

2455-3

Workshop on Portable X-ray Analytical Instruments for Cultural Heritage

29 April - 3 May, 2013

**External-beam IBA methods for non-destructive analysis of Cultural Heritage
Artefacts**

Massimo Chiari
*I.N.F.N., Firenze
Italy*

External-beam IBA methods for non-destructive analysis of Cultural Heritage artefacts

Massimo Chiari / *I.N.F.N. Florence*

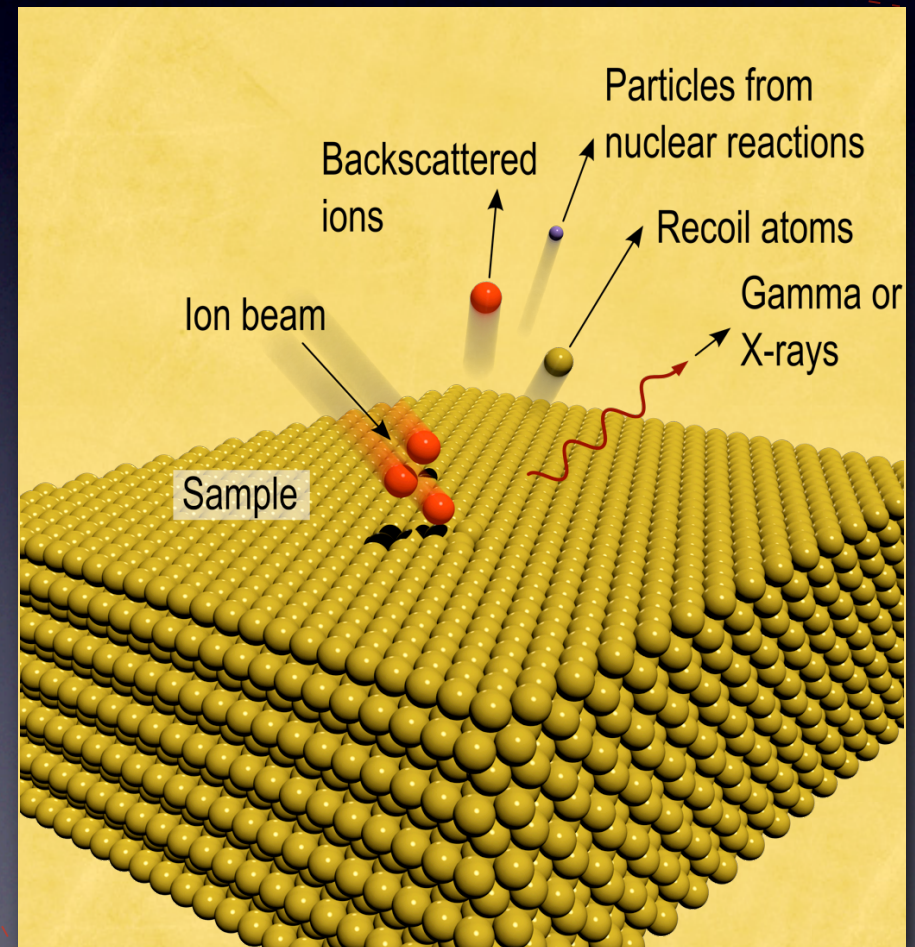
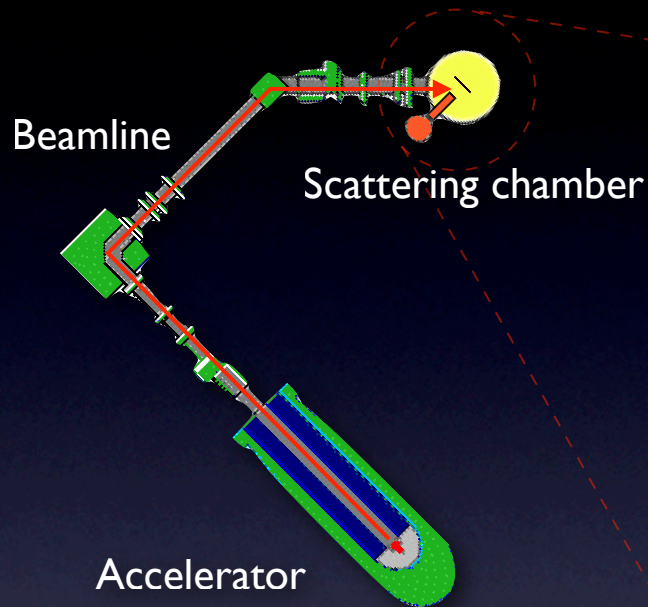
@ *chiari@fi.infn.it*

S *max0068*

Outline

- Introduction to Ion Beam Analysis
- External beams
- IBA applications to Cultural Heritage
- Essential bibliography

Ion Beam Analysis techniques



Beam IN	Beam OUT	Analytical technique
ion	ion	RBS, NRA
ion	target	ERDA, SIMS, SNMS
ion	X-ray	PIXE
ion	Gamma-ray	PIGE, Activation Analysis
ion	h ν	Ionoluminescence (IL)

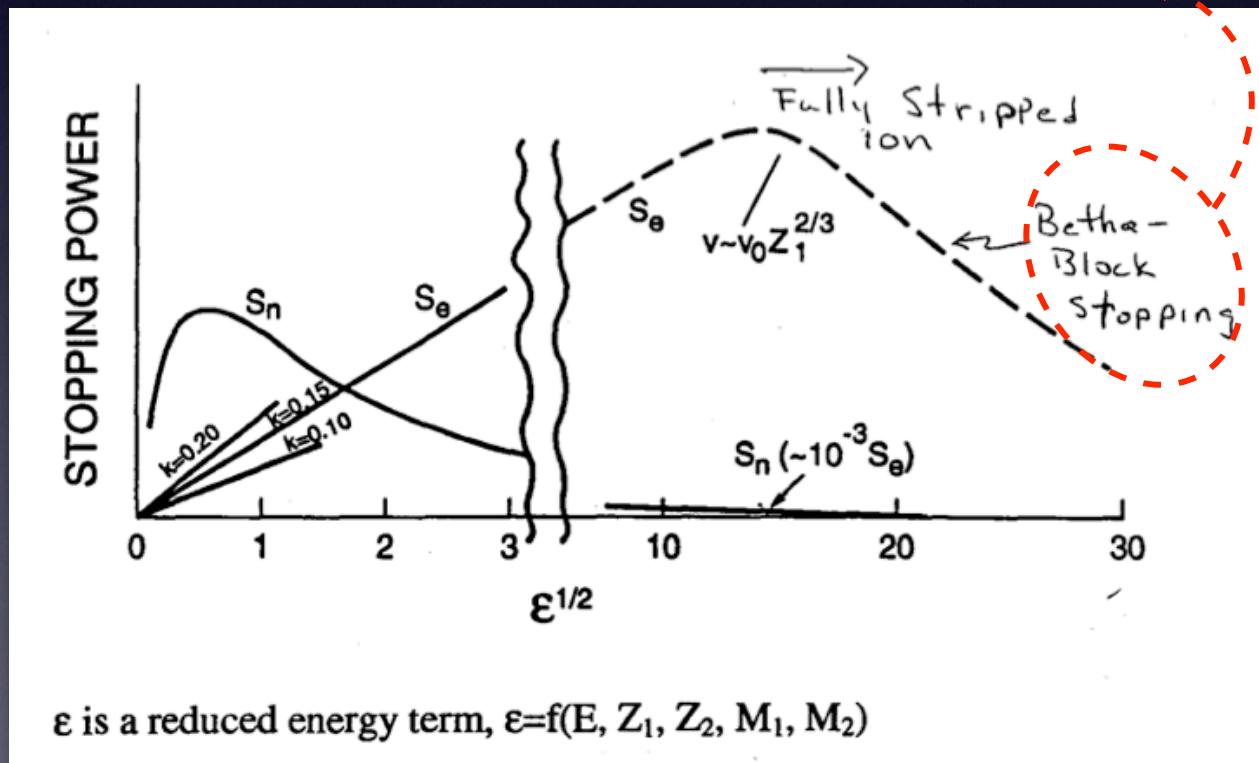
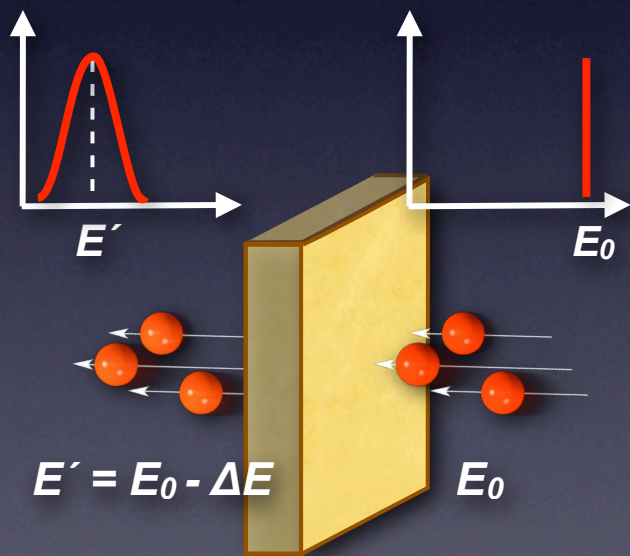
General features of IBA

- Multielemental
- Quantitative analysis (“traceability”)
- High sensitivity (1-100 ppm in at/cm³; 10¹¹-10¹² in at/cm²)
- Surface analysis (10 Å - 10 μm)
- Depth profiling
- Non-destructive
- No sample pre-treatment
- Microanalysis (lateral resolution <1 μm)
- 2D mapping

Stopping power

The “stopping power” (the energy lost per unit length, dE/dx) is a macroscopic and measurable quantity, describing the average interaction of the ion with the material:

$$-\frac{dE}{dx} = \frac{4\pi}{m_e c^2} \cdot \frac{n z^2}{\beta^2} \cdot \left(\frac{e^2}{4\pi\epsilon_0}\right)^2 \cdot \left[\ln \left(\frac{2m_e c^2 \beta^2}{I \cdot (1 - \beta^2)} \right) - \beta^2 \right]$$



ϵ is a reduced energy term, $\epsilon = f(E, Z_1, Z_2, M_1, M_2)$

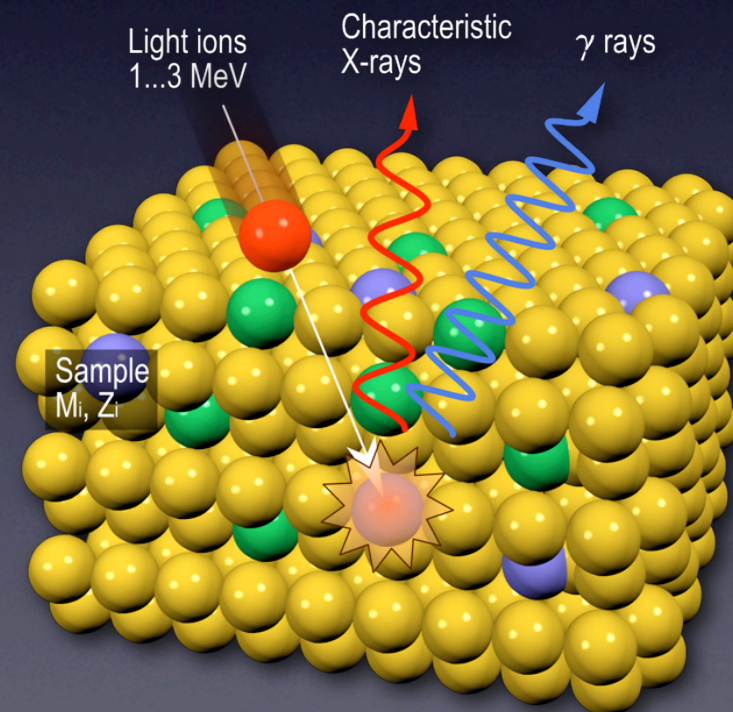
PIXE

Particle Induced X-ray Emission

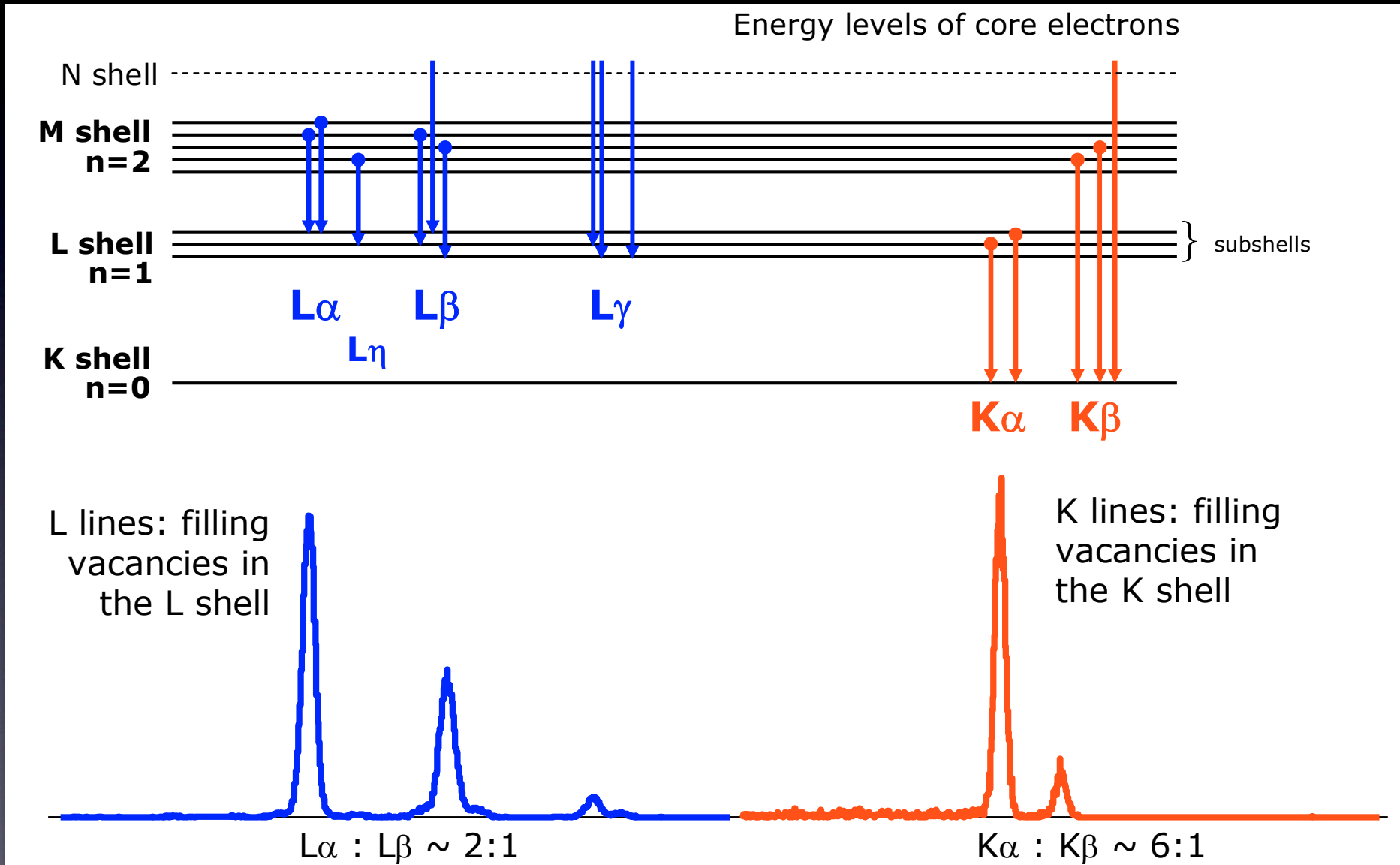
Emission of characteristic X-rays following ionization from incident ions



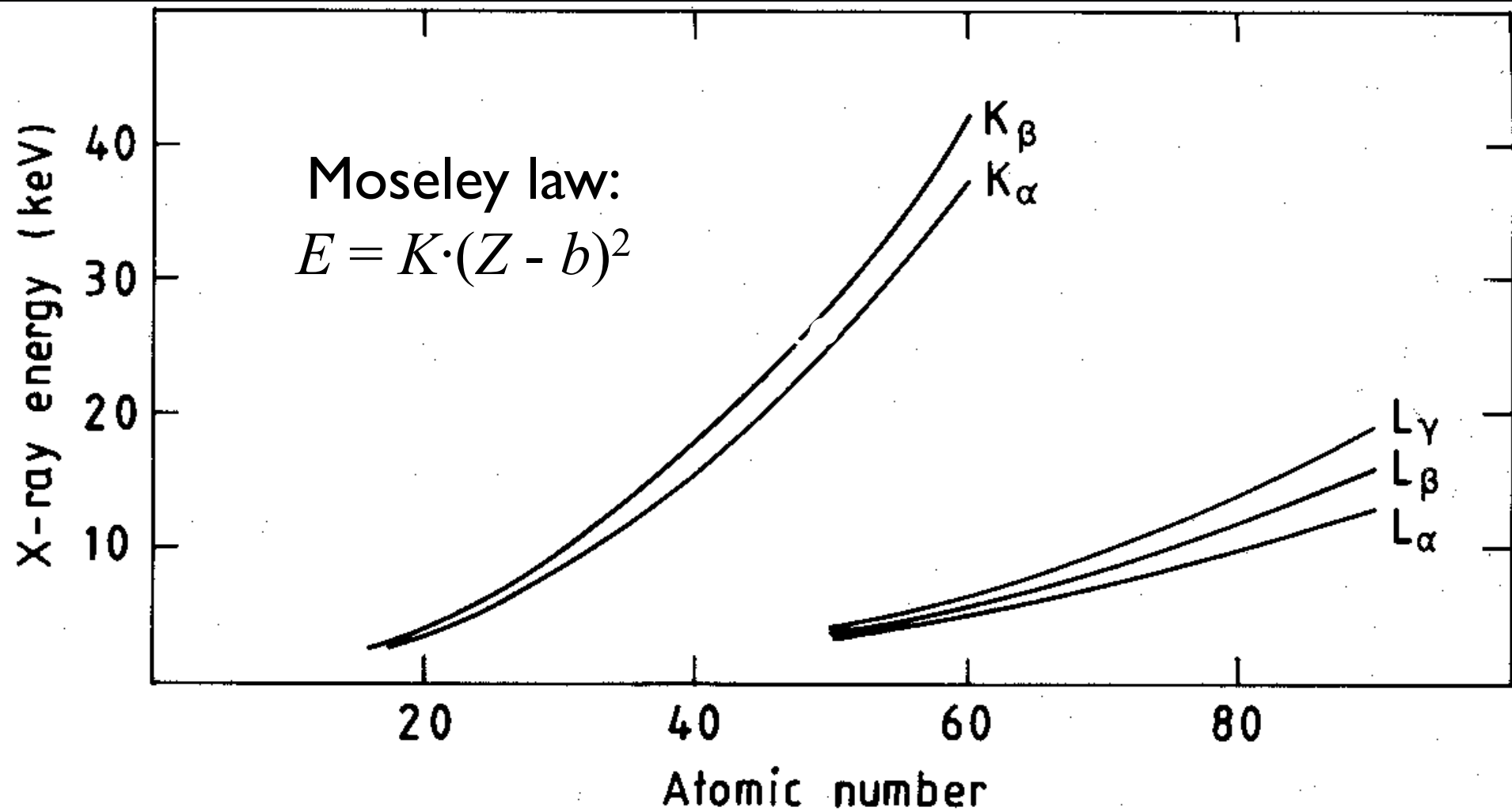
Z > 10



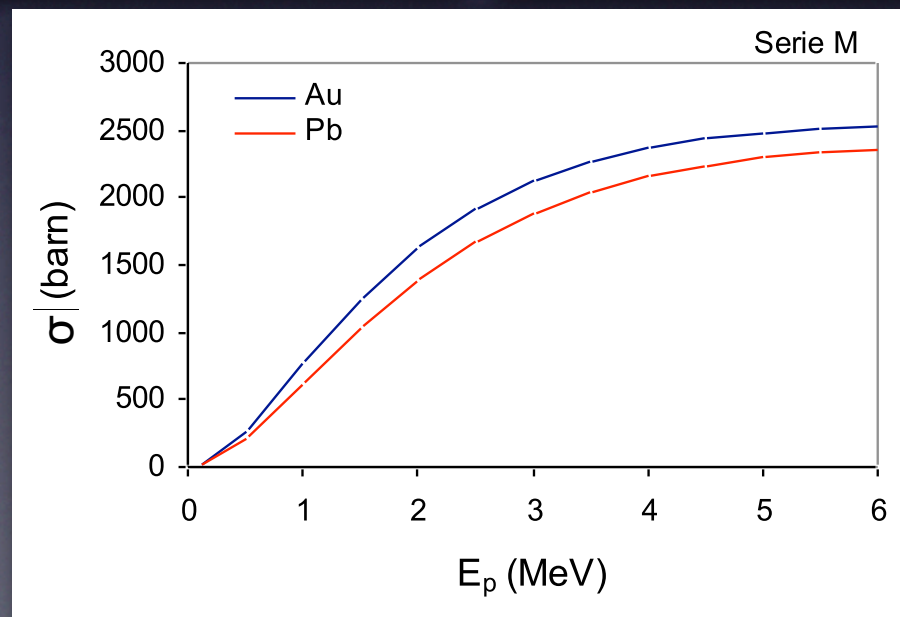
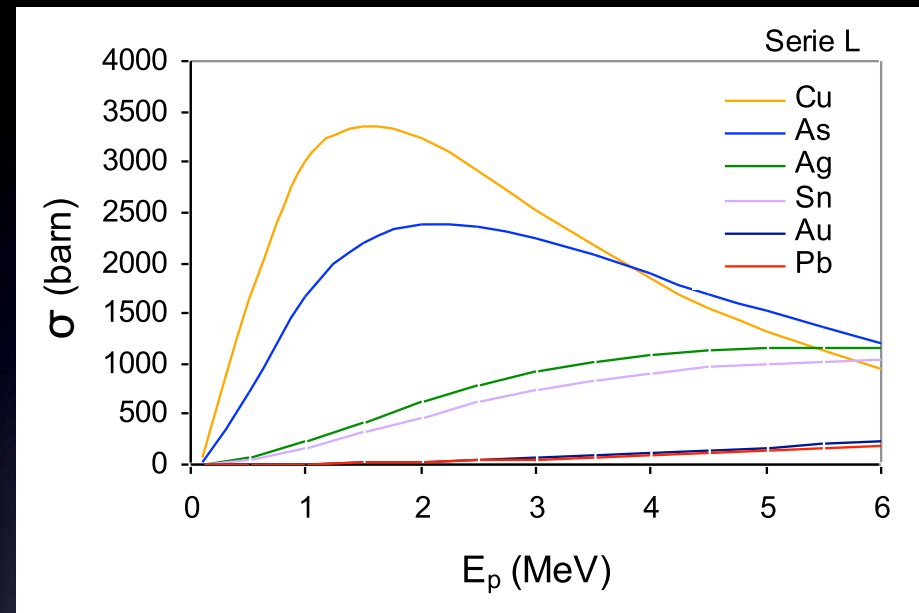
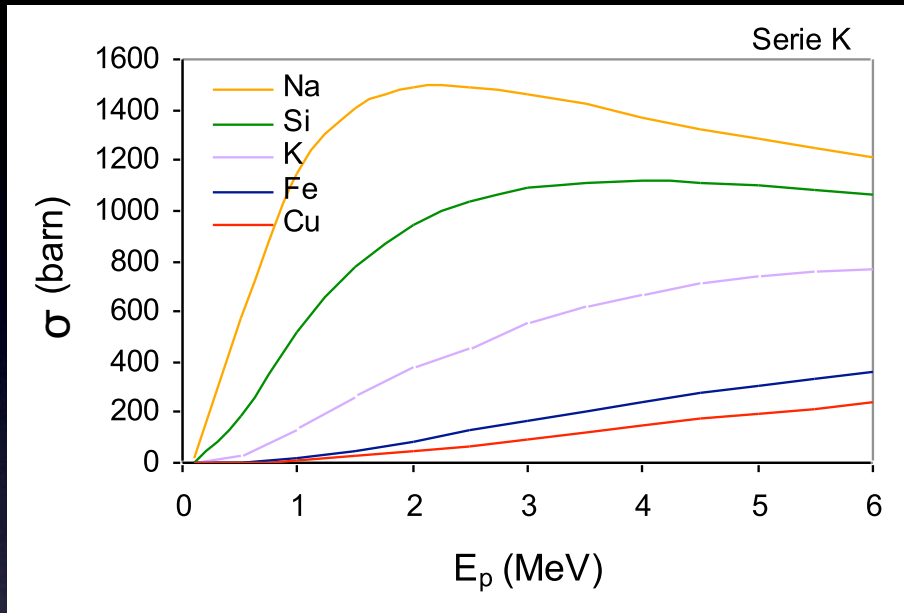
Energy of characteristic X-rays



Energy of characteristic X-rays



X-ray production cross sections



Advantages of PIXE

- very fast, high-sensitivity, **non-destructive** analysis
- **quantitative** analysis
- minimum energy of detected X-rays typically ~ 1 keV
 - ➔ *all the elements with $Z \geq 11$ are quantifiable simultaneously*

Limitations of PIXE

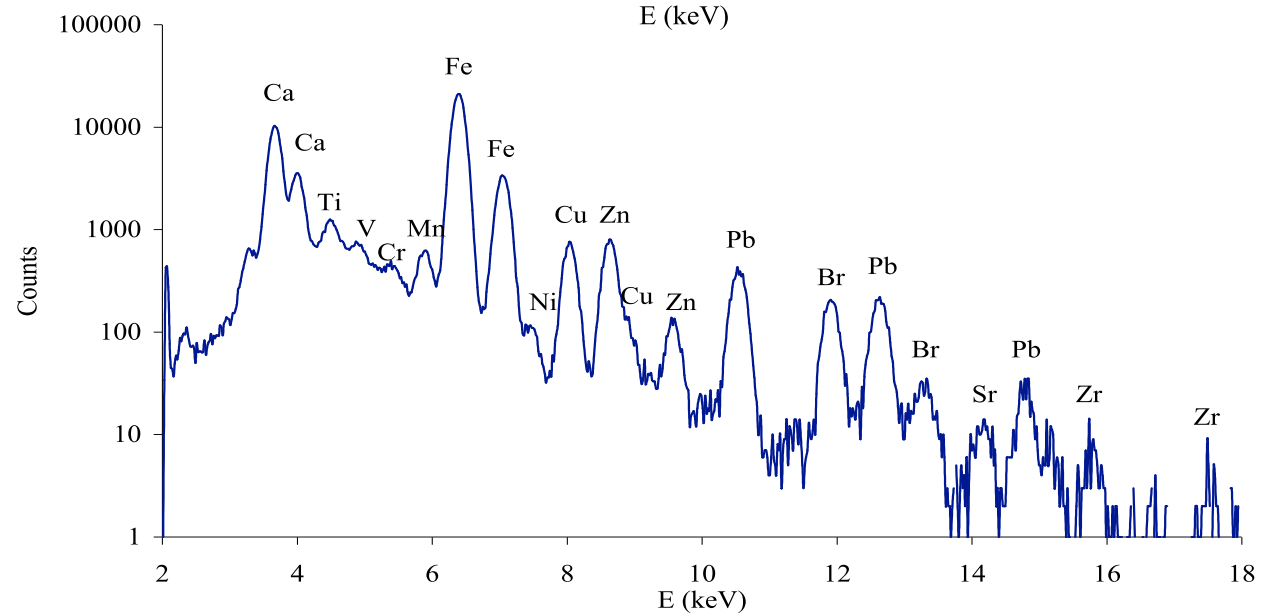
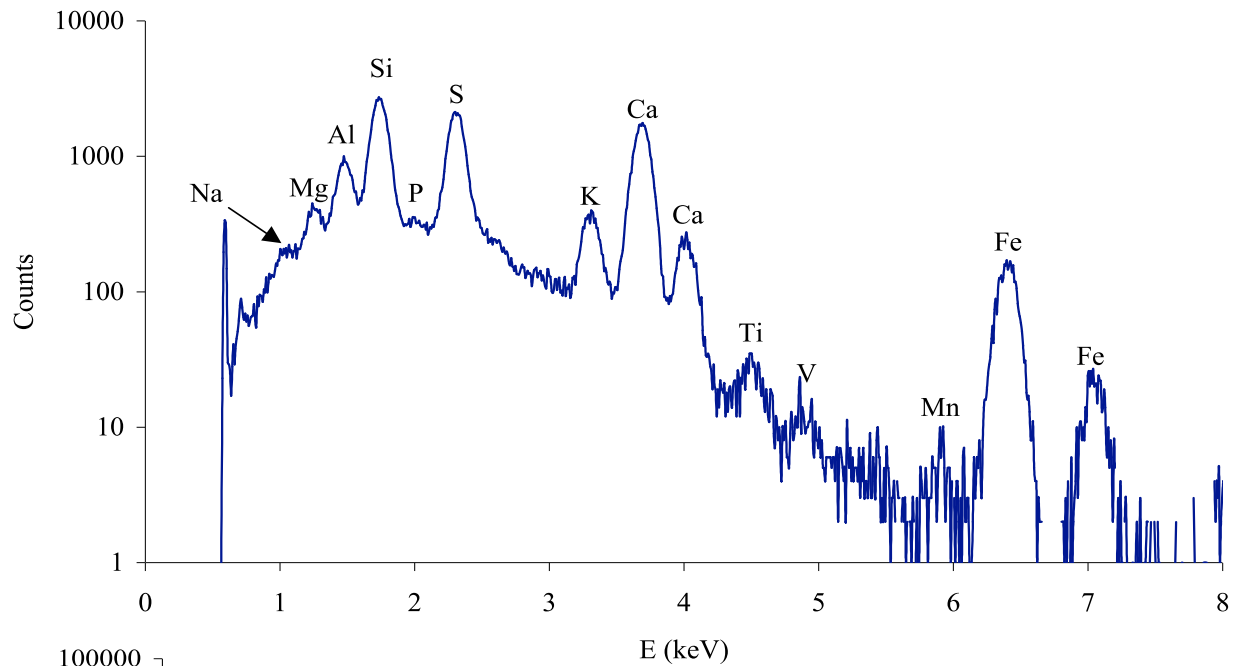
- no information on the organic components
- no direct information on chemical bonds

but... hypothesis on stoichiometry through quantitative and multielemental analysis

- no direct information on the stratigraphy and the depth distribution of the elements

but... Differential PIXE

Example of PIXE spectra



Quantitative analysis (thin target)

$$Y_0(Z) = N_P \cdot N_Z \cdot t \cdot \sigma_{Z,E0} \cdot (\alpha_Z \cdot \varepsilon_Z \cdot \Delta\Omega / 4\pi)$$

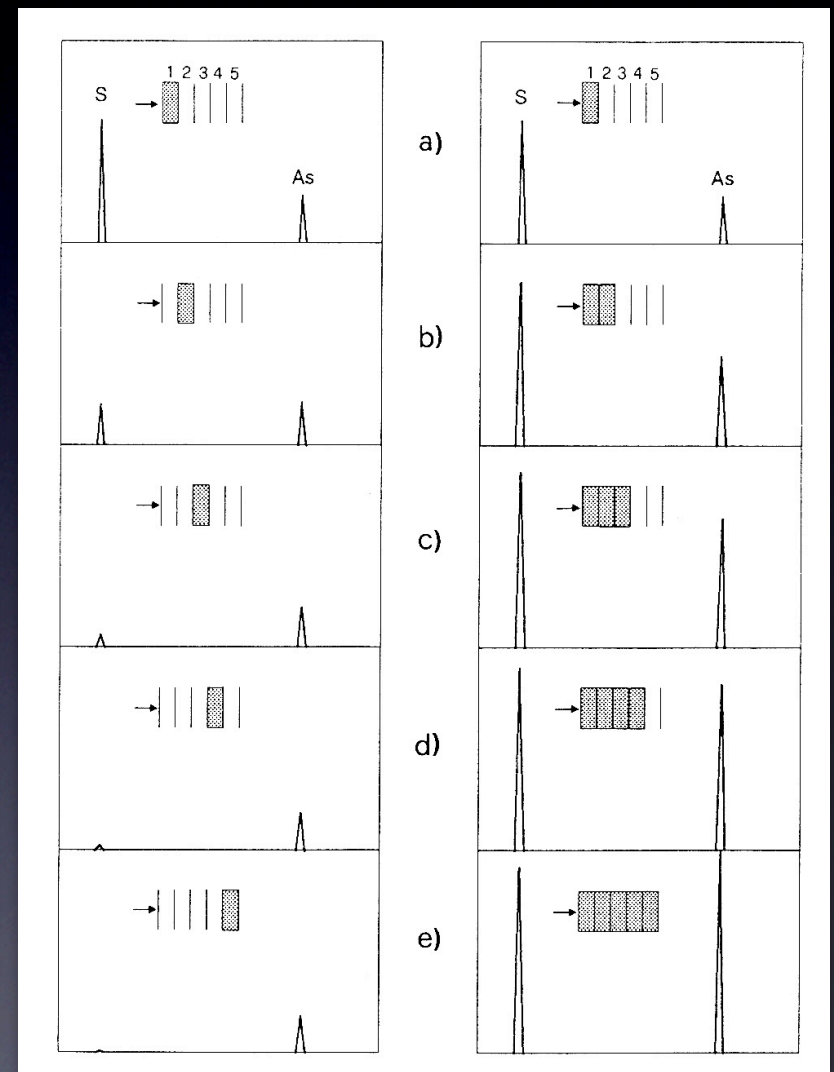
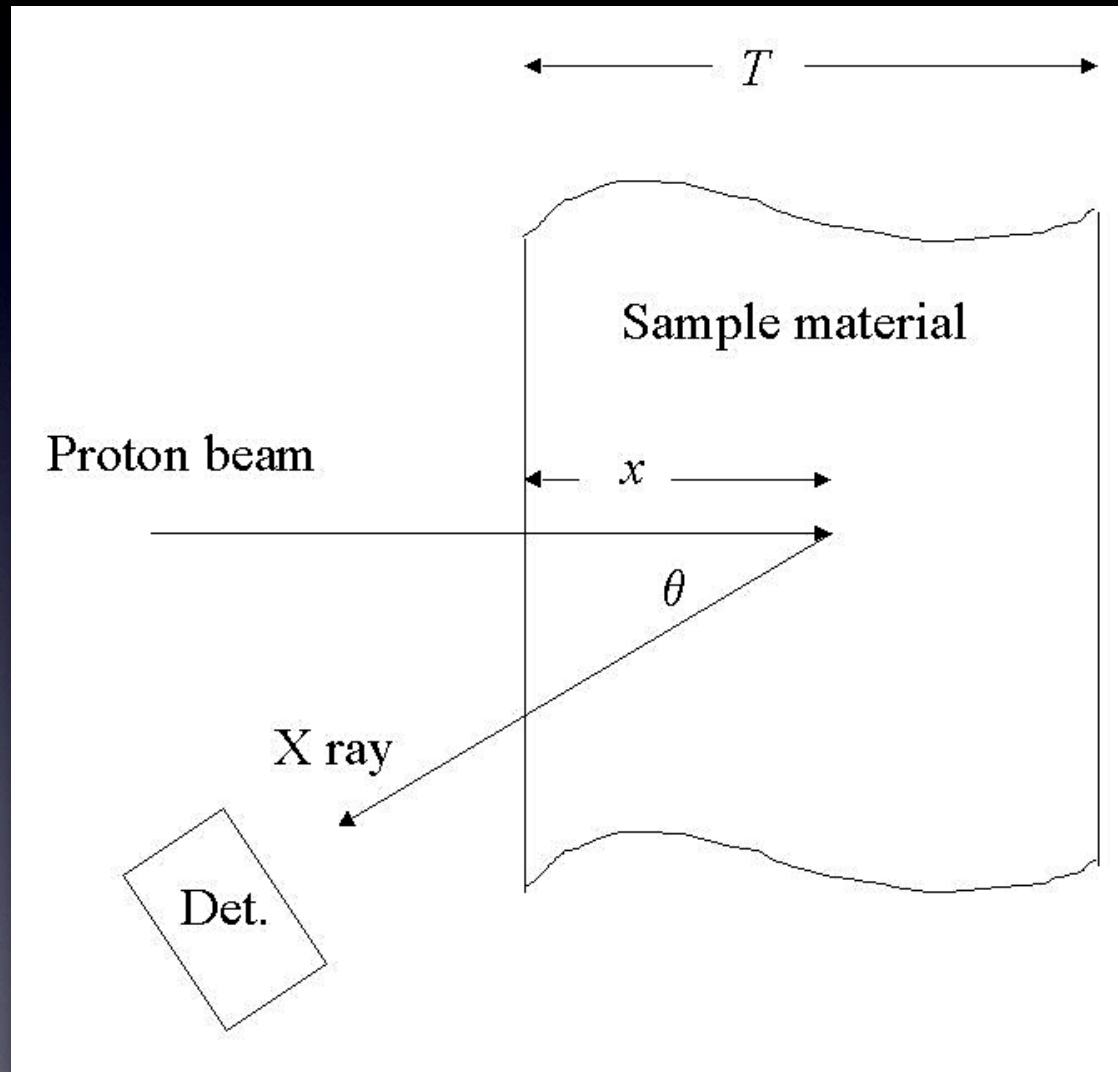
$$Y_0(Z) = (Q/e)(N_A/A)(\rho_Z t) \cdot \sigma_{Z,E0} \cdot (\alpha_Z \cdot \varepsilon_Z \cdot \Delta\Omega / 4\pi)$$

defining $\eta_Z = (1/e)(N_A/A) \cdot \sigma_{Z,E0} \cdot (\alpha_Z \cdot \varepsilon_Z \cdot \Delta\Omega / 4\pi)$

$$Y_0(Z) = \eta_Z \cdot Q \cdot (\rho_Z t)$$

$$(\rho_Z t) = Y_0(Z) / (\eta_Z \cdot Q)$$

Thick targets



Quantitative analysis (thick targets)

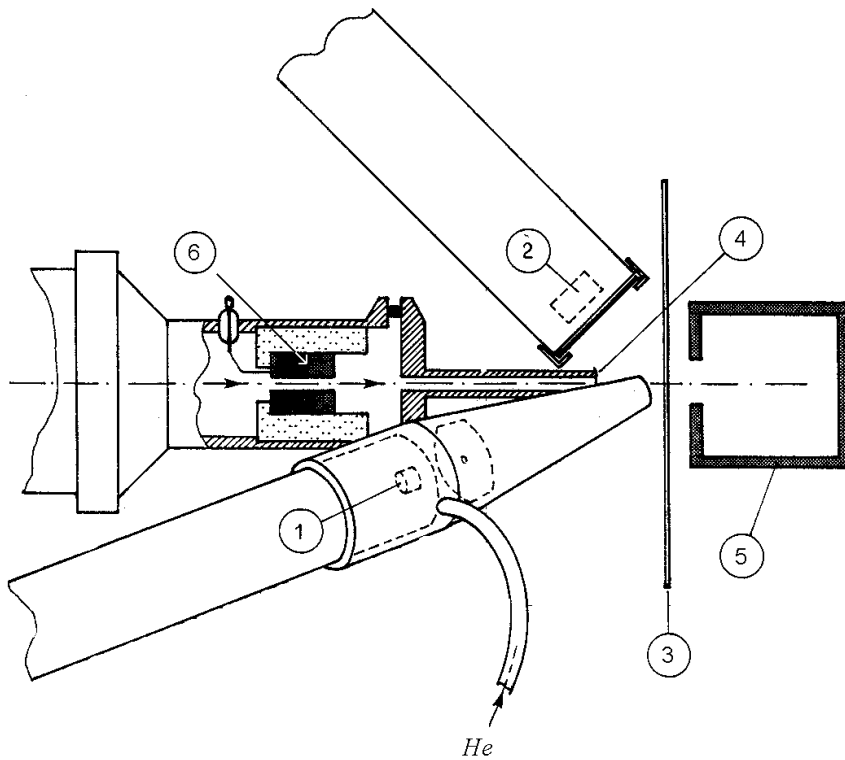
$$Y(Z) = (Q/e)(N_A/A)(\alpha_Z \cdot \epsilon_Z \cdot \Delta\Omega/4\pi) \cdot \rho_Z \int_0^T \sigma_{Z,E} \cdot \exp(-\mu \cdot x/\cos\theta) \cdot dx$$

$$Y(Z) = (Q/e)(N_A/A)(\alpha_Z \cdot \epsilon_Z \cdot \Delta\Omega/4\pi)(\rho_Z/\rho) \int_{E_0}^{E_F} \sigma_{Z,E} \cdot \exp(-\mu \cdot x/\cos\theta) \cdot dE/S(E)$$

$$F(Z) = Y_0(Z)/Y(Z) = \frac{\rho \cdot T \cdot \sigma_{Z,E_0}}{\int_{E_0}^{E_F} \sigma_{Z,E} \cdot \exp(-\mu \cdot x/\cos\theta) \cdot dE/S(E)}$$

$$(\rho_Z t) = F(Z) \cdot Y(Z) / (\eta_Z \cdot Q)$$

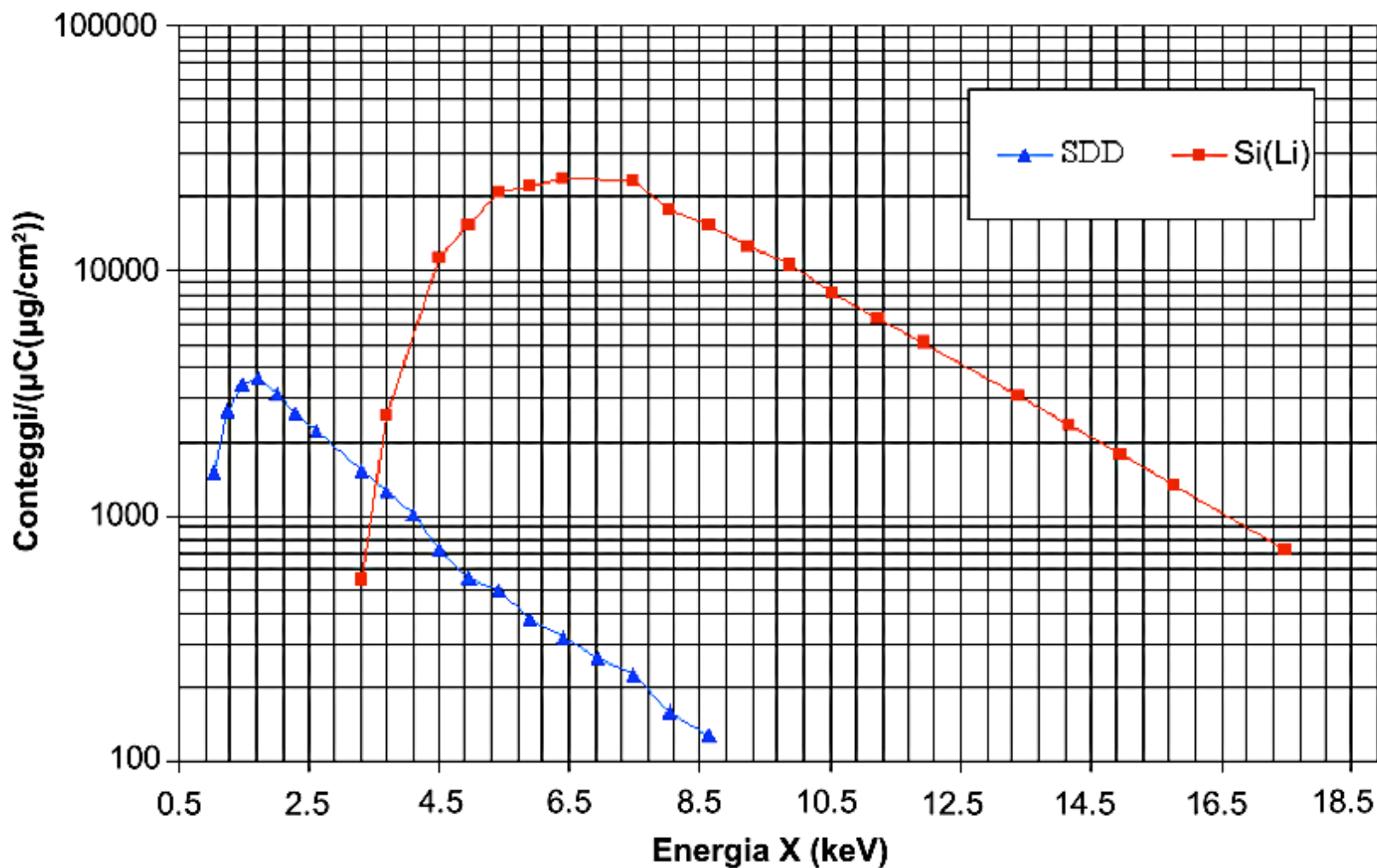
2-detectors PIXE set-up



- 1,2) X ray detectors 3) sample to analyse
 4) beam exit window 5) Faraday cup
 6) graphite collimator

Target	X-rays	What is needed	Detector features
Low-Z elements	Low energy High cross sections	Minimum dead layers Small solid angles	Thin entrance window Small active area
Medium-high-Z elements	High energy Low cross sections	Large solid angles Efficiency	Large active area Large active thickness

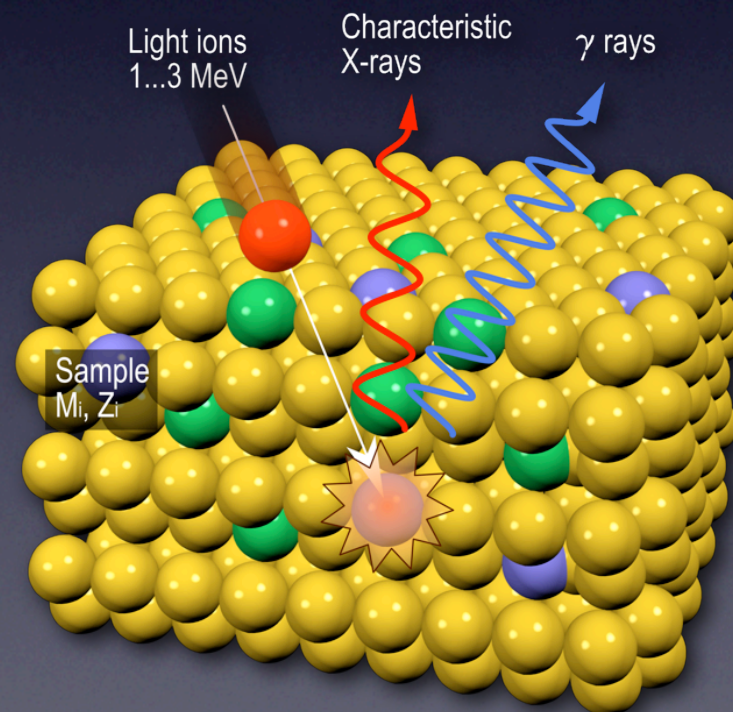
Detection efficiency for a 2-detectors PIXE set-up



PIGE

Particle Induced Gamma-ray Emission

Prompt emission of gamma-rays during the ion beam irradiation



**Li, B, F,
Na, Al...**

Nuclear reactions with prompt emission of gamma -rays

Radiative capture (direct reaction)	$a + A \rightarrow B^* \rightarrow B + \gamma$	$^{27}\text{Al}(p,\gamma)^{28}\text{Si}$
Inelastic scattering	$a + A \rightarrow A^* + a'$ $\quad \searrow$ $\quad A + \gamma$	$^{27}\text{Al}(p,p'\gamma)^{27}\text{Al}$
Rearrangement collisions	$a + A \rightarrow C^* + c$ $\quad \searrow$ $\quad C + \gamma$	$^{27}\text{Al}(p,\alpha\gamma)^{24}\text{Mg}$

List of proton-induced reaction

Element	E_γ (keV)	Nuclear origin	Transition	Detection limit (%)	Possible interferences
Li	429	${}^7\text{Li}(p, n\gamma){}^7\text{Be}$	429 + 0	0.01	As(427), B(429)
	478	${}^7\text{Li}(p, p'\gamma){}^7\text{Li}$	478 + 0	5×10^{-4}	Be(478), Mn(478)
Be	415	$\text{Be}^9(p, \gamma)\text{B}^{10}$	2154 + 1740	0.1	Ag(415)
	718	$\text{Be}^9(p, \gamma)\text{B}^{10}$	718 + 0		B(718)
	1023	$\text{Be}^9(p, \gamma)\text{B}^{10}$	1740 + 718		Ti(1022)
	1437	$\text{Be}^9(p, \gamma)\text{B}^{10}$	3590 + 2150		
	3562	$\text{Be}^9(p, \alpha\gamma)\text{Li}^6$	3562 + 0		
B	429	${}^{10}\text{B}(p, \alpha\gamma){}^7\text{Be}$	429 + 0	5×10^{-3}	As(427), Li(429)
	478	${}^{10}\text{B}(p, \alpha\gamma){}^7\text{Be} + {}^7\text{Li}$	478 + 0		Li(478), Mn(478)
	718	${}^{10}\text{B}(p, p'\gamma){}^{10}\text{B}$	718 + 0		Be(718)
	2124	${}^{10}\text{B}(p, p'\gamma){}^{10}\text{B}$	2124 + 0		
	4433	${}^{11}\text{B}(p, \gamma){}^{12}\text{C}$	4433 + 0		N(4433)

List of proton-induced reaction

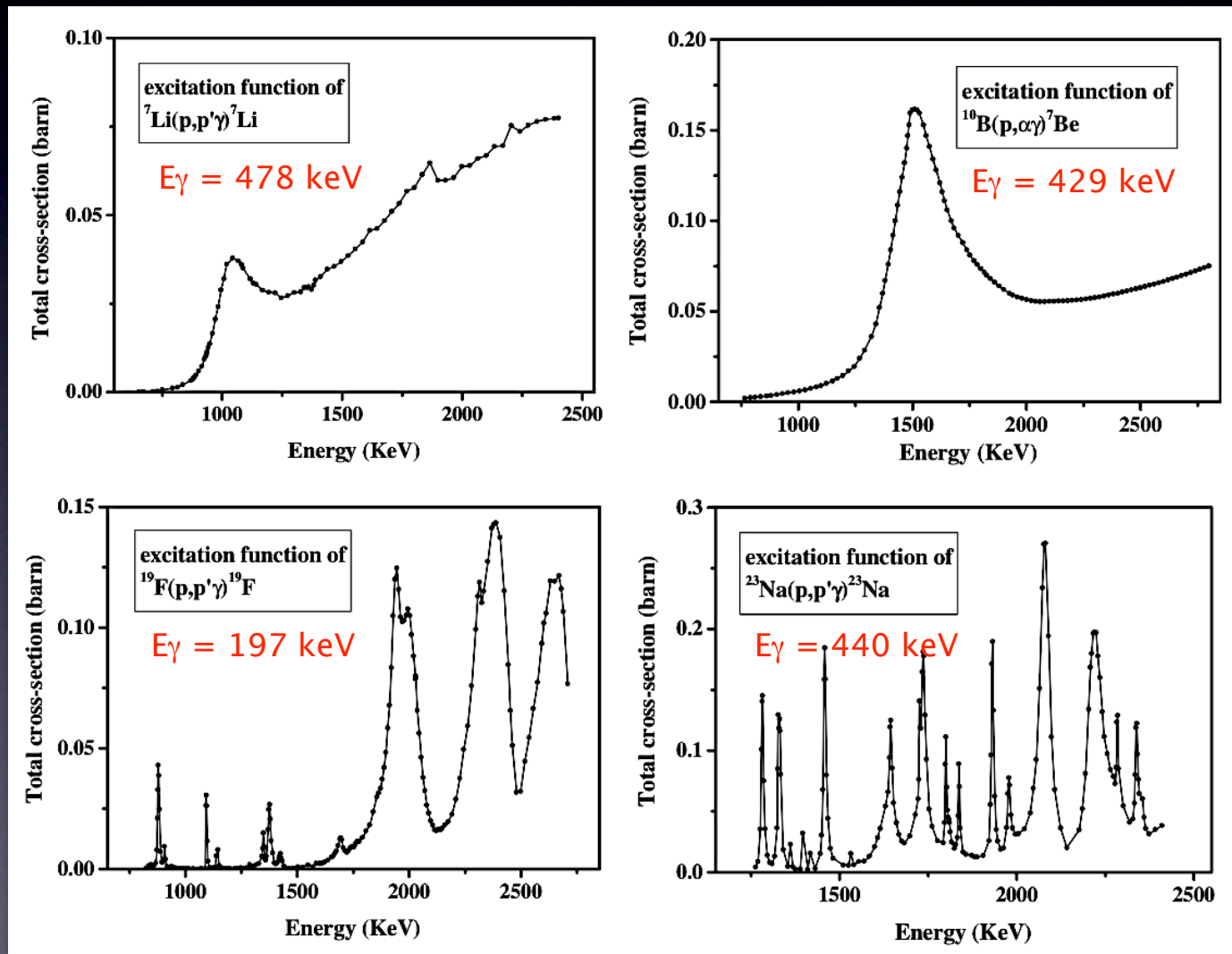
Element	E_γ (keV)	Nuclear origin	Transition	Detection limit (%)	Possible interferences
C	2357	$^{12}\text{C}(p,\gamma)^{13}\text{N}$	2357 + 0	1	
N	1400	$^{14}\text{N}(p,\gamma)^{15}\text{O}$			Cr(1400)
	4433	$^{15}\text{N}(p,\alpha\gamma)^{12}\text{C}$	4433 + 0		B(4433)
O	110	$^{18}\text{O}(p,\gamma)^{19}\text{F}$	110 + 0		F(110), W(111)
	197	$^{16}\text{O}(p,\gamma)^{17}\text{F}$	197 + 0		F(197), Co(197), Ni(197), Ga(197), Ti(199), Ge(199)
	496	$^{16}\text{O}(p,\gamma)^{17}\text{F}$	496 + 0	5	Ga(493)
F	110	$^{19}\text{F}(p,p'\gamma)^{19}\text{F}$	110 + 0	2×10^{-4}	O(110), W(111)
	197	$^{19}\text{F}(p,p'\gamma)^{19}\text{F}$	197 + 0	5×10^{-5}	Co(197), Ni(197), Ga(197) Ti(199), Ge(199), O(197)
Na	439	$^{23}\text{Na}(p,p'\gamma)^{23}\text{Na}$	439 + 0	10^{-3}	Se(439)
	1368	$^{23}\text{Na}(p,\gamma)^{24}\text{Mg}$	1368 + 0		Mg(1368), Al(1368)
	1633	$^{23}\text{Na}(p,\alpha\gamma)^{20}\text{Ne}$	1633 + 0		

List of proton-induced reaction

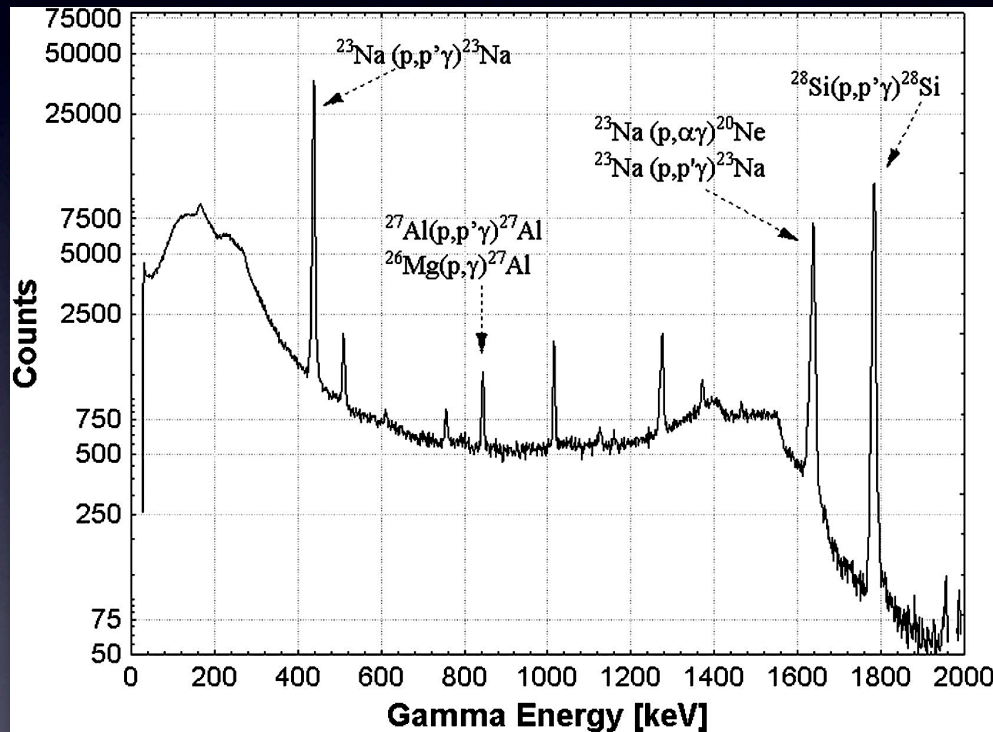
Element	E_{γ} (keV)	Nuclear origin	Transition	Detection limit (%)	Possible interferences
Mg	170	$^{26}\text{Mg}(p,\gamma)^{27}\text{Al}$	1014 + 844	5×10^{-3}	Al(170)
	390	$^{25}\text{Mg}(p,p'\gamma)^{25}\text{Mg}$	975 + 585		
	585	$^{25}\text{Mg}(p,p'\gamma)^{25}\text{Mg}$	585 + 0		
	844	$^{26}\text{Mg}(p,\gamma)^{27}\text{Al}$	844 + 0		Al(844)
	975	$^{25}\text{Mg}(p,p'\gamma)^{25}\text{Mg}$	975 + 0		
	1014	$^{26}\text{Mg}(p,\gamma)^{27}\text{Al}$	1014 + 0		Ti(1012), Al(1014)
	1368	$^{24}\text{Mg}(p,p'\gamma)^{24}\text{Mg}$	1368 + 0		Na(1368), Al(1368)
Al	170	$^{27}\text{Al}(p,p'\gamma)^{27}\text{Al}$	1014 + 844	2×10^{-3} 5×10^{-3}	Mg(170)
	844	$^{27}\text{Al}(p,p'\gamma)^{27}\text{Al}$	844 + 0		Mg(844)
	1014	$^{27}\text{Al}(p,p'\gamma)^{27}\text{Al}$	1014 + 0		Ti(1012), Mg(1014)
	1368	$^{27}\text{Al}(p,\alpha\gamma)^{24}\text{Mg}$	1368 + 0		Na(1368), Mg(1368)
	1779	$^{27}\text{Al}(p,\gamma)^{28}\text{Si}$	1779 + 0		Si(1779), P(1779)
Si	1273	$^{29}\text{Si}(p,p'\gamma)^{29}\text{Si}$	1273 + 0	3	
	1779	$^{28}\text{Si}(p,p'\gamma)^{28}\text{Si}$	1779 + 0		Al(1779), P(1779)

PIGE cross sections

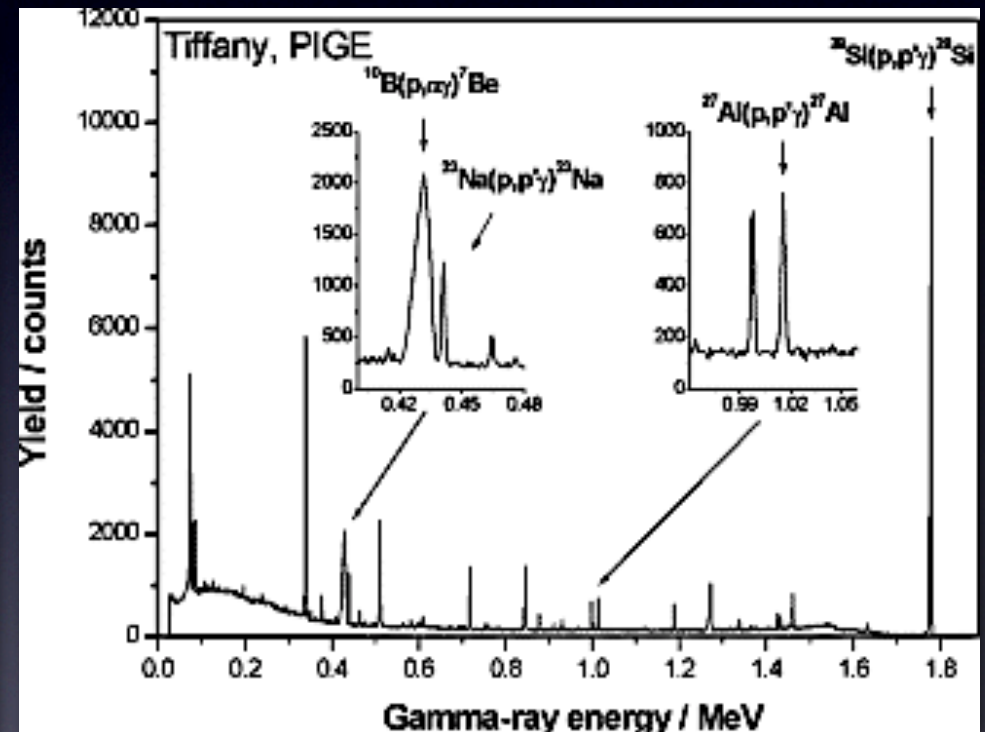
The cross sections are the superimposition of resonances (Breit-Wigner) on a continuum due to direct nuclear reactions



Examples of PIGE spectra



Soda-lime glass



Borosilicate glass

PIGE elemental analysis

The elemental concentrations (N_T) are obtained from measured quantities (i.e. gamma-ray peak areas) using physical models implementing the sample structure and the physical microscopic data and processes:

$$Y_{\gamma}(E_0, \theta) = \varepsilon_{\text{abs}}(E_{\gamma}) \cdot N_p \cdot \int_0^{E_0} N_T \cdot \sigma(E_0, \theta) / S(E) dE$$

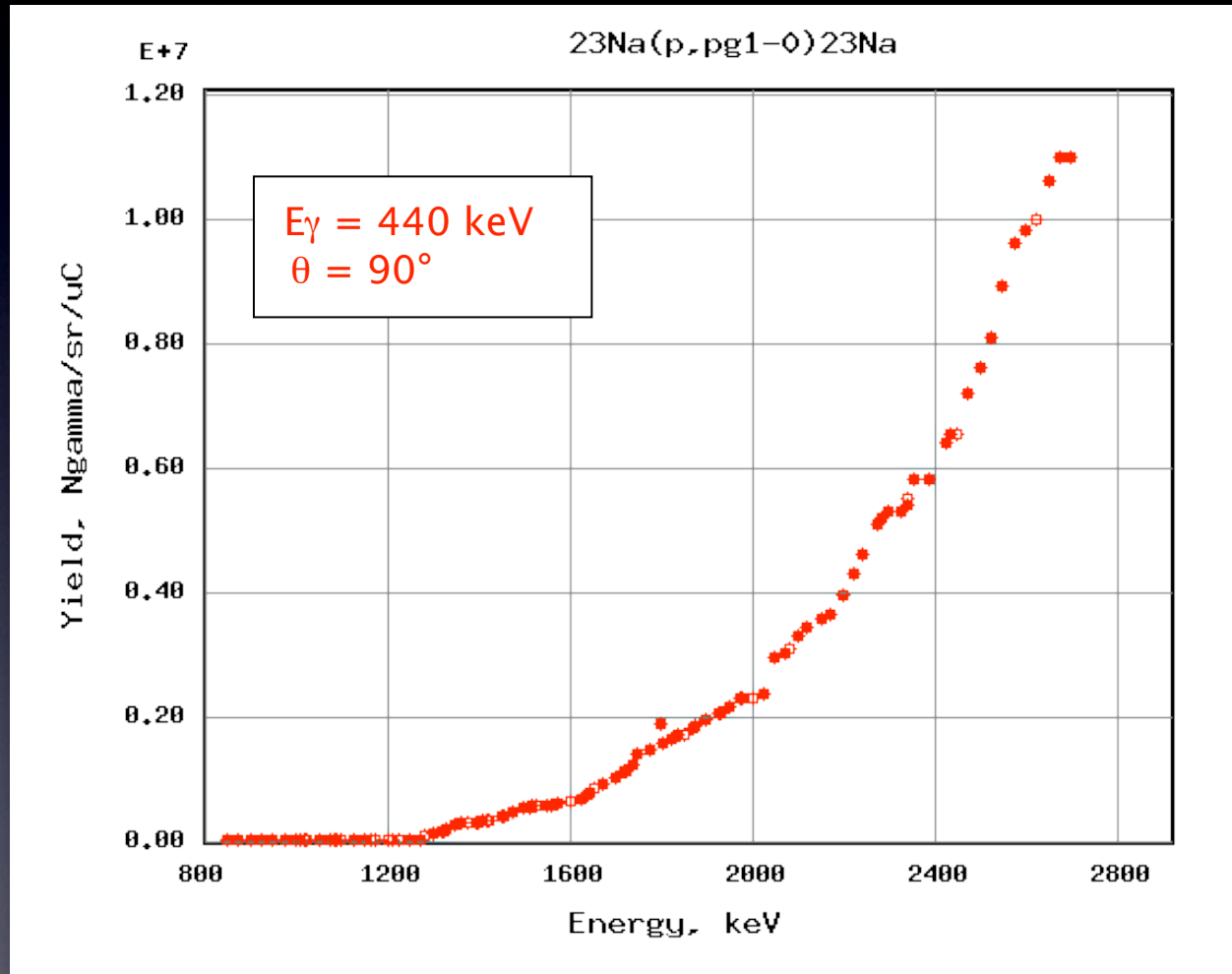
The principal needed microscopic data are **stopping powers** and **differential cross sections** of the interaction (as well as the detector absolute efficiency)

PIGE analysis in cultural heritage

- The PIGE technique can be used for (semi-)quantitative determination of light elements like Na, Al or Si in infinitely thick targets
- The unknown elemental concentrations are typically deduced by comparing the γ -ray yields with those of **thick** standards of similar composition, without the detailed knowledge of the cross section
- The crucial point is the difference between the stopping power of the unknown sample and that of the standard

Reaction	E γ (keV)
$^{23}\text{Na} (p, p', \gamma) ^{23}\text{Na}$	440
$^{25}\text{Mg} (p, p', \gamma) ^{25}\text{Mg}$	585
$^{24}\text{Mg} (p, p', \gamma) ^{24}\text{Mg}$	1369
$^{27}\text{Al} (p, p', \gamma) ^{27}\text{Al}$	844, 1014
$^{28}\text{Si} (p, p', \gamma) ^{28}\text{Si}$	1779
$^{31}\text{P} (p, p', \gamma) ^{31}\text{P}$	1266

Thick target gamma-ray yields



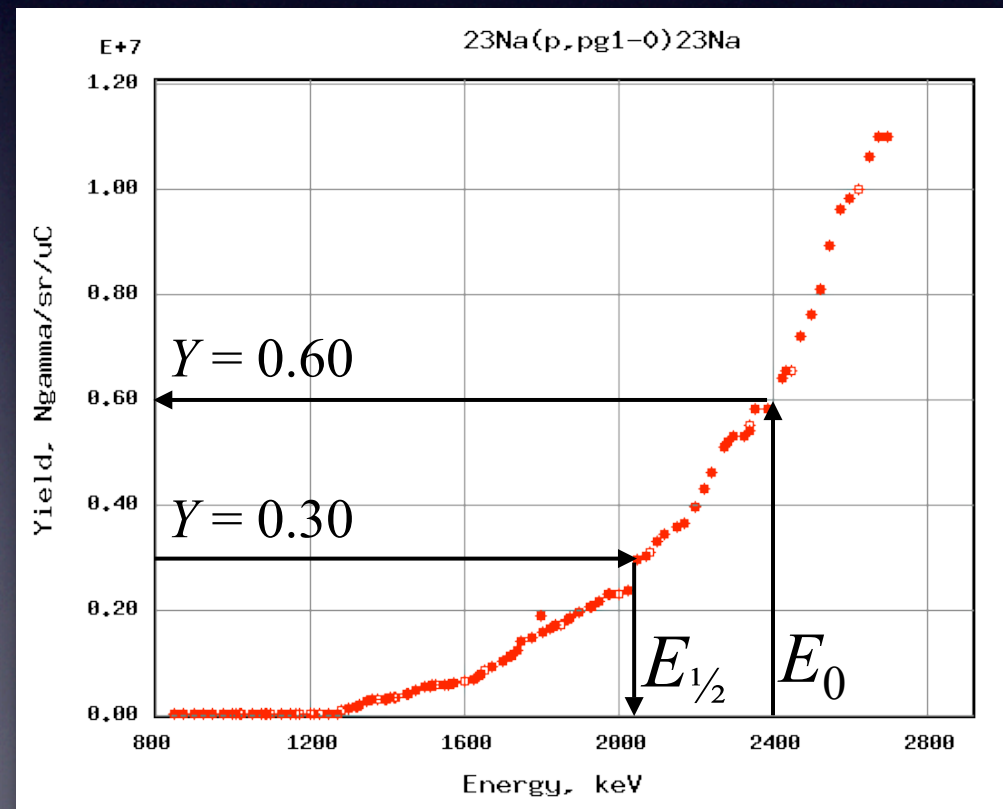
PIGE quantitative analysis: comparison with thick standards

$$C_{camp} = C_{rif} \cdot Y_{camp}(E_0)/Y_{rif}(E_0) \cdot S_{camp}(E_{1/2})/S_{rif}(E_{1/2})$$

$E_{1/2}$ such as:

$$Y(E_0) = 2 \cdot Y(E_{1/2})$$

The “ $E_{1/2}$ ” method is valid as long as the excitation function varies slowly with the energy



RBS

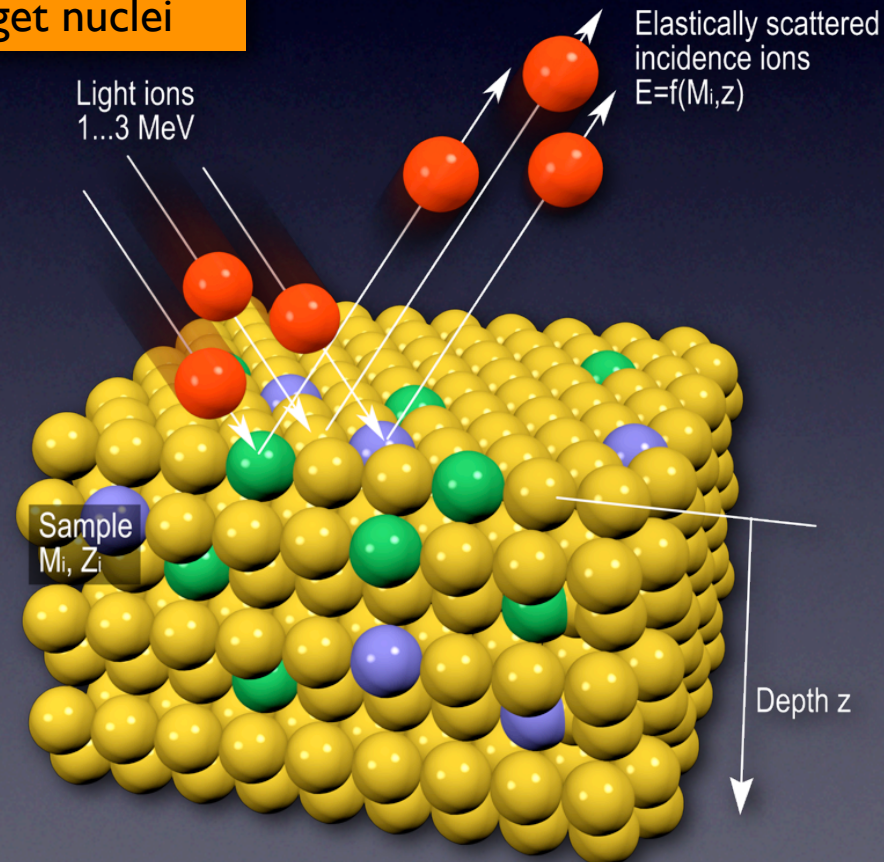
Rutherford Backscattering Spectrometry

Elastic scattering at backward angles of the impinging ions by the target nuclei



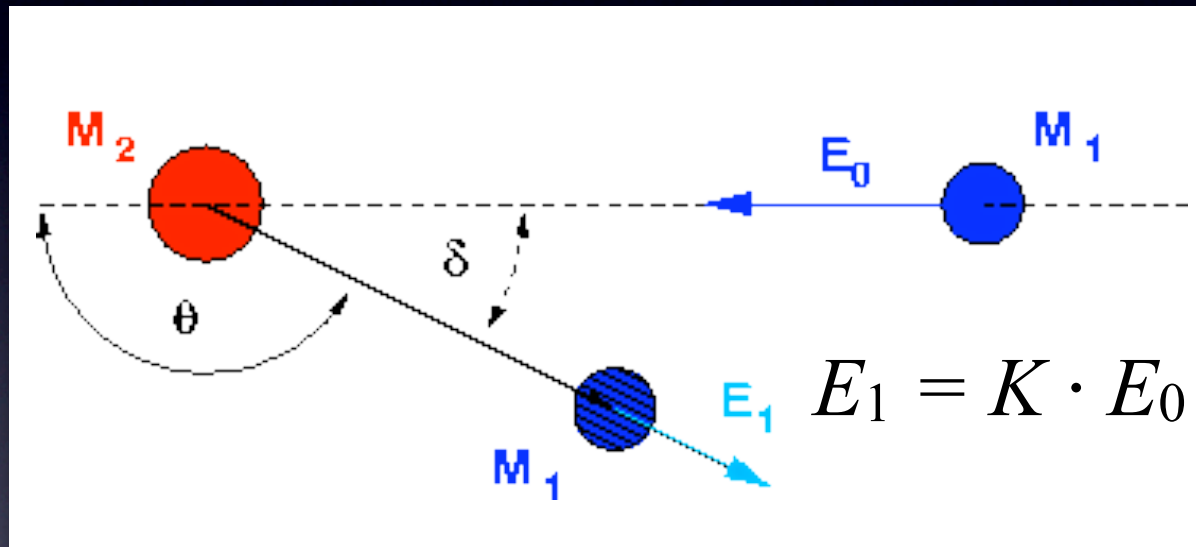
$A > A_p$

✓ depth profile



Principles of RBS

For a given scattering angle θ , the energy E_1 of the incident ion (mass M_1) after the collision is only a function of the mass M_2 of the target nuclei

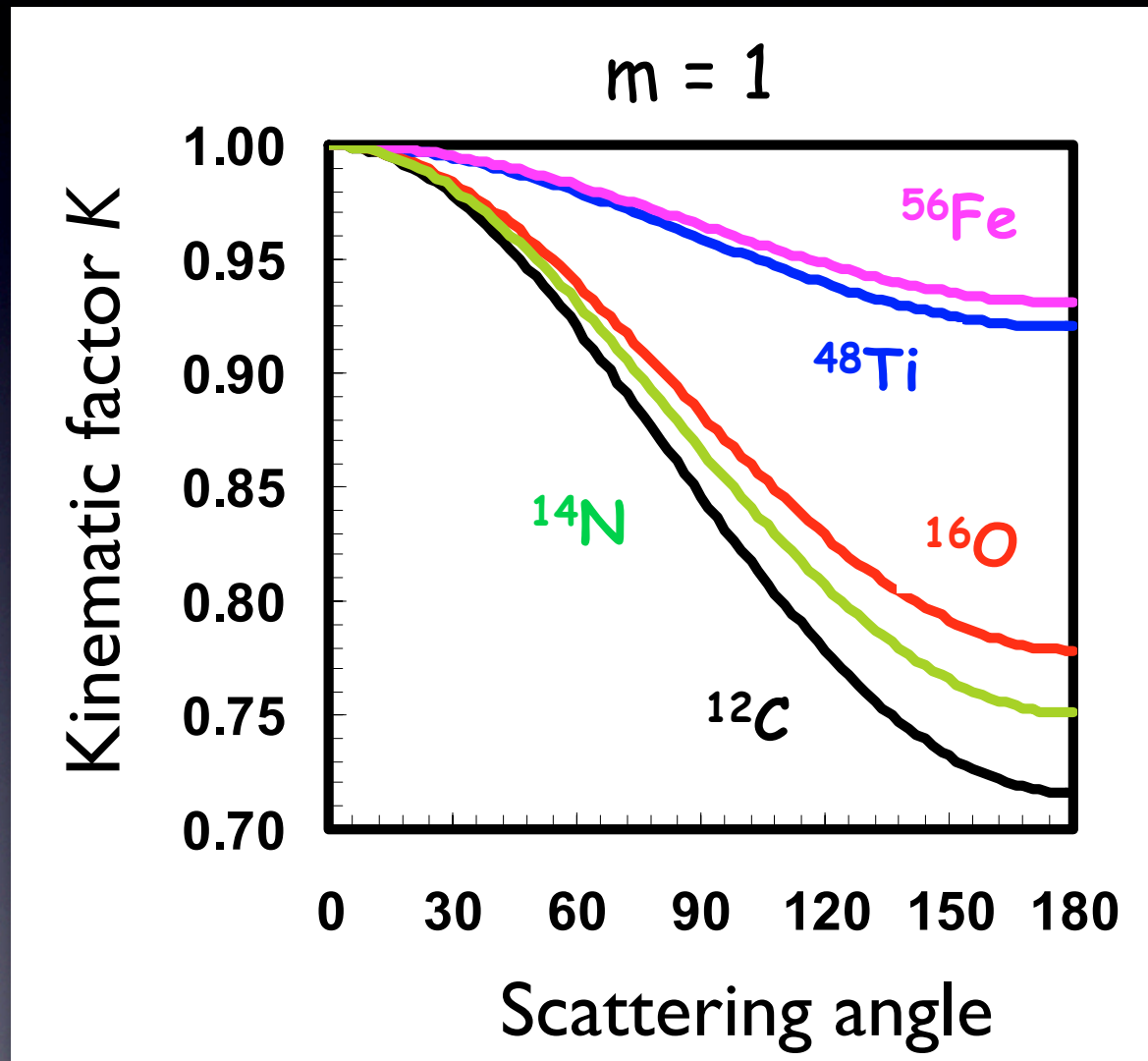


$$K = \frac{\left[\sqrt{\left(\frac{M_2}{M_1}\right)^2 - \sin^2 \theta} + \cos \theta \right]^2}{\left(\frac{M_2}{M_1} + 1\right)^2}$$

larger ΔE for smaller M
(light nuclei)

Larger ΔE for larger θ
(backscattering)

The kinematic factor K



Elastic scattering cross section

The Rutherford formula:

$$\left(\frac{d\sigma}{d\Omega}\right)_{Ruth} = \left(\frac{Z_1 Z_2 e^2}{E}\right)^2 \frac{4}{\sin^4(\theta)} \frac{\left[\sqrt{1 - \left(\frac{M_1}{M_2} \sin \theta\right)^2} + \cos \theta\right]^2}{\sqrt{1 - \left(\frac{M_1}{M_2} \sin \theta\right)^2}}$$

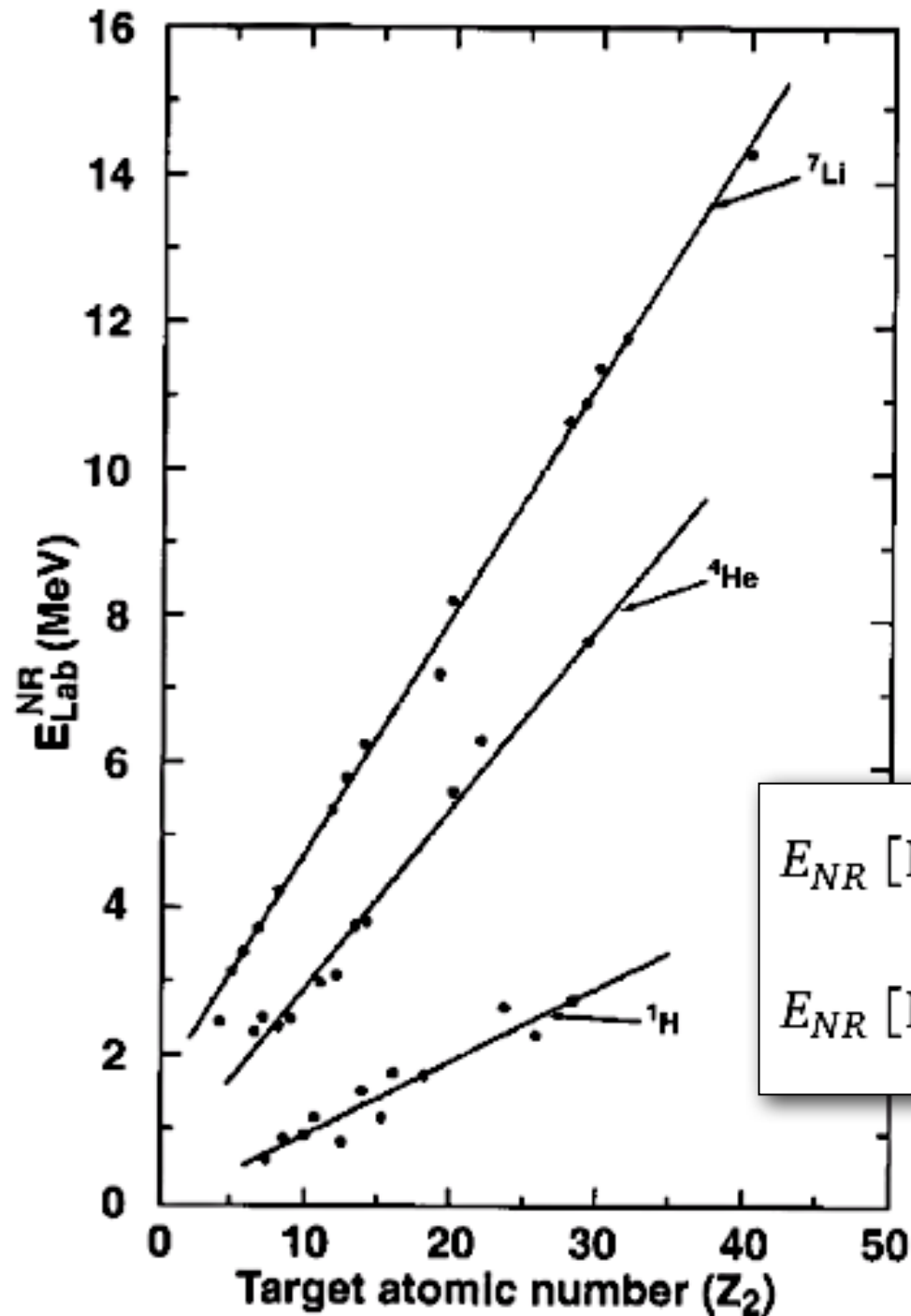
Z_1 Atomic number of the incident ion

Z_2 Atomic number of the target nucleus

E Energy of the incident ion

M_1 Mass of the incident ion

M_2 Mass of the target nucleus

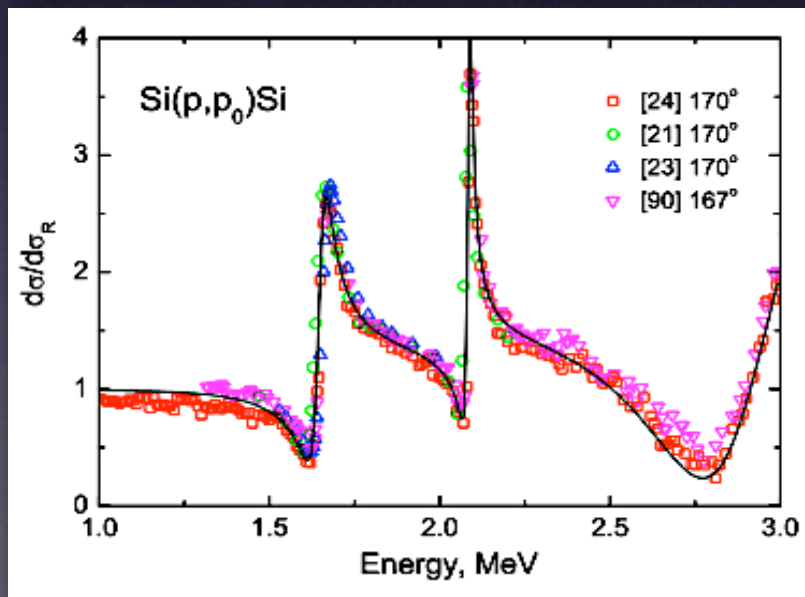
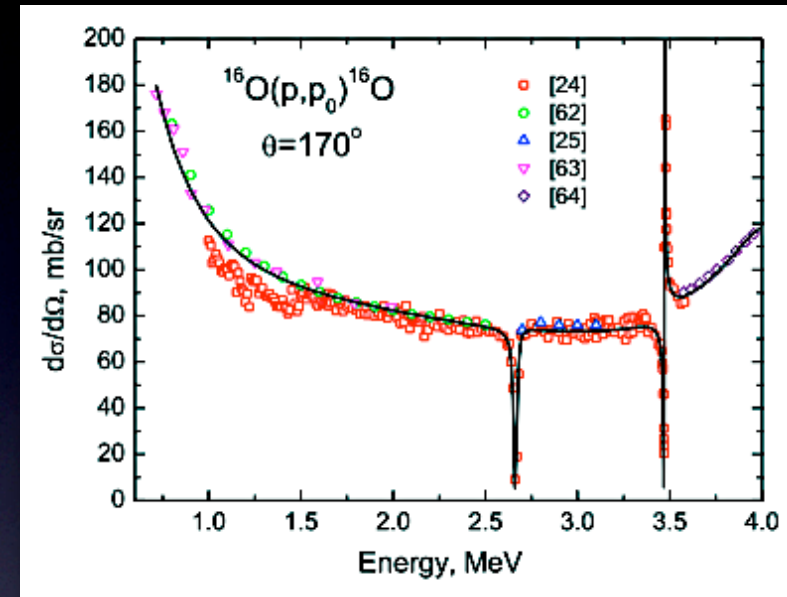
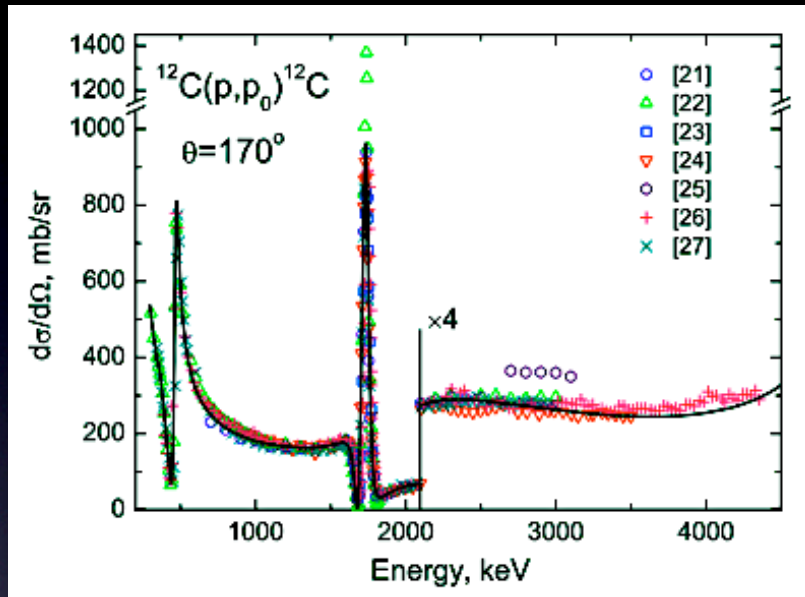


Energy (in the laboratory) at which the elastic scattering cross section deviates more than 4% from the Rutherford value (for scattering angles $> 160^\circ$)

$$E_{NR} \text{ [MeV]} = \frac{M_1 + M_2}{M_2} \frac{Z_2}{10} \quad \text{for } Z_1 = 1$$

$$E_{NR} \text{ [MeV]} = \frac{M_1 + M_2}{M_2} \frac{Z_1 Z_2}{8} \quad \text{for } Z_1 > 1$$

Non-Rutherford cross sections

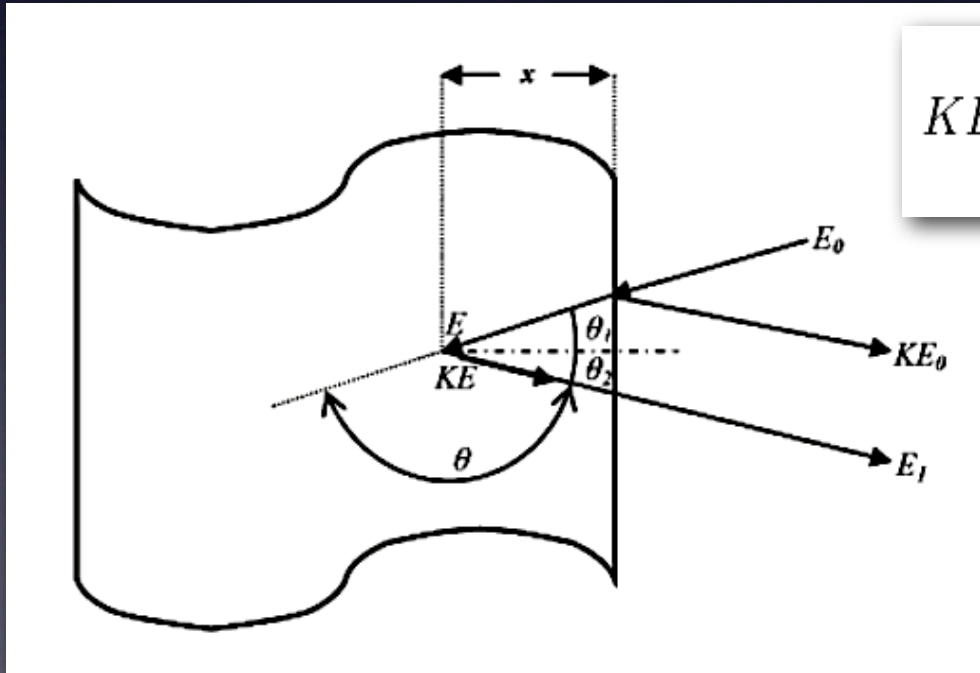


Evaluated and recommended experimental cross sections available from IBANDL web site (www-nds.iaea.org/ibandl/)

Depth scale of RBS

The signal from an atom at the sample surface will appear in the energy spectrum at a position KE_0 .

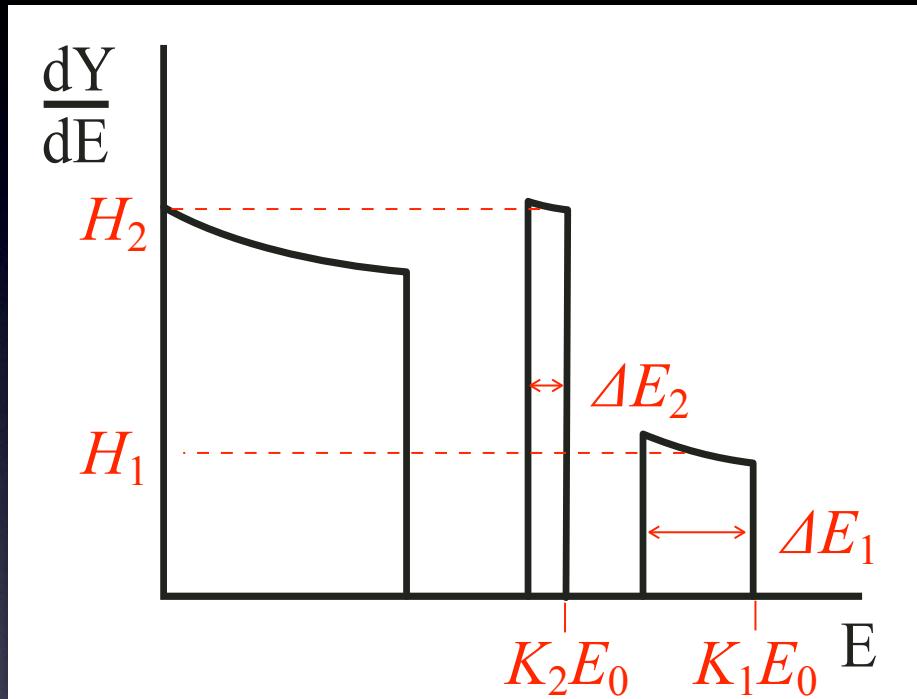
The signal from atoms of the same mass below the sample surface will be shifted by the amount of energy lost while the projectiles pass through the sample, both before (ΔE_{in}) and after a collision (ΔE_{out}).



$$KE_0 - E_1 = \left[\frac{Kx}{\cos \theta_1} \cdot \left(\frac{dE}{dx} \right)_{in} + \frac{x}{\cos \theta_2} \cdot \left(\frac{dE}{dx} \right)_{out} \right]$$

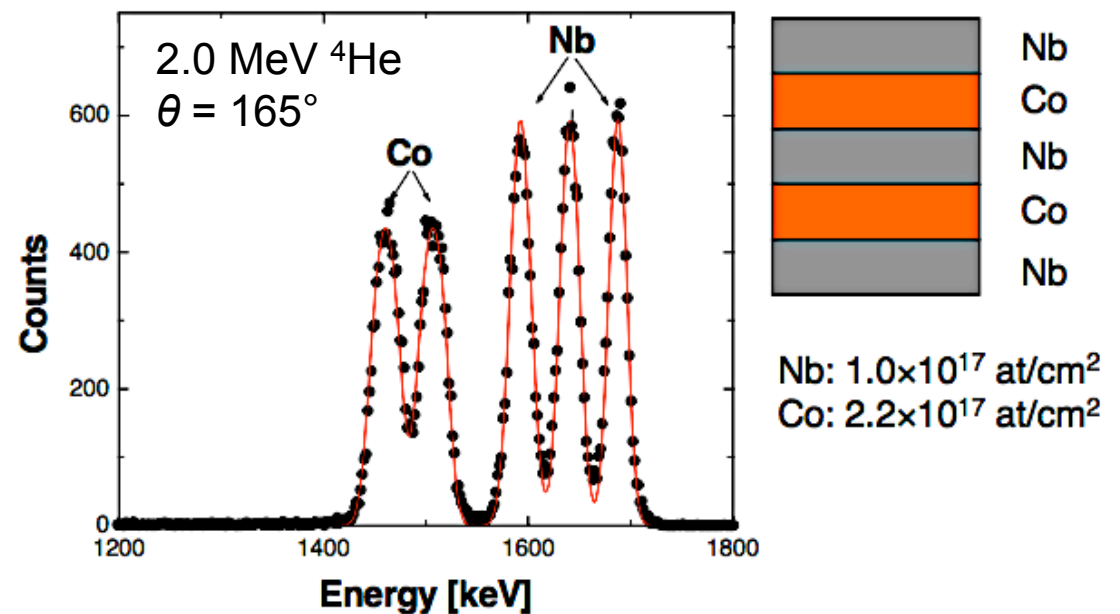
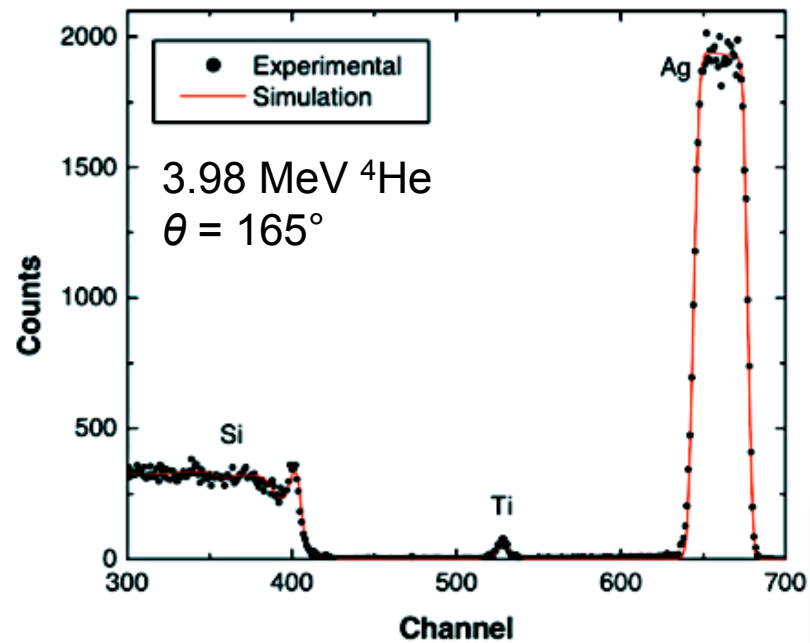
There exists a relation between the measured energy E_1 and the depth x at which the scattering took place

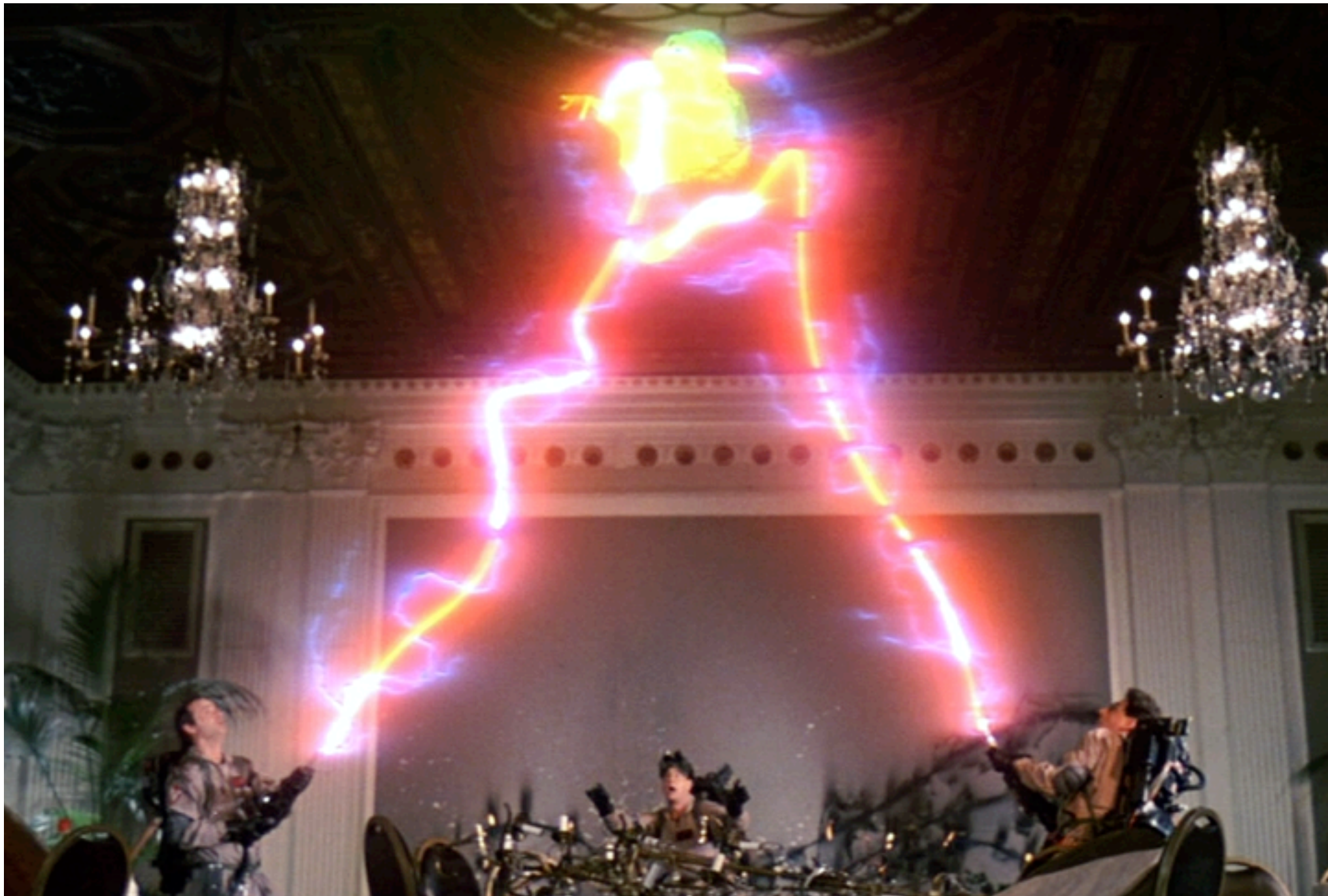
The features of RBS spectra



- Position of the signal (mass perception *i.e.* *kinematic factor*)
- Width of the signal (depth/thickness perception *i.e.* *stopping force*)
- Height of the signal (quantitative analysis of elemental composition *i.e.* *scattering cross section*)

Examples of RBS spectra



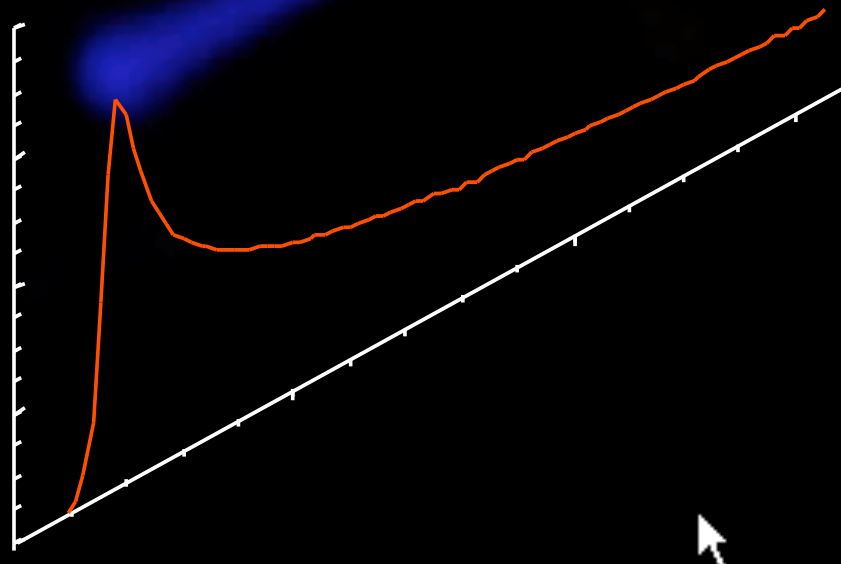


Do extracted ion beams look like these?

External beam

Advantages

direct analysis of artefacts having any shape and any size
no sampling
no charging, no preparation (conductive coating etc.)
no heating, reduced damage risk
easy sample positioning
fast and efficient



Disadvantages

energy loss
energy straggling
beam lateral spread
x-ray attenuation

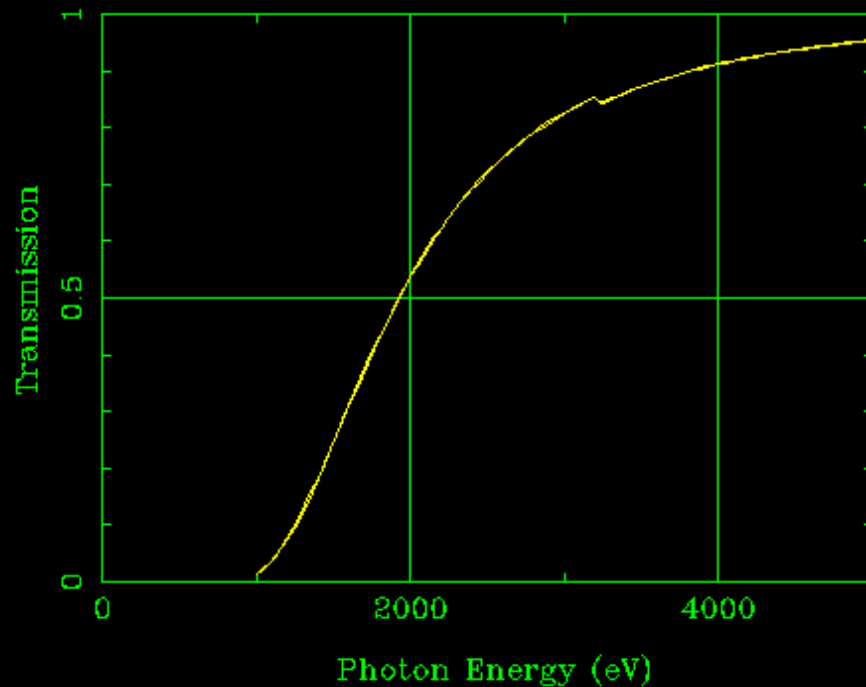
Typical extraction windows

Material	Thickness (μm)	ΔE (keV)	σ_E (keV)	σ_θ ($\mu\text{m}/\text{mm}$)
Al	10	235	16	14
Kapton	8	130	9	6
Zr	2	75	7.3	15
Si ₃ N ₄	0.1	8	5	<1
	0.5	40	9	<2

Choice of external atmosphere

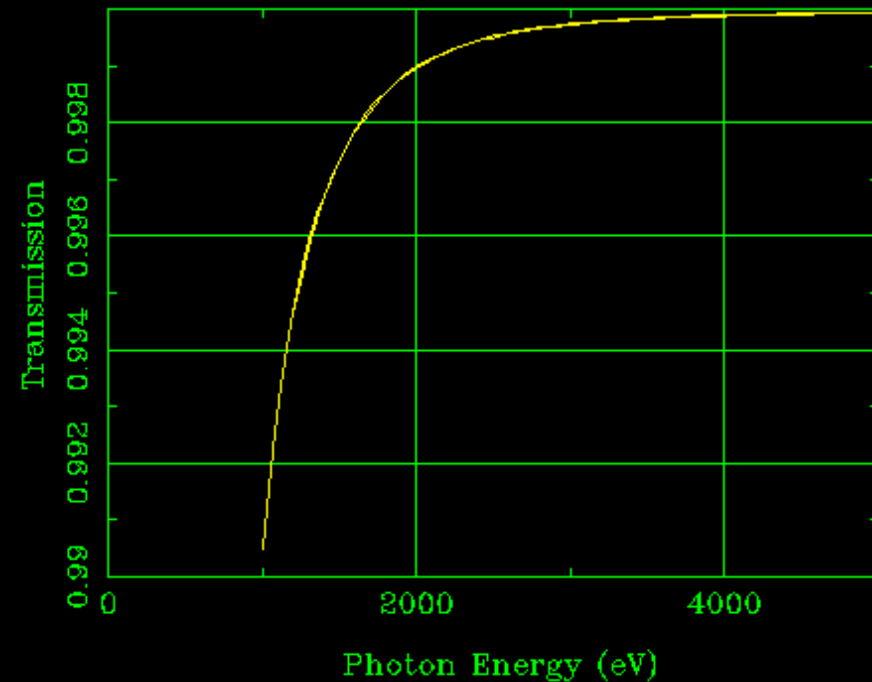
Air

N1.5620.42C.0003Ar.0094 Pressure=760. Path=1. cm



Helium

He Pressure=760. Path=1. cm

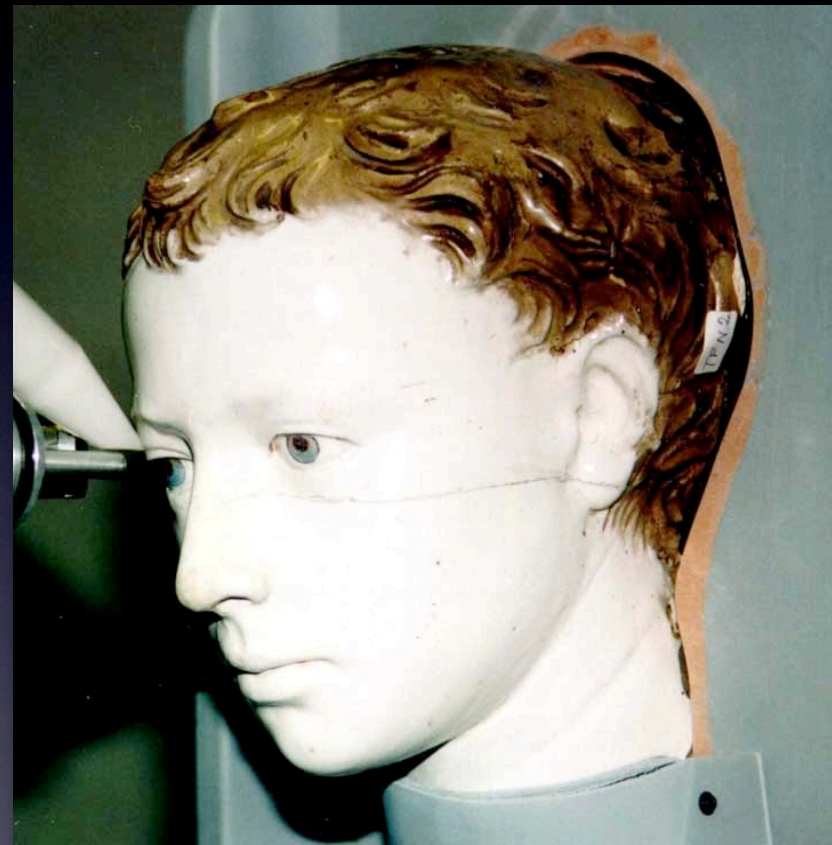


The use of an helium-saturated atmosphere in front of the X-ray detector is mandatory

External beam IBA of ancient manuscripts, ...ceramics,



PIXE analysis of the frontispiece of
Pl.16,22, from Biblioteca
Laurenziana in Florence



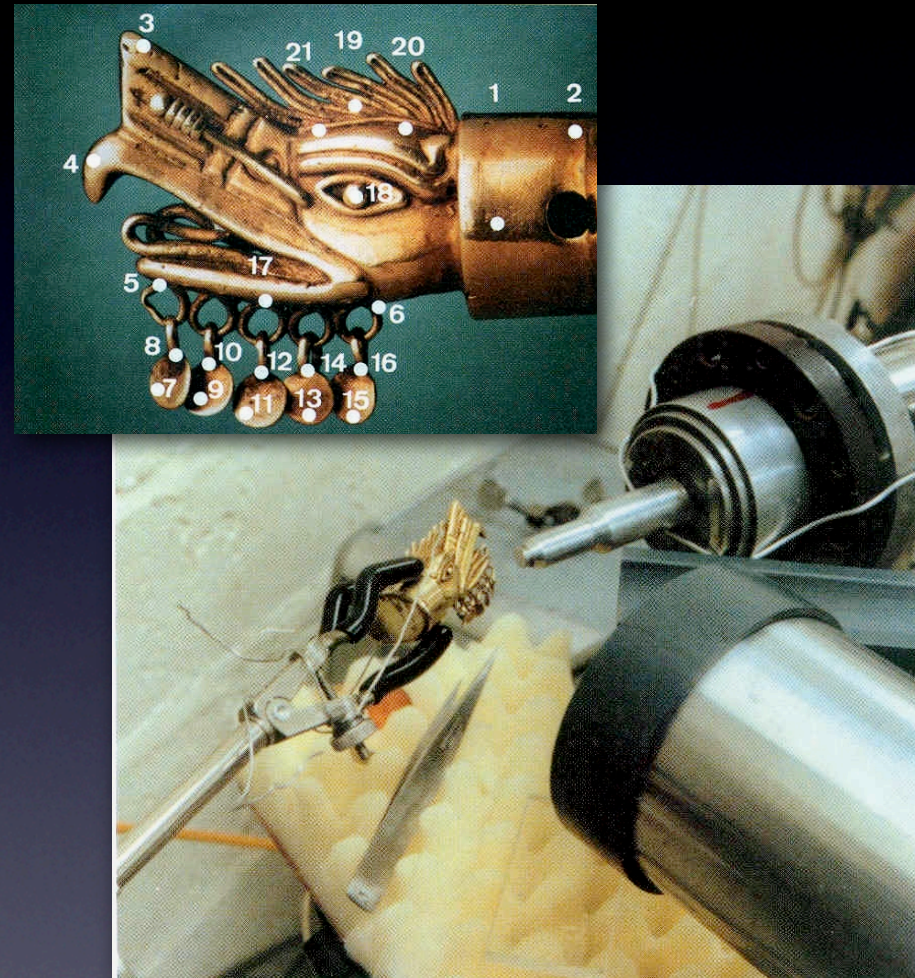
Analysis of the Ritratto di fanciullo
by Luca Della Robbia – before
restoration at the Opificio delle
Pietre Dure in Florence

... drawings,



Micro-PIXE measurements of
Portrait of Lucas de Leyde by
Alfred Dürer
A.Duval et al., (Louvre laboratory)

... jewels,



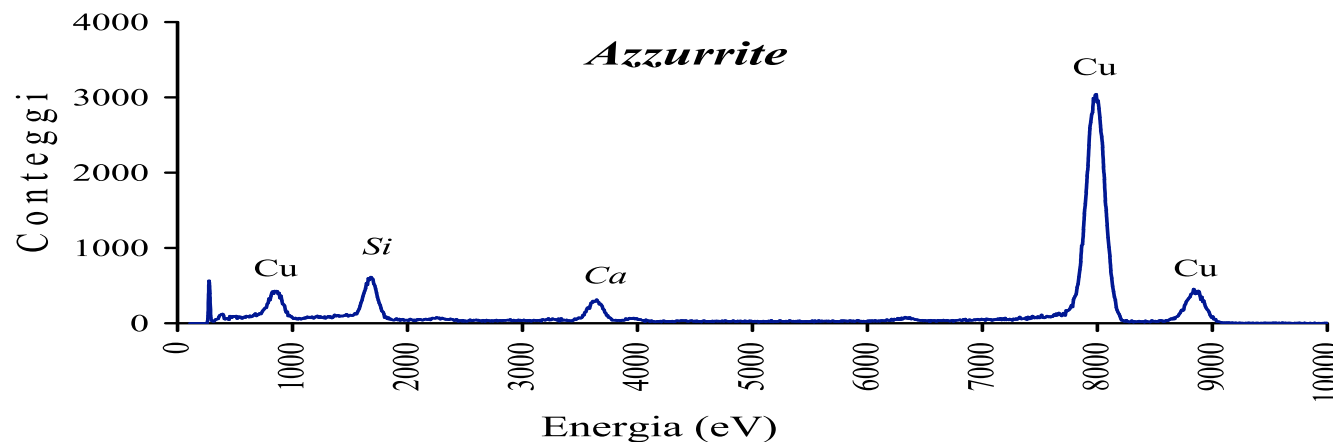
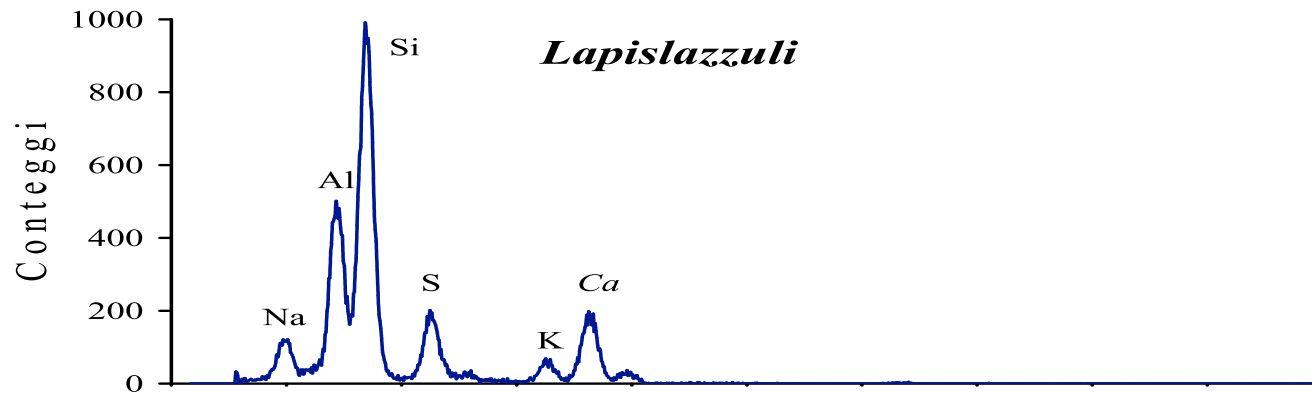
Micro-PIXE measurements of a
Mexican gold alloy ornament
G.Demortier and J.L.Ruvalcaba Sil
(Namur)

...paintings



PIXE analysis of a painting by
Lucas Cranach the Elder
C. Neelmeijer et al.
(Rossendorf Forschungszentrum, Dresda)

Blue Temperas



Example of
PIXE spectra
of two blue
pigments

Blue Temperas

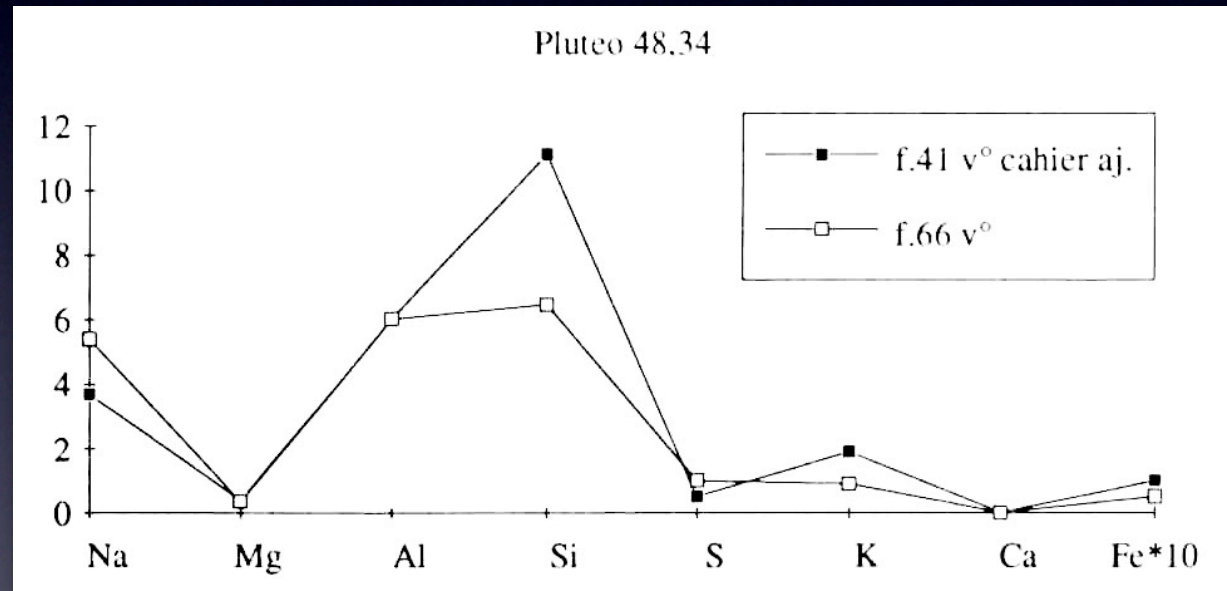
Extensive use of lapislazuli starting from XI century



Pluteo 48, 34, f. 66 v°



Pluteo 48, 34, f. 41 v°



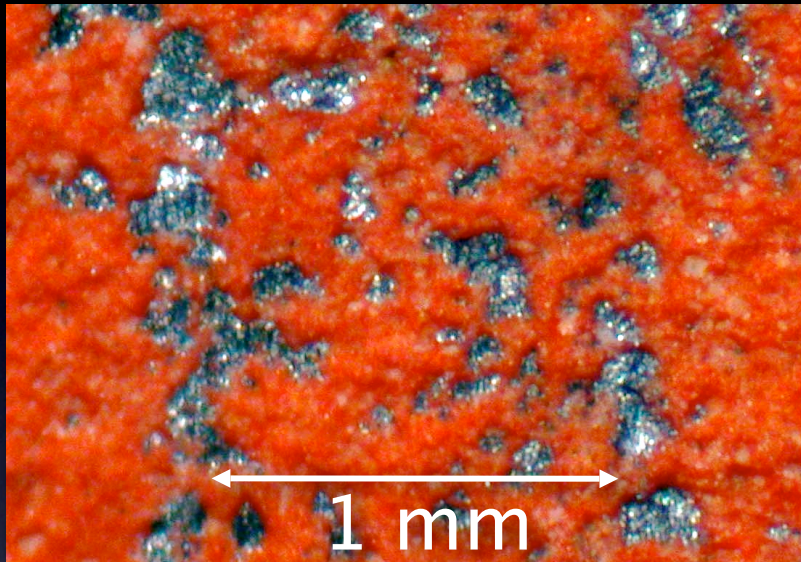
Metal point drawings

LEONARDO DA VINCI
STUDY OF A DRAPERY
Roma, Istituto Nazionale
per la Grafica

metal point, lead white
red prepared paper



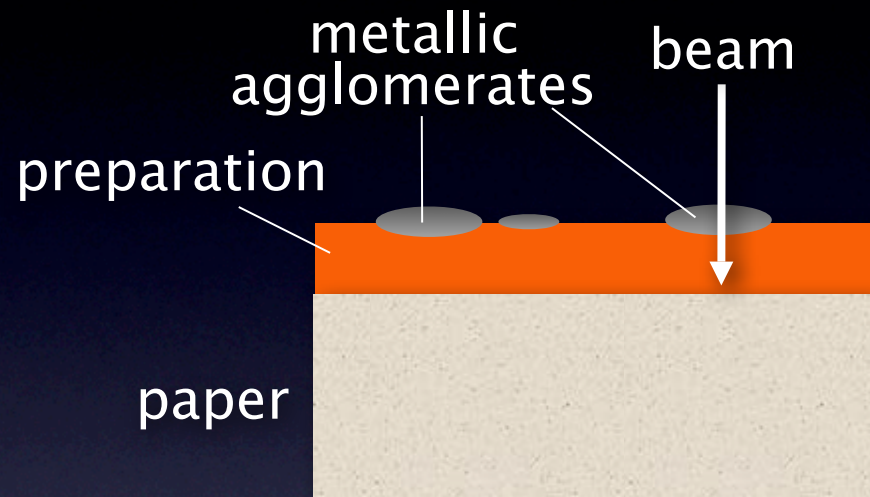
Characteristics of metal point drawings



The extension of the metallic agglomerates on the surface is some tens of μm



The beam size does not allow a detailed analysis

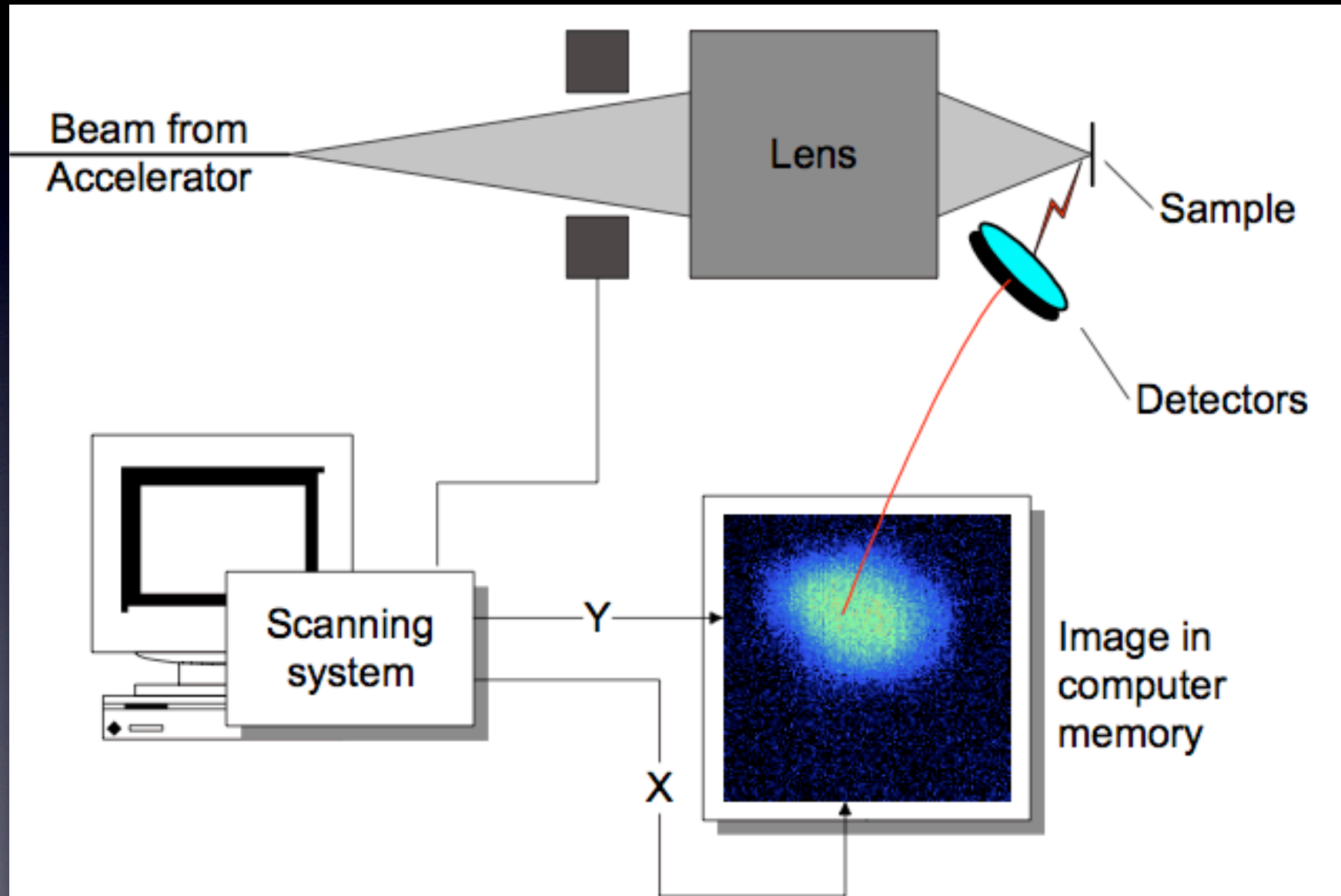


The beam can pass through the trace and hit the preparation



The contribution of the preparation must be taken into account

Nuclear microscopy

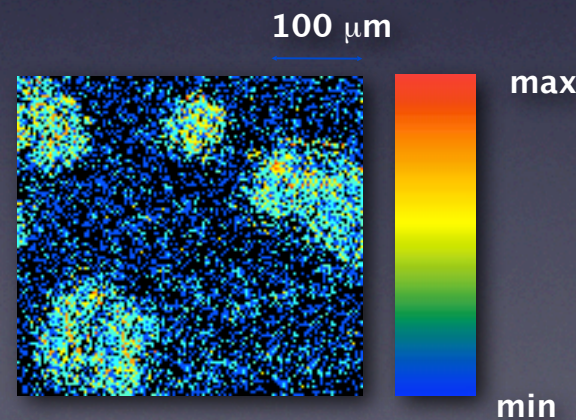
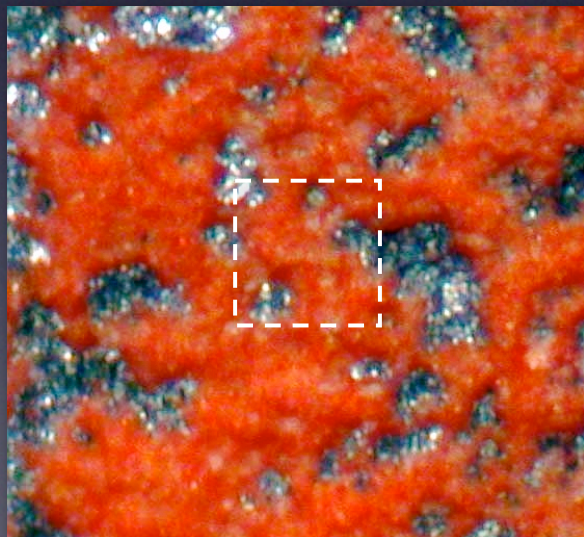


MicroPIXE analysis of metal point drawings

Four metallic points:
silver, lead, gold, copper

Red preparation:
cinnabar, yellow ochre,
lead white, bone white

Au Cu Pb

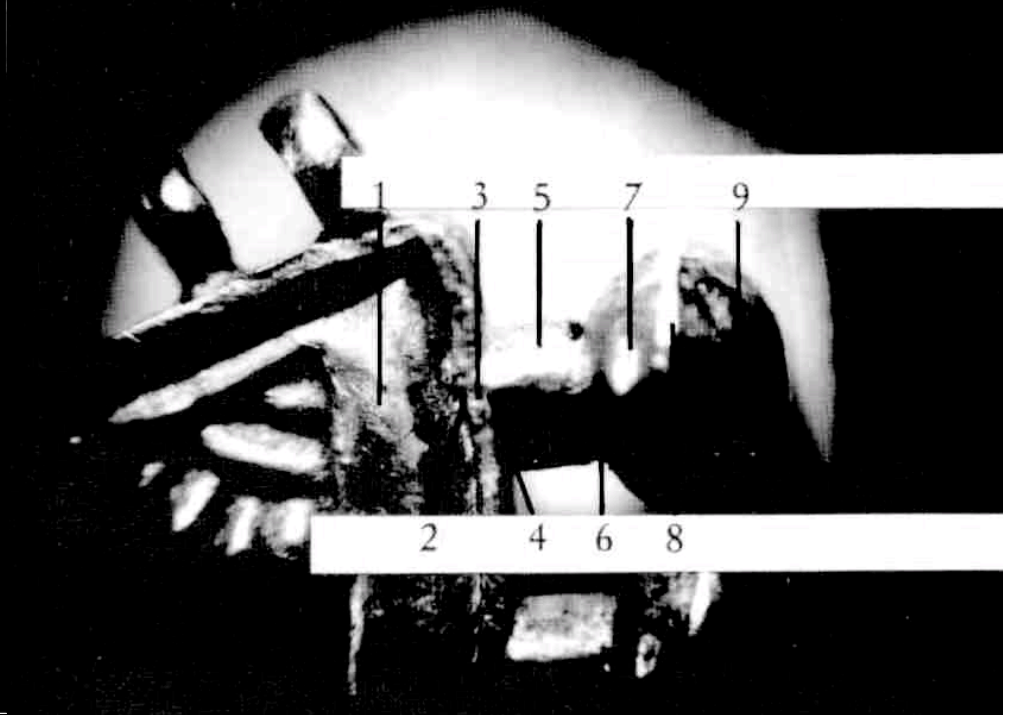
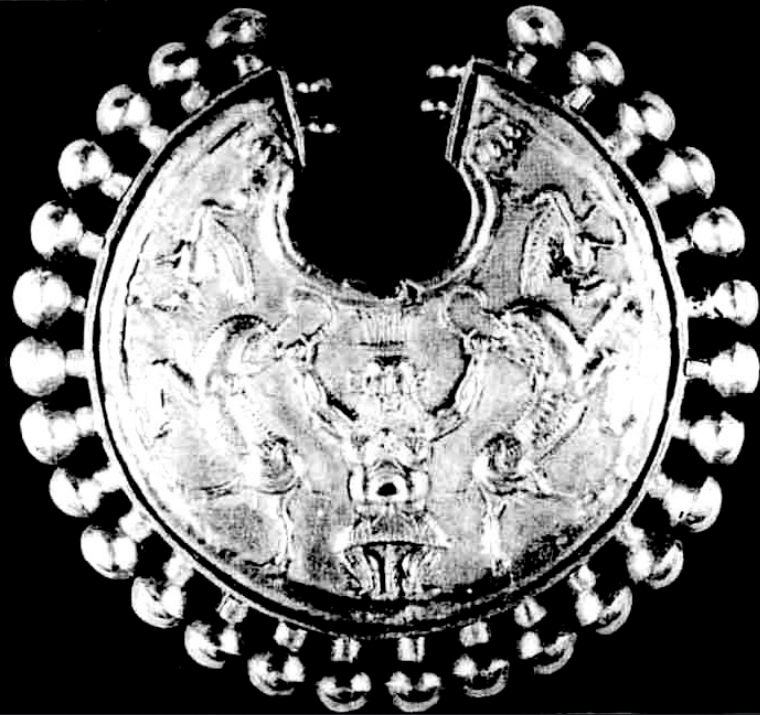


Elemental map on
 $0.4 \times 0.4 \text{ mm}^2$

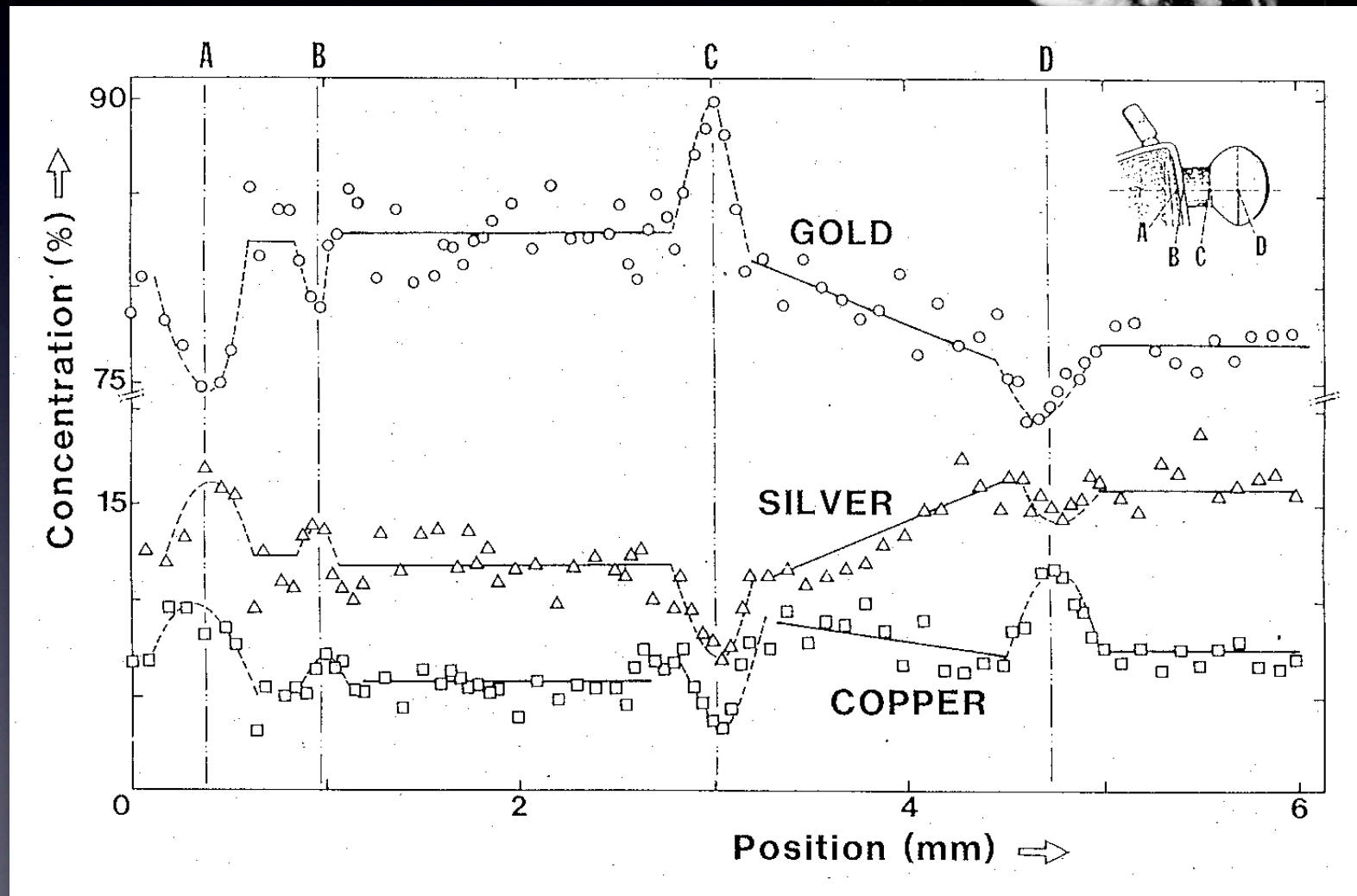
Lead stylus
(Pb, trace of Sn)

Red preparation
(S, Hg, Pb, Fe, P, Ca)

Micro-PIXE measurements of an Achemenide pendant (IV century BC)



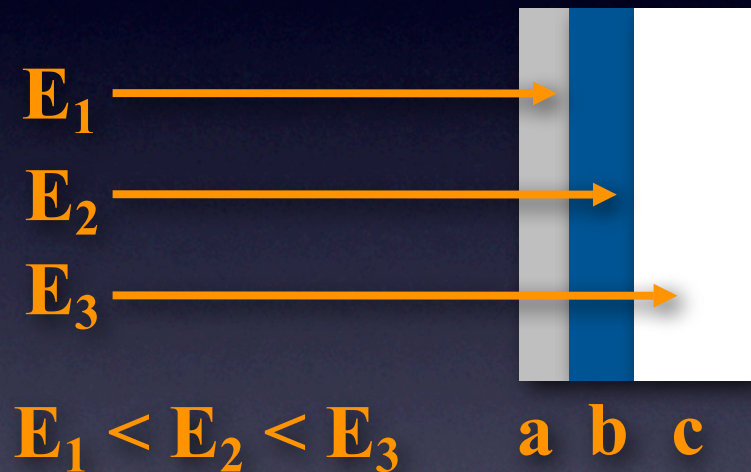
Micro-PIXE measurements of an Achemenide pendant (IV century BC)



Differential PIXE

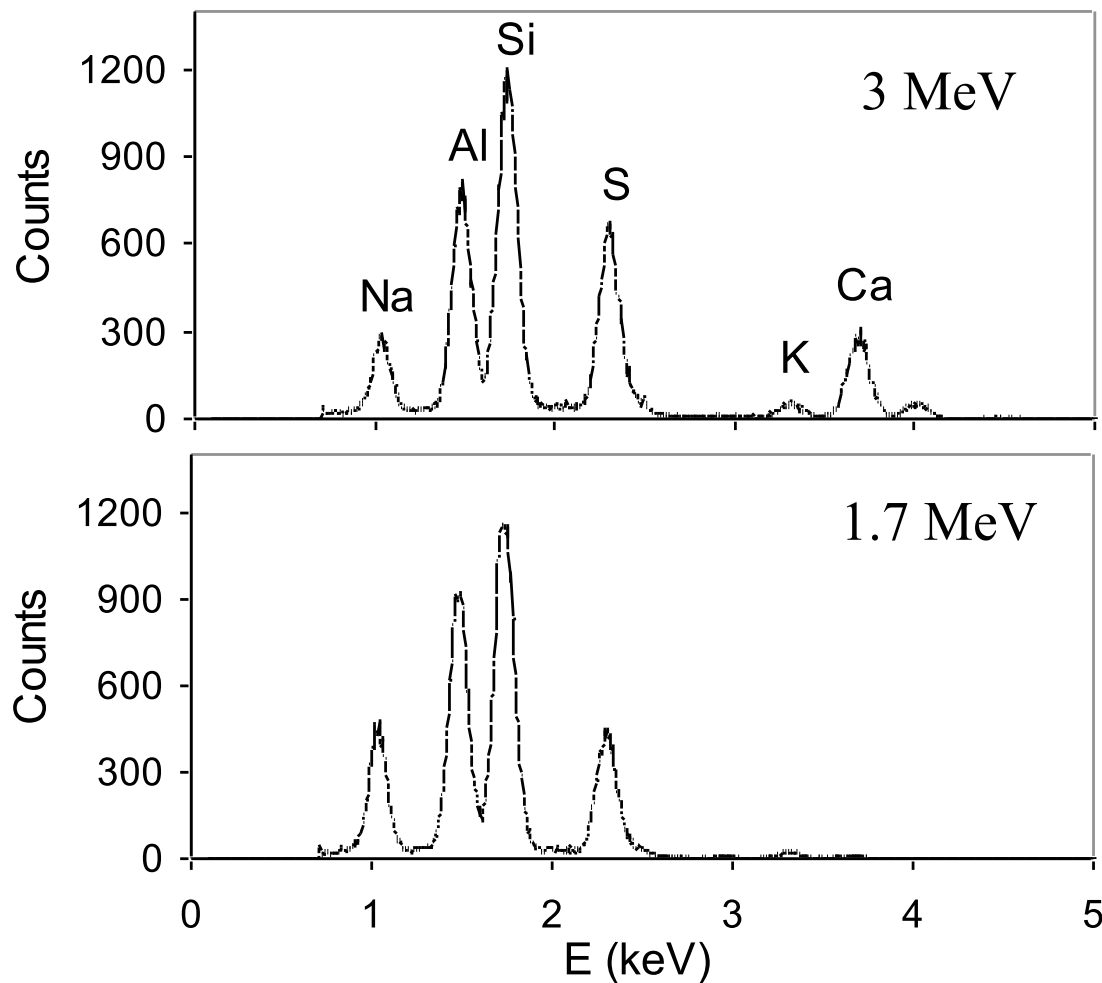
Consists in performing measurements on the same area with beams of different energies

At different energies
proton beam ranges
are different



By comparing X-ray spectra taken at different energies,
stratigraphic information can be obtained

PIXE spectra at different energies

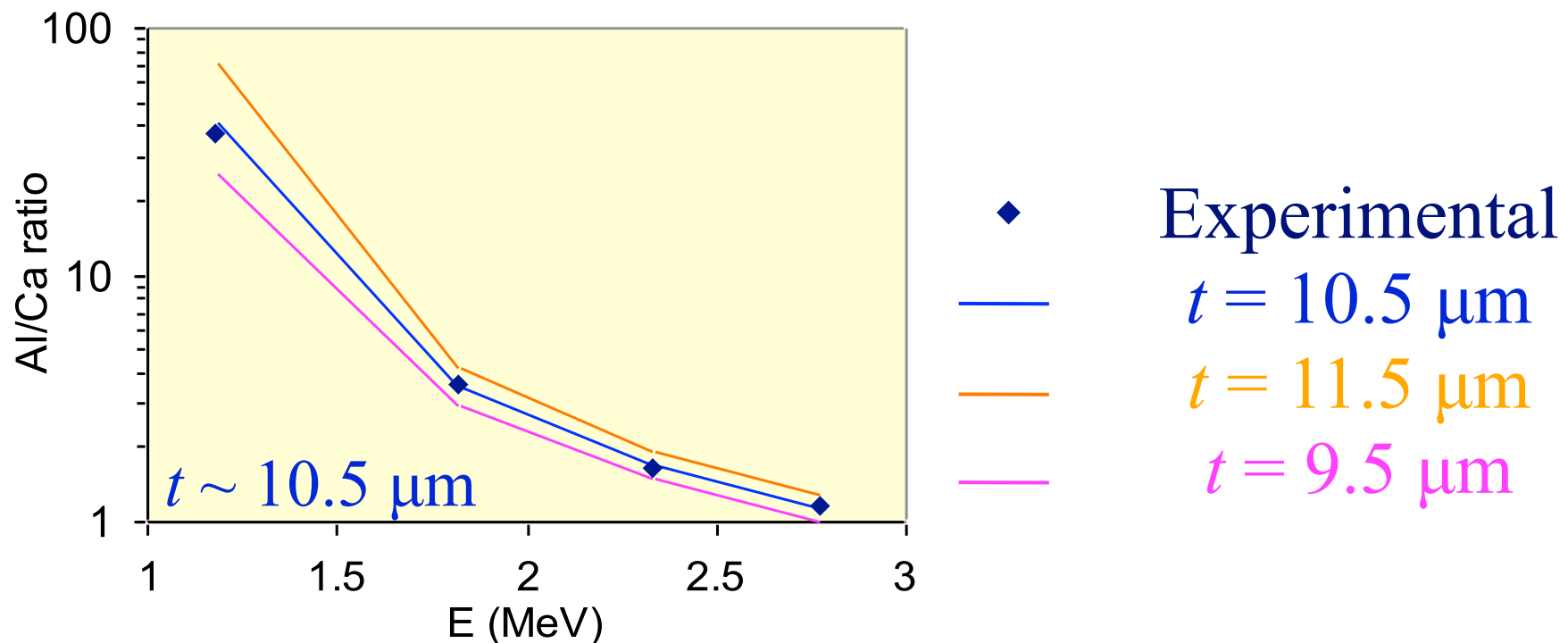


Blue paint layer
(lapislazuli) on a substrate
of calcium sulphate

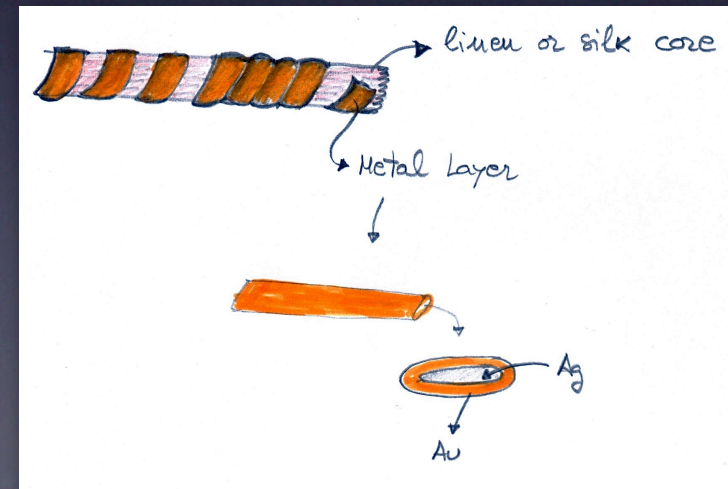
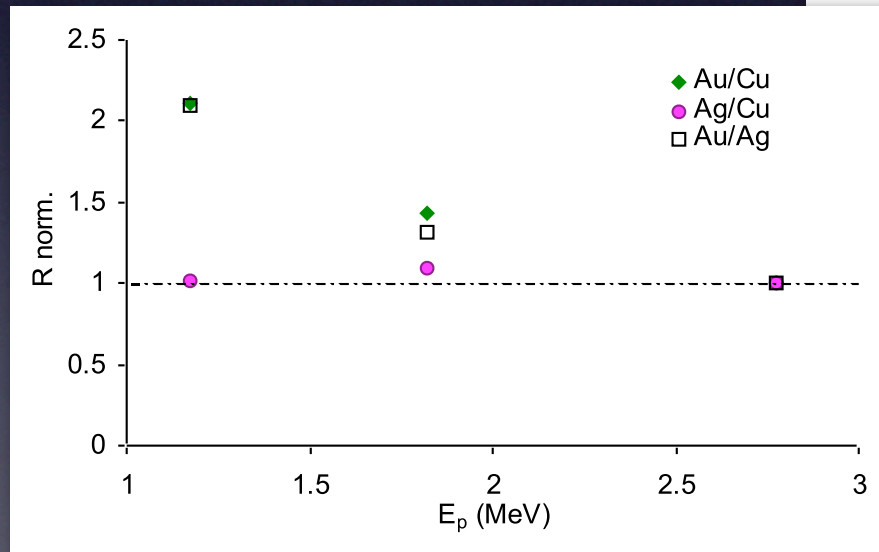
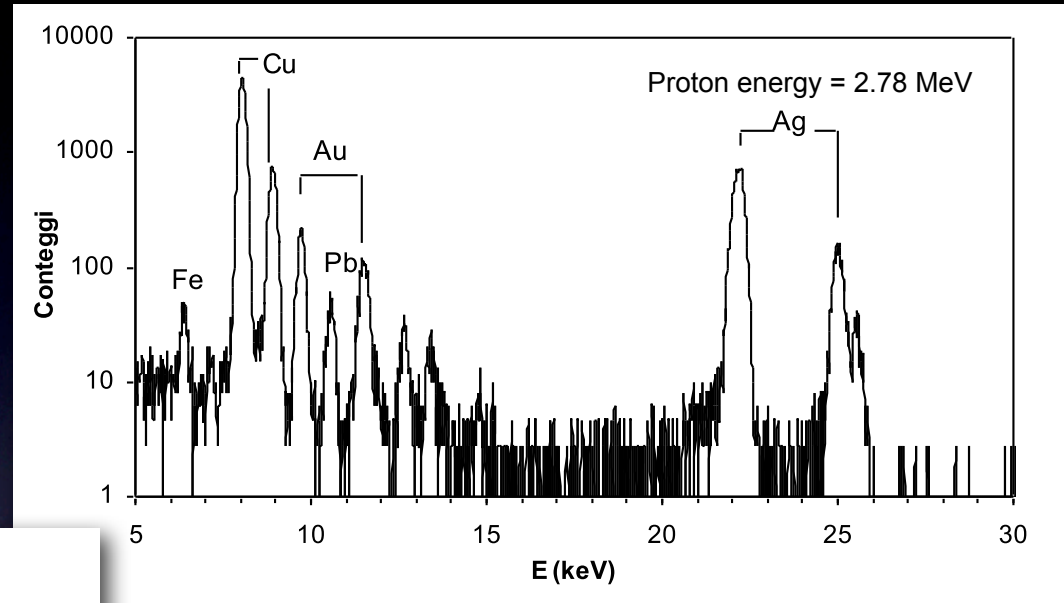
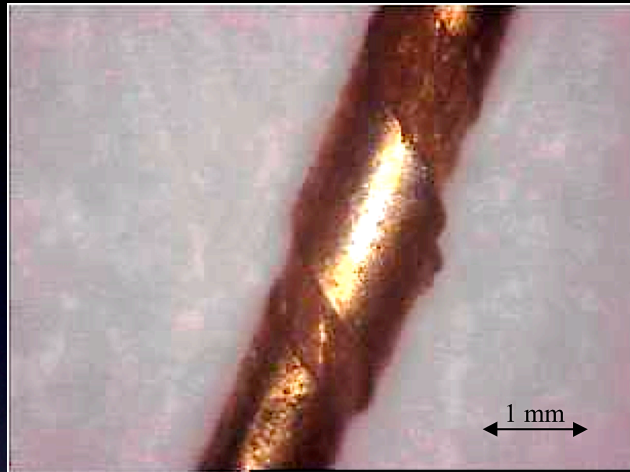


Estimate of the paint layer thickness

$$\frac{Y_{\text{Al}}}{Y_{\text{Ca}}} = \frac{C_{\text{Al}}}{C_{\text{Ca}}} \frac{\int_{E_p}^{E_p - \Delta E_{\text{lap}}(t)} \sigma_X^{(\text{Al})}(E) e^{-\mu_{\text{lap}}^{(\text{Al})} \frac{x(E)}{\cos \theta}} \frac{dE}{S_{\text{lap}}(E)}}{e^{-\mu_{\text{lap}}^{(\text{Ca})} \frac{t}{\cos \theta}} \int_{E_p - \Delta E_{\text{lap}}(t)}^0 \sigma_X^{(\text{Ca})}(E) e^{-\mu_{\text{white}}^{(\text{Ca})} \frac{x'(E)}{\cos \theta}} \frac{dE}{S_{\text{white}}(E)}}$$

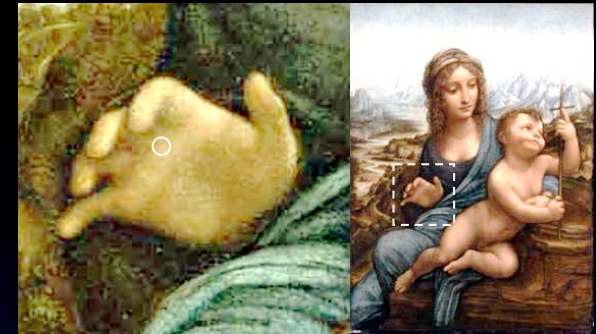
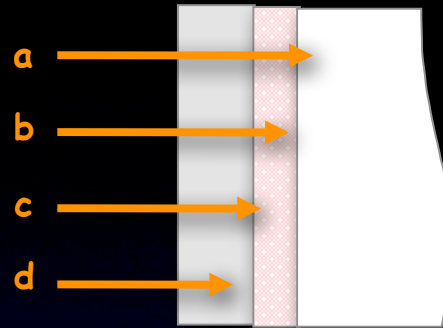
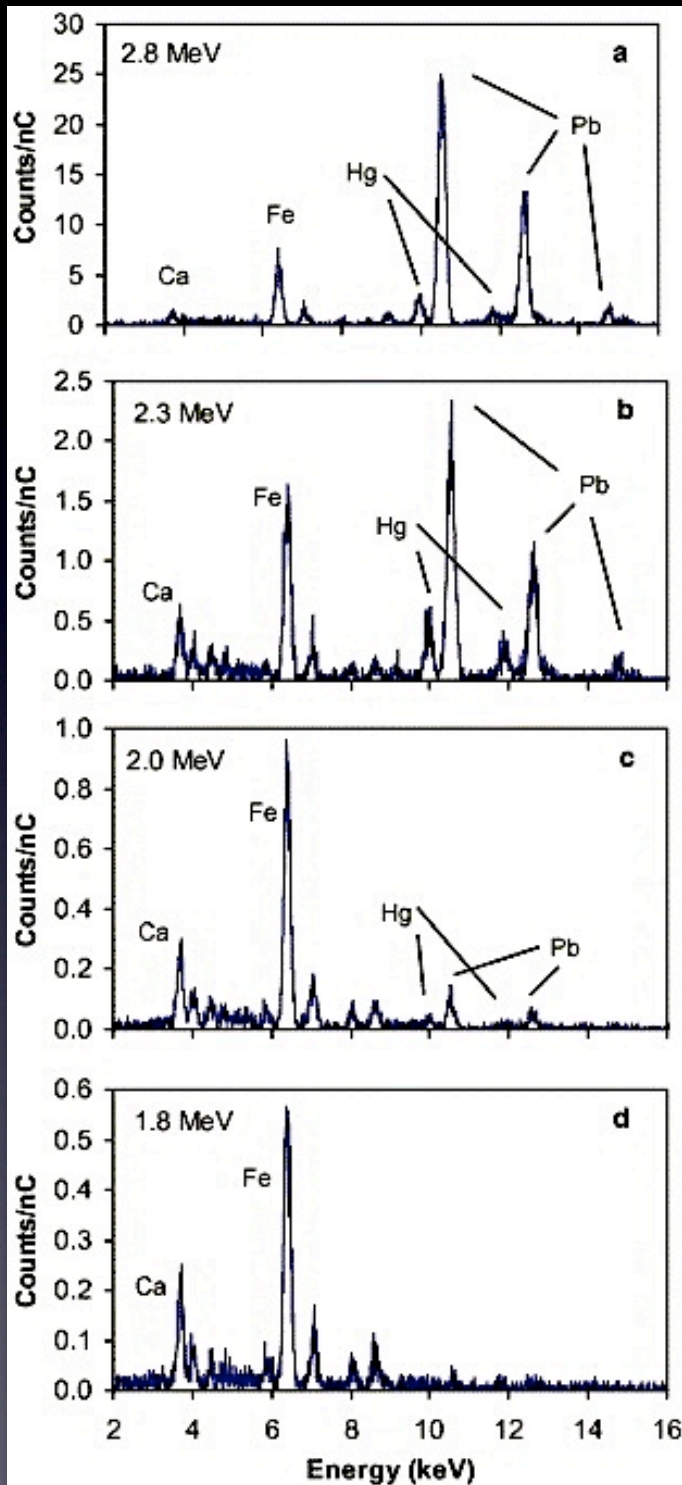


Metal threads (Alhambra, Granada)



Enrichment of gold on the surface

“Incarnato”



paint layer:

cinnabar (HgS, red pigment)+lead white

preparation:

lead white

Ca and Fe are in the varnish

Lapis-lazuli pigment in paint layers



“Maddonna dei fusi”, Leonardo da Vinci (1501)

Lapis-lazuli is a blue pigment, mainly composed of lazurite ($3\text{Na}_2\text{O} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot 2\text{Na}_2\text{S}$)

Limited possibility of identifying lapis-lazuli by PIXE in canvas and wood paintings:

- *low-energy X-rays absorption in the varnish and in the paint layer itself*
- *signal interference from other pigments*

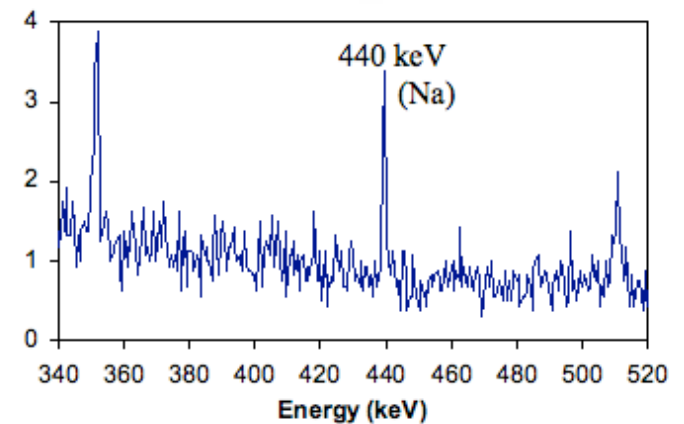
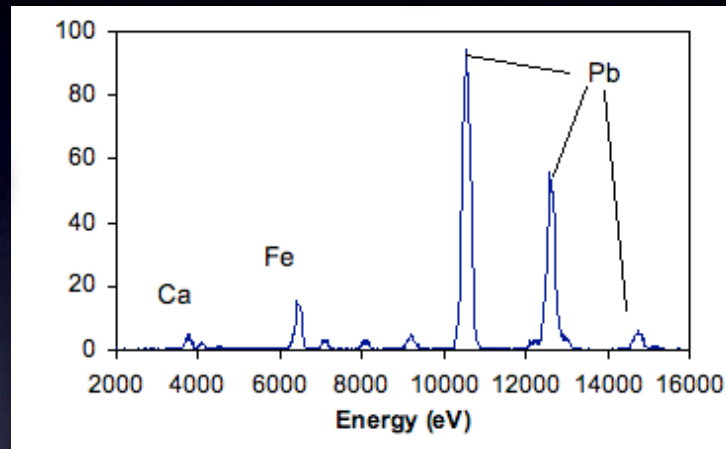
Lapis-lazuli pigment in paint layers

PIXE spectra

PIGE spectra

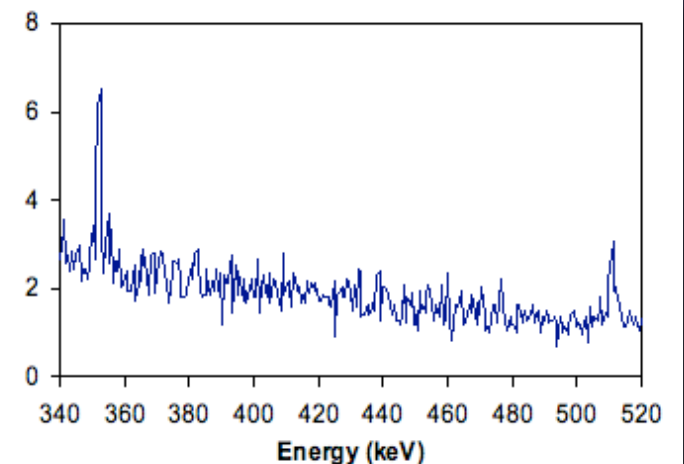
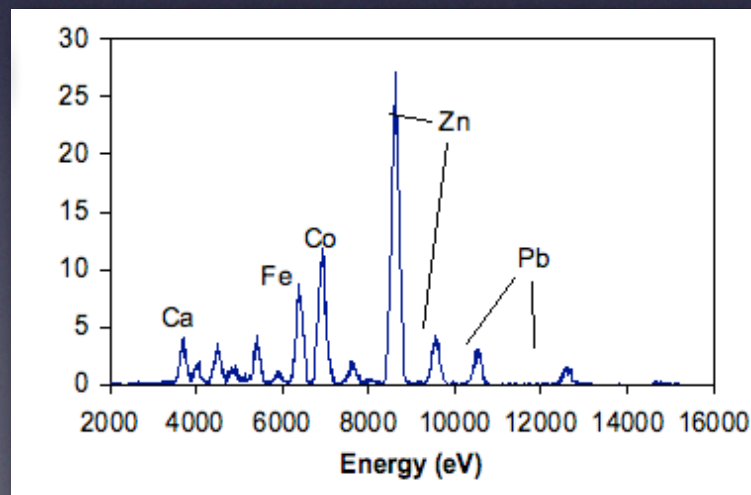
Original

Blue pigment mixed with Lead white (Ca and Fe from the varnish)



Restored

Cobalt blue and Zinc white (used only from XIX century!)



Analysis of ancient Roman glasses



Roman glass mosaic *tesserae* from Villa Adriana, Tivoli (Italy)

Quantification of Na is of great importance for the characterization of ancient glasses

Two basic typologies of Western glass:

- **natron** (high Na_2O , low K_2O and MgO)
Roman and High Middle Ages
- **plant ash** (low Na_2O , high K_2O)
since Middle Ages

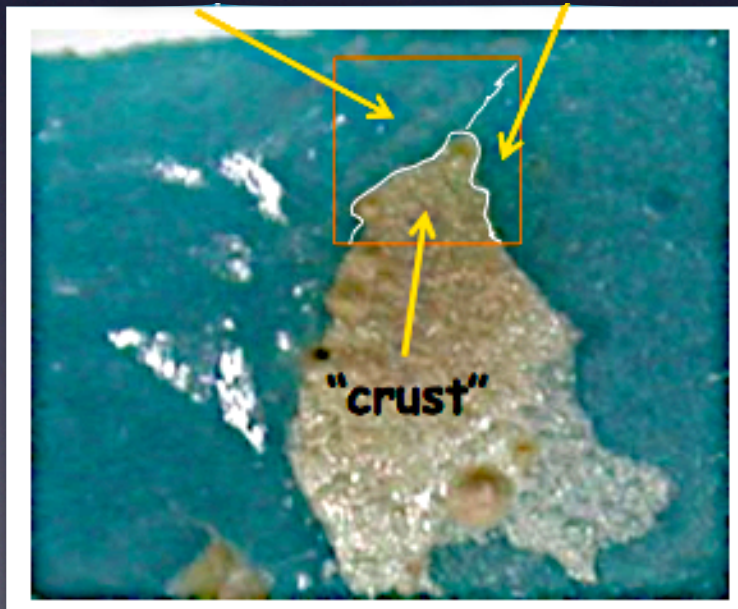
Sodium in Roman glasses

X-rays from the lightest elements strongly absorbed by crusts and *patinae*

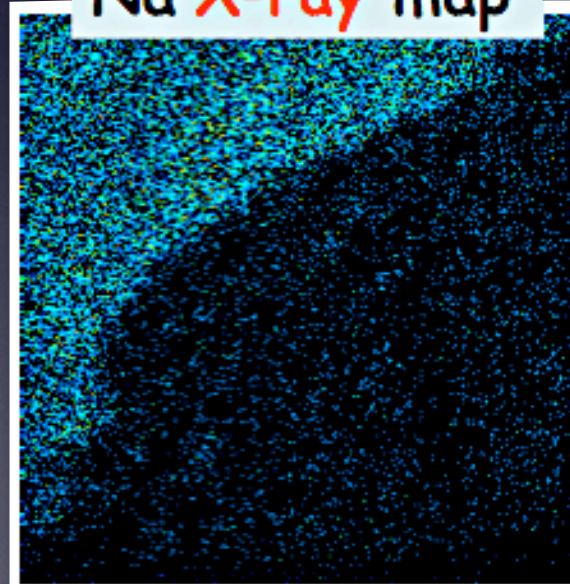


Roman glass mosaic *tesserae*

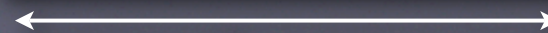
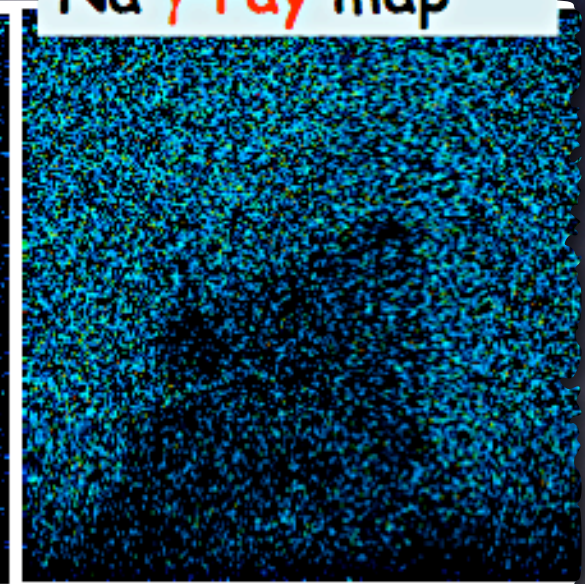
“freshly cut” zone coloured but more opaque zone



Na X-ray map



Na γ -ray map

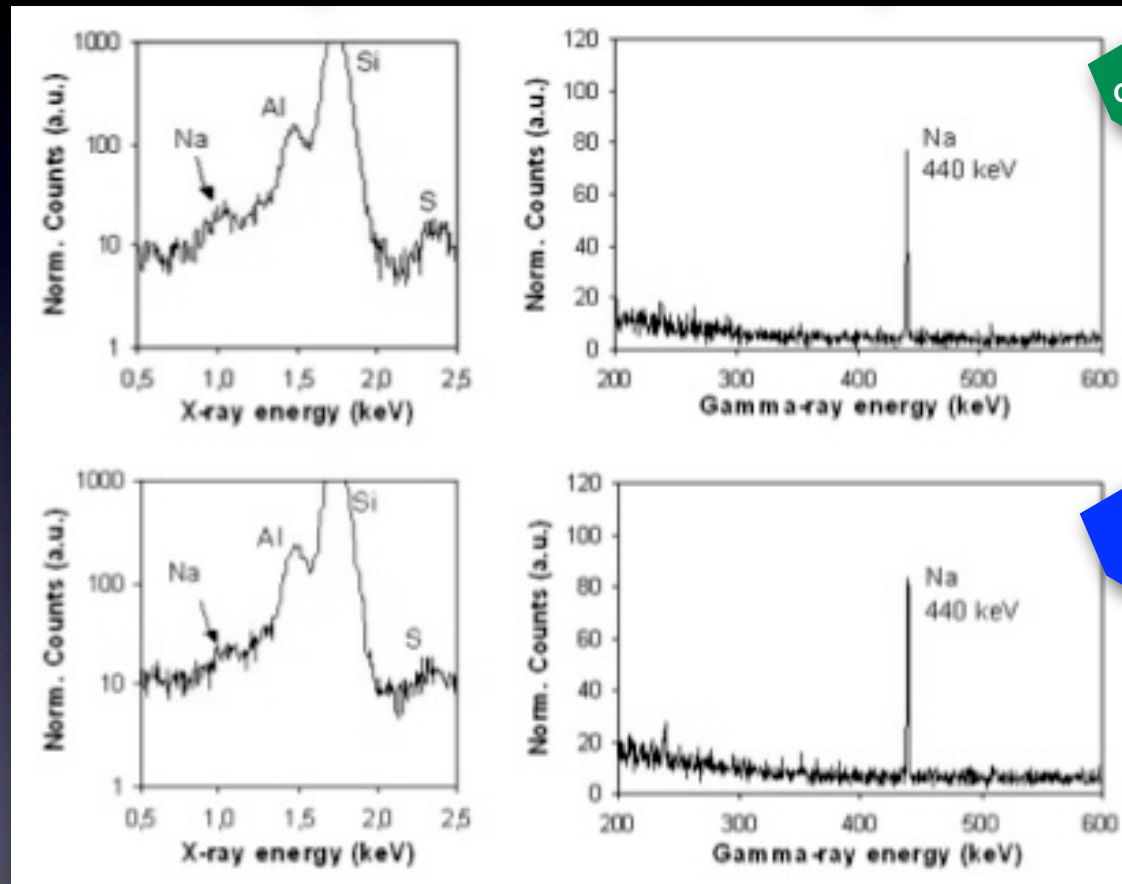


2 mm

Sodium in Roman glasses

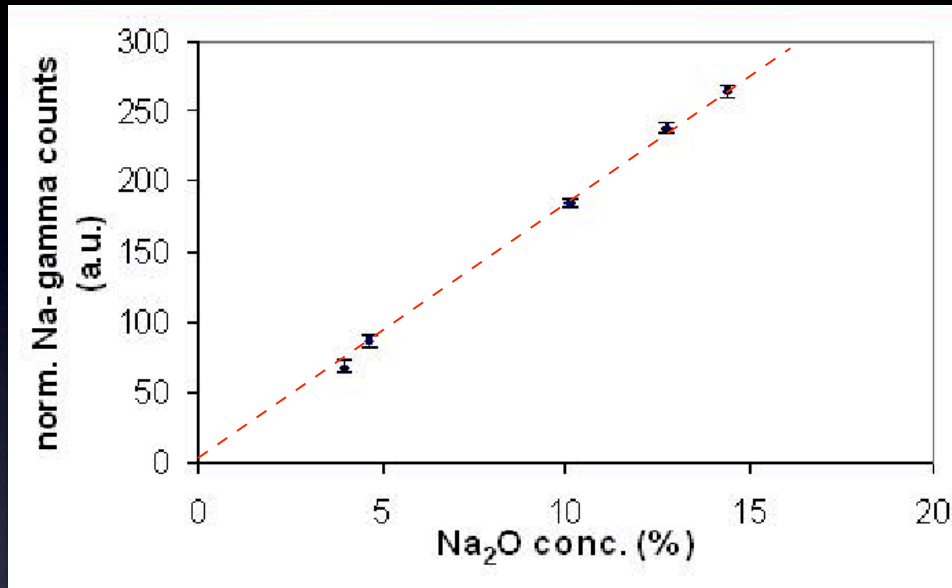
PIXE spectra

PIGE spectra



X-rays from the lightest elements strongly absorbed by crusts and *patinae*

Sodium in Roman glasses

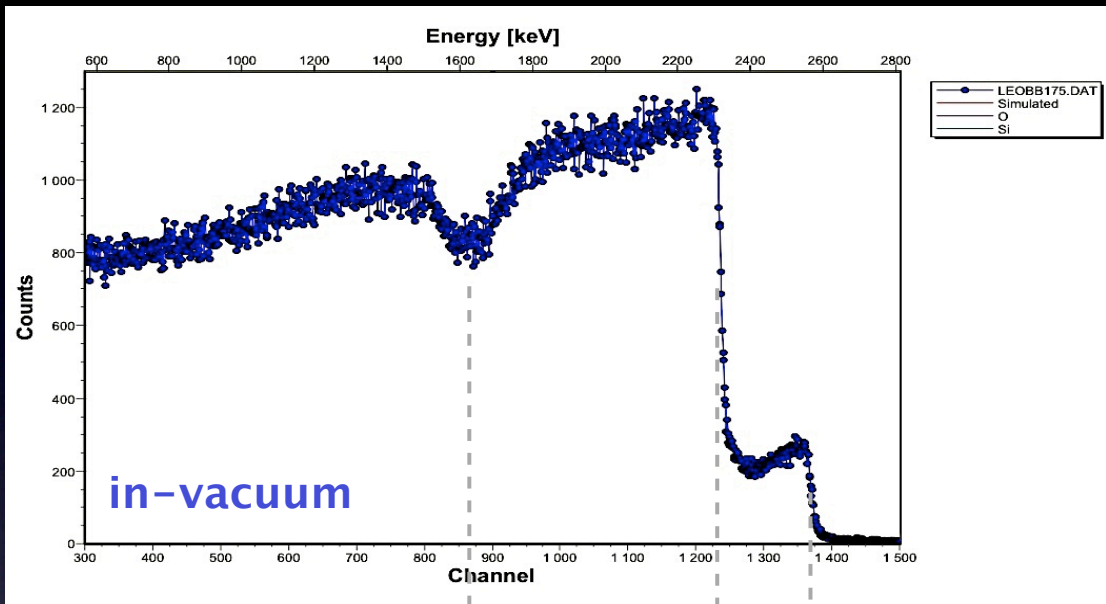


Estimate of Na content by comparing gamma-ray yields to those of thick glass standards (NIST SRM) with certified Na₂O concentration

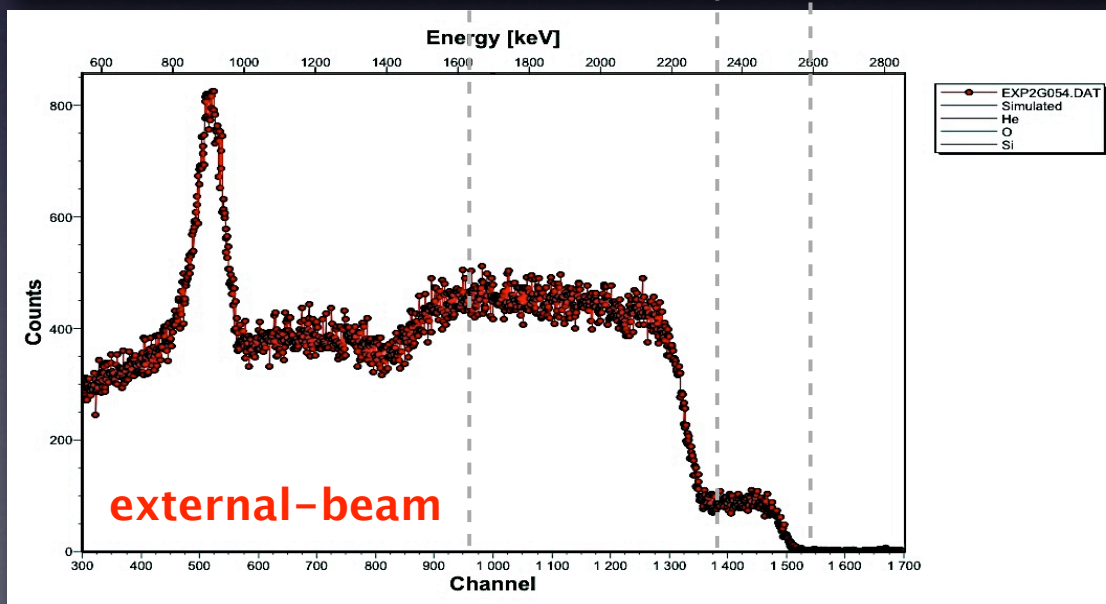
Concentration ranges perfectly compatible with the typical Roman soda-lime-silica glass

glass colour	main oxides (%)			
	Na ₂ O	SiO ₂	CaO	PbO
green	~20	55-60	5-9	1-3
blue	~20	60-65	5-9	<0.1
turquoise	~20	55-60	5-9	<0.3
yellow	~15	55-60	5-9	5-8
red	~10	35-40	5-9	30-35

RBS: External vs Vacuum



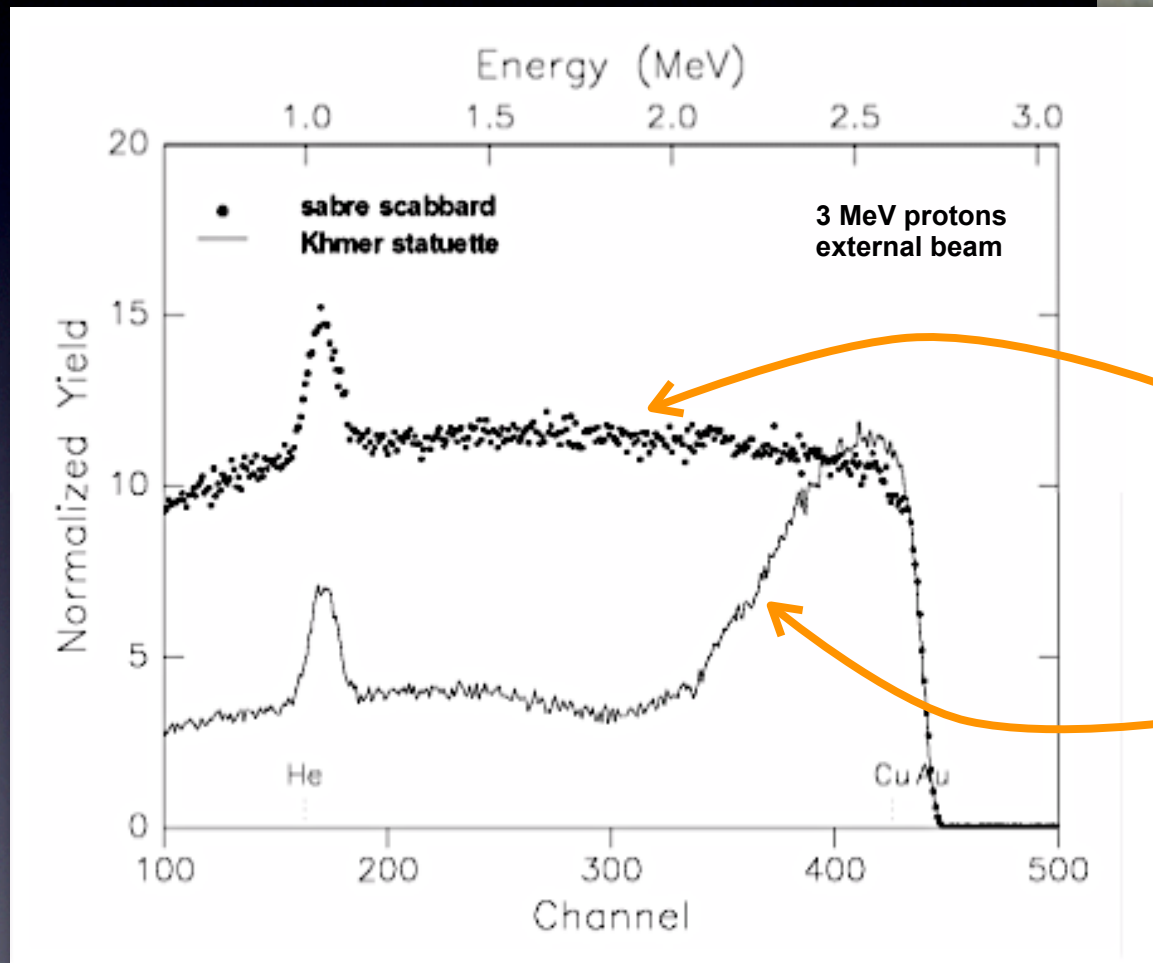
- 3 MeV protons on target
- $\theta = 150^\circ$
- SiO_2 target



- Energy loss and energy straggling * in exit window and external path in atmosphere

* 15 keV FWHM for 500 nm Si_3N_4 + 10 mm He

Gold alloy or gilding

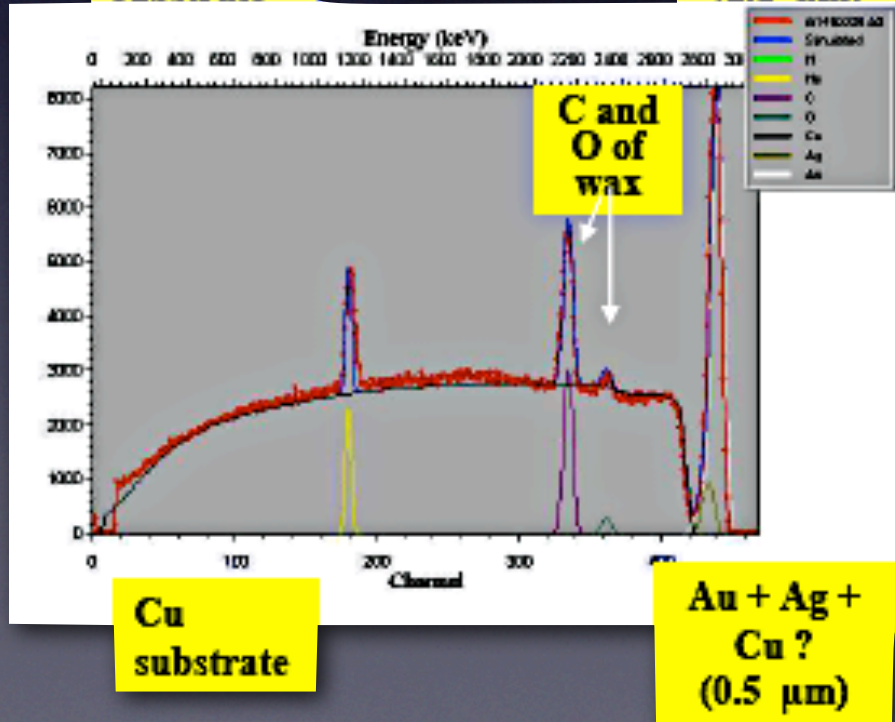
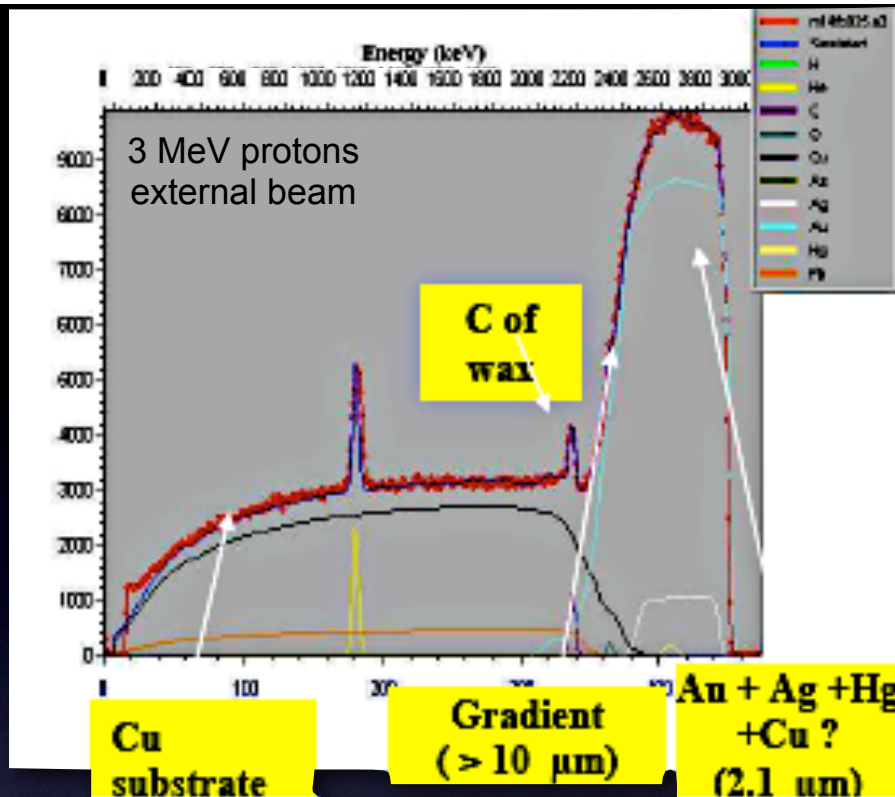


The scabbard is made of a thick Au-Cu alloy (thickness > 10 μm).

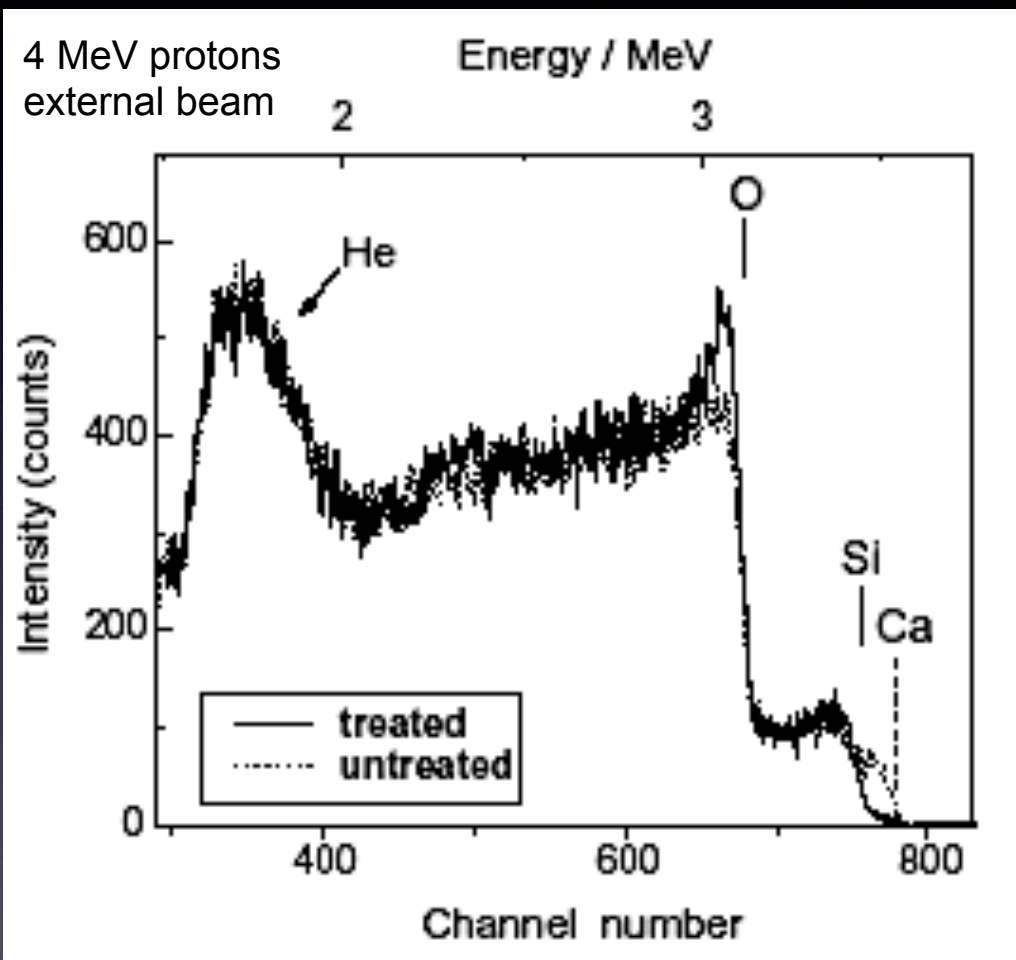
For comparison, the gilding of the statuette is about 2 μm thick.

From the width of the Au signal it is possible to obtain the thickness of the gilding (dE/dx is known).

RBS analysis of a gilded bronze chandelier (XI century) from the Cathedral of Hildesheim, Germany



RBS study of glass corrosion



Leaching of Ca, K, Na from the interaction with moisture or water and formation of surface hydrate silicates (cfr. PIGE analysis)

Complementary PIXE/RBS

In samples with a layered structure the elemental depth profile is needed to correctly calculate absorption effects in PIXE

PIXE strenghts

- High sensitivity
- Excellent specificity

RBS strenghts

- Traceable accuracy
- Excellent depth resolution

RBS weaknesses

- Low sensitivity
- Poor mass resolution

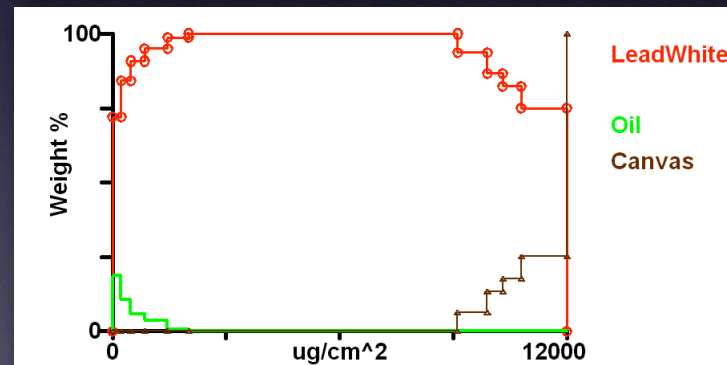
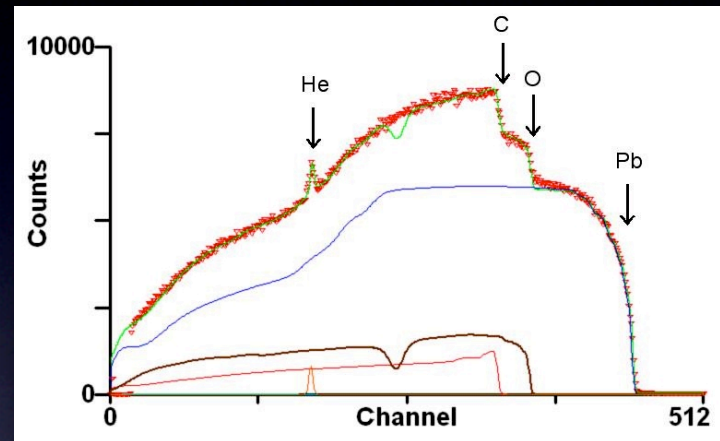
PIXE weaknesses

- Poor traceability
- Poor depth resolution

Characterization of paint layers by simultaneous PIXE/RBS analysis



"La Bohémienne", Frans Hals (1630)



The canvas is schematized as carbon plus chalk (CaCO₃)

Ochre pigment (ematite) detected and quantified thanks to simultaneous PIXE/RBS measurements: $440 \cdot 10^{15}$ atoms/cm² Fe₂O₃ in $7000 \cdot 10^{15}$ atoms/cm² of oil (C₁₃O₅)

Thanks for
your attention!

Bibliography

- R. Tesmer, M. Nastasi ed.s *“Handbook of Modern Ion Beam Materials Analysis”* MRS
- S.A.E. Johansson, J.L. Campbell, K.G. Malmqvist ed.s *“Particle-induced X-ray emission spectrometry (PIXE)”* John Wiley & sons
- W.-K. Chu, J.W. Mayer, M.-A. Nicolet *“Backscattering Spectrometry”* Academic Press
- G. Deconninck et al. *“Prompt gamma-ray spectroscopy and its use in elemental analysis”* At. Energy Rev. suppl. no. 2 (1981) 151