

Introduction to IBA – The RBS and ERD techniques

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Outline

- Ion Beam Analysis
- Theoretical background
- Rutherford Back Scattering
- Elastic Recoil Detection Analysis
- Conclusions



Pros / Cons

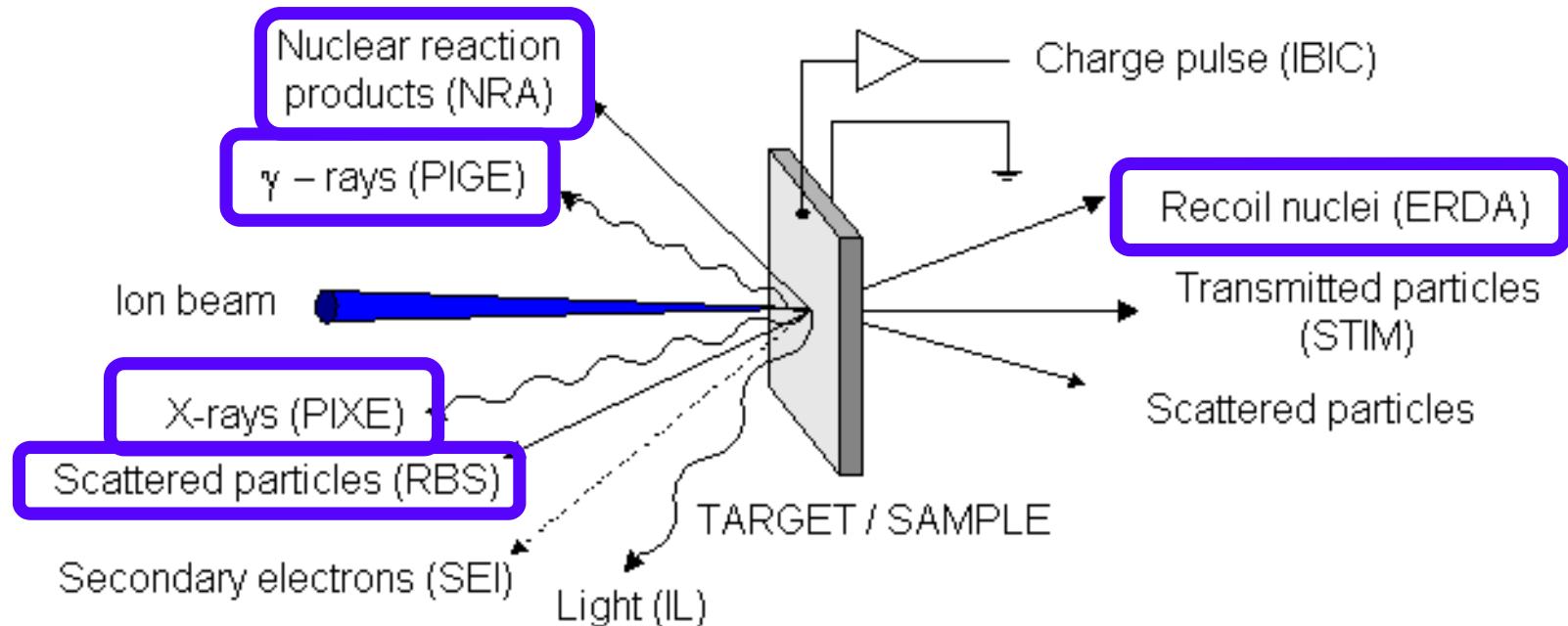
- They are generally least destructive and are suitable for use with delicate materials.
- They are to a certain extent multielementary and produce high-accuracy quantitative results.
- They require little or no preparation of the sample with the result that a specimen (like an artifact) could be directly analyzed.
- Only very small quantities (mg) of sample are needed.
- They permit the analysis of a very small portion of the sample by reducing the diameter of the ion beam to less than 0.5 mm.

- Some damage cannot be avoided (thermal, carbon buildup etc.)!
- A VdG type of accelerator is required.
- In most of the cases the experiments are carried out in vacuum chambers.
- Several experimental issues need to be addressed, thus a minimum knowledge of nuclear physics (experimental and theoretical) is mandatory.
- No direct information about the chemical environment can be produced.
- The analysis concerns only a few microns below the surface of the samples.
- In most of the cases, a combination of techniques is required to solve a problem, and this implies time consuming experiments!



Ion Beam Analysis

Ion Beam Analysis (IBA) is based on the **interaction**, at both the atomic and the nuclear level, between **accelerated charged particles** and the bombarded material. When a charged particle moving at high speed strikes a material, it interacts with the electrons and nuclei of the material atoms, slows down and possibly deviates from its initial trajectory. This can lead to the **emission of particles or radiation** whose energy is characteristic of the **elements** which constitute the **sample material**



Theoretical Background I

Nuclear Reaction:

The interaction between two nuclei which results in the emission of nuclei and/or gamma rays.

Cross Section:

The probability of a nuclear reaction to occur

$$\sigma = \frac{N_{\text{det}}}{\Omega \cdot N_{\text{inc}} \cdot N_{\text{tar}}}$$



Theoretical Background II

Scattering:

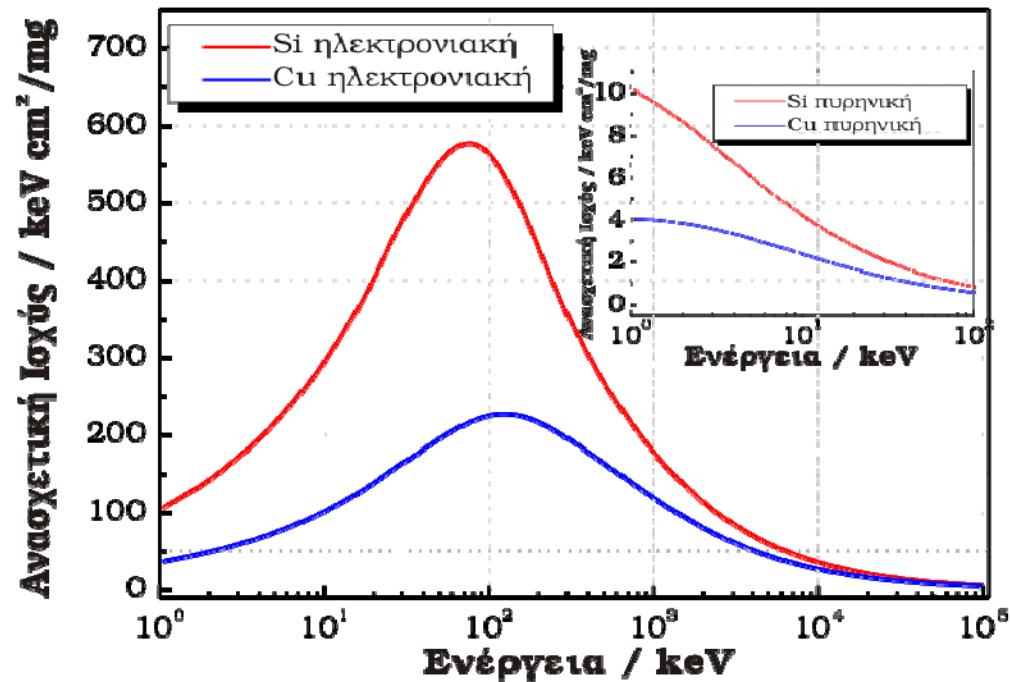
When a charged particle impinges on a material, it interacts with the electrons and the nuclei of the material. The result of the interaction is the loss of energy and the change of trajectory of the initial ion.

Energy Straggling:

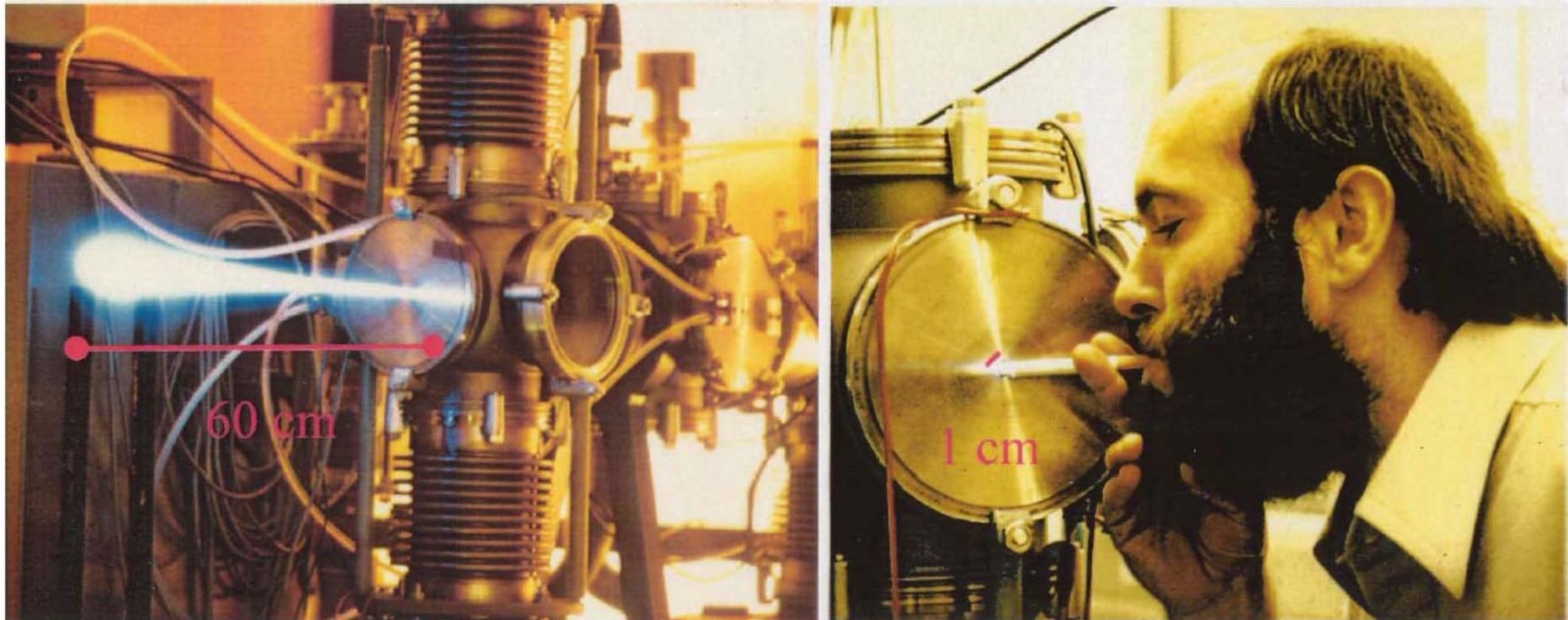
Loss of kinetic energy per length unit

Inelastic collisions with the electrons and the nuclei

$$S(E) = S_e(E) + S_n(E)$$



Theoretical Background II



7 MeV beam energy
 $R \approx 60$ cm

0.3 MeV beam energy
 $R \approx 1$ cm



Depth Profiling

YES

- Rutherford Backscattering Spectroscopy (RBS)
- Nuclear Backscattering Spectroscopy (NBS)
- Elastic Recoil Detection Analysis (ERDA)
- Nuclear Reaction Analysis (NRA)

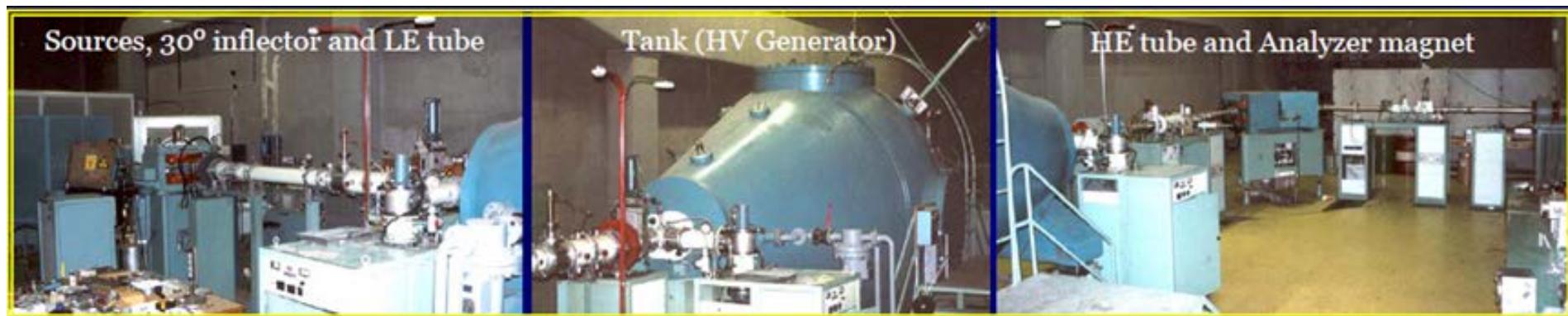
- Particle Induced γ –Ray Emission (PIGE)

NO

- Charged Particle Activation Analysis (CPAA)
- Particle Induced X-Ray Emission (PIXE)
- Neutron Activation Analysis (NAA)
- Secondary Ion Mass Spectroscopy (SIMS)



The 5.5 MV VdG Tandem accelerator @ I.N.P.



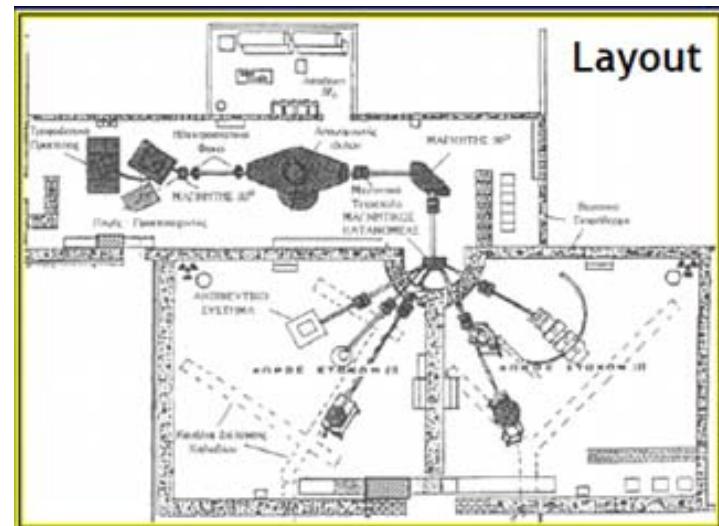
Sources: Sputter & Duoplasmatron	
Accelerated beams	Source Current (max)
p	40 µA
d	20 µA
⁴ He	1 µA
Li	1 µA
C	15 µA
O	15 µA
F	1 µA
Ti, V, ... Os	0.5 µA

Operation (no PAC) 2500 hrs/year, 65% external users

Basic Research (60%)
Nuclear Astrophysics
Neutron Physics
Nuclear Reactions on light systems

Applied Research (40%)

Materials, Archaeometry,
Environmental studies

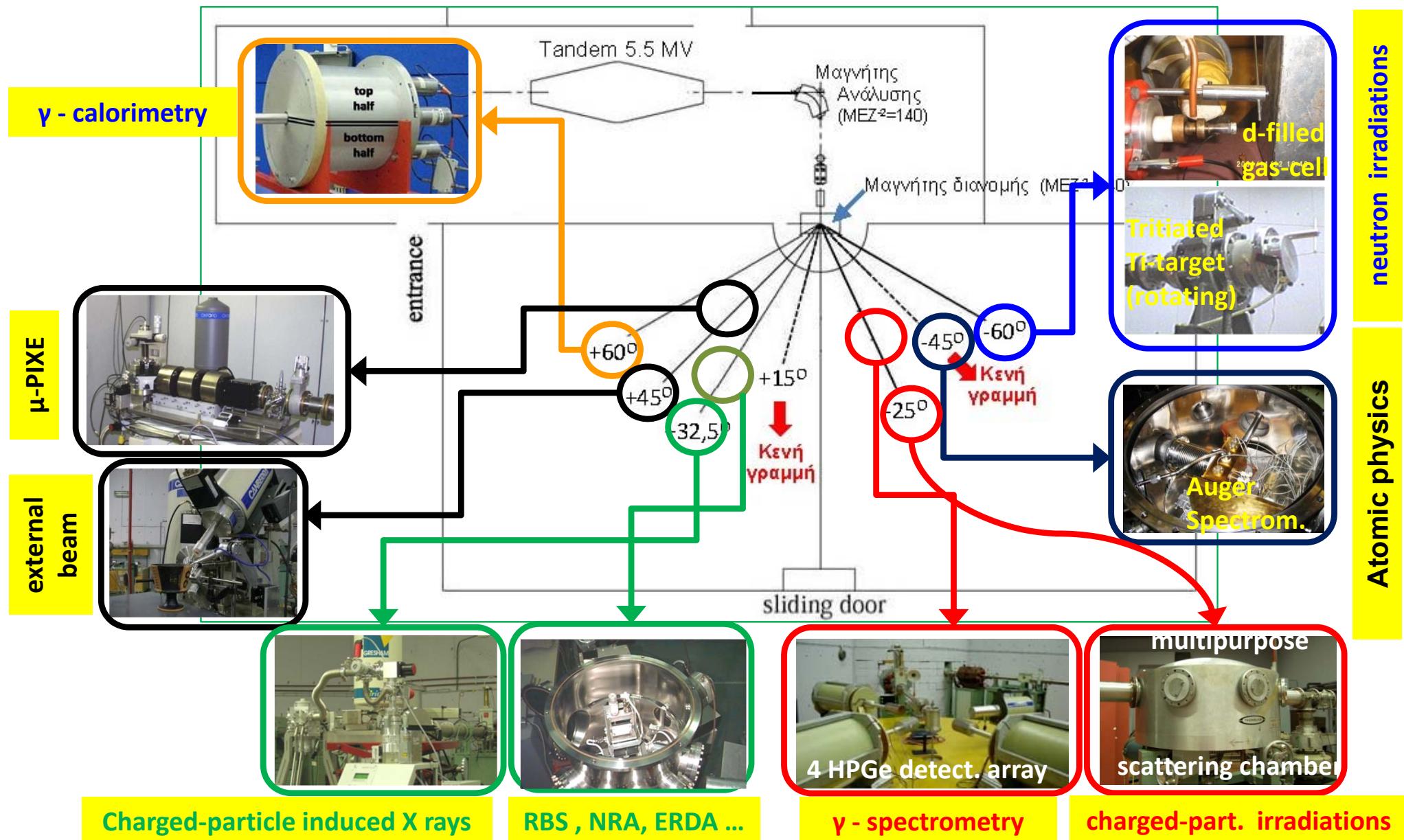


THE ONLY ACCELERATOR FACILITY EXISTING IN GREECE



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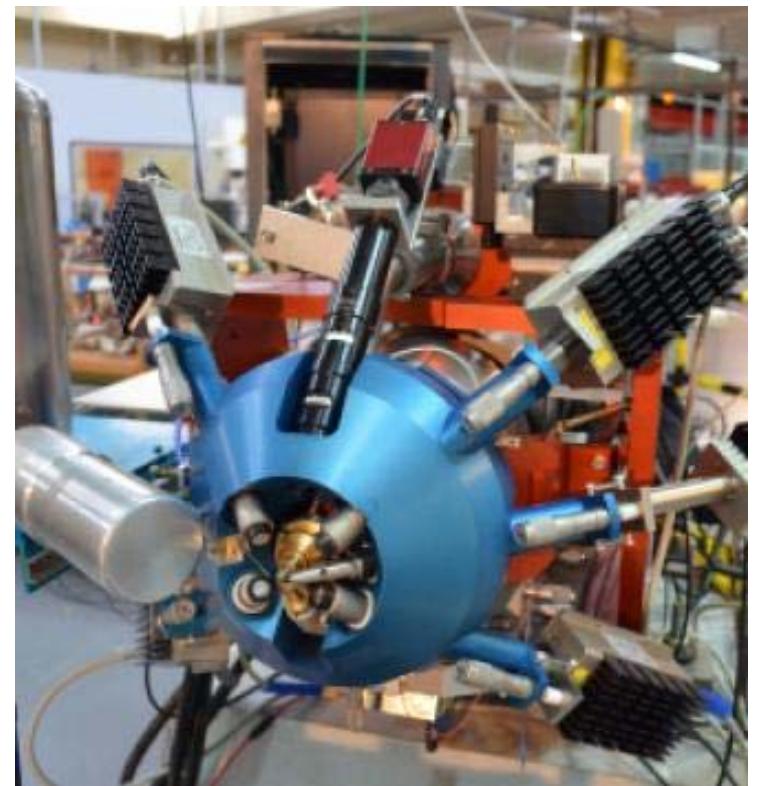
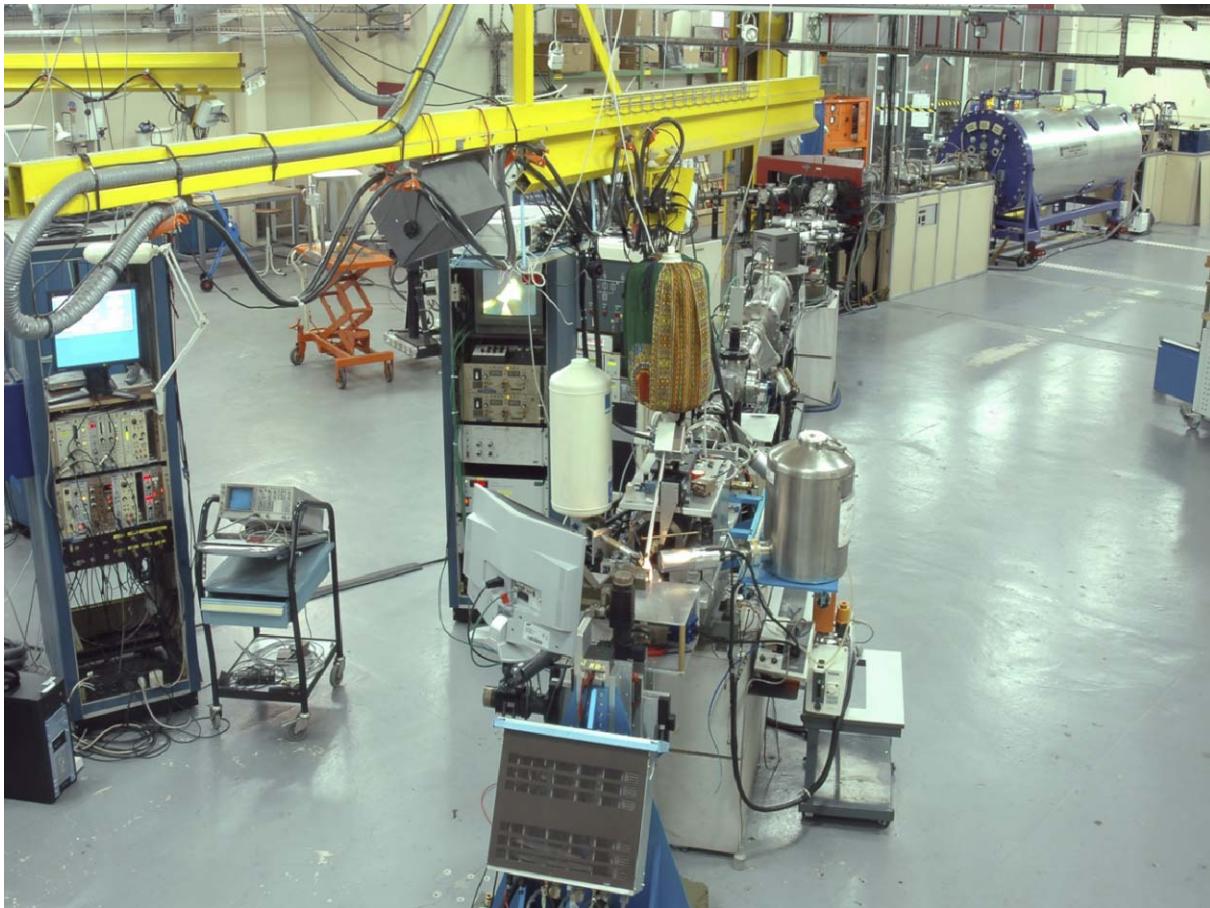
Tandem Layout



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AGLAЕ

Accelerateur Grand Louvre d' Analyse Elementaire



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Sample Size Selection

There are three possibilities

Under Vacuum

Small samples (1 to 10 cm)
Can withstand vacuum (no wood)
Preferably good electrical conductivity
Greater accuracy

External Beam

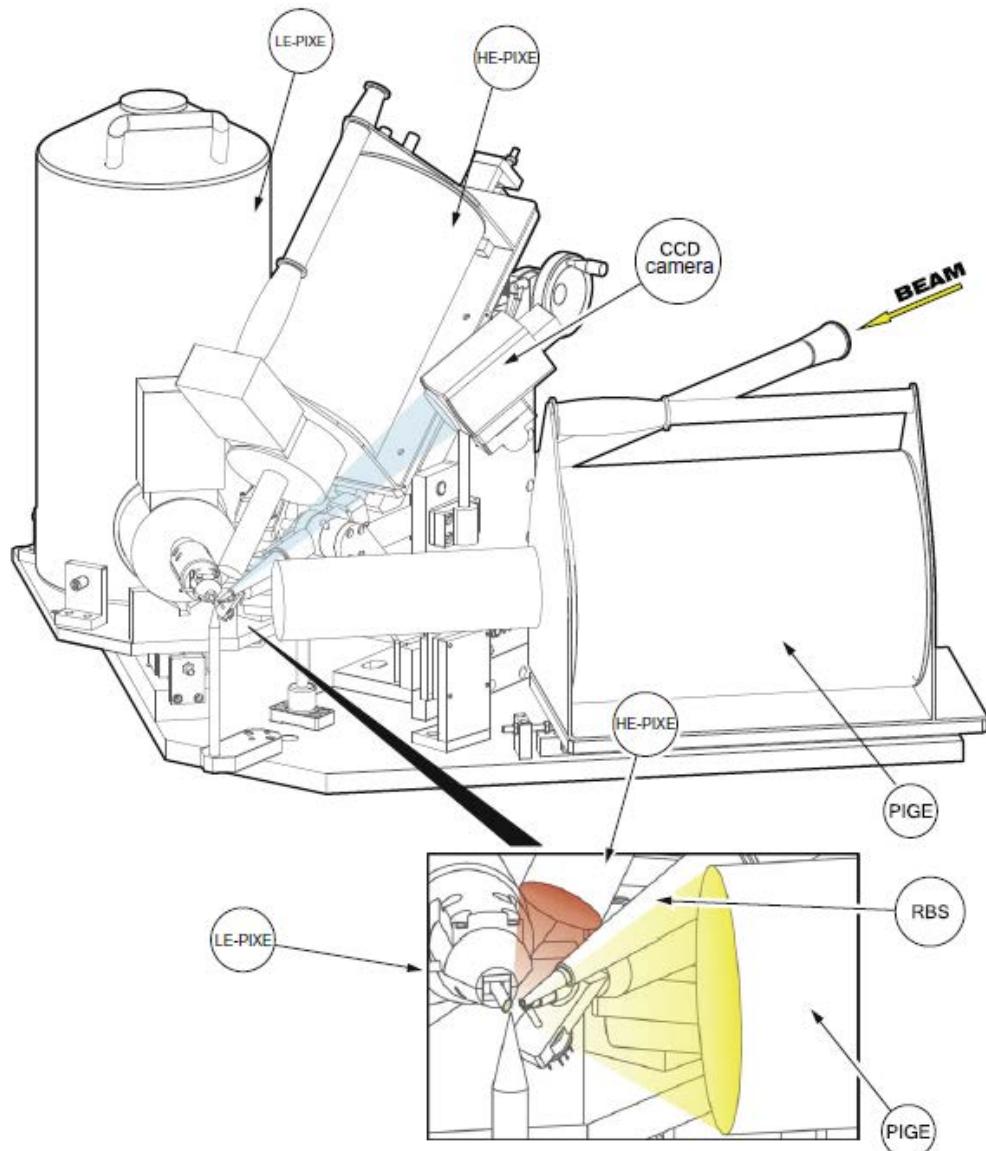
No size limitation
No vacuum conditions
Flow of He
Limited accuracy

Microbeam

Small samples (less than 1 cm)
Elemental mapping possibilities



External Beam Setup

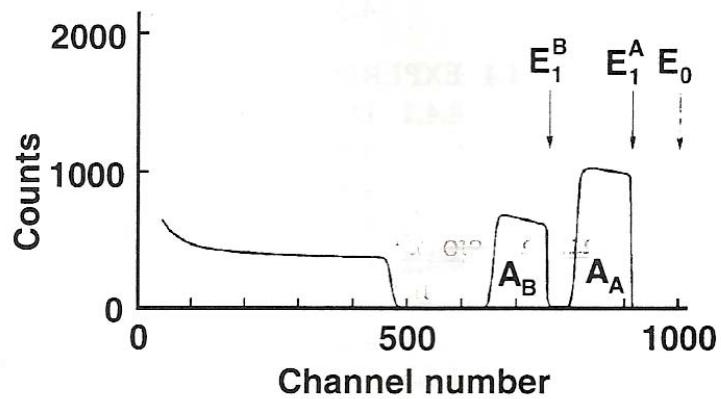
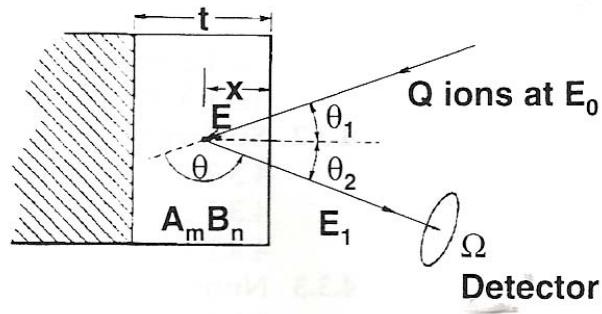


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Rutherford Backscattering Spectroscopy

Rutherford backscattering (RBS) is ideal for depth-profiling heavy elements on lighter substrates.

The beam ions impinge on the sample and they are Elastically Back Scattered



Identification of material

The identification of the sample
Is done with the use of basic ideas:
Conservation of Energy and Momentum

High sample Z



Higher Energy at
the scattered beam



Rutherford Back Scattering

Quantification

$$N_{tar} = \frac{N_{det}}{\Omega \cdot N_{inc} \cdot \sigma}$$

Unknown

The detected ions (known)

Detector's solid angle (known)

Number of beam's particles (known)

Cross section (Analytical form unknown)

EXCEPT for

RUTHERFORD Cross Section

$$\frac{d\sigma}{d\Omega} = \frac{z \cdot Z \cdot e^2}{4\pi\epsilon_0} \left(\frac{1}{4E_0} \right)^2 \frac{1}{\sin^4 \frac{\theta}{2}}$$



Detection Apparatus

Beams used

- Protons from 0.5 to 3 MeV Probe larger depths
- Heavier ions (^{12}C , ^{16}O) 10 to 20 MeV Probe only surface layers
 - Higher mass resolution
 - Higher depth resolution

Most commonly used detectors are Surface Barrier Detectors (SSB)

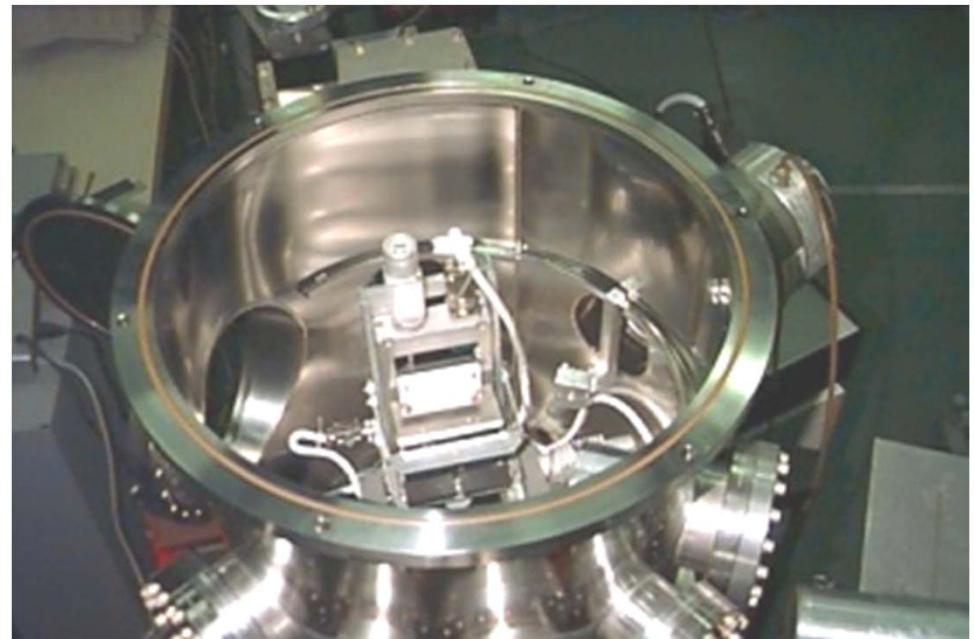
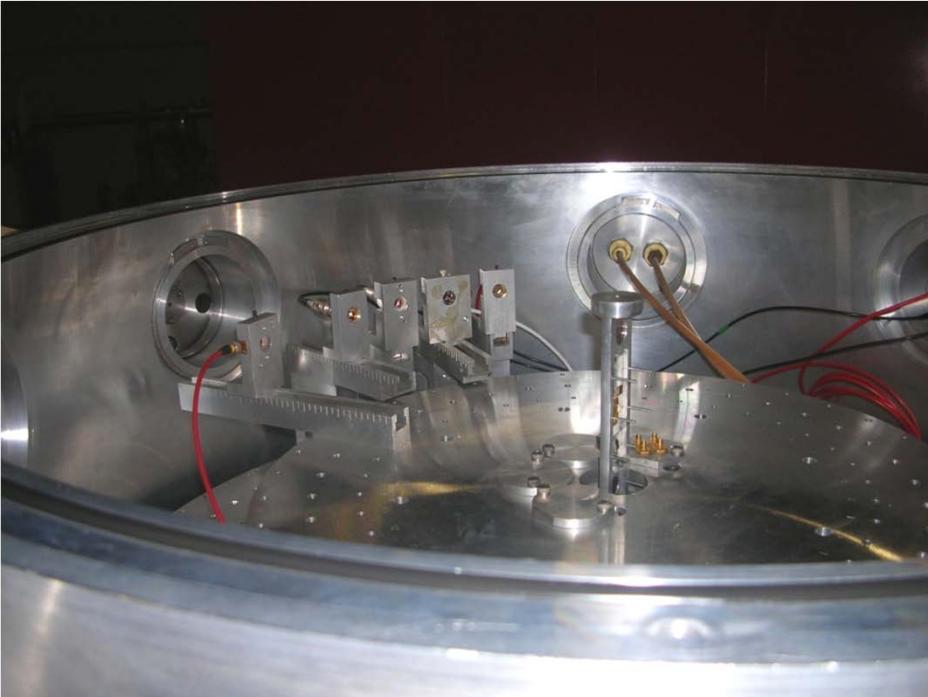
- Various thicknesses (μm) and apertures (mm^2)
- They work only under high vacuum
- Can detect the energy of the particle (resolution $\sim 15 \text{ keV}$)
- Can't detect the mass of the particle

Sample considerations

- Small size (few cm)
- Capable of being under vacuum (no wood e.t.c.)
- Preferable good electrical conductivity



Experimental Setup

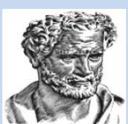


Motor driven goniometer

Great angular accuracy (0.01 deg.)
Up to 4 targets
Water cooling available

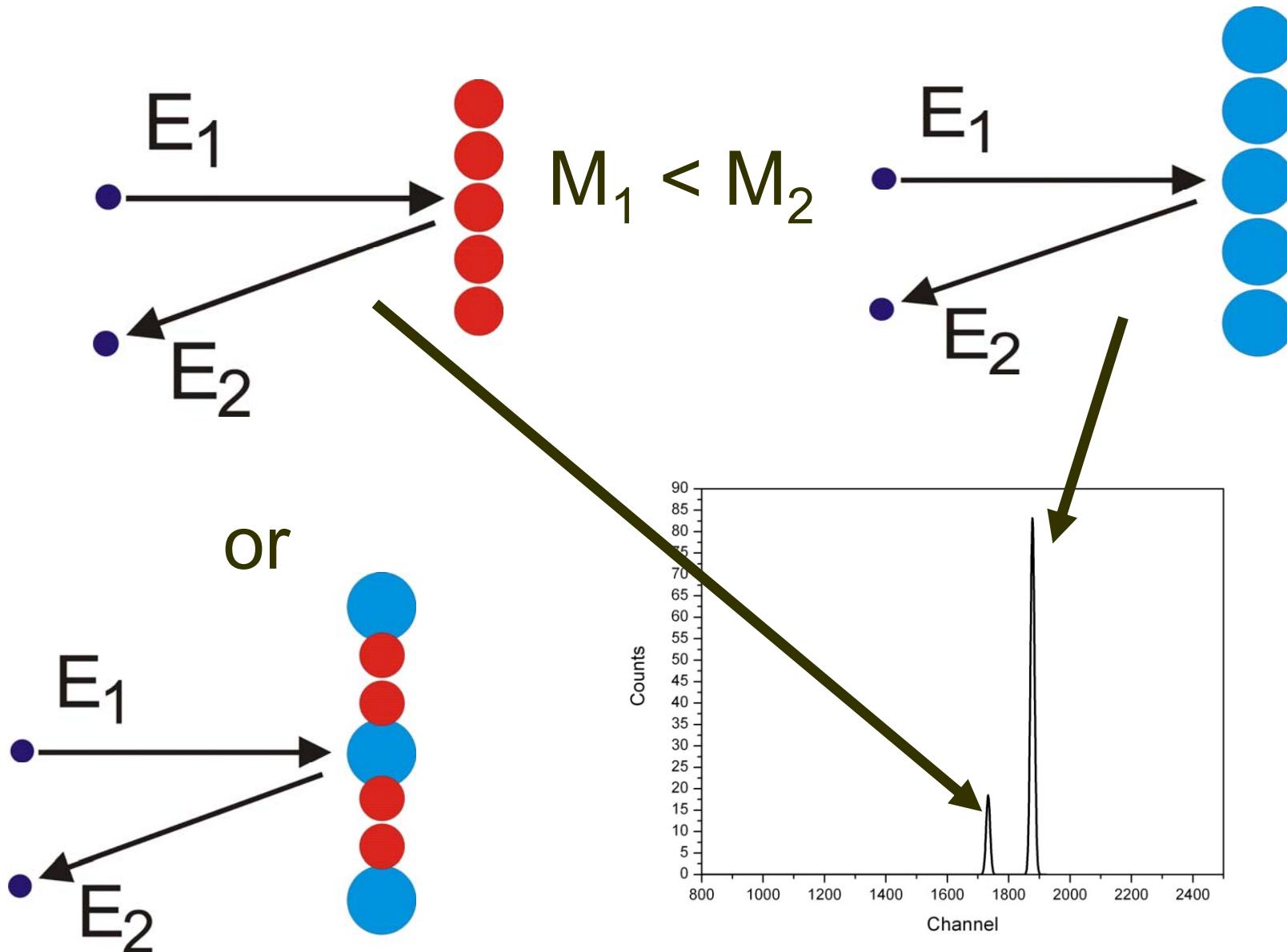
Motor driven goniometer

Suitable for channeling studies
4 – axis target movement
Place for PIGE detector



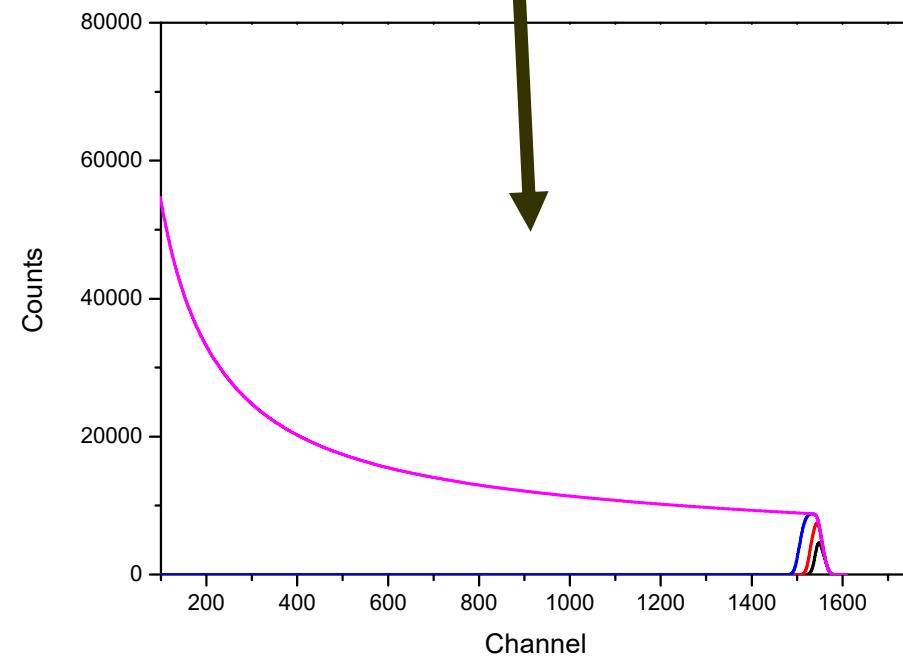
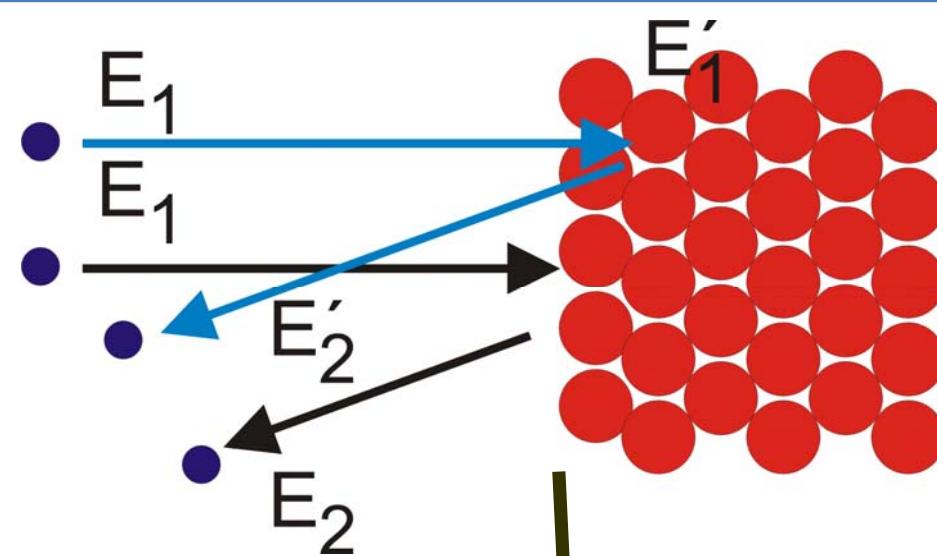
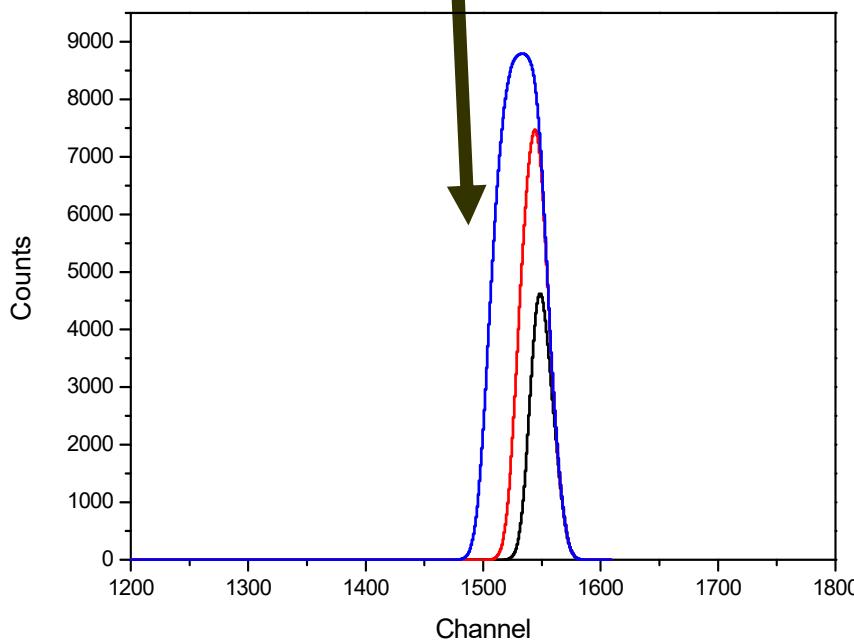
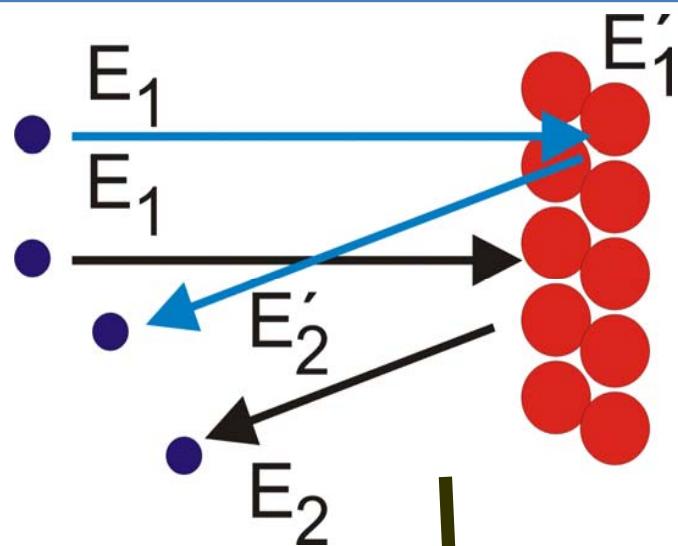
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Conceptual Examples



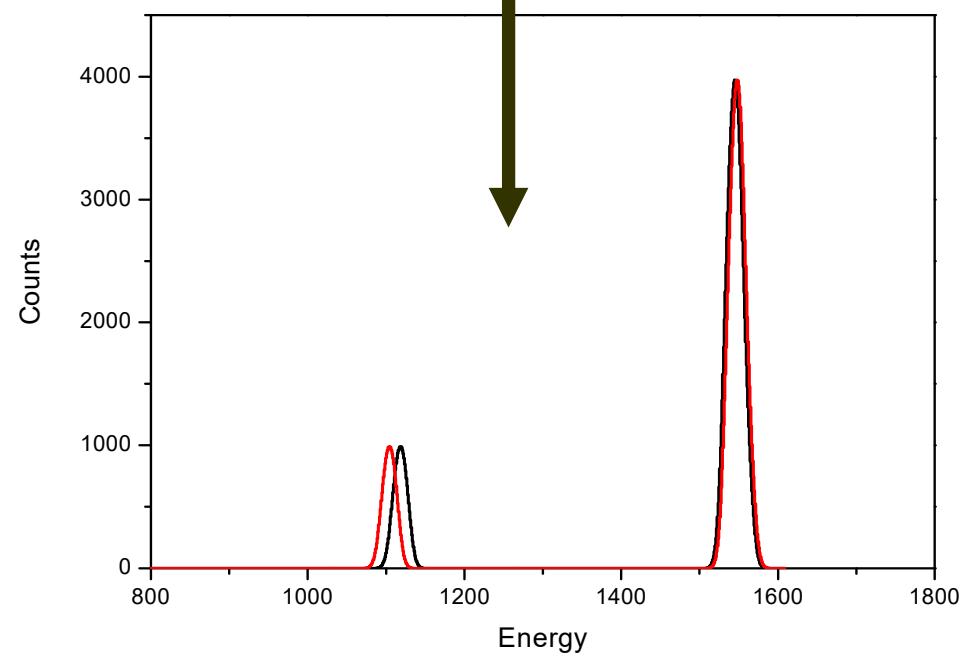
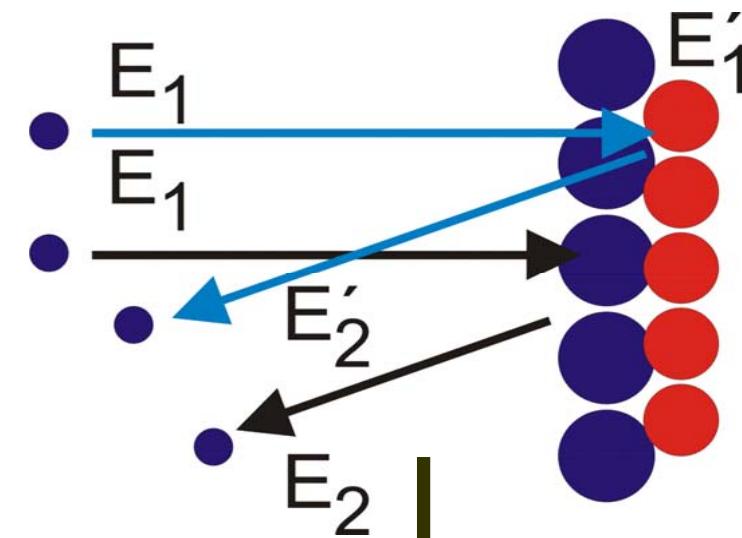
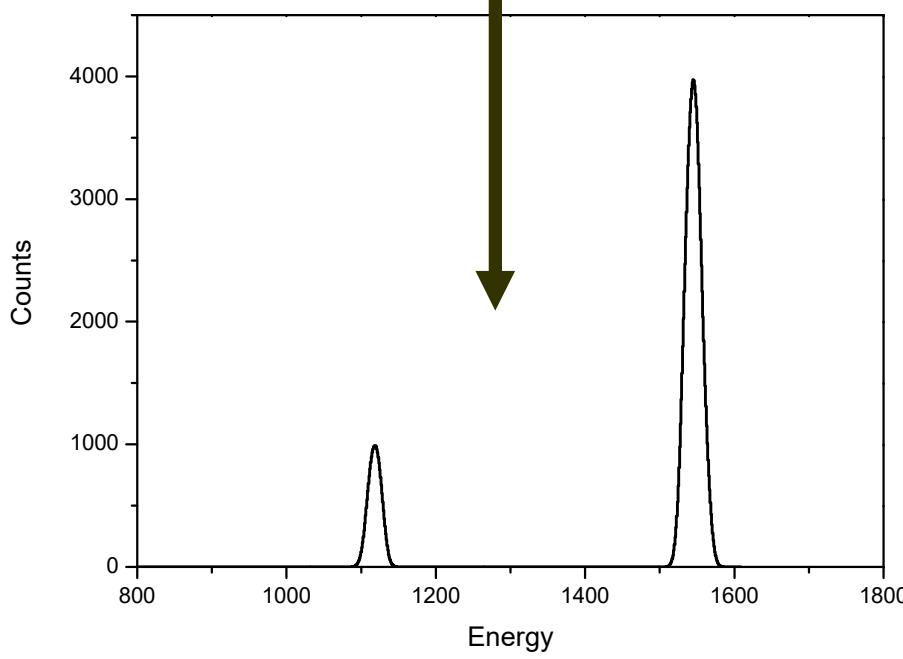
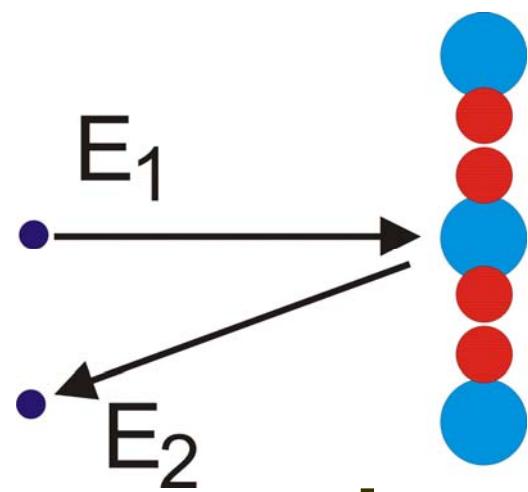
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Conceptual Examples



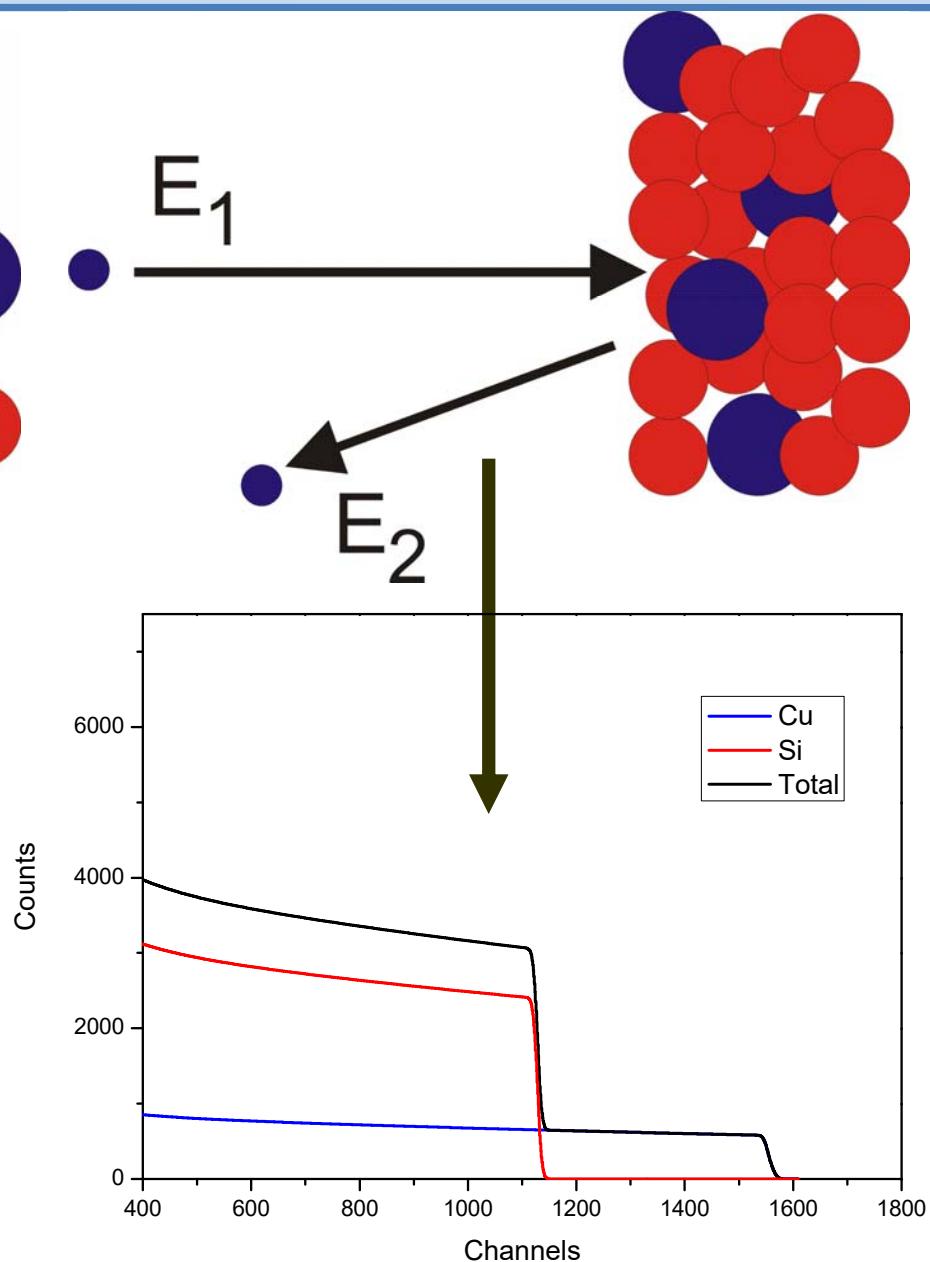
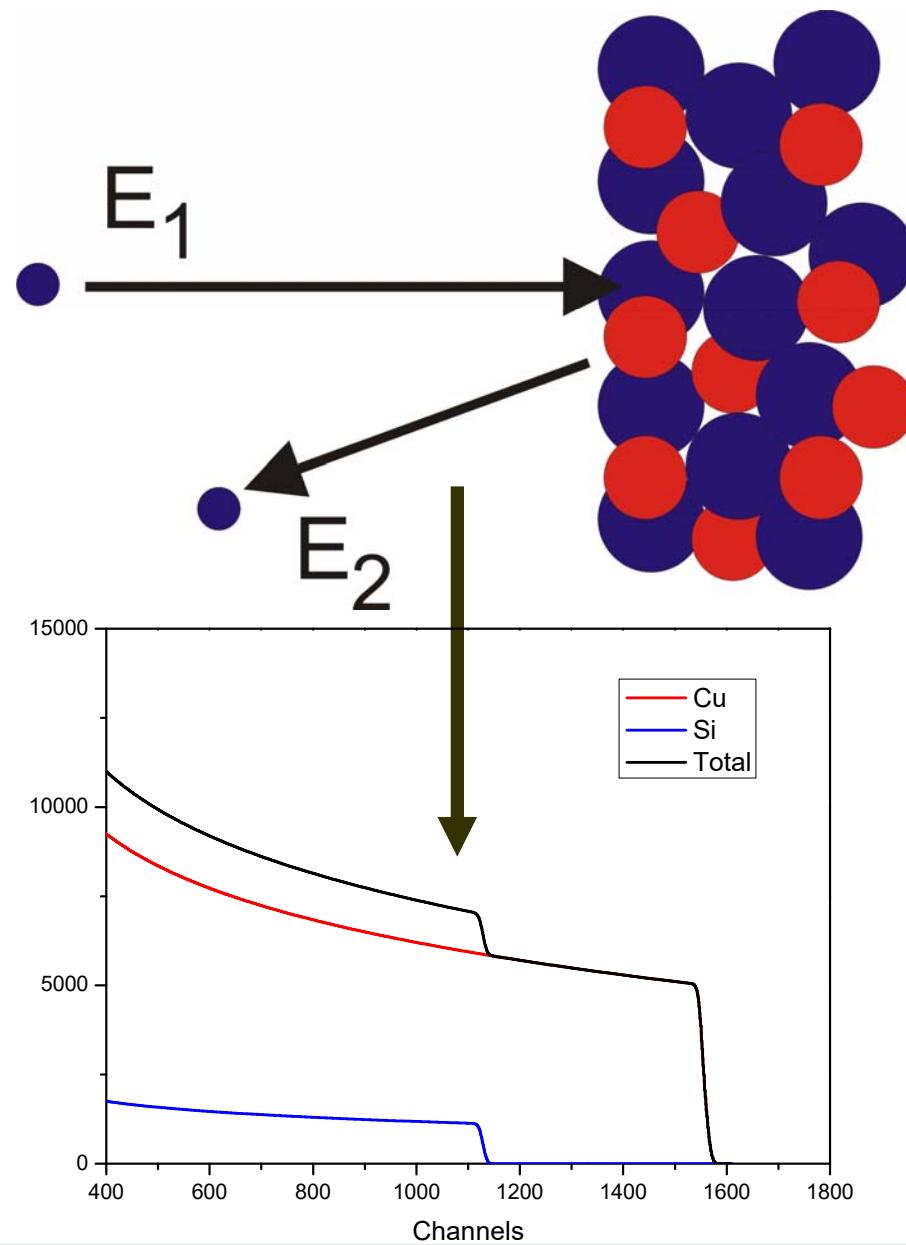
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Conceptual Examples



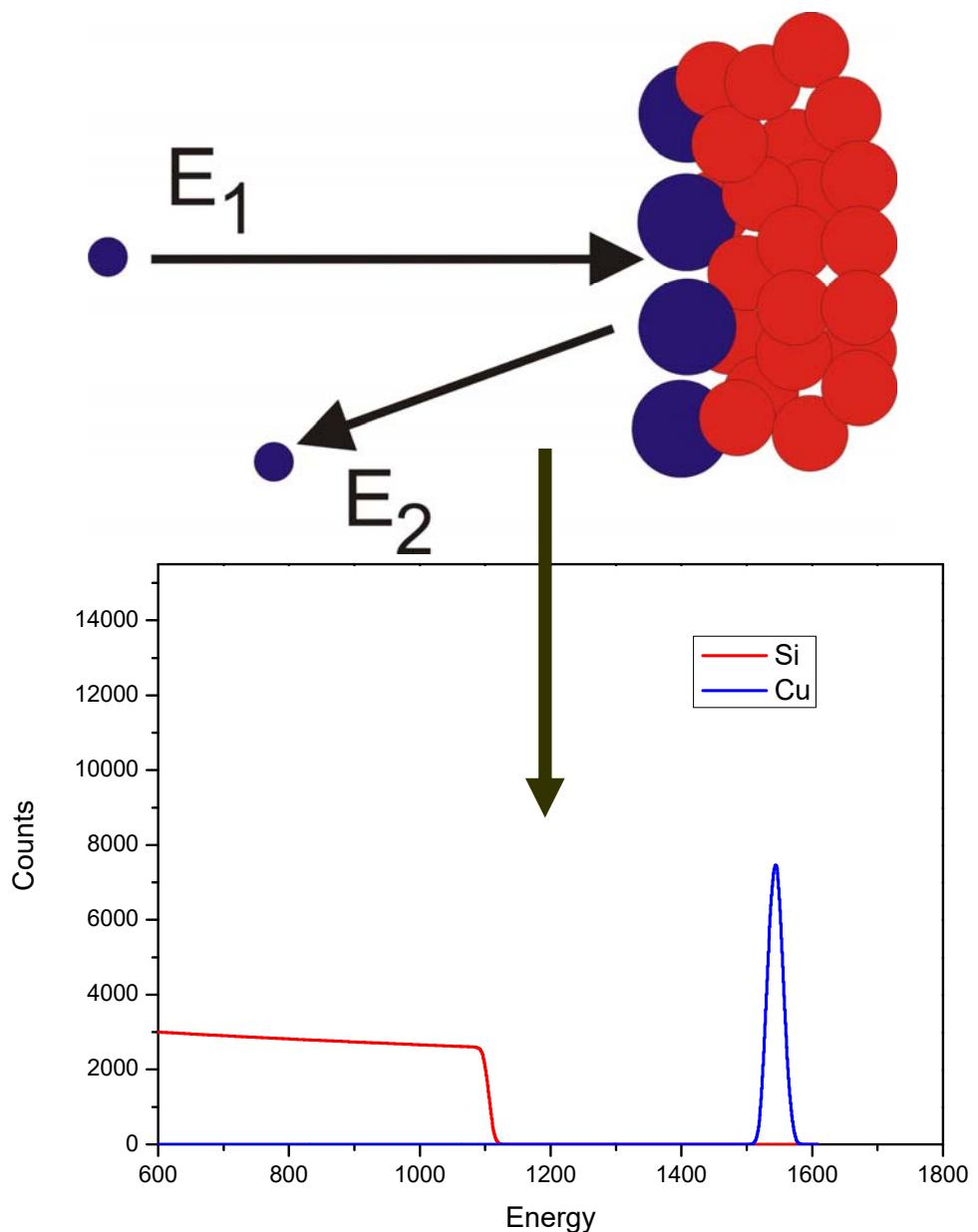
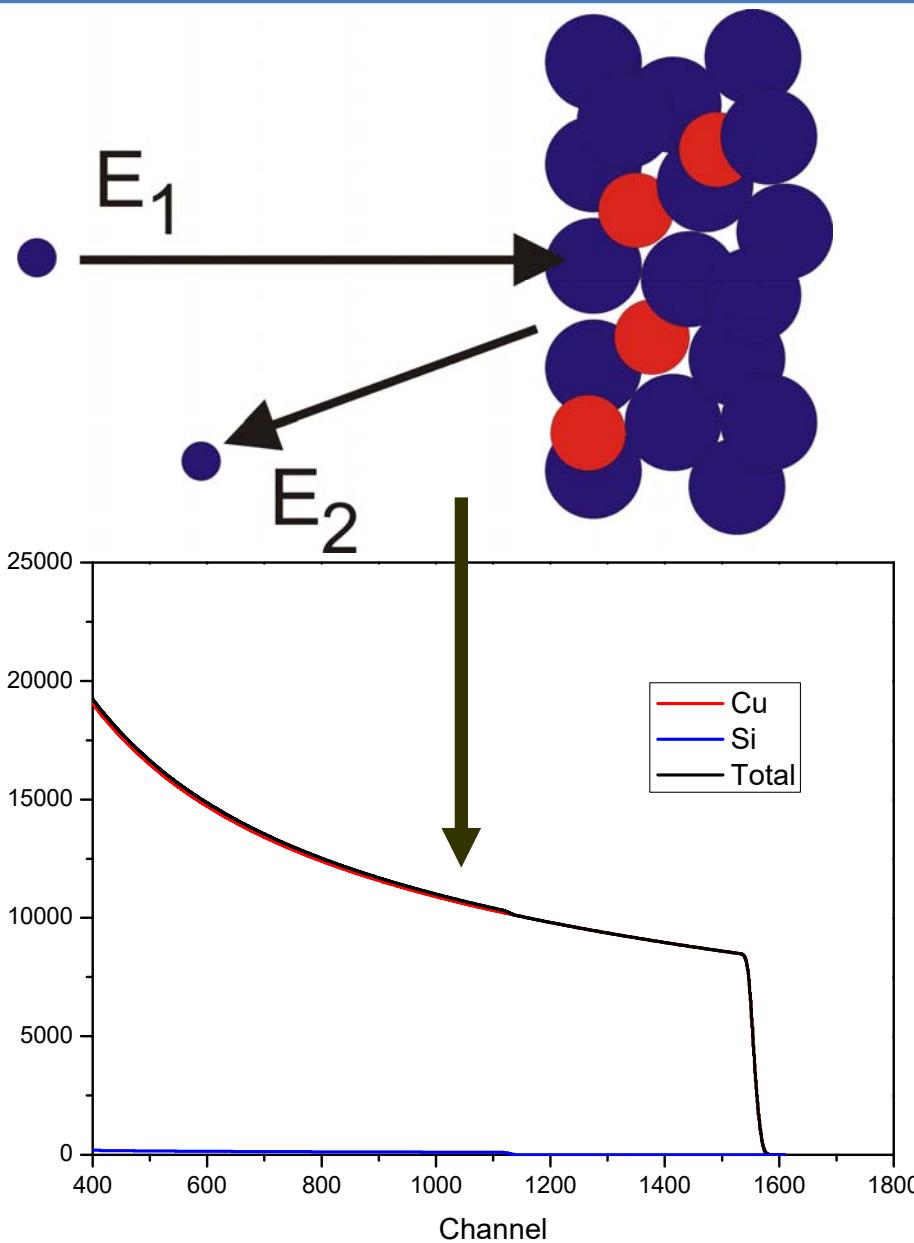
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Conceptual Examples



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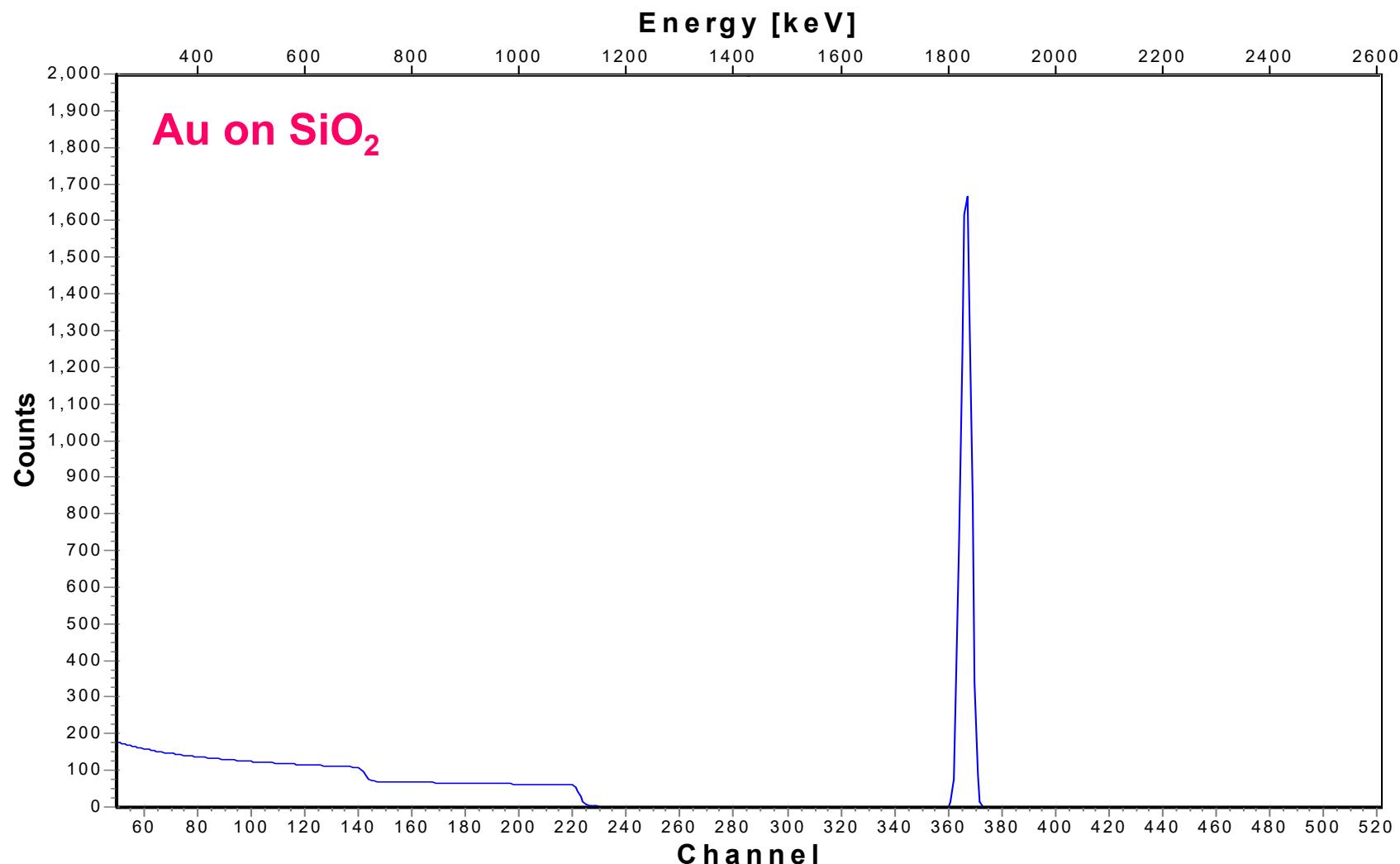
Conceptual Examples



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Conceptional Examples

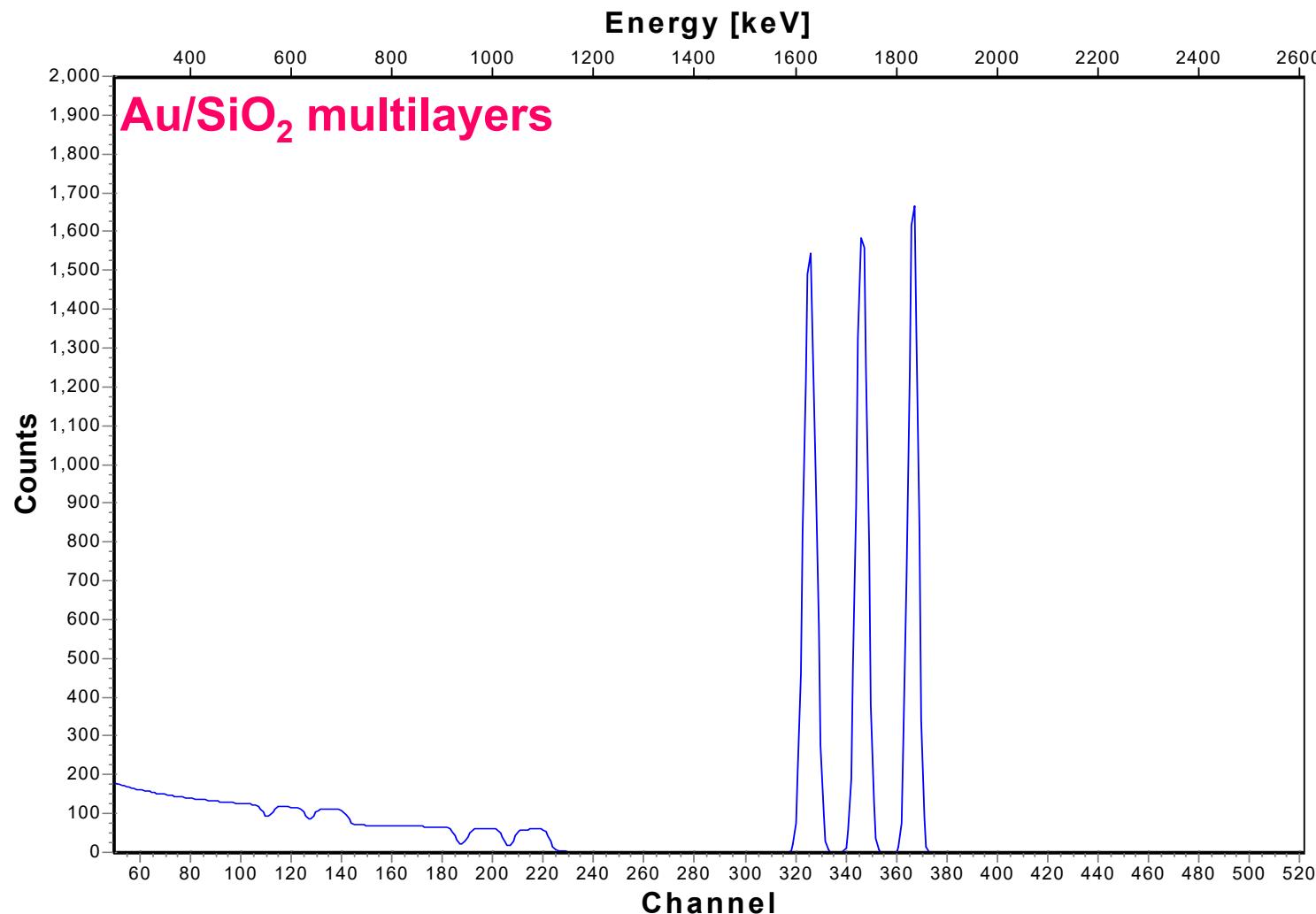
2 MeV ${}^4\text{He}$, $\theta=165^\circ$



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Conceptional Examples

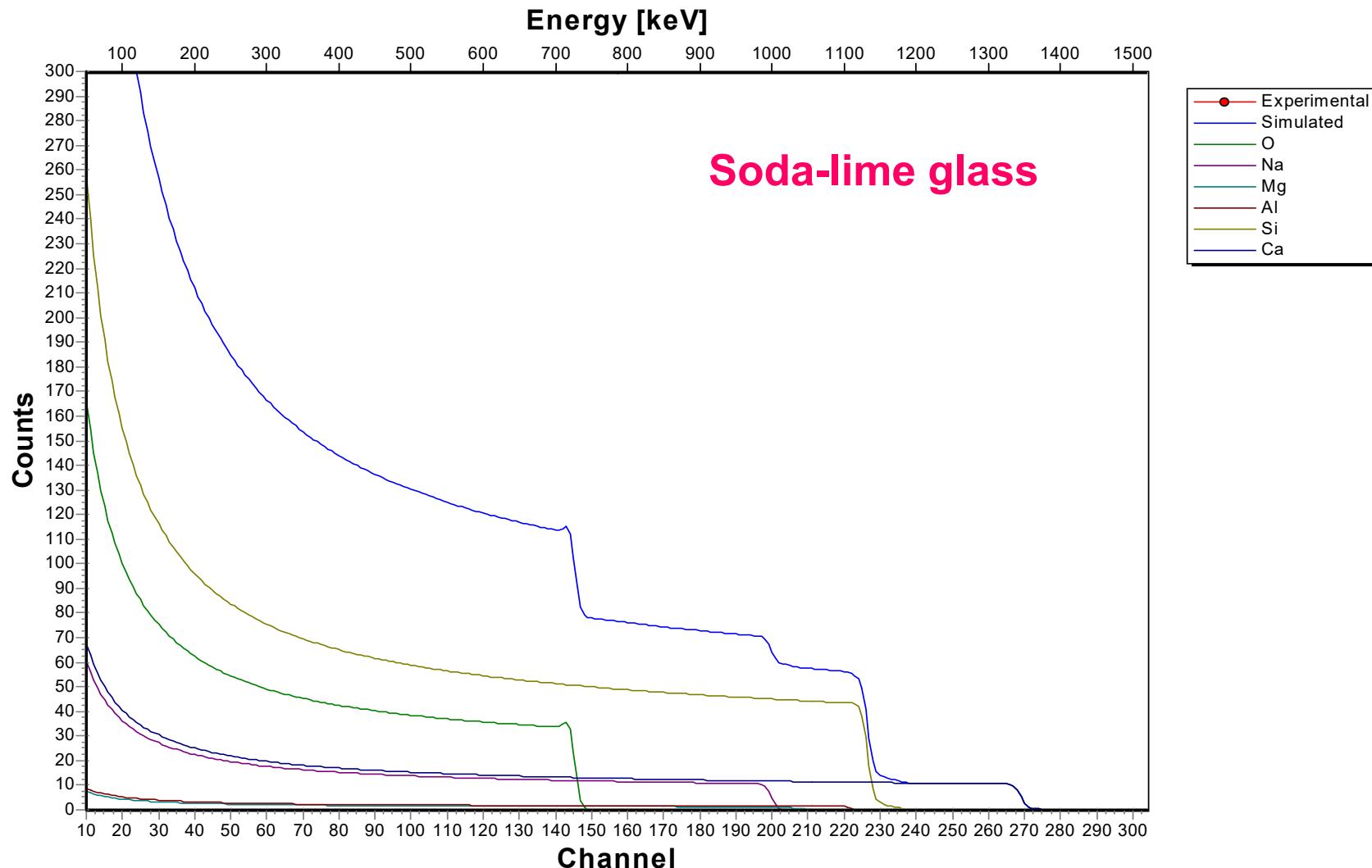
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Conceptional Examples

2 MeV ${}^4\text{He}$, $\theta=165^\circ$

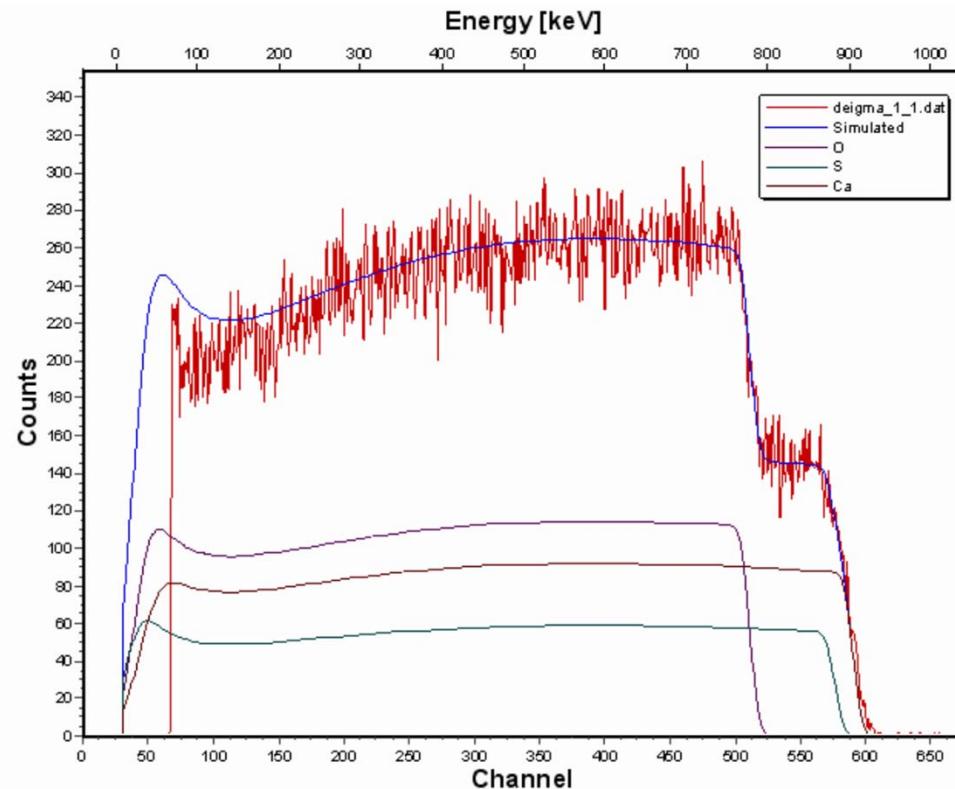


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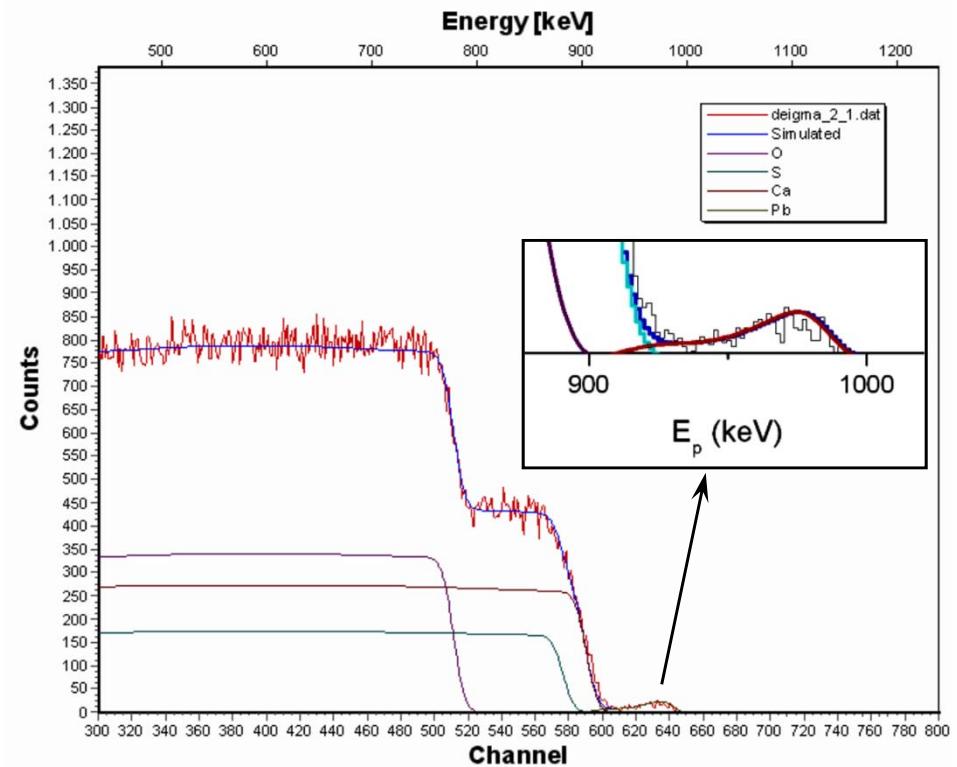
Examples

Sulphate mineral surfaces (e.g. gypsum) and dissolved heavy metal ions (e.g. Pb^{2+})

Pb (10 ppm) for 30 min



Pb (10 ppm) for 3 days



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Examples

Proton beam examination of glass – an analytical contribution for preventive conservation
M. Mader et al. NIMB 226 (2004) 110

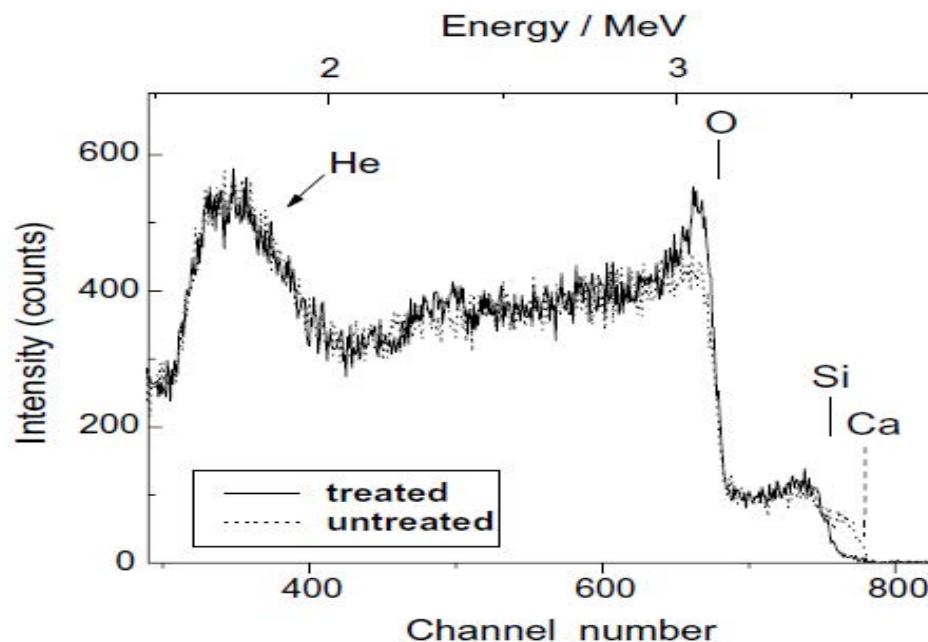


Fig. 2. RBS spectra of glass M3 (60 wt% SiO_2 , 15 wt% K_2O , 25 wt% CaO) before and after chemical treatment in 0.1 mol/l HCl solution. After 20 hours leaching the thickness of the surface layer is 3.4 μm .

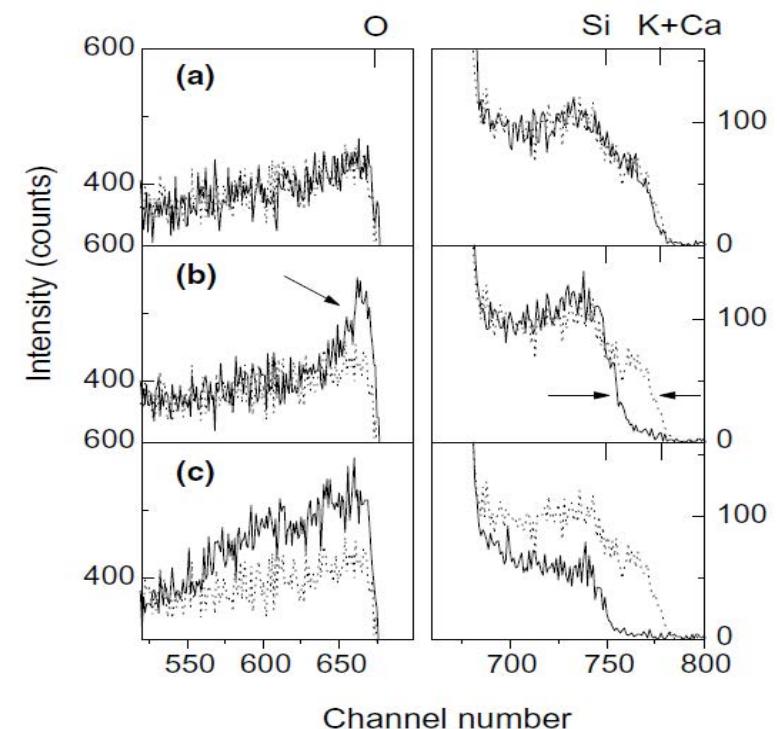
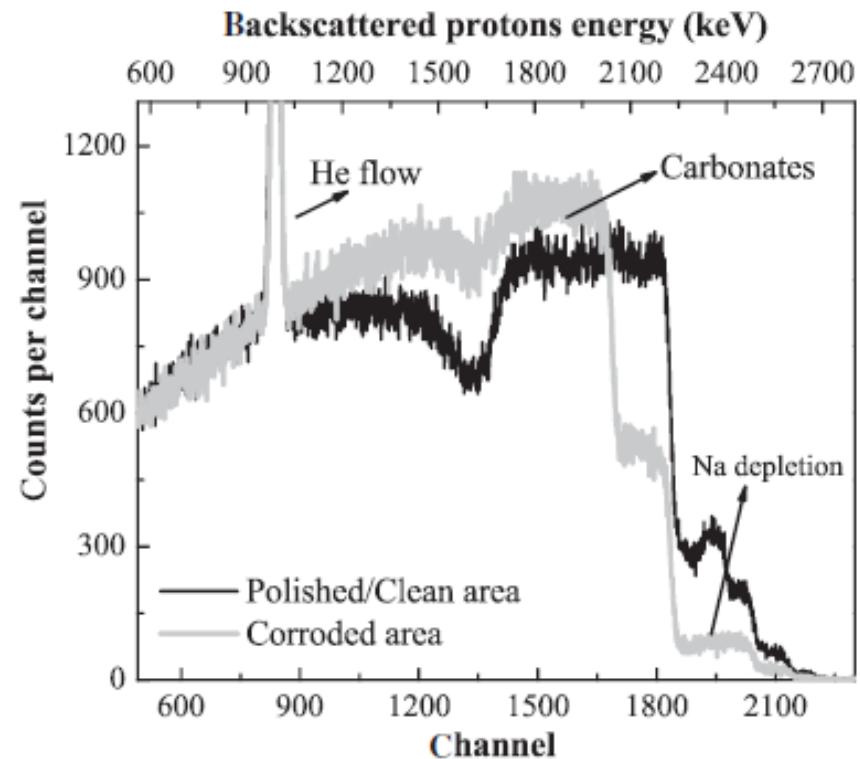
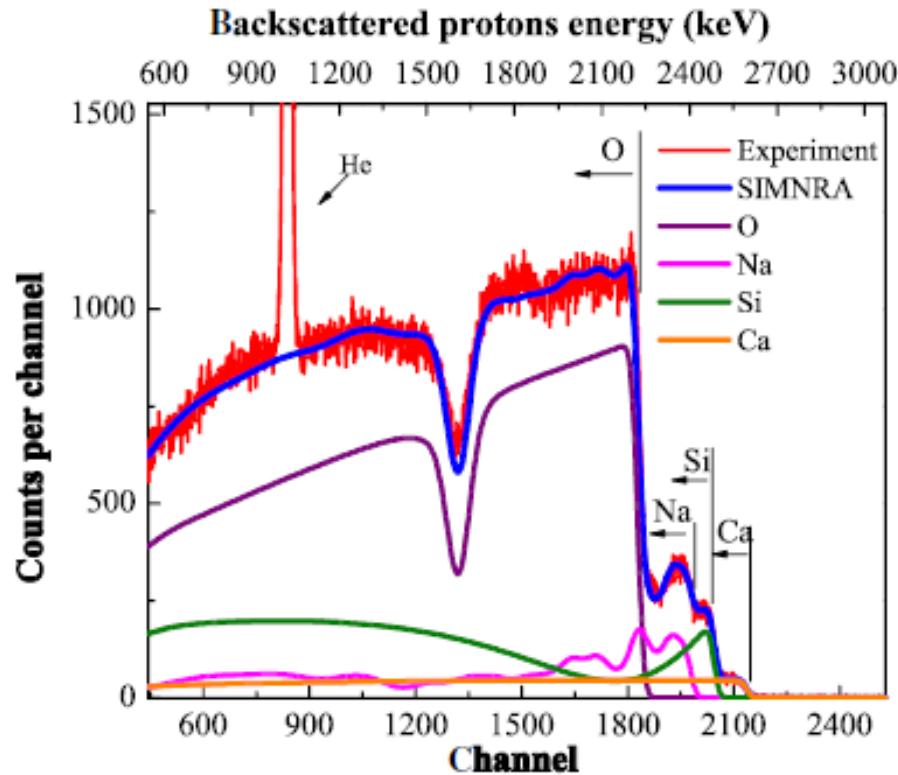


Fig. 3. RBS spectra (regions of interest) obtained from glass M3 (see Table 1) before (dotted lines) and after (full lines) treatment in diluted HCl: leaching times (a) 90 min, (b) 20 h, (c) 195 h cause surface layer thickness of (a) 0.45 μm , (b) 3.4 μm , (c) 10.4 μm , respectively. For example (b), the arrows indicate an enhanced concentration of oxygen as well as a shift of the high energy K+Ca edge (see text).



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External Beam Examples



NIST 620 quantitative results obtained through PIXE and RBS analysis.

Symbol	PIXE	RBS	Certified
O		45.3 ± 4.5	47.0 (oxides)
Na	10.6 ± 1.1	7.2 ± 0.7	10.70 ± 0.03
Si	36.5 ± 1.7	39.1 ± 4.0	33.80 ± 0.04
Ca	5.05 ± 0.25	6.4 ± 0.6	5.08 ± 0.04



External Beam Examples

Review of accelerator gadgets for art and archaeology
T. Calligaro et al. NIMB 226 (2004) 29

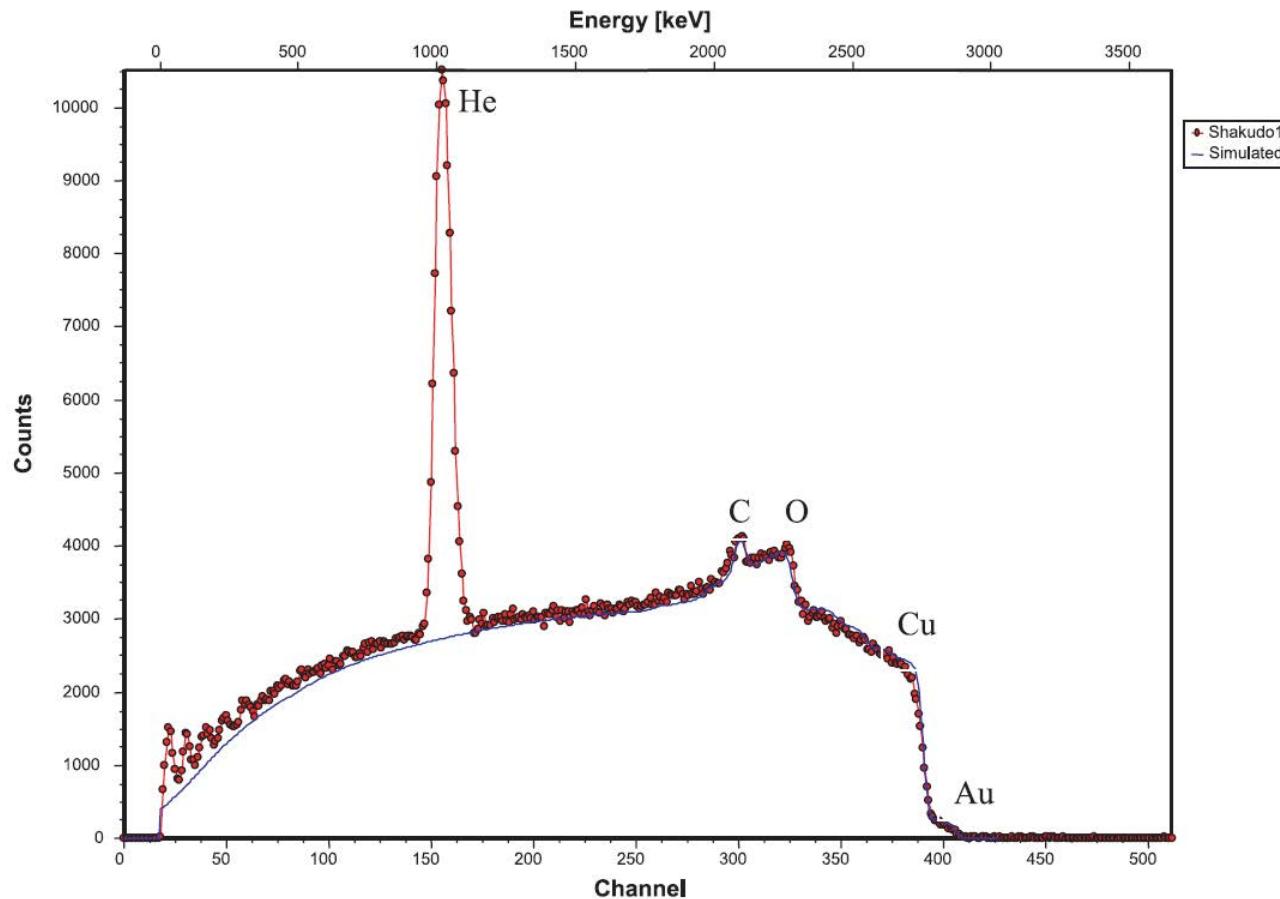


Fig. 4. RBS spectrum of the hilt of a historical Japanese sword. The hilt is made of a traditional Au/Cu alloy named *Shakudo* covered with a patina composed mainly of copper oxide and a varnish layer. The spectrum is obtained with an external 3-MeV proton beam in a helium atmosphere, producing an intense He peak which can be used for dose monitoring and energy calibration.



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External Beam Examples

Review of accelerator gadgets for art and archaeology

T. Calli

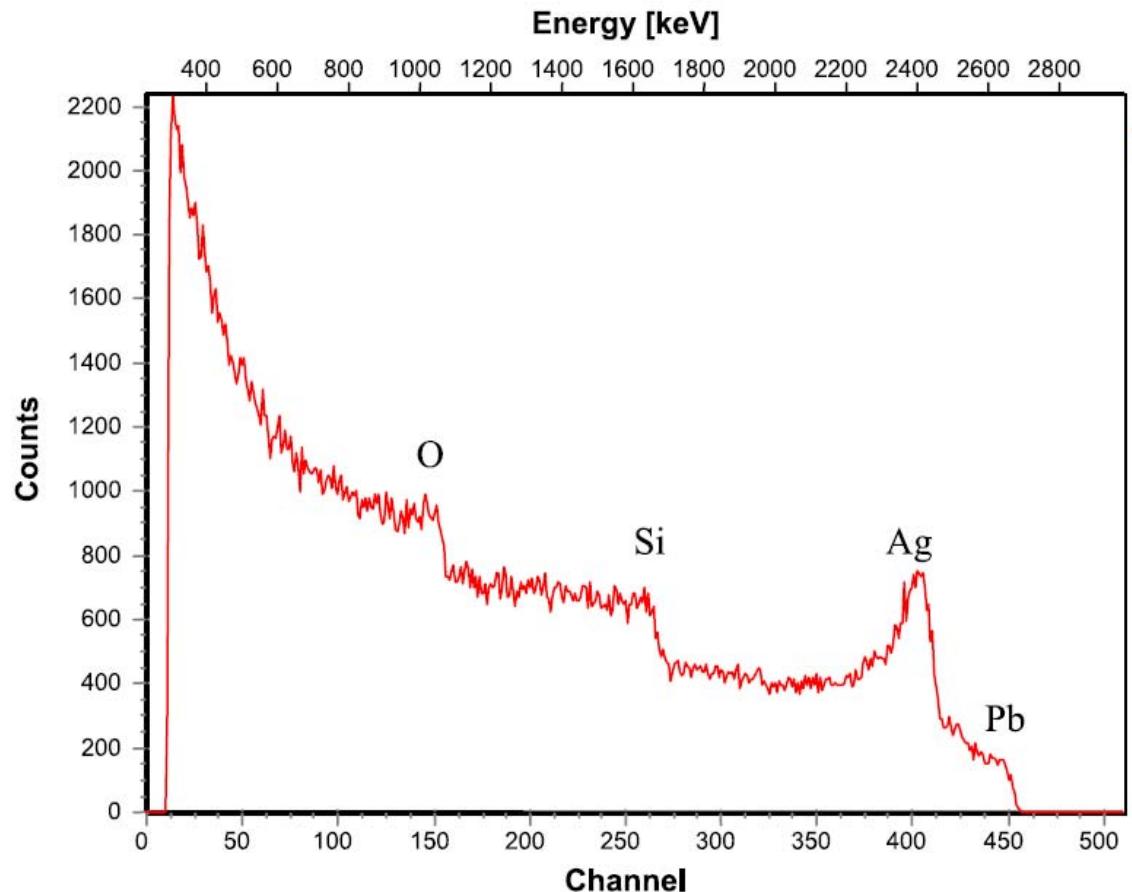


Fig. 7. RBS spectrum of a fragment of a lustred Islamic ceramic obtained with 3-MeV alpha external beam. The lustre is a lead-rich glassy layer containing silver aggregates.



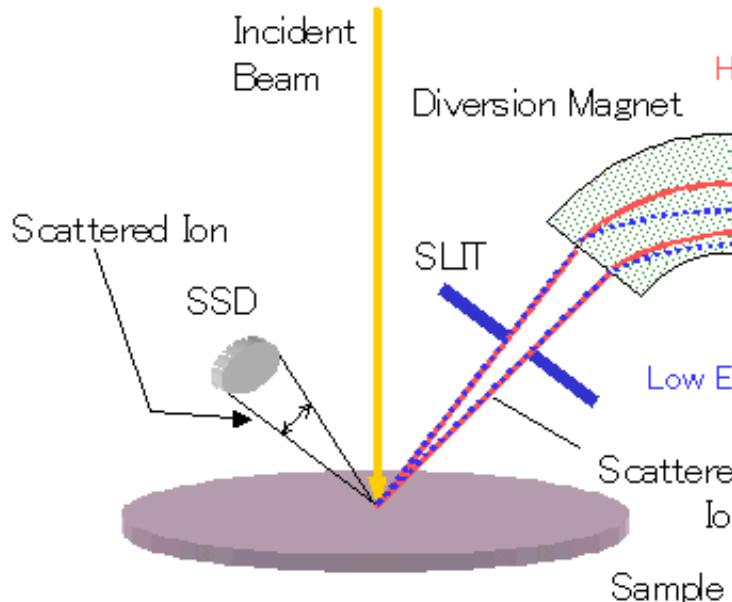
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Developments in RBS

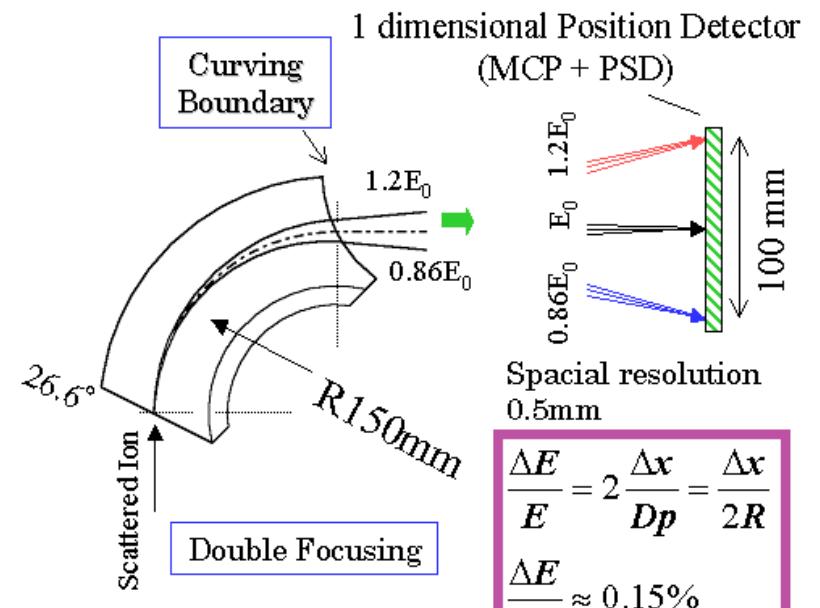
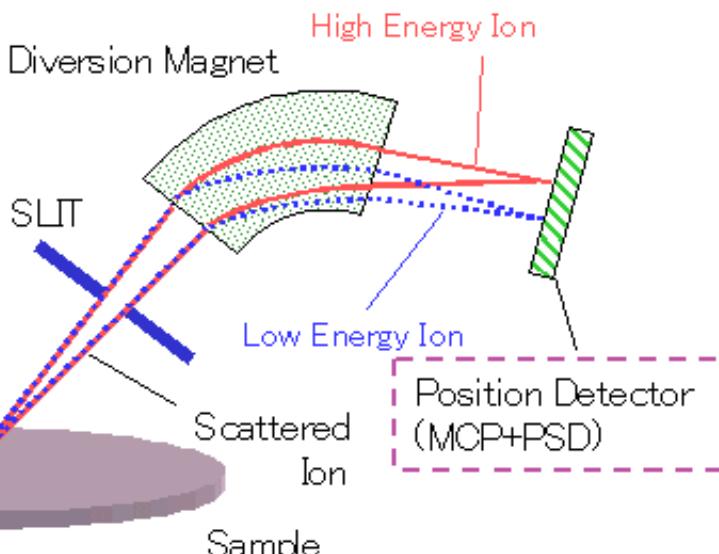
High Resolution RBS

The resolution is enhanced with the aid of a magnetic field

Conventional RBS



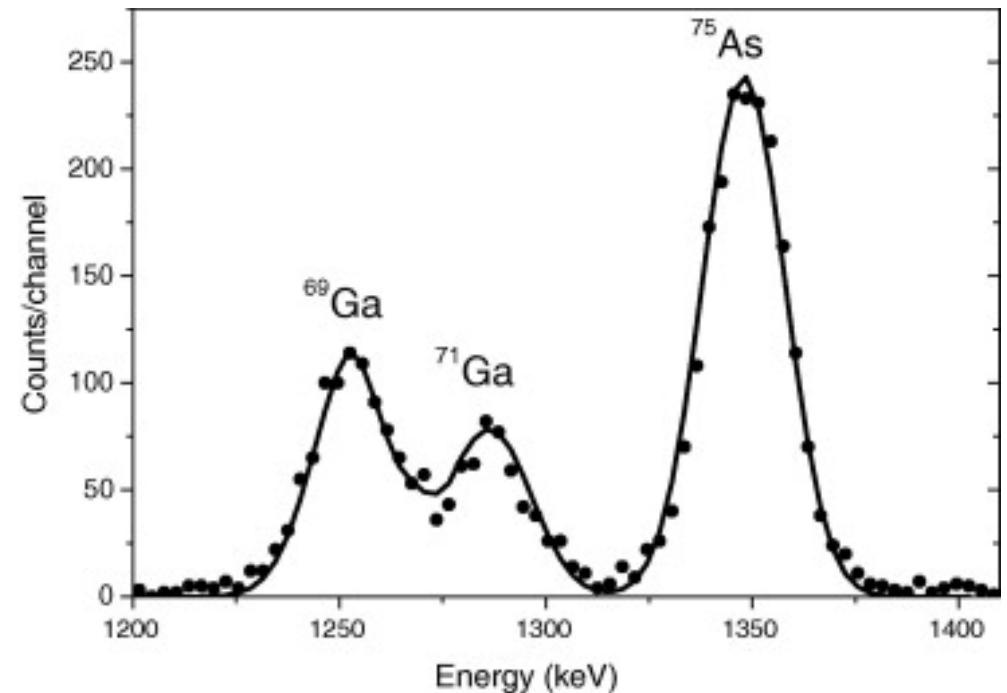
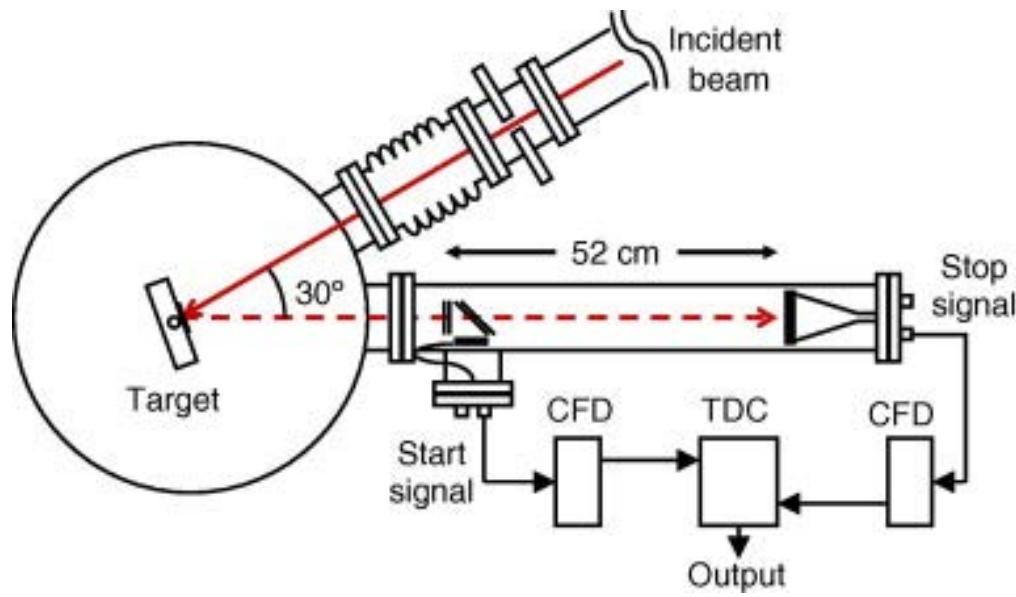
HRBS



Developments in RBS

Time of Flight HI - RBS

Mass resolution is enhanced due to the TOF technique

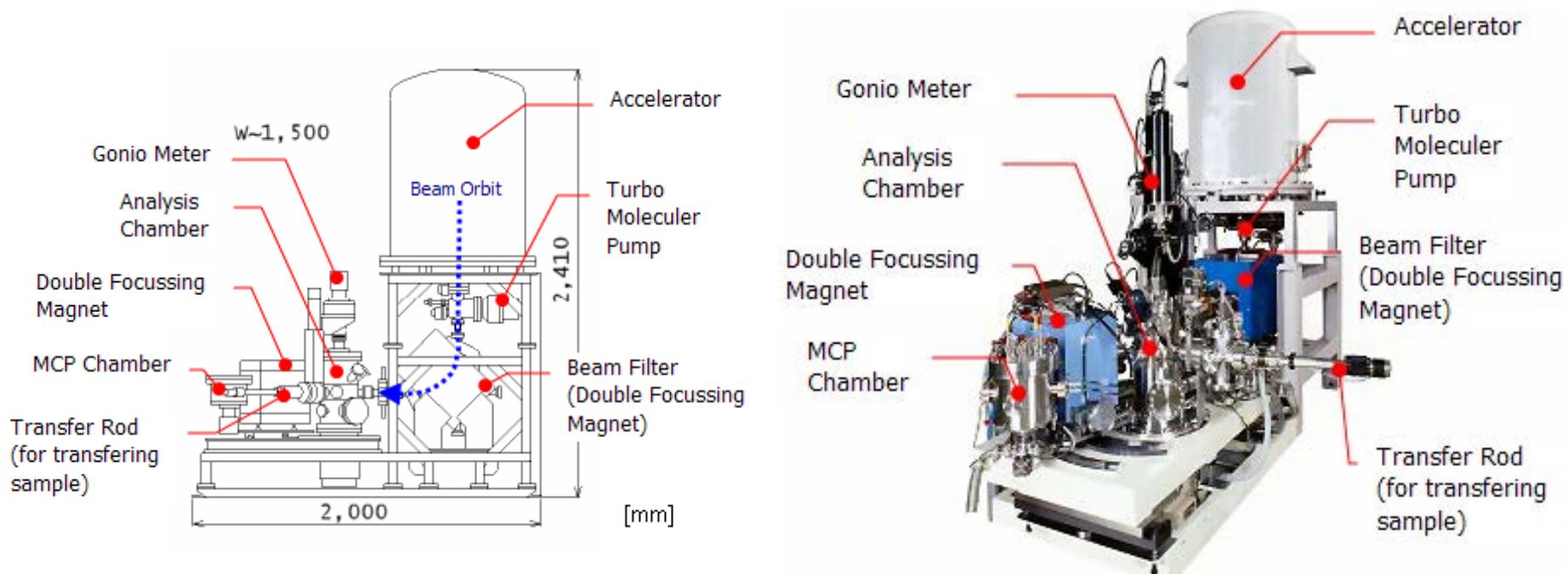


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Developments in RBS

“Benchtop” RBS

300 - 500 keV proton beam

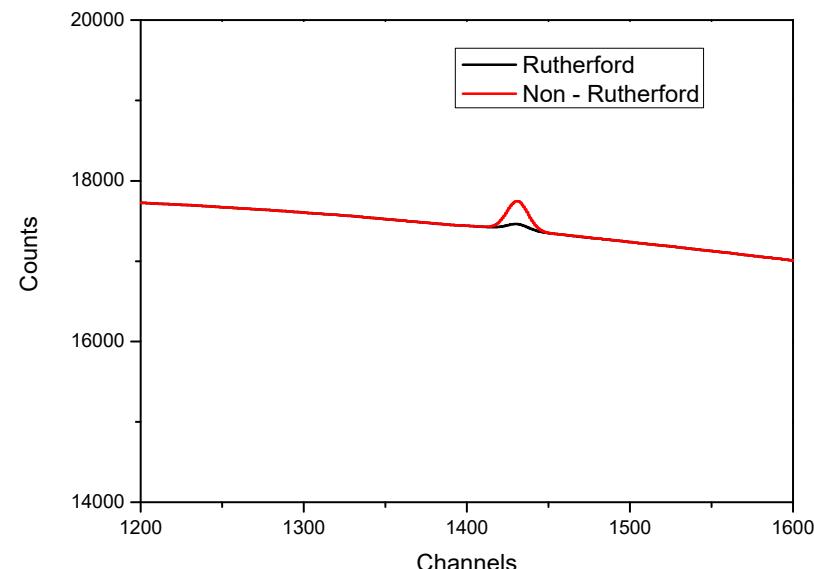
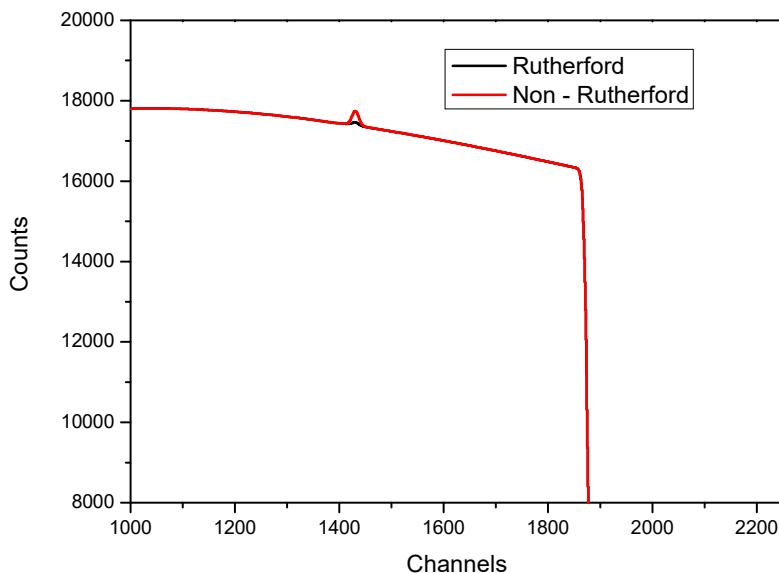
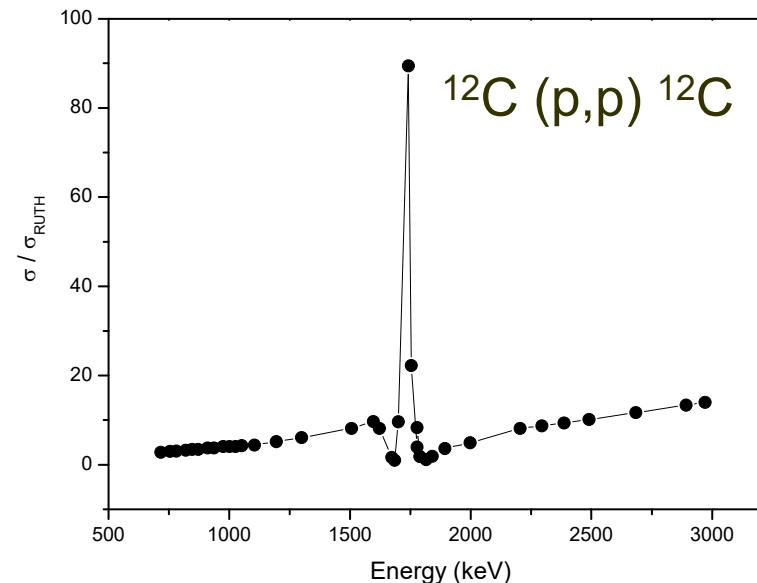


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Elastic Back Scattering

Same as RBS with the difference
That the cross section is not obeying
Rutherford's law

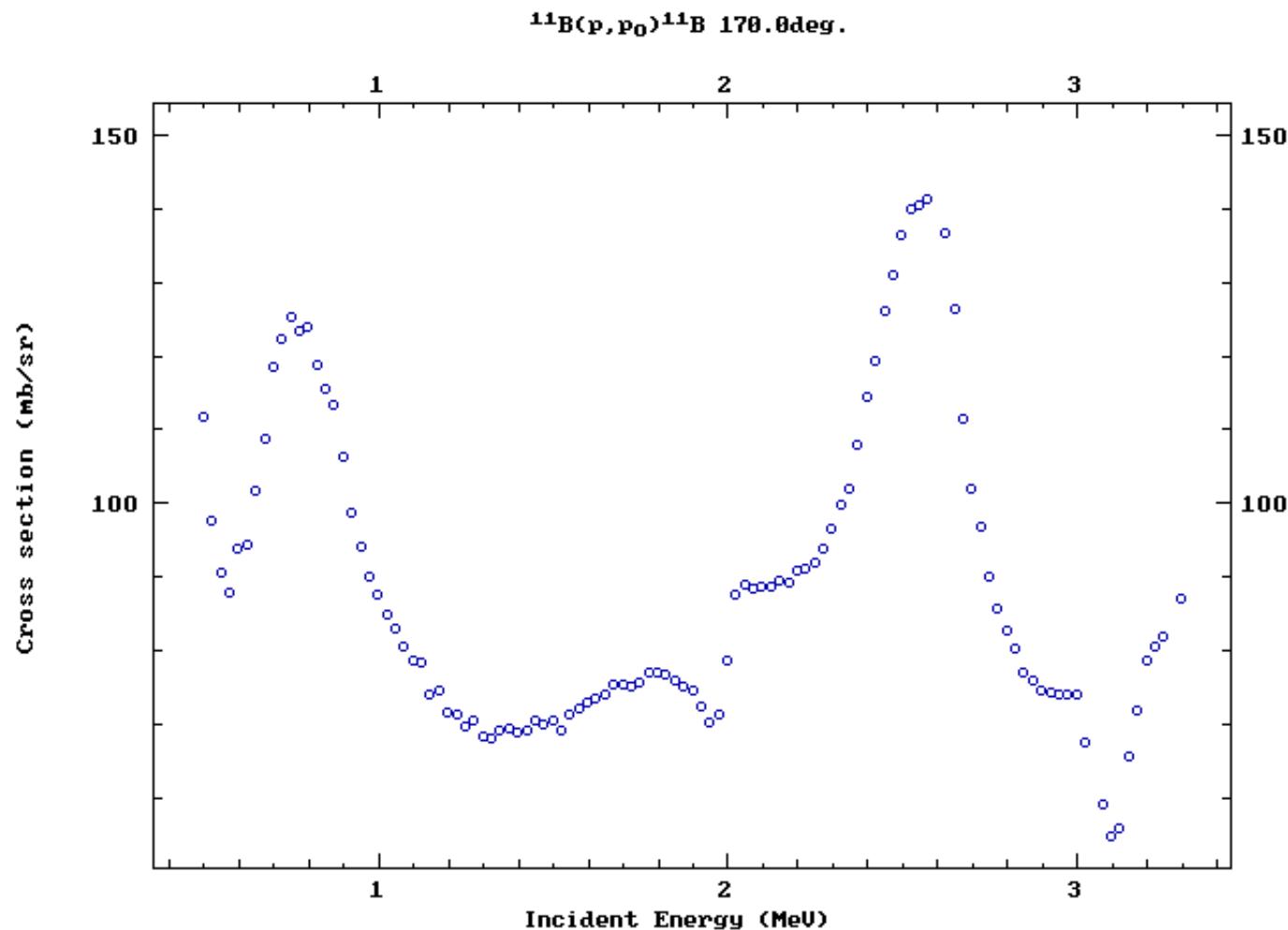
Applicable only for **specific**
combinations of beam and target
nuclei and at very **specific energies**



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Elastic Back Scattering

Applicable only for **specific combinations** of beam and target nuclei and at very **specific energies**



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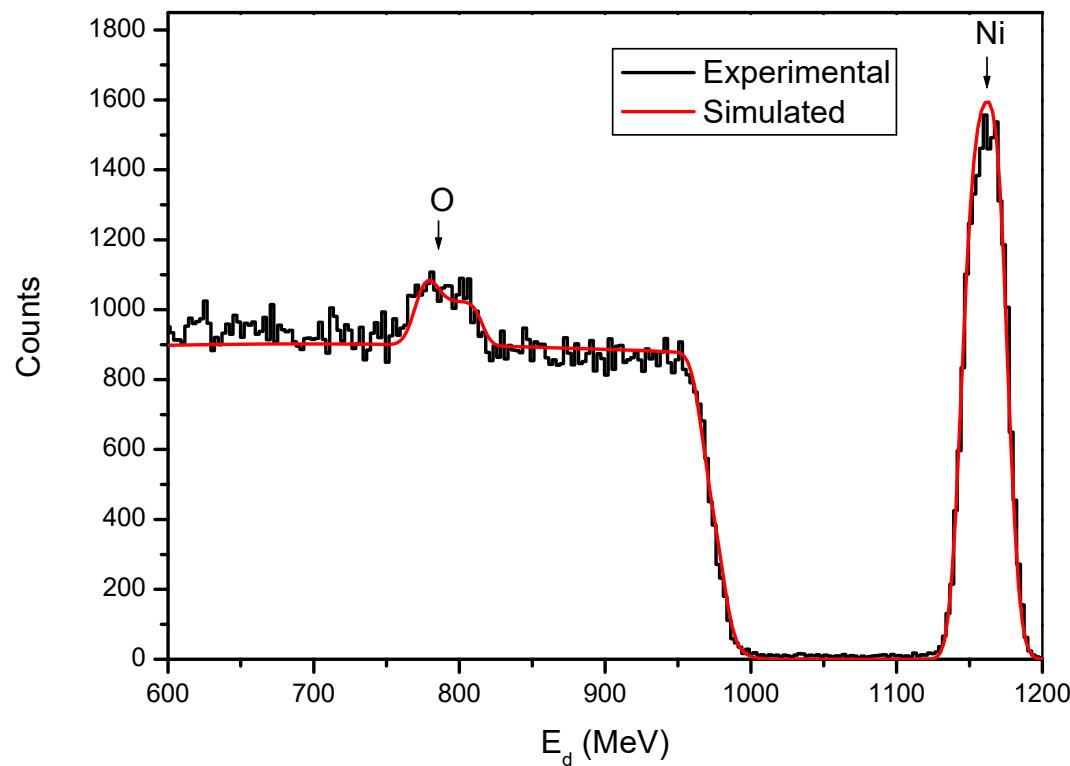
Examples

Metal oxide thin film technology

SIMNRA simulation of a NRA spectrum of a NiO thin film

Deuteron beam of 1.35 MeV

Detection angle of 170°



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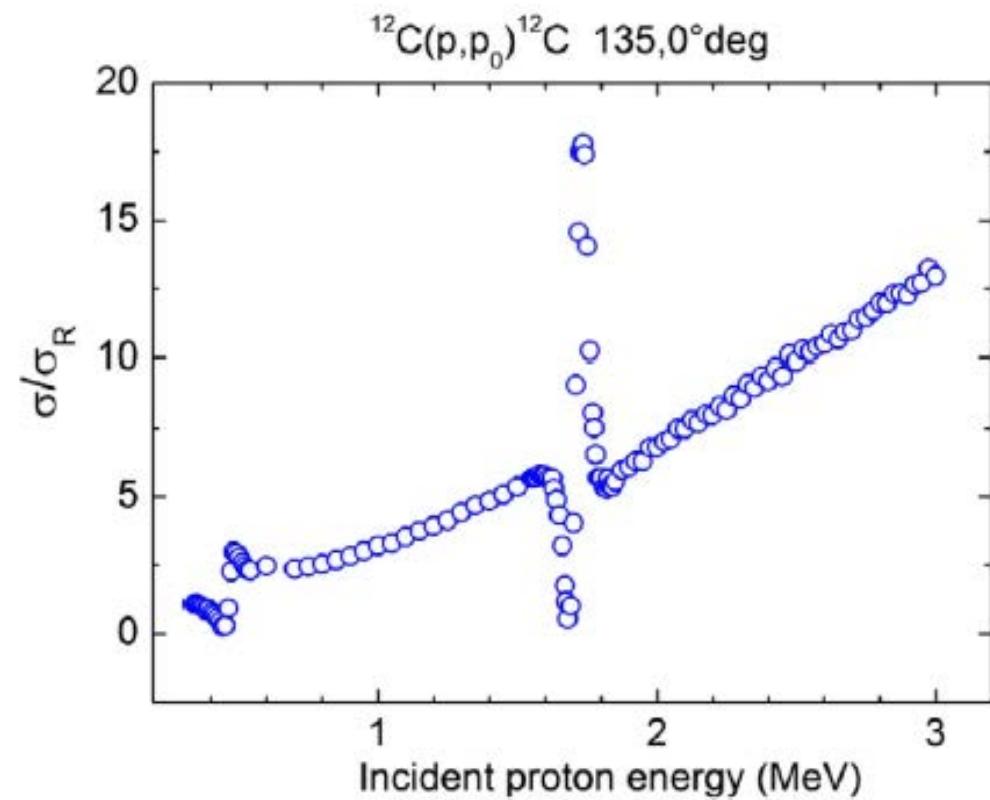
Examples

External-RBS, PIXE and NRA analysis for ancient swords

H. C. Santos *et al.* NIMB B 345 (2015) 42



Differences in manufacturing between
a Damascus and a Japanese blade



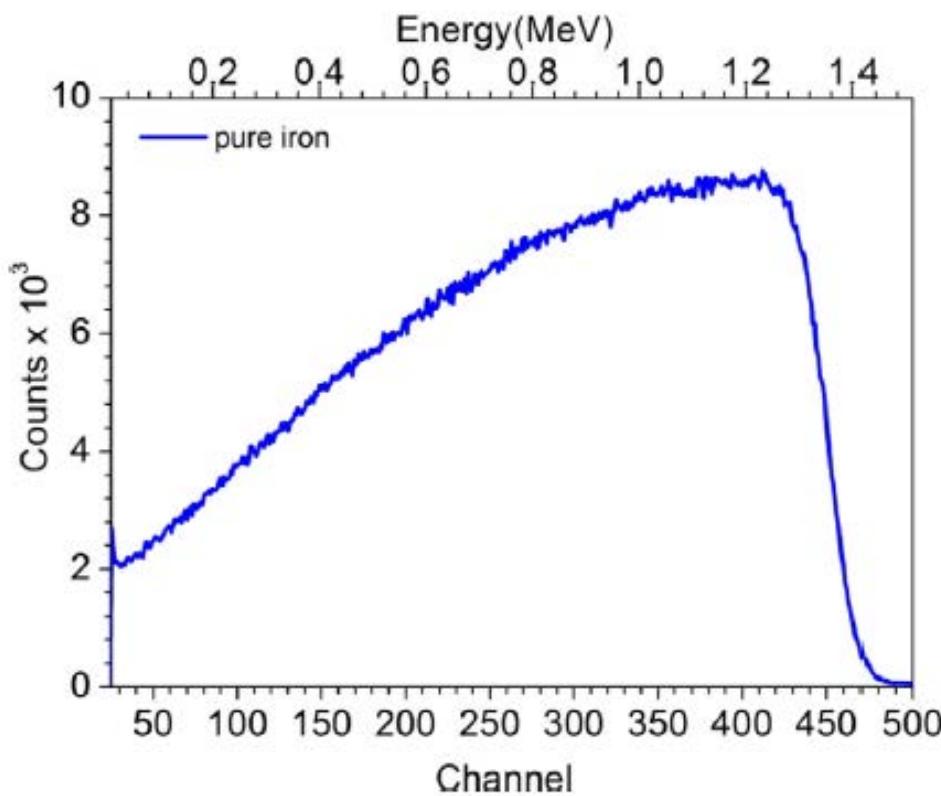
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Examples

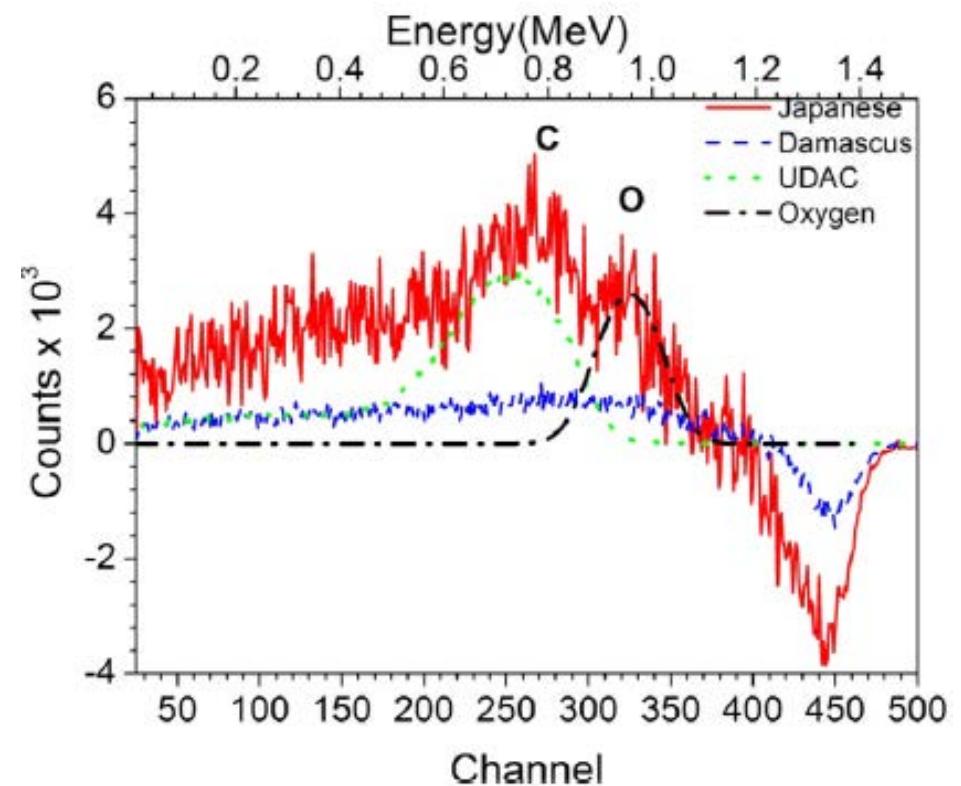
External-RBS, PIXE and NRA analysis for ancient swords

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Typical RBS spectrum



Spectrum after subtraction



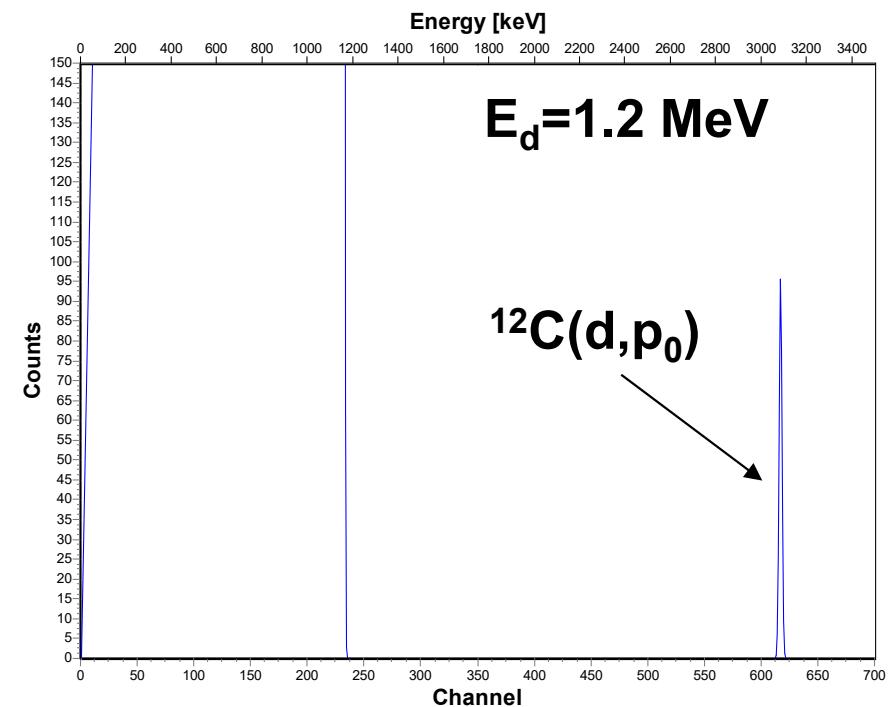
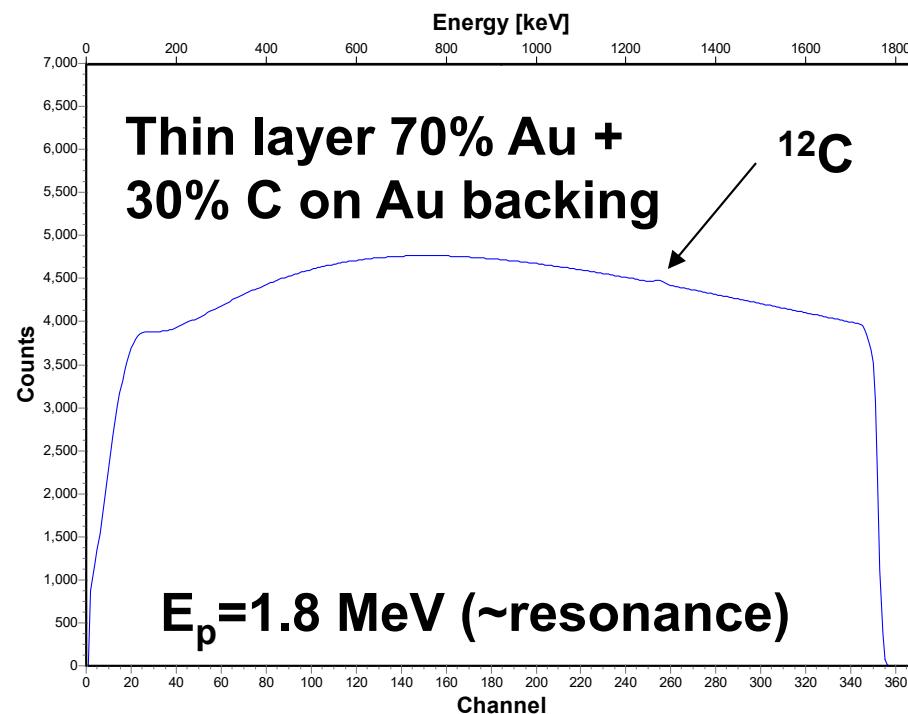
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Nuclear Reaction Analysis

Use of nuclear reactions, (d,p) , (d,α) , (p,α) , (α,p) etc.

Usually with high enough Q-values

e.g. The ‘carbon problem’: RBS is weak, EBS can be applied only in certain cases (no other light elements present, no high-Z matrix, very case-specific measurements):



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Examples

Analysis of Mexican obsidians by IBA techniques
G. Murillo et al. NIMB B 136-I 38 (1998) 888

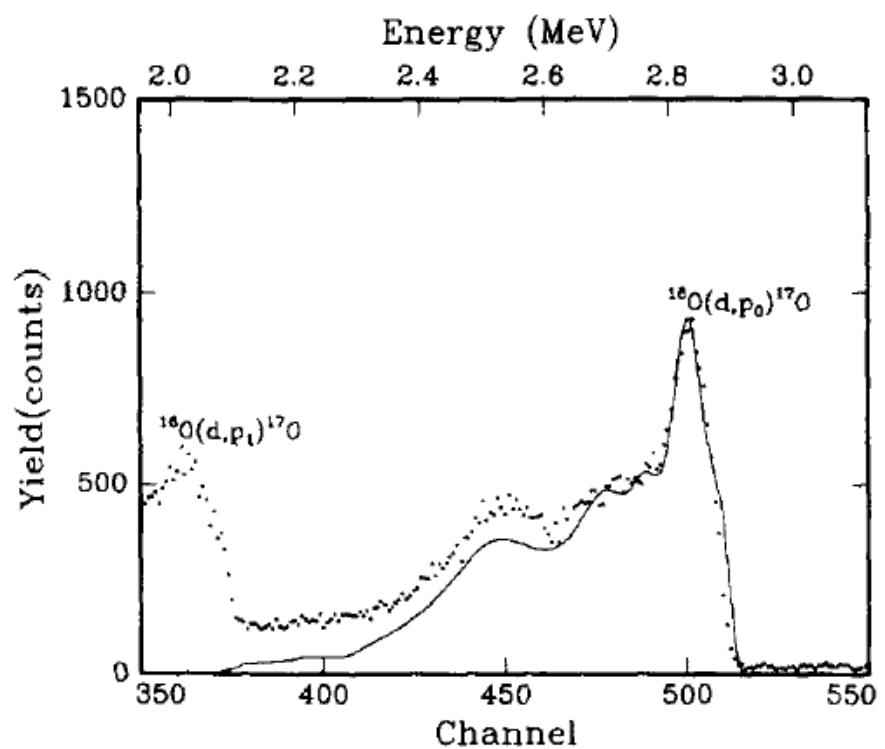
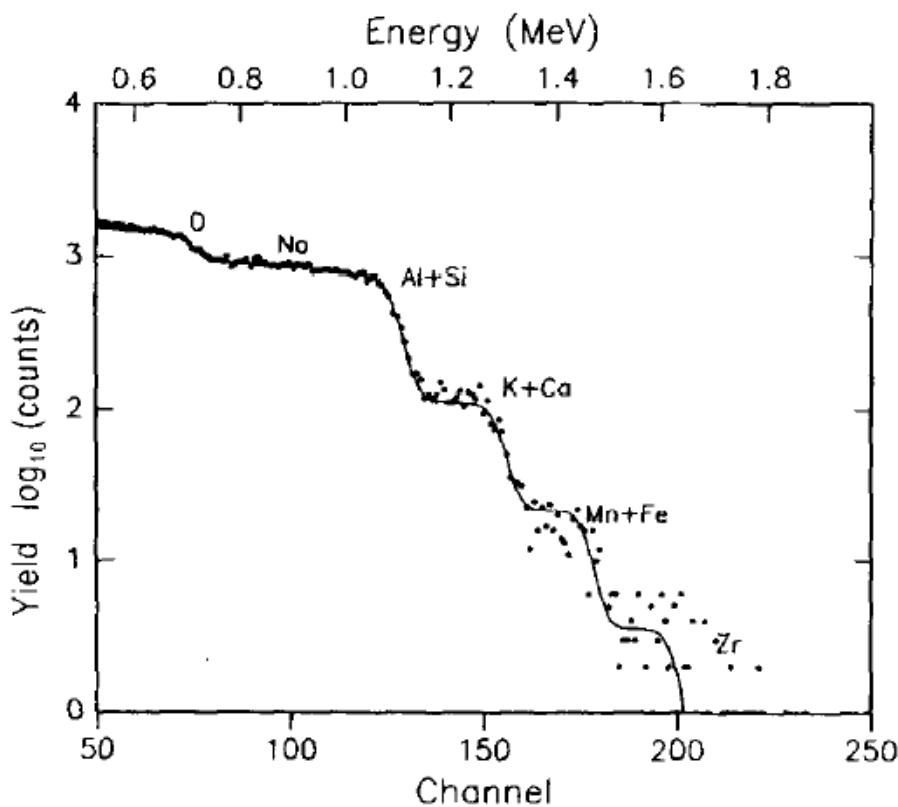


Fig. 4. Typical ion energy spectrum of an obsidian sample, used

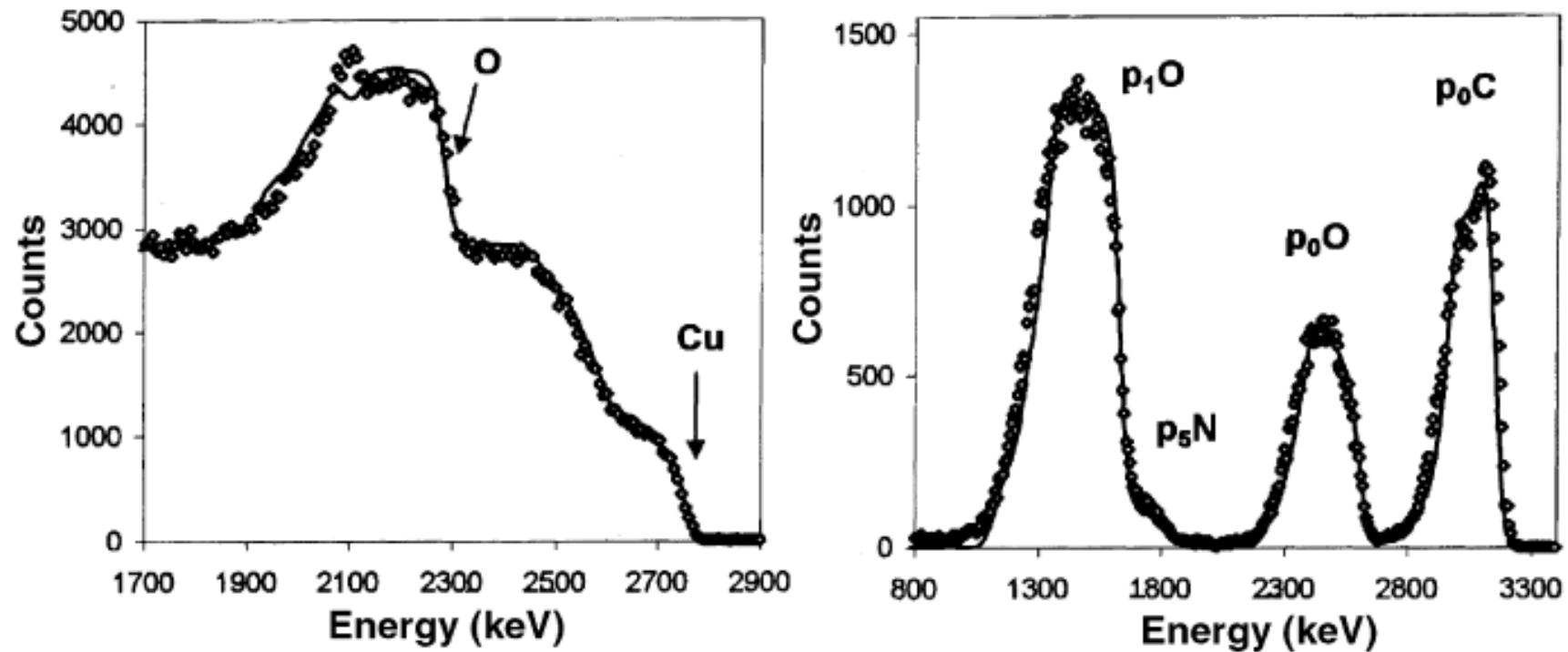


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Examples

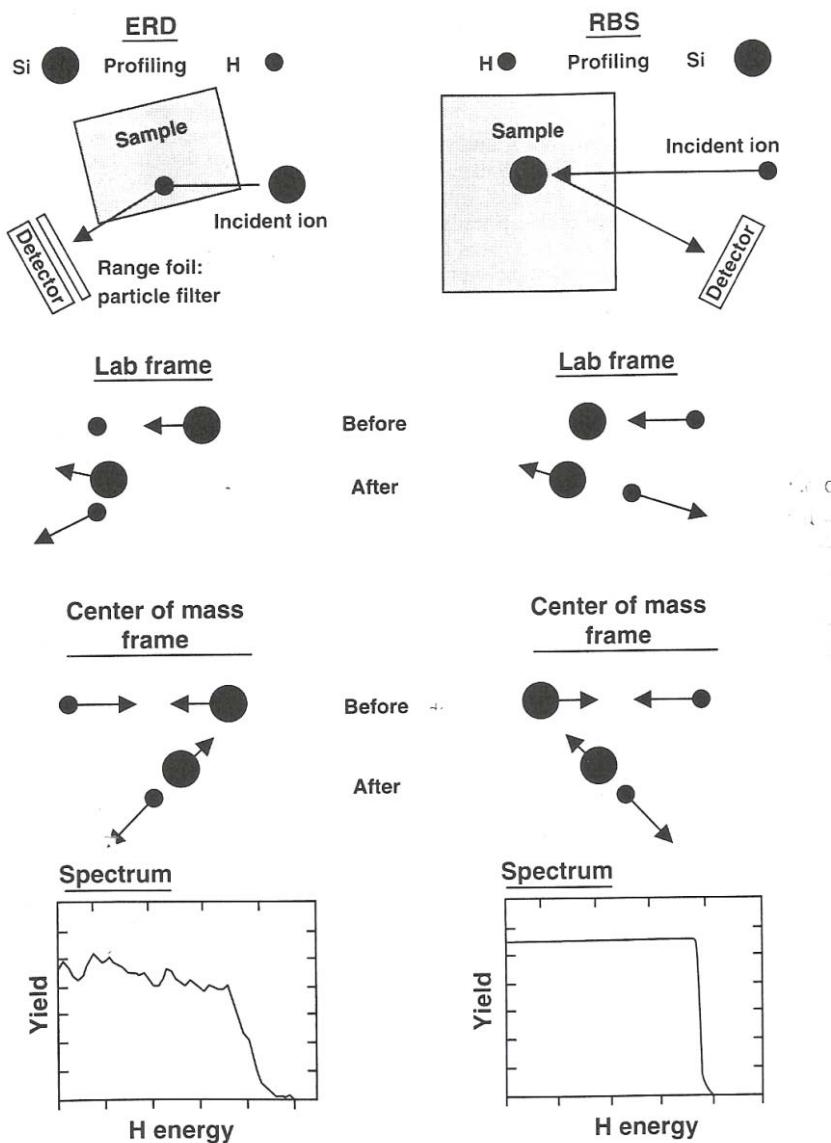
RBS and NRA with external beams for archaeometric applications
E. Ioannidou al. NIMB B 161±163 (2000) 730±736

Examination of patina layers on ancient steel



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Elastic Recoil Detection



Suitable for the detection of
light elements on **heavy** substrates

Heavy ions are used as the ion beam

Detection of the light nuclei at
forward angles

In simple ERDA experiments necessary to
use **filters** in front of the detector in order to
stop the heavy beam ions



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Elastic Recoil Detection

IBA and SIMS coupling to study glass alteration mechanisms
S. Djanarthany et al. *Journal of Non-Crystalline Solids* 353 (2007) 4830

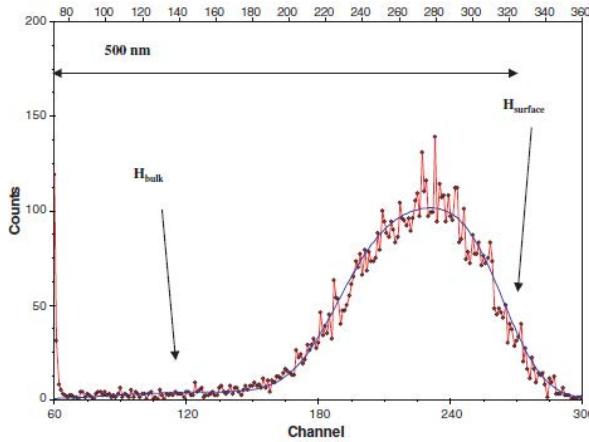


Fig. 3(a). ERDA data for A3 glass leached for 1 month at 96 °C.

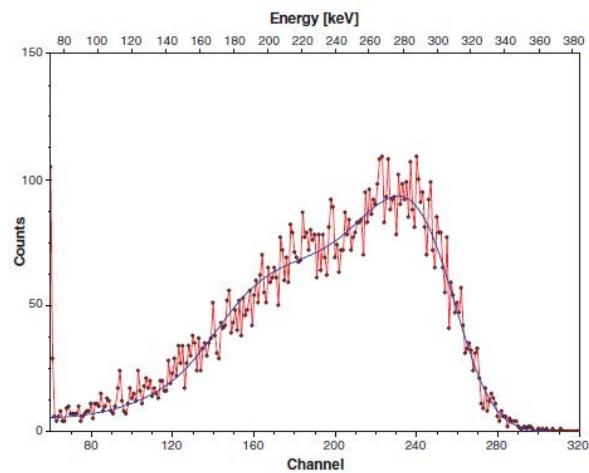


Fig. 3(b). ERDA data for B7 glass leached for 1 month at 96 °C.

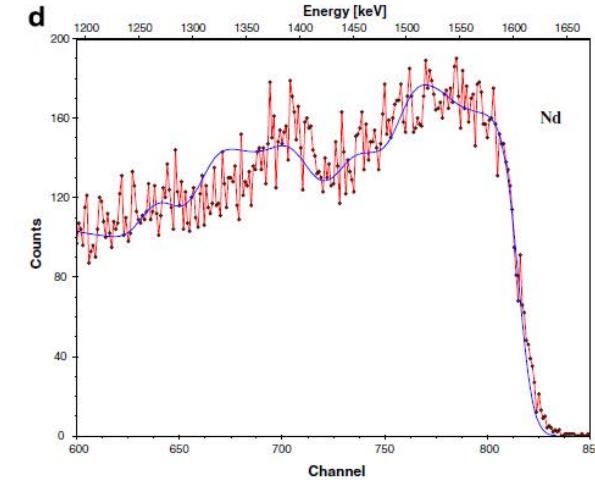
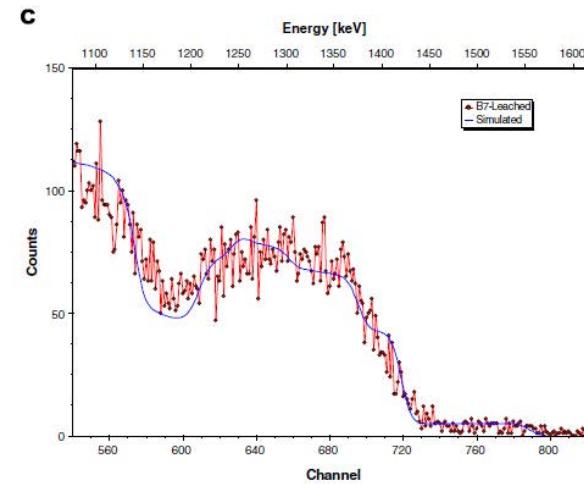
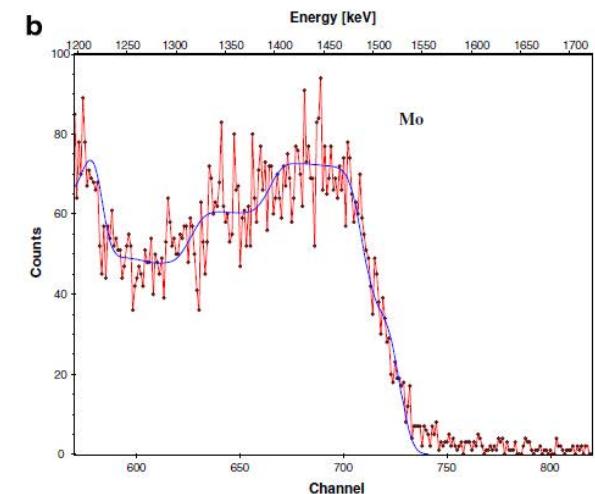
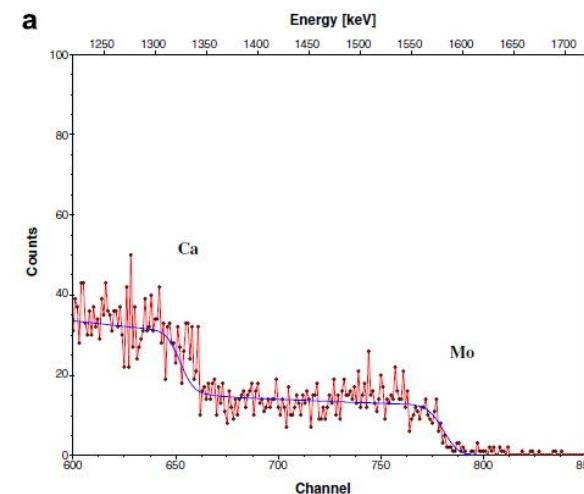
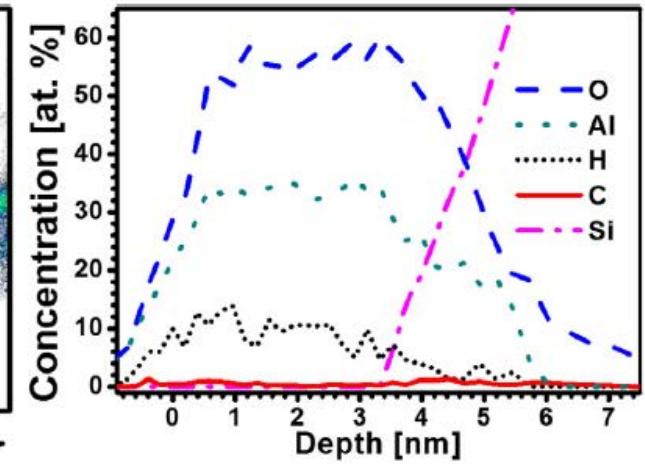
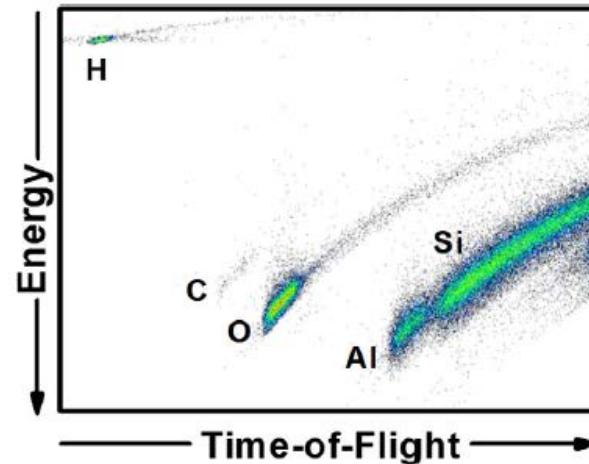
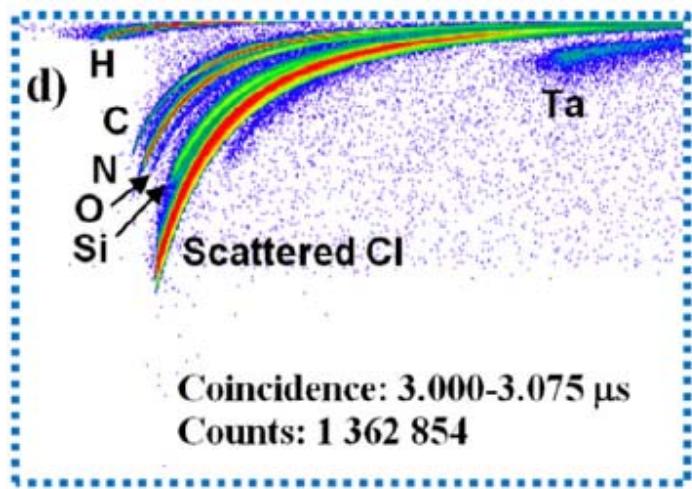
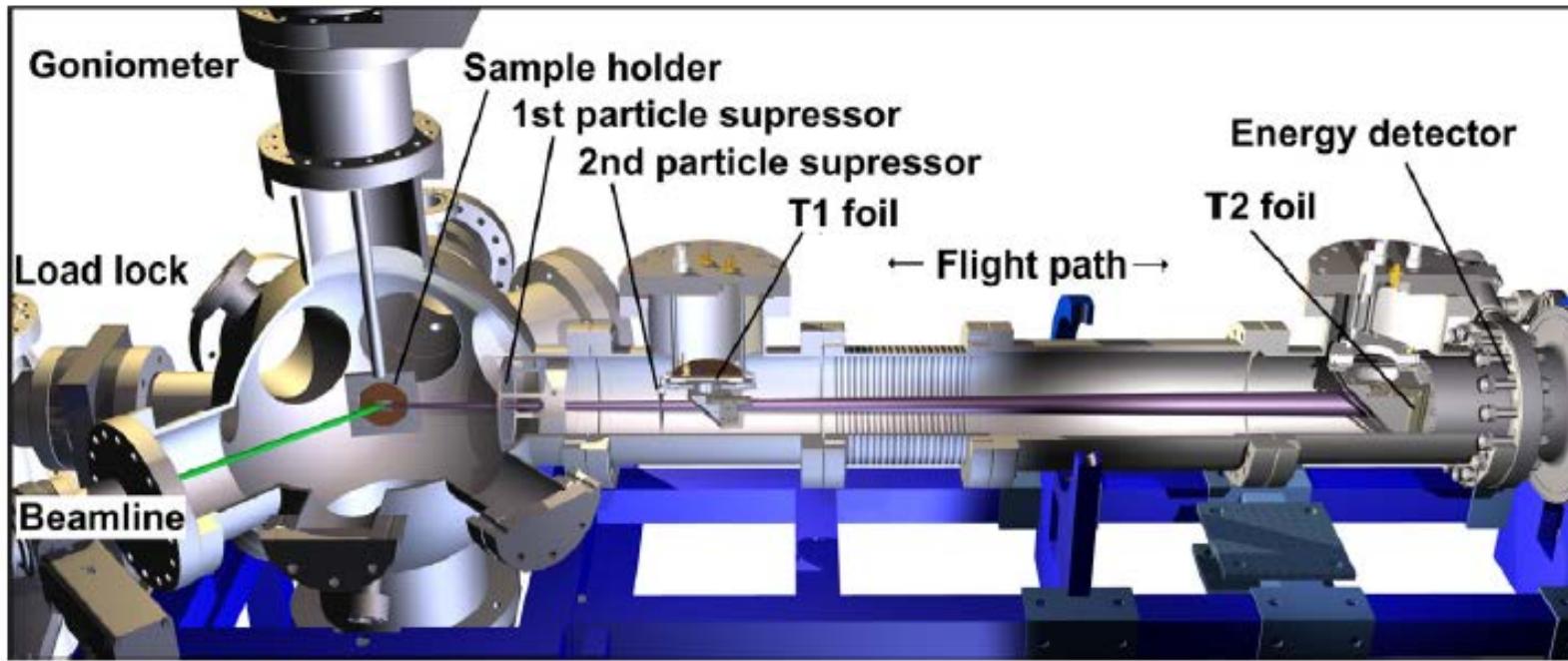


Fig. 2. RBS data for A3 glass (a) unleached, (b) leached for 1 month at 96 °C, (c) RBS data for B7 glass leached for 1 month at 96 °C and (d) RBS data for C11 glass leached for 1 month at 96 °C.



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Elastic Recoil Detection



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Synopsis

Main Applications	Relevant Techniques	Detection Capability
Material Science	Rutherford Backscattering Spectrometry (RBS)	$Z > 1$
Archaeometry and Cultural Heritage	Elastic Recoil Detection (ERD)	$Z < 17$ (typically including H)
Earth and Environmental Sciences	Particle Induced X-ray Emission (PIXE)	$Z > 11$
Biological Sciences	Particle Induced Gamma-ray Emission (PIGE)	$Z < 17$ (Li, B, F, Na, Mg, Al, Si)
Nuclear Safety and Radioprotection	Nuclear Reaction Analysis (NRA)	$Z < 17$ (often for C, N, O and isotope detection)
Fundamental Nuclear and Atomic Physics	Scanning Transmission Ion Microscopy (STIM)	Sample density
Forensic	Ionoluminescence (IL)	Defects and Sample Structure



Conclusions

Summary

1. Rutherford backscattering (RBS) is ideal for depth-profiling heavy elements on lighter substrates.
2. Elastic recoil detection analysis (ERDA) is excellent for depth-profiling very light elements in thin films.
3. Nuclear reaction analysis (NRA), is excellent for high resolution depth-profiling of specific isotopes.

Present Situation

1. A lot of work is being done in PIGE and NRA.
2. Micro-beams and measurements in air (Louvre) have enhanced IBA capabilities.

Future Perspectives

1. New techniques are always evolving (e.g. HR-RBS).
2. PIGE analytical algorithms?
3. CAN WE SOLVE ALL THE PROBLEMS??? NO (BUT MANY YES...)

