X-ray spectrometry at external-beam IBA facilities for cultural heritage applications

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Outline

• Introduction to Ion Beam Analysis and to Particle Induced X-ray Emission technique

• External beams

• External-beam PIXE analysis of Cultural Heritage

• Synergy with other IBA techniques
Ion Beam Analysis techniques

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<th>Analytical technique</th>
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<td>ion</td>
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<td>Ionoluminescence (IL)</td>
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</tbody>
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IN
Beam
OUT
Analytical

technique
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PIGE, Activation Analysis
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hν
Ionoluminescence (IL)
General features of IBA

- Multielemental
- Quantitative analysis (“traceability”)
- High sensitivity (1-100 ppm in at/cm$^3$; $10^{11}$-$10^{12}$ in at/cm$^2$)
- Surface analysis (10 Å - 10 μm)
- Depth profiling
- Non-destructive
- No sample pre-treatment
- Microanalysis (lateral resolution <1 μm)
- 2D mapping
Particle Induced X-ray Emission (PIXE)

Emission of characteristic X-rays following ionization from incident ions for Z > 10.
Energy of characteristic X-rays

Energy levels of core electrons

- **K shell**
  - $n=0$
  - **K lines**: filling vacancies in the K shell
  - $K\alpha : K\beta \sim 6:1$

- **L shell**
  - $n=1$
  - **L lines**: filling vacancies in the L shell
  - $L\alpha : L\beta \sim 2:1$

- **M shell**
  - $n=2$

**Transitions to the M shell** give a series of M lines

Energy depends on Z. Useful ranges:
- **K**: F, D, Ag
- **L**: Cu, U
- **M**: Pt - U
Energy of characteristic X-rays

Moseley law:

\[ E = K \cdot (Z - b)^2 \]
X-ray production cross sections

Series L:
- Cu (orange)
- As (blue)
- Ag (green)
- Sn (purple)
- Au (light blue)
- Pb (red)

Series M:
- Au (blue)
- Pb (red)
Adavantages of PIXE

Among IBA techniques, PIXE is a “killer application” for the non-destructive analysis of cultural heritage object since it is highly sensitive over a broad range of elements and it can be performed with external beams while maintaining the object in atmosphere, avoiding the need of picking up samples and greatly easing the object positioning, thus precious and big artefacts can be studied

• Very fast, high-sensitivity, non-destructive analysis

• Quantitative analysis

• Minimum energy of detected X-rays tipically \( \sim 1 \) keV

\( \Rightarrow \) all the elements with \( Z \geq 11 \) are quantifiable simultaneously
Limitations of PIXE

- No information on the organic components
- No information on chemical states
- No structural information
- Surface analysis (problems with altered objects)
- No direct information on the stratigraphy and the depth distribution of the elements
What PIXE can do for cultural heritage?

- Materials identification
  - analysis of major elements by PIXE (and PIGE)
- Materials provenance
  (sources of raw materials and trade routes)
  - analysis of trace elements by PIXE
- Manufacture technology
  - high spatial resolution: lateral by µ-PIXE (in-depth by RBS)
Example of PIXE spectra

PIXE spectra of obsidian

counts

E keV

Si  Al  Na  K  Fe  Zn  Pb  Rb  Zr  Y  Nb

low energy PIXE spectrum
main constituents

high energy PIXE spectrum
trace elements
PIXE quantitative analysis (thin targets)

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

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

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

\[ Y_0(Z) = \eta_Z \cdot Q \cdot (\rho_Z \cdot t) \]

Sensibility (counts/μC/(μg/cm²))

\( (\rho_Z \cdot t) = Y_0(Z) / (\eta_Z \cdot Q) \)
PIXE quantitative analysis by comparison with thin elemental standards

\[ Y_0(Z)_{\text{sample}} = \eta_Z \cdot Q_{\text{sample}} \cdot (\rho_Z t)_{\text{sample}} \]

\[ Y_0(Z)_{\text{std}} = \eta_Z \cdot Q_{\text{std}} \cdot (\rho_Z t)_{\text{std}} \]

\[ (\rho_Z t)_{\text{sample}} = (\rho_Z t)_{\text{std}} \cdot \left[ \frac{Y_0(Z)_{\text{sample}}}{Y_0(Z)_{\text{std}}} \right] \cdot \left( \frac{Q_{\text{std}}}{Q_{\text{sample}}} \right) \]
Quantitative analysis by comparison with thin elemental standards

\[ Y_0(Z)_{\text{sample}} = A_{Z,\text{sample}} \cdot \frac{1}{1 - DT_{\text{sample}}} \cdot \frac{1}{1 - PU_{\text{sample}}} \]

\[ Y_0(Z)_{\text{std}} = A_{Z,\text{std}} \cdot \frac{1}{1 - DT_{\text{std}}} \cdot \frac{1}{1 - PU_{\text{std}}} \]

- X-ray peak area
- Dead time fraction
- Pile-up fraction
Thick targets
Quantitative analysis (thick targets)

\[ Y(Z) = \left( \frac{Q}{e} \right) \left( \frac{N_A}{A} \right) \left( \alpha_Z \varepsilon_Z \Delta \Omega / 4\pi \right) \rho_Z \int_{0}^{T} \sigma_{Z,E} \exp(-\mu \cdot x / \cos \theta) \cdot dx \]

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

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

\[ (\rho_Z t) = F(Z) \cdot Y(Z) / (\eta_Z \cdot Q) \]
Do extracted ion beams look like these?
**External ion 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
External ion 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

<table>
<thead>
<tr>
<th>Material</th>
<th>Thickness (μm)</th>
<th>ΔE (keV)</th>
<th>(\sigma_E) (keV)</th>
<th>(\sigma_\theta) (μm/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>10</td>
<td>235</td>
<td>16</td>
<td>14</td>
</tr>
<tr>
<td>Kapton</td>
<td>8</td>
<td>130</td>
<td>9</td>
<td>6</td>
</tr>
<tr>
<td>Zr</td>
<td>2</td>
<td>75</td>
<td>7.3</td>
<td>15</td>
</tr>
<tr>
<td>Si</td>
<td>0.1</td>
<td>8</td>
<td>5</td>
<td>&lt;1</td>
</tr>
<tr>
<td></td>
<td>0.5</td>
<td>40</td>
<td>9</td>
<td>&lt;2</td>
</tr>
</tbody>
</table>

- Typical extraction windows for 0.5 μm Si₃N₄ and 7.5 μm Upilex.
The use of an helium-saturated atmosphere in front of the X-ray detector is mandatory.
X-ray detector efficiency

Low energy:
\[ \varepsilon \approx \exp[-\mu(Z, E_X) \cdot t_{\text{window}}] \]

High energy:
\[ \varepsilon \approx 1 - \exp[-\mu(Z, E_X) \cdot L_{\text{detector}}] \]
Typical PIXE detectors

**Silicon Drift Detector (SDD)**
- Active area: 7 - 100 mm$^2$
- Thickness: 300 - 450 μm
- Energy resolution <140 eV
- High count-rate (100 kHz)
- Peltier cooling (-10, -20 °C)

**Lithium-drifted Si / Ge, Si(Li) / Ge(Li)**
- Active area: 10 - 100 mm$^2$
- Thickness: 3 - 5 mm
- Energy resolution <180 eV
- Liquid N2 cooling (77 K)
- Ge(Li): high-Z material, but “escape peak”
# 2-detectors PIXE set-up

1. X-ray detectors
2. Sample to analyse
3. Beam exit window
4. Faraday cup
5. Graphite collimator

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<table>
<thead>
<tr>
<th>Target</th>
<th>X-rays</th>
<th>What is needed</th>
<th>Detector features</th>
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<tr>
<td>Low-Z elements</td>
<td>Low energy</td>
<td>Minimum dead layers</td>
<td>Thin entrance window</td>
</tr>
<tr>
<td></td>
<td>High energy</td>
<td>Small solid angles</td>
<td>Small active area</td>
</tr>
<tr>
<td></td>
<td>High cross sections</td>
<td>Efficiency</td>
<td>Large active area</td>
</tr>
<tr>
<td>Medium-high-Z elements</td>
<td>High energy</td>
<td>Large solid angles</td>
<td>Large active area</td>
</tr>
<tr>
<td></td>
<td>Low cross sections</td>
<td>Efficiency</td>
<td>Large active thickness</td>
</tr>
</tbody>
</table>
### Target
- **Low-Z elements**
- **Medium-high-Z elements**

### X-rays
- Low energy
- High energy
- Low cross sections
- High cross sections

### What is needed
- Minimum dead layers
- Small solid angles
- Large solid angles
- Efficiency

### Detector features
- Thin entrance window
- Small active area
- Large active area
- Large active thickness

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#### Diagram:
- 1. X-ray detectors
- 2. Sample to analyse
- 3. Beam exit window
- 4. Faraday cup
- 5. Graphite collimator

#### 2-detectors PIXE set-up
### 2-detectors PIXE set-up

![Diagram of the PIXE set-up](image)

#### Table: Target X-rays and Detector Features

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The working principle of Silicon Drift Detectors

- The Silicon Drift Detector (SDD) was first proposed in the early '80s by Emilio Gatti and Pavel Rehak [Gatti & Rehak, NIM 225 (1983) 608] as a position sensitive semiconductor detector for high energy charged particles, based on a novel charge transport scheme where the field responsible for the charge transport is independent of the depletion field.

Schematic diagram of the Silicon Drift Detector for X-ray spectroscopy with radiation entrance window of the detector consisting of a continuous shallow p+ implant.

Energy potential for electrons inside a SDD with homogeneous entrance window.
The SSD for X-ray spectroscopy

- The SDD is employed in high-resolution X-ray spectroscopy because of the low capacitance of the collecting electrode (0.5-1 pF/cm²) and the low leakage current (1-2 nA/cm² at room temperature) resulting in improved energy resolution.

$$\text{ENC} = \left[ \frac{k_1 \cdot \langle e_{w}^2 \rangle \cdot (C_d + C_i + C_p)^2}{\tau} + k_3 A_{1/f} (C_d + C_i + C_p)^2 + 2k_2qI_l\tau \right]^{1/2}$$

### Energy resolution

<table>
<thead>
<tr>
<th>Temperature</th>
<th>SDD Roentec 10 mm², 0.3 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>T = -27.5°</td>
<td>SDD Si(Li)</td>
</tr>
<tr>
<td>T = -20°</td>
<td>140-160 eV</td>
</tr>
<tr>
<td>T = -10°</td>
<td>180-200 eV</td>
</tr>
<tr>
<td>Ambient T</td>
<td>1 μs 6 μs 50 kHz 5 kHz -10/-20 °C -195 °C</td>
</tr>
</tbody>
</table>
Commercial SDD

- Starting from the first SDDs, 5 or 10 mm² area, 0.3 mm thick, now several companies are selling SDDs with a wide range of characteristics and designs, and competitive prices.
Large area SDD

- A single SDD with active area up to 150 mm$^2$ is now commercially available (Ketek Gmbh)

- Larger areas can be obtained using arrays of individual systems or integrated multi-channel SDDs

Ring-shaped multi cell SDD with 12 x 5 mm$^2$ hexagonal cells (PN Detectors, Germany)

Multi SDD PIXE system (1 low and 4 high energy), total solid angle 500 msr, at AGLAE, Paris (Ketek Gmbh, Germany)

4-channel SDD (30 mm$^2$ each) PIXE system at Surrey Ion Beam Centre (SGX Sensortech, UK)
Increased thickness SDD

- Single SDD with active thickness of 0.7 mm (Ketek Gmbh) or 1 mm (Hitachi High-Technologies Science America, Inc) are now commercially available

QE (0.3 mm) = 90% @10 keV; 25% @20 keV
QE (0.5 mm) = 100% @10 keV; 40% @20 keV
QE (1.0 mm) = 100% @10 keV; 60% @20 keV

32 keV Ba spectra from USGS GSP1 – Rh tube, 50 kV, 1 mA, 0.5 mm Cu + 6 mm Al Filter, 0.5 μs peaking time, 300 sec. livetime.
(Gordon Myers, Hitachi High-Technologies Science America, Inc.)
Sensitivity curve for large area SDD and Si(Li)
Backscattered protons effects on SDD

- PIXE detectors with thin entrance window used in presence of a large backscattered protons flux from the sample can suffer unrecoverable damages (**long-term effects**) and worsening of the energy resolution under beam irradiation (**short-term effects**).
Magnetic proton deflector

- The use of a properly designed magnetic deflector to filter out the backscattered protons without substantial limitations to the SDD intrinsic efficiency at low X-ray energies is mandatory to prevent any long-term damages and to avoid the worsening of the energy resolution.

NdFeB permanent magnets, B = 0.5 T

- 6 cm for H7, p < 4 MeV
- 7 cm for R10, p < 6 MeV
- 8 cm for H30, p < 3 MeV
Detection efficiency for a 2-detectors PIXE set-up
The “funny filter” concept was introduced by Harrison and Eldred in 1973, when PIXE was starting to develop as an analytical technique.

\[ \alpha_1(Z), \text{ transmission coefficient of absorber 1} \]
\[ \alpha_2(Z), \text{ transmission coefficient of absorber 2} \]
\[ R, \text{ ration between absorber 2 area and detector area} \]

\[ \alpha_Z = \alpha(Z) = \alpha_1(Z) \cdot [R + (1 - R) \cdot \alpha_2(Z)] \]
Mainly due to Secondary Electron Bremsstrahlung radiation
(for $E < 10$ keV)

$T_m = 4m_eE_0/m_p$
PIXE spectrum background

Possible contribution from Compton interaction of gamma-rays in the detector active volume (for $E > 10$ keV)

Compton background due to gamma-rays is reduced by a factor 3 using thinner PIXE detectors
Minimum Detection Limit (MDL)

$N_B$ is the number of counts integrated in a region of the background under the peak as wide as the peak FWHM

$$Y_{X_j}^{MDL} = 3\sigma_F = 3\sqrt{N_B}$$

In terms of areal density (i.e. $\mu g/cm^2$) the PIXE MDL can be calculated as:

$$MDL_Z = \frac{3 \cdot \sqrt{N_B}}{\eta_Z \cdot Q}$$
Nuclear microscopy
External beam PIXE 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)
PIXE analysis of a painting by Lucas Cranach the Elder
C. Neelmeijer et al.
(Rossendorf Forschungszentrum, Dresda)
Example of PIXE spectra of two blue pigments
Blue Temperas

Extensive use of lapislazuli starting from XI century

Pluteo 48, 34, f. 66 v°

Pluto 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
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.
MicroPIXE analysis of metal point drawings

Four metallic points: silver, lead, gold, copper

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

Lead stylus (Pb, trace of Sn)

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

Elemental map on 0.4 × 0.4 mm²

100 µm
Micro-PIXE measurements of an Achemenide pendant (IV century BC)
Micro-PIXE measurements of an Achemenide pendant (IV century BC)
Whereas PIXE does not provide elemental depth profiles, “Differential PIXE” measurements can provide semi-quantitative stratigraphical information on cultural heritage objects (works mainly by the groups in Florence, Ljubljana and Namur).

“Differential PIXE” consists in performing measurements on the same area with beams of different energies.

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

\[
\frac{Y_{\text{Al}}}{Y_{\text{Ca}}} = \frac{C_{\text{Al}}}{C_{\text{Ca}}} \frac{\int_{E_p-\Delta E_{\text{lap}(t)}}^{E_p} \sigma^{(\text{Al})}_X(E) e^{-\mu^{(\text{Al})}_\text{lap} \frac{x(E)}{\cos \theta}} \frac{dE}{S_{\text{lap}}(E)} }{e^{-\mu^{(\text{Ca})}_\text{lap} \frac{t}{\cos \theta}} \int_{E_p-\Delta E_{\text{lap}(t)}}^{0} \sigma^{(\text{Ca})}_X(E) e^{-\mu^{(\text{Ca})}_\text{white} \frac{x'(E)}{\cos \theta}} \frac{dE}{S_{\text{white}}(E)} } }
\]

- Experimental
- \( t = 10.5 \, \mu m \)
- \( t = 11.5 \, \mu m \)
- \( t = 9.5 \, \mu m \)

\( t \sim 10.5 \, \mu m \)
Metal threads (Alhambra, Granada)

Enrichment of gold on the surface
Ca and Fe are in the varnish
"Incarnato" paint layer:
cinnabar (HgS, red pigment) + lead white

preparation:
lead white

Ca and Fe are in the varnish
Particle Induced Gamma-ray Emission (PIGE) technique is an invaluable tool, complementary to PIXE, to quantify low-Z elements (Li, B, F, Na, Mg, Al, Si, ...) in cultural heritage objects. In this respect PIGE can be considered a “sidekick” of PIXE.
Lapis-lazuli pigment in paint layers

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

“Maddonna dei fusi”, Leonardo da Vinci (1501)
Lapis-lazuli pigment in paint layers

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

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

PIXE spectra
PIGE spectra
Analysis of ancient Roman glasses

Quantification of sodium is of great importance for the characterisation of ancient glasses.

There are two basic typologies of Western glasses:

- **natron** (high Na\(_2\)O, low K\(_2\)O and MgO content) since Middle Ages
- **plant ash** (low Na\(_2\)O, high K\(_2\)O content) since Middle Ages
Evolution of glass manufacture technology

- **476 AD** end of Roman Empire
- **1000** strong demand for stained glass windows
- **1600** modern chemistry synthetic NaCO3
- **1800**

Glass type:
- natron : NaCO3
- soda (Na)
- soda-potash (Na/K)
- potash (K)

Plant ashes : Salicornia
beech tree ashes Na<<K
Sodium in Roman glasses

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

“freshly cut” zone

coloured but more opaque zone

Roman glass mosaic tesserae

“crust”

2 mm

Na X-ray map

Na γ-ray map
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 Na

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

<table>
<thead>
<tr>
<th>glass colour</th>
<th>main oxides (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Na₂O</td>
</tr>
<tr>
<td>green</td>
<td>~20</td>
</tr>
<tr>
<td>blue</td>
<td>~20</td>
</tr>
<tr>
<td>turquoise</td>
<td>~20</td>
</tr>
<tr>
<td>yellow</td>
<td>~15</td>
</tr>
<tr>
<td>red</td>
<td>~10</td>
</tr>
</tbody>
</table>
Complementary PIXE/RBS

Whereas PIXE and Rutherford Backscattering Spectroscopy (RBS) separately give only partial information, in samples with a layered structure these analyses can be performed simultaneously and their synergic use permits to derive detailed data about composition and elemental depth distribution of the analysed material (aka “Total IBA”)

**PIXE strengths**
- High sensitivity
- Excellent specificity

**RBS strengths**
- Traceable accuracy
- Excellent depth resolution

**PIXE weaknesses**
- Poor traceability
- Poor depth resolution

**RBS weaknesses**
- Low sensitivity
- Poor mass resolution
**“Total IBA” of 1-layered brass test sample**

<table>
<thead>
<tr>
<th></th>
<th>DataFurnace</th>
<th>SIMNRA</th>
<th>GUPIX</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Gilding</strong></td>
<td>Cu 94% Zn 2%, Fe 1%, 510 µg/cm</td>
<td>Cu 89% Zn 11%, 390 µg/cm</td>
<td></td>
</tr>
<tr>
<td><strong>Mordant</strong></td>
<td>H 40-49%, C 41-49%, O 10-11%, 1.0 mg/cm</td>
<td>H 51%, C 41%, O 8%, 1.0 mg/cm</td>
<td></td>
</tr>
<tr>
<td><strong>Bulk</strong></td>
<td>Cu 65% Zn 35%</td>
<td>Cu 65% Zn 35%</td>
<td>Cu 66%, Zn 34%, H&amp;Q 0.946</td>
</tr>
</tbody>
</table>

**Graphs and Data**

- **Count vs. Channel**
- **Log(Line Area)**
- **Atomic % vs. ug/cm²**
Analysis of stratigographies in cultural heritage by PIXE/RBS analysis

"View from the Window at Le Gras", the oldest surviving camera photograph ("heliography") created by Nicéphore Niépce (in 1826 or 1827) at Saint-Loup-de-Varennes

PIXE/RBS analyses reveal that the corrosion products are lead oxides in a Sn/Pb matrix.
Characterization of paint layers by simultaneous PIXE/RBS analysis

Ochre pigment (ematite) detected and quantified thanks to simultaneous PIXE/RBS measurements: $440 \cdot 10^{15}$ atoms/cm$^2$ Fe$_2$O$_3$ in $7000 \cdot 10^{15}$ atoms/cm$^2$ of oil (C$_{13}$O$_5$)
Thanks for your attention!