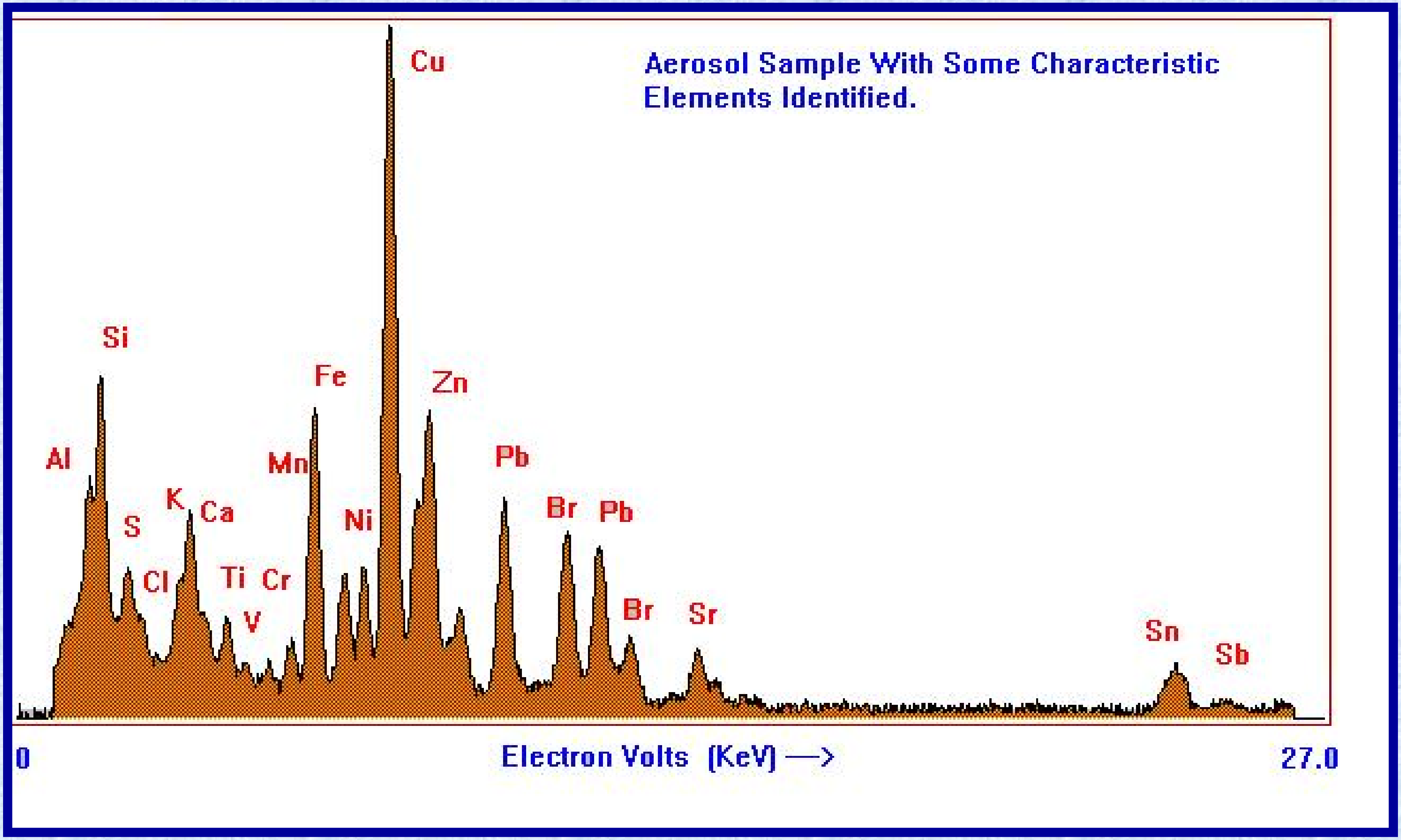


SIMS image of gold in FeS_2, pyrite

PIXE - Proton Induced X-Ray Emission

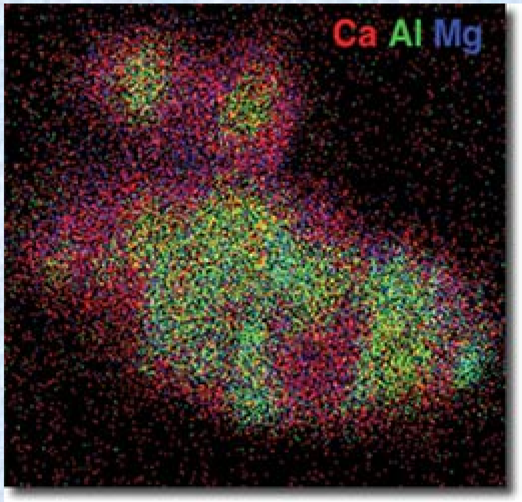


PIXE - Proton Induced X-Ray Emission

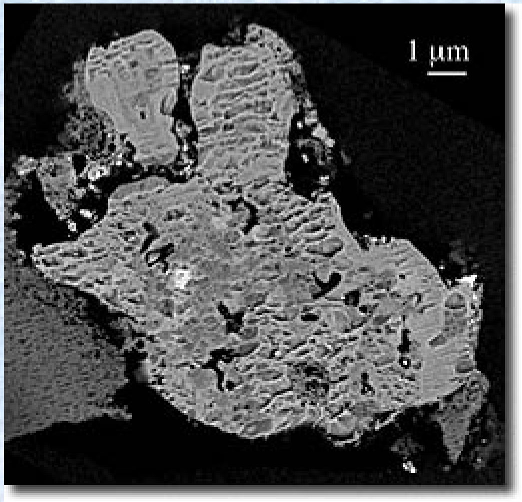


PIXE - Proton Induced X-Ray Emission

PIXE – Aerogel Comet Mission

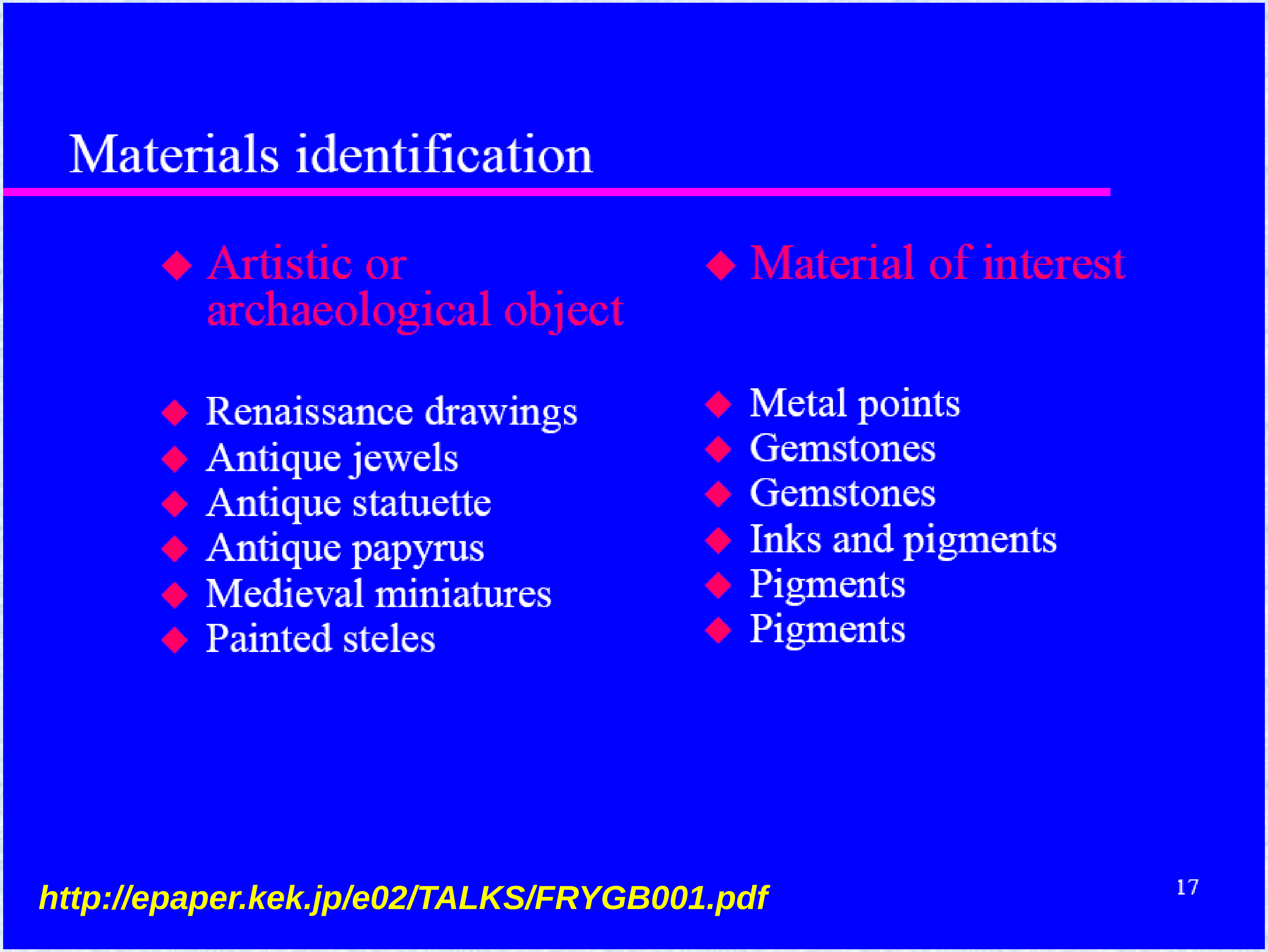


Stardust fire and ice: Livermore scientists were able to determine that this tiny mineral is osbornite using X-ray fluorescence maps (above) and backscattered electron images (below). Osbornite forms much closer to the sun than where Comet Wild 2 formed on the outskirts of Neptune



The primary objective of the Stardust mission is to capture both cometary samples and interstellar dust. When the Stardust Spacecraft encounters the Comet Wild 2, the impact velocity of the particles will be up to 6 times the speed of a rifle bullet. Although the captured particles will each be smaller than a grain of sand, high-speed capture could alter their shape and chemical composition - or even vaporize them entirely.

PIXE - Proton Induced X-Ray Emission

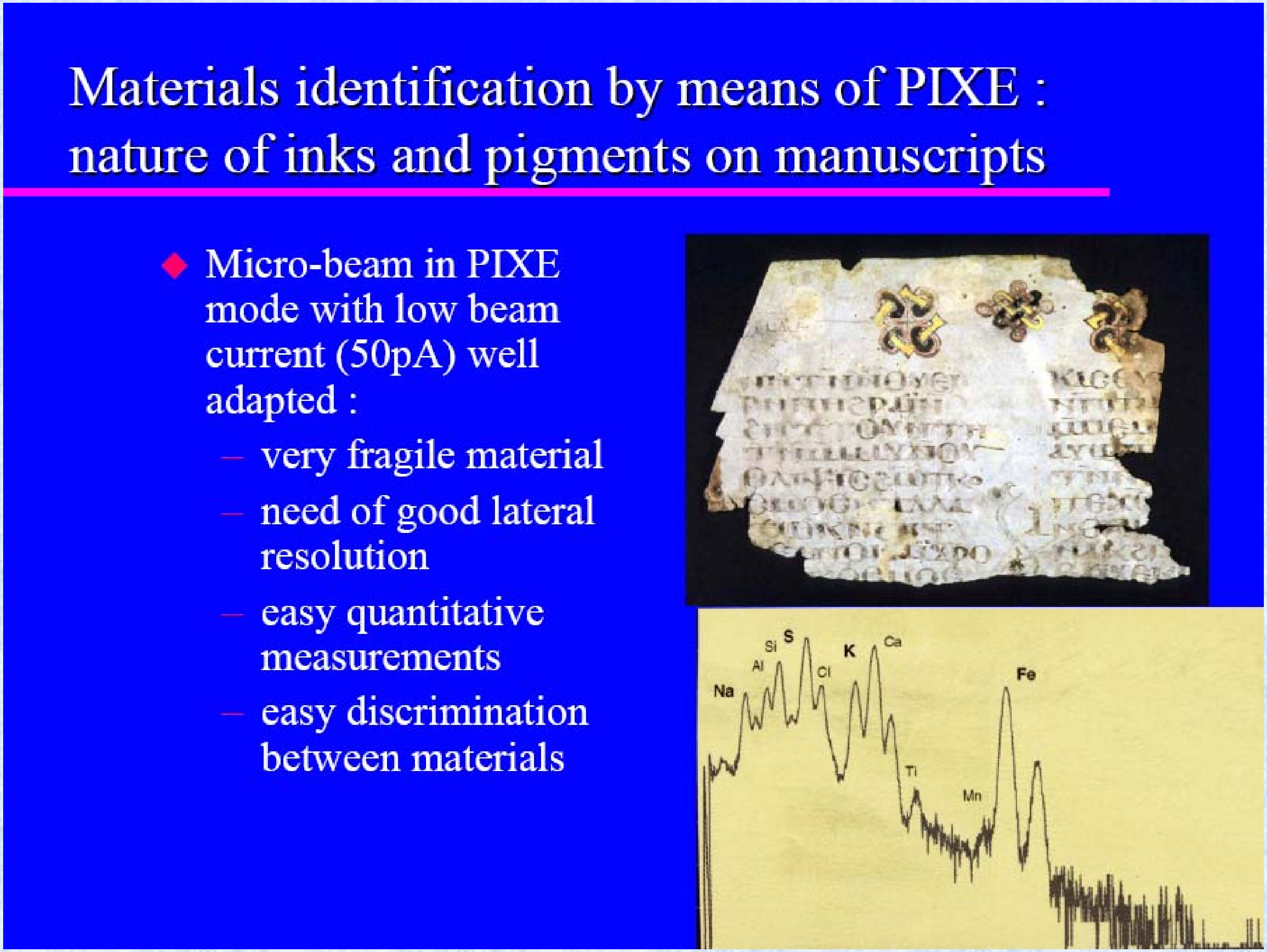


Materials identification

◆ Artistic or archaeological object	◆ Material of interest
◆ Renaissance drawings	◆ Metal points
◆ Antique jewels	◆ Gemstones
◆ Antique statuette	◆ Gemstones
◆ Antique papyrus	◆ Inks and pigments
◆ Medieval miniatures	◆ Pigments
◆ Painted steles	◆ Pigments

http://epaper.kek.jp/e02/TALKS/FRYGB001.pdf

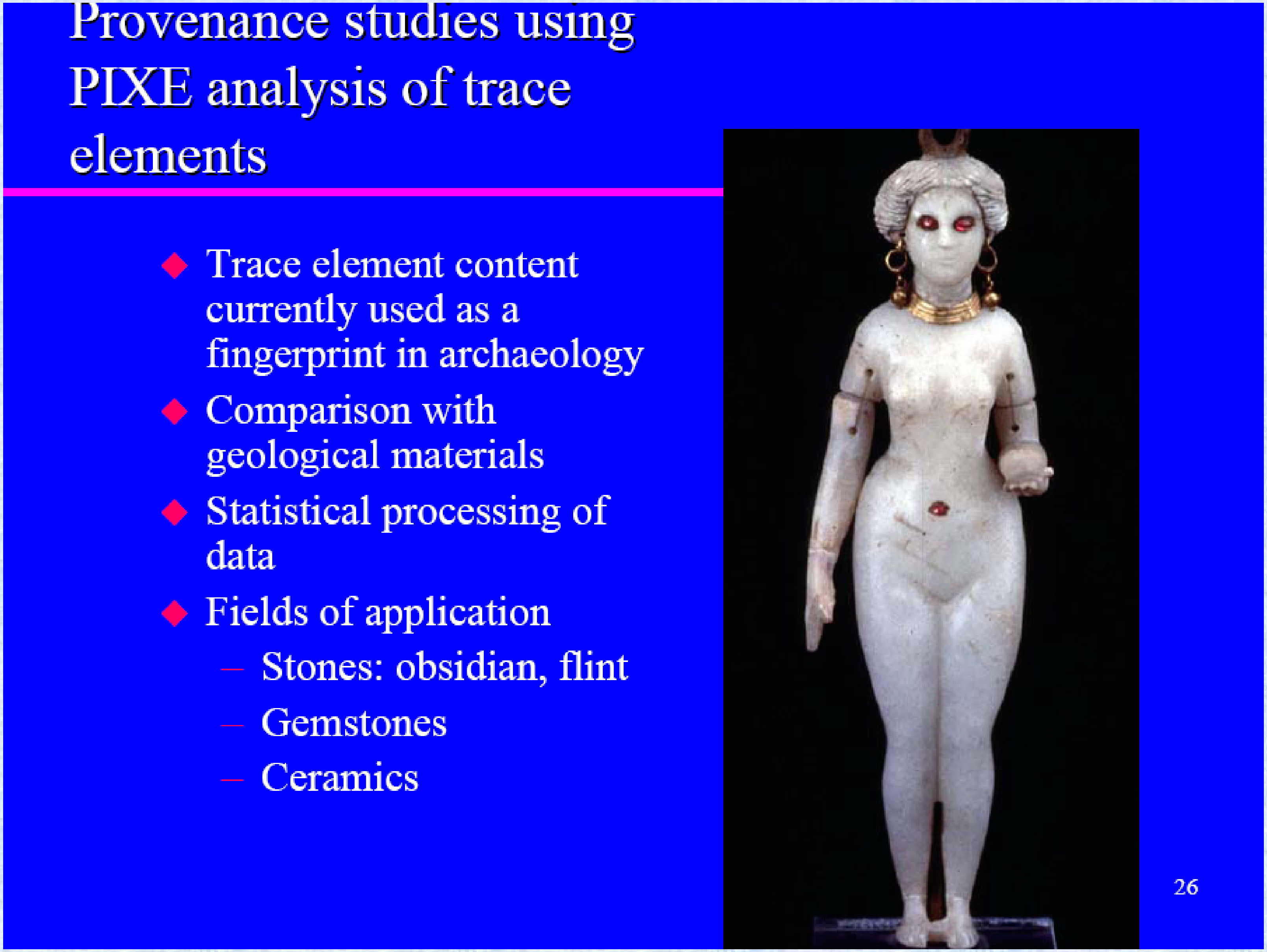
PIXE - Proton Induced X-Ray Emission



Materials identification by means of PIXE : nature of inks and pigments on manuscripts

- Micro-beam in PIXE mode with low beam current (50pA) well adapted :
  - very fragile material
  - need of good lateral resolution
  - easy quantitative measurements
  - easy discrimination between materials

PIXE - Proton Induced X-Ray Emission



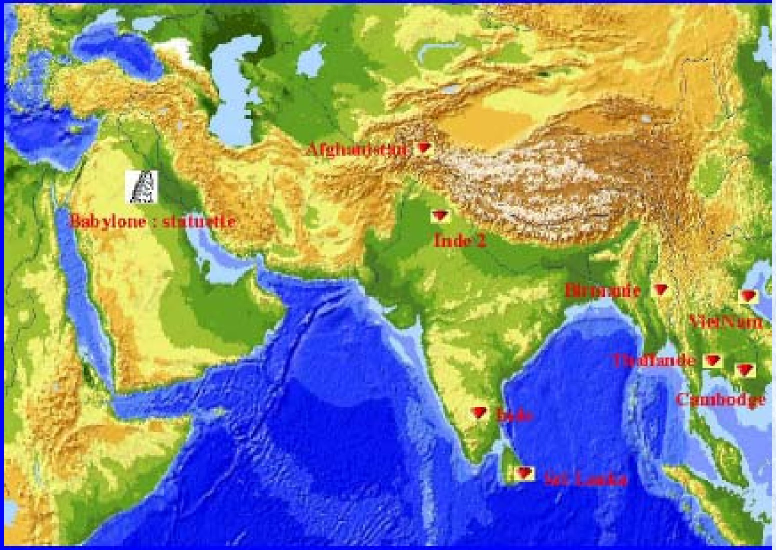
Provenance studies using PIXE analysis of trace elements

- Trace element content currently used as a fingerprint in archaeology
- Comparison with geological materials
- Statistical processing of data
- Fields of application
  - Stones: obsidian, flint
  - Gemstones
  - Ceramics

PIXE - Proton Induced X-Ray Emission

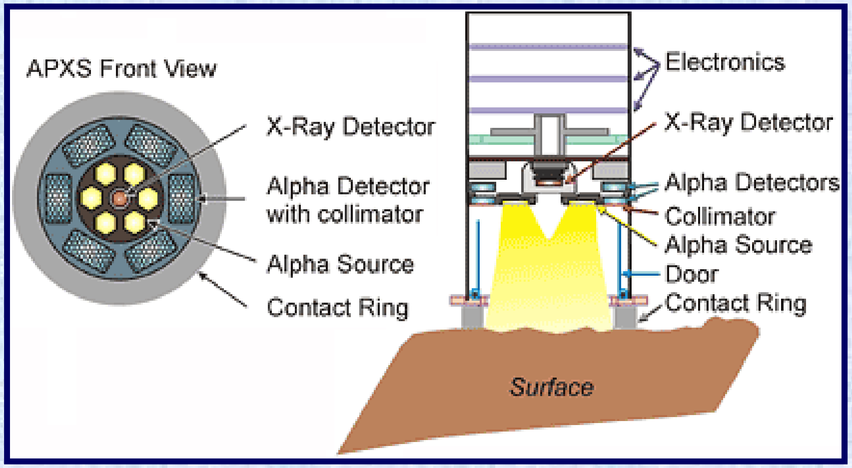
PIXE - Proton Induced X-Ray Emission

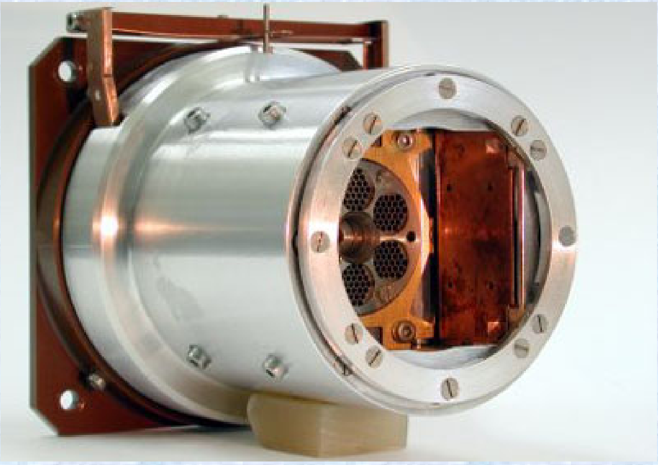
Provenance
of statuette rubies

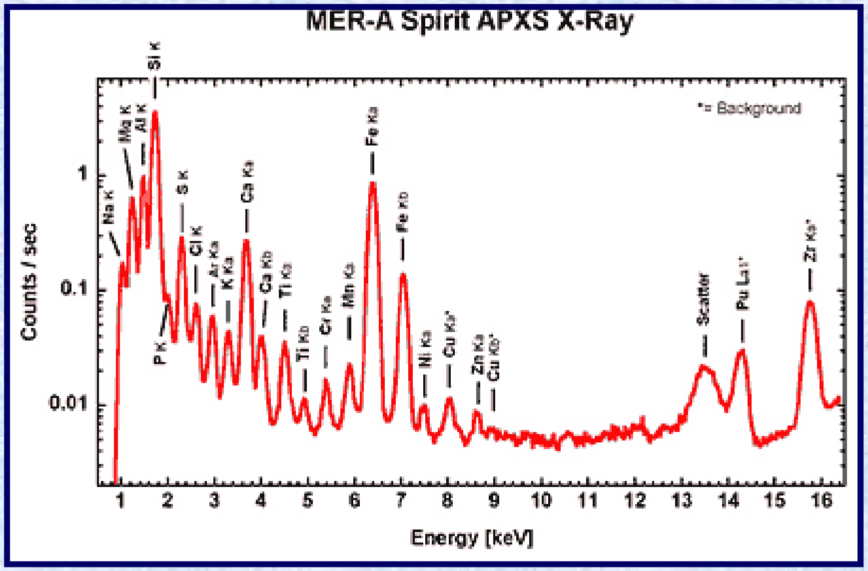


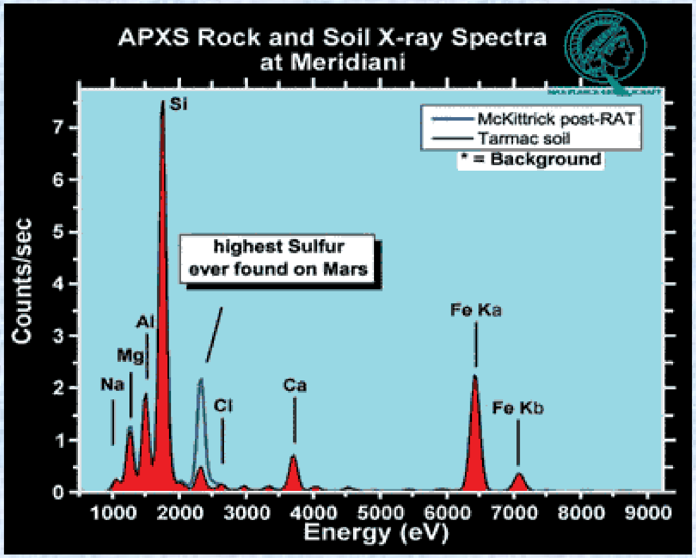
APXS - Alpha Particle X-Ray Spectroscopy

Alpha Particle X-ray Spectrometer works by exposing Mars materials to energetic alpha particles and x-rays from a radioactive ^{244}Cm source, and then measuring the energy spectra of backscattered alphas and emitted-x rays.

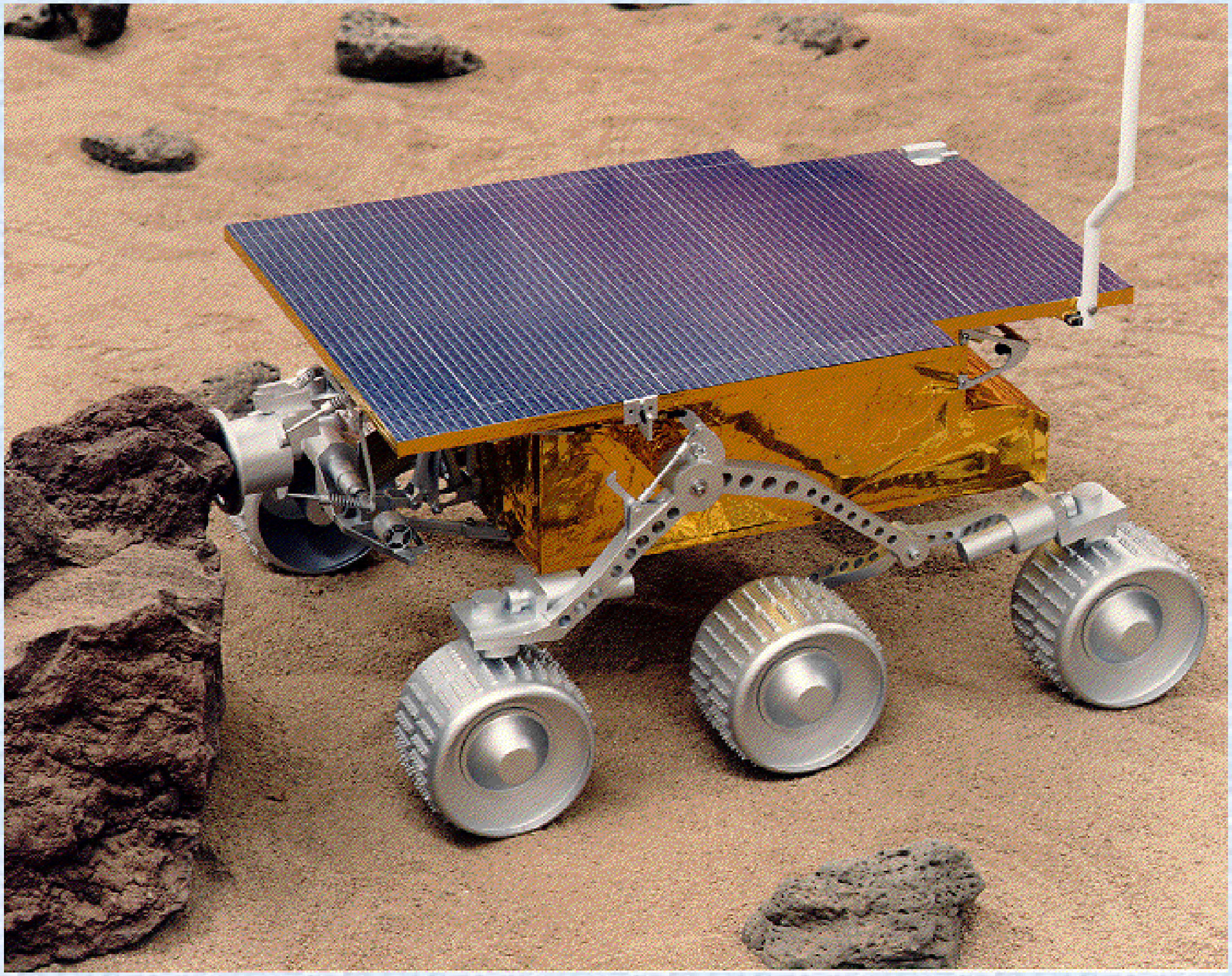


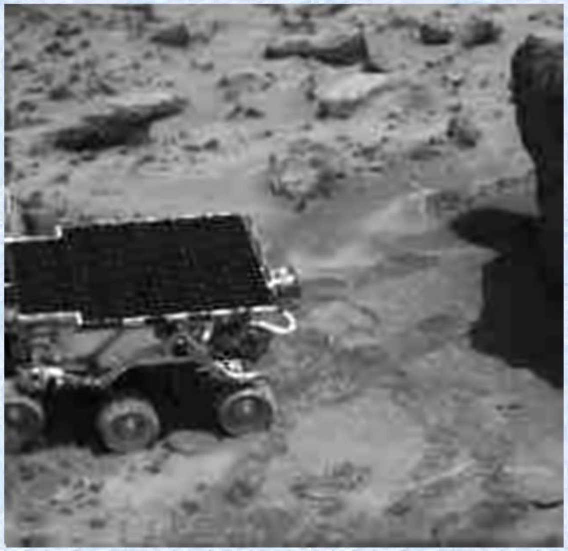






APXS - Alpha Particle X-Ray Spectroscopy





Mössbauer Spectroscopy

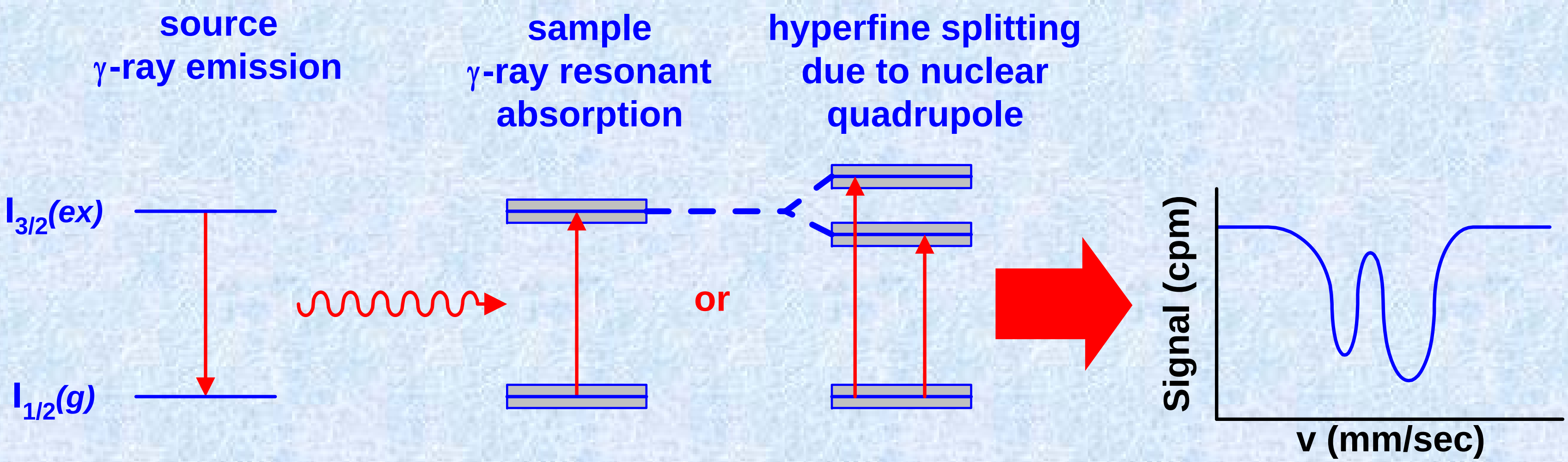
- for low energy γ-ray emission ($E\gamma < 50$ keV), photon emission and absorption are **recoiless** (no **phonons** are created are destroyed)
- emission width is then governed by lifetime uncertainty ($\Delta E \Delta t \geq \hbar$)
- for $\Delta t \approx 100 \times 10^{-9}$ sec, $\Delta E \approx 1 \times 10^{-9}$ eV
- use **Doppler shift** to vary Eγ

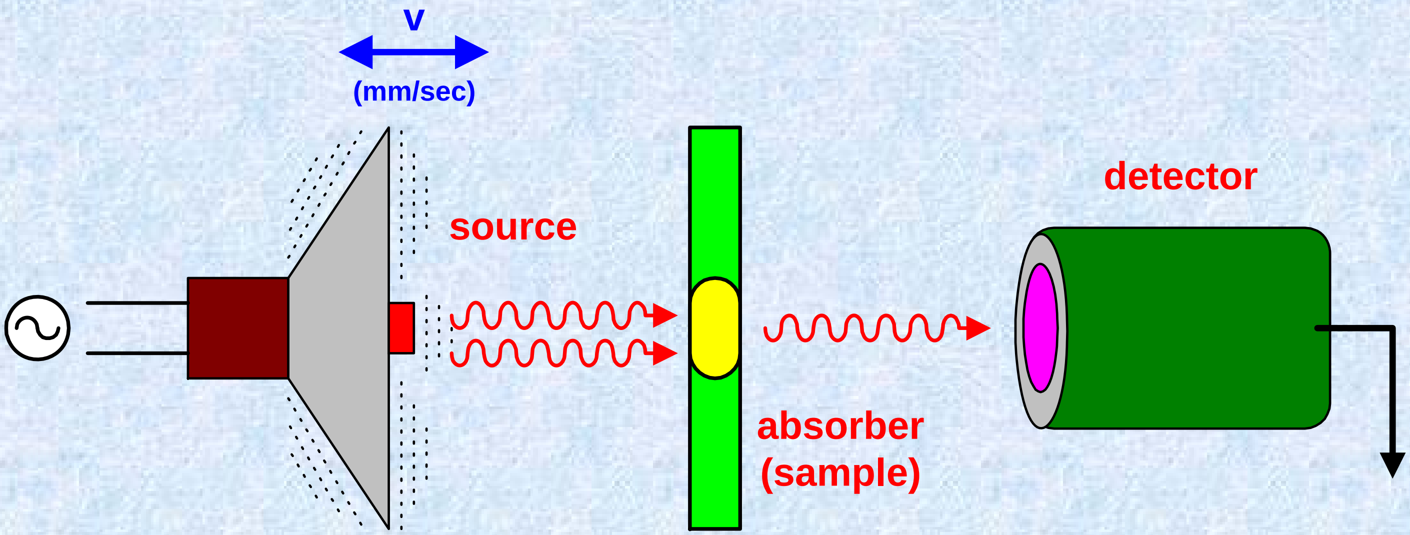
$$\boxed{E_\gamma = E_\gamma^0 \left(1 - \frac{v}{c} \cos\Theta\right)}$$

- typical values:

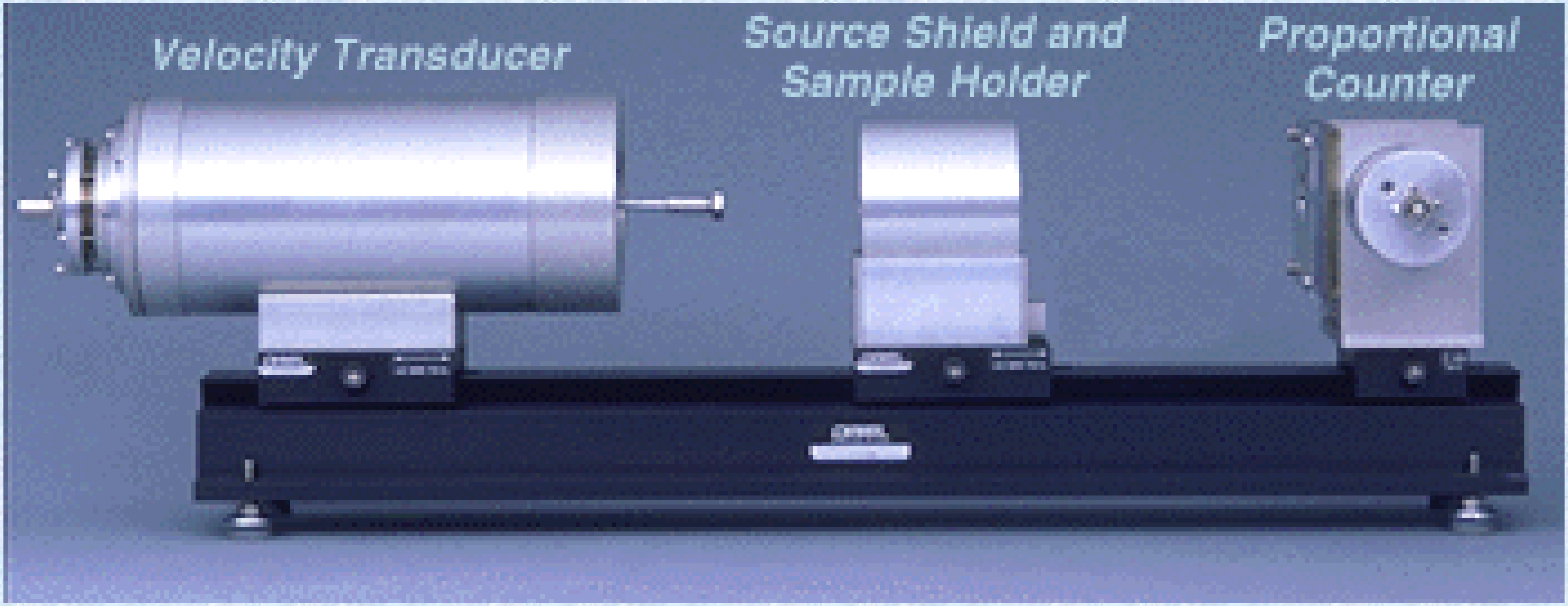
  $v \approx 1$cm/sec $\quad \Delta E \approx 10^{-6}$ eV $\quad f \approx 100$ MHz

Mössbauer Spectroscopy

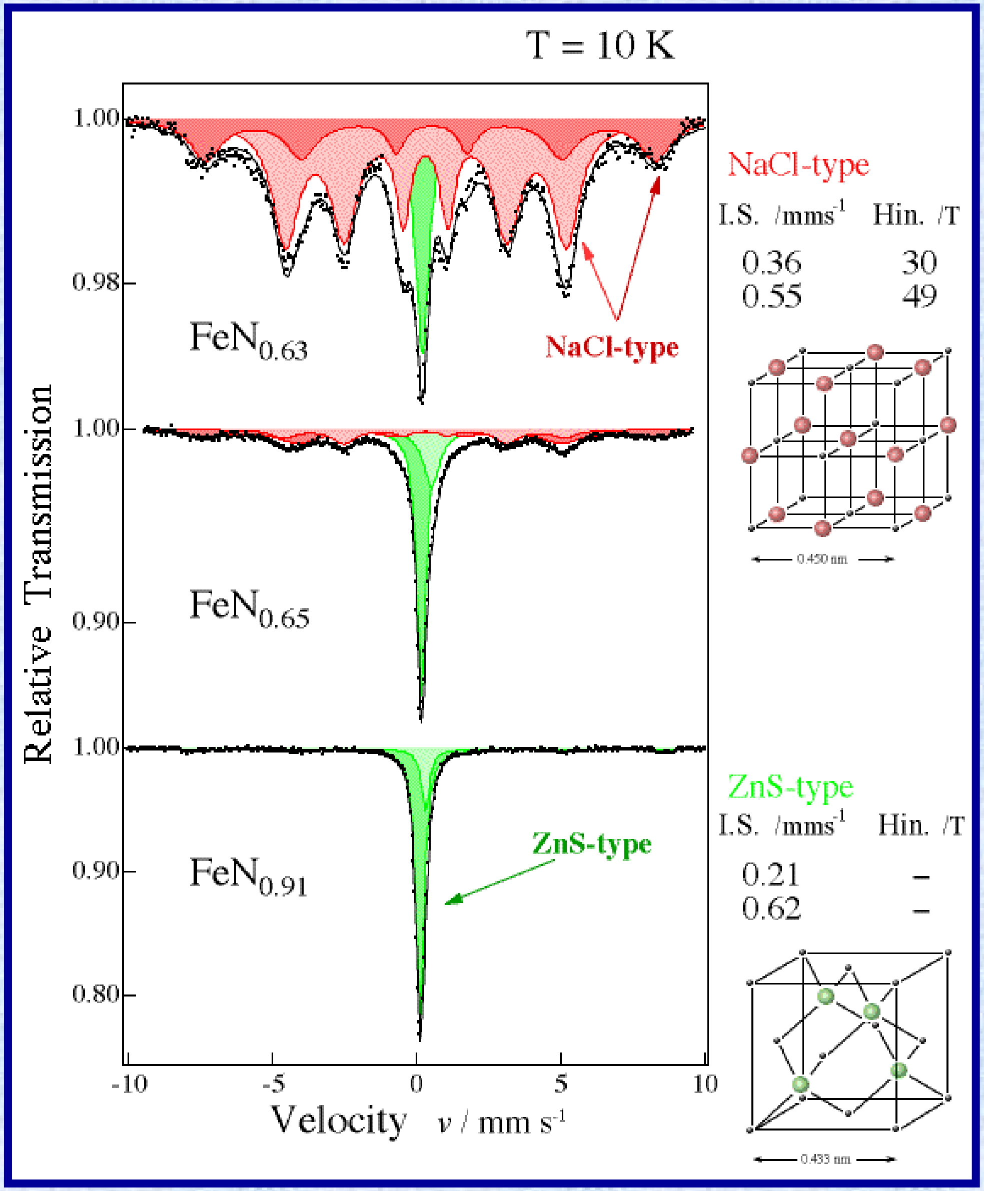




Mössbauer Spectroscopy



Mössbauer Spectroscopy



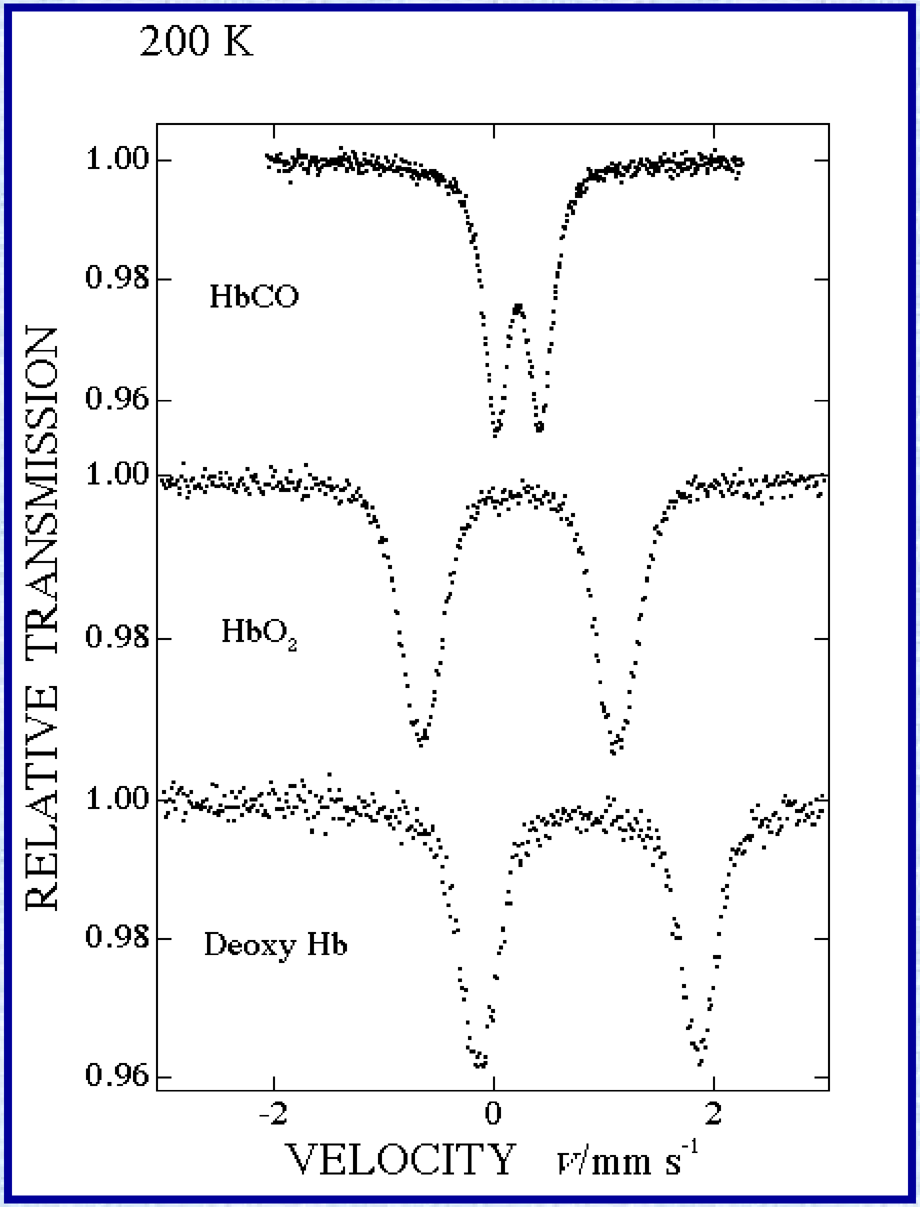
NaCl-type

I.S. /mms^{-1}	Hin. /T
0.36	30
0.55	49

ZnS-type

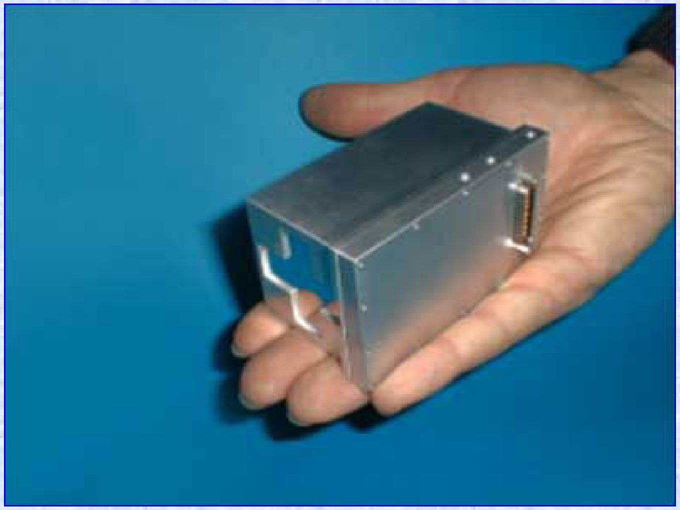
I.S. /mms^{-1}	Hin. /T
0.21	–
0.62	–

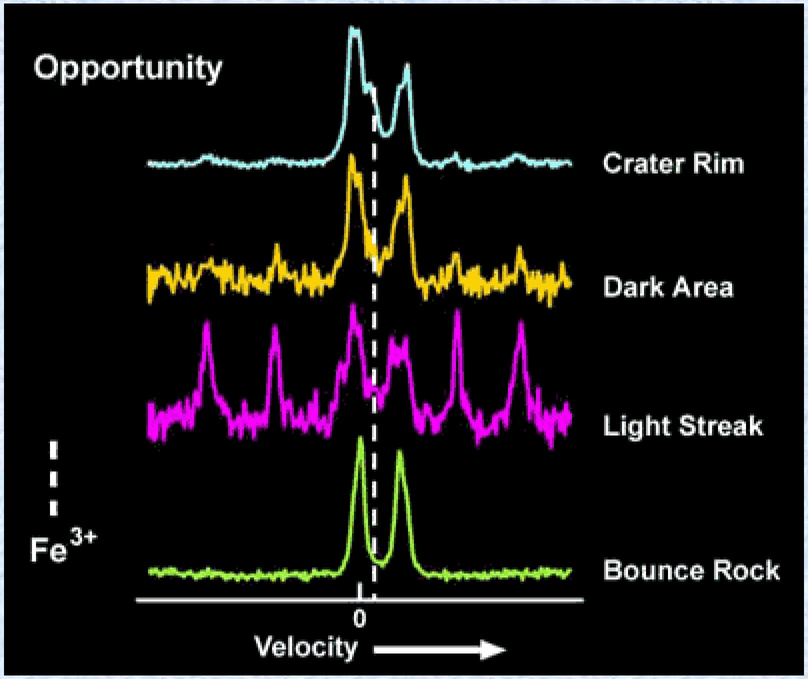
Mössbauer Spectroscopy

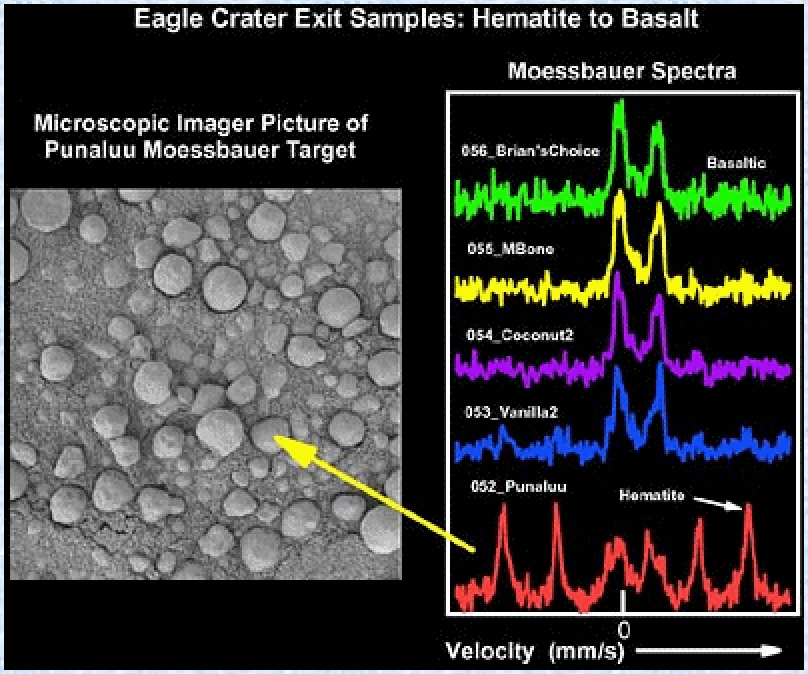


Mossbauer spectrum of hemoglobin

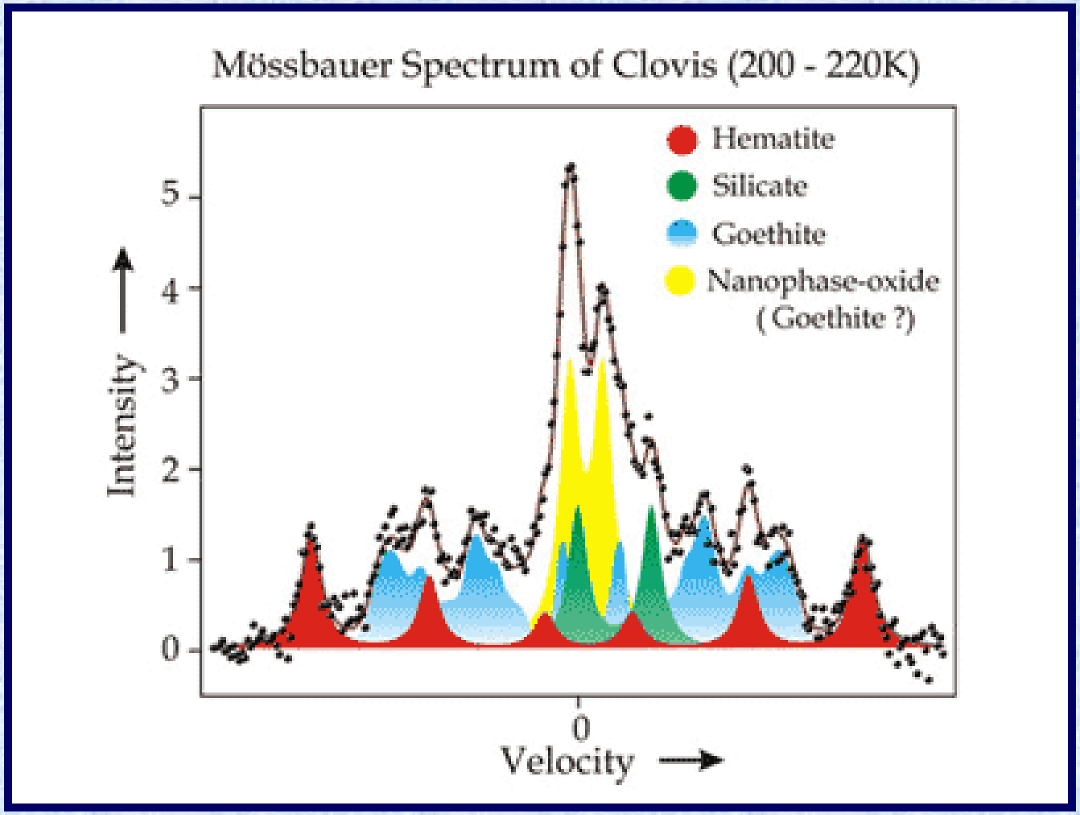
Mössbauer Spectroscopy on Mars







Mössbauer Spectroscopy on Mars



Hematite (α-Fe_2O_3) deposits are mostly sedimentary in origin, They are found throughout the world and are the most important iron ore in the world today.

Goethite (α-$FeOOH$) is a common iron mineral. It often forms by weathering of other iron-rich minerals, thus is a common component of soils.

Magnetite (Fe_3O_4) is a naturally occurring metallic mineral that is occasionally found in sufficient quantities to be an ore of iron.

Limonite is a mixture of hydrated iron oxides, primarily goethite.

Chapter 10 - Glossary

Advanced Light Sources
Alpha Particle X-ray Spectrometry (AXPS)
Auger Electron Spectroscopy
Electron Microprobe
Electron Spectroscopy for Chemical Analysis (ESCA)
electron-beam curing
electron-beam sterilization
Energy Dispersive X-ray Analysis
Extended X-ray Absorption Fine Structure (EXAFS)
fast neutron activation analysis
fill level gauge
food irradiation
industrial CT scanning
industrial radiography
instrumental neutron activation analysis
K-edge, L-edge
laser ablation
Moseley's Law
Mössbauer Spectroscopy
neutron absorption
non-destructive testing (NDT)
portable XRF analyzers
prompt gamma neutron activation analysis
Proton Induced X-Ray Emission (PIXE)
radiochemical neutron activation analysis
radiography
Secondary Ion Mass Spectrometry (SIMS)
Synchrotron
thickness gauges
Time-of-Flight SIMS (TOF-SIMS)
X-ray Absorption Near Edge Structure (XANES)
X-Ray Crystallography
X-Ray diffractometer
X-Ray Fluorescence Spectrometry (XRF)
X-Ray line spectrum
X-Ray Photoemission Spectroscopy (XPS)
X-Ray Powder Diffraction (XRD)
X-Ray tubes