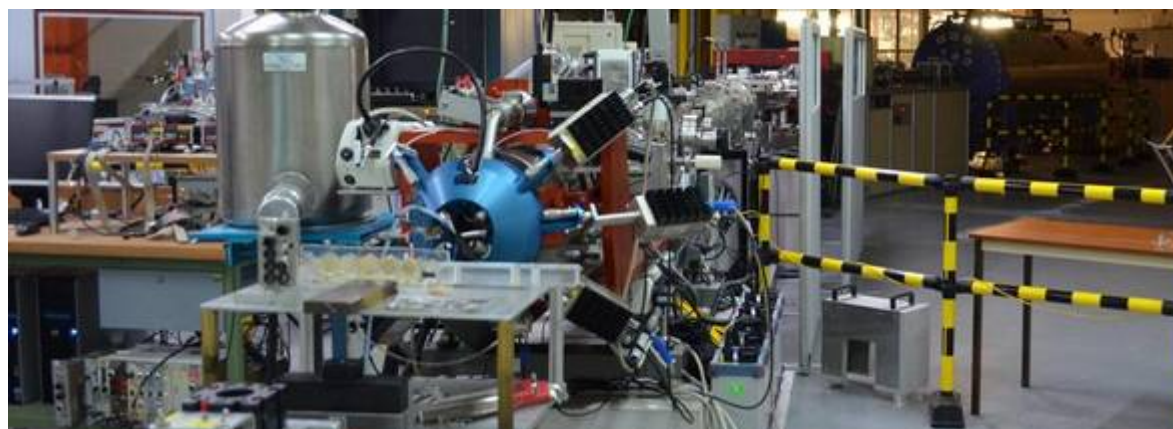




The accelerator under the Louvre the *New AGLAE*



Claire Pacheco^{1;2}, Quentin Lemasson^{1;2}, Brice Moignard^{1;2},
Laurent Pichon^{1;2}, Marie Radepont^{1;3}, Didier Gourier^{2;4}

¹Centre de Recherche et de Restauration des Musées de France, Palais du Louvre, Paris, France

²FR 3506 New AGLAE – CNRS/Ministère de la Culture et de la Communication, C2RMF, France

³Centre de Recherche sur la Conservation (CRC) – USR3224 – CNRS/MNHN/MCC, Paris, France

⁴Institut de Recherche de Chimie Paris, CNRS, Chimie ParisTech, Paris, France

Contact email: claire.pacheco@culture.gouv.fr



CENTRE DE
RECHERCHE
ET DE
RESTAURATION
DES MUSÉES
DE FRANCE



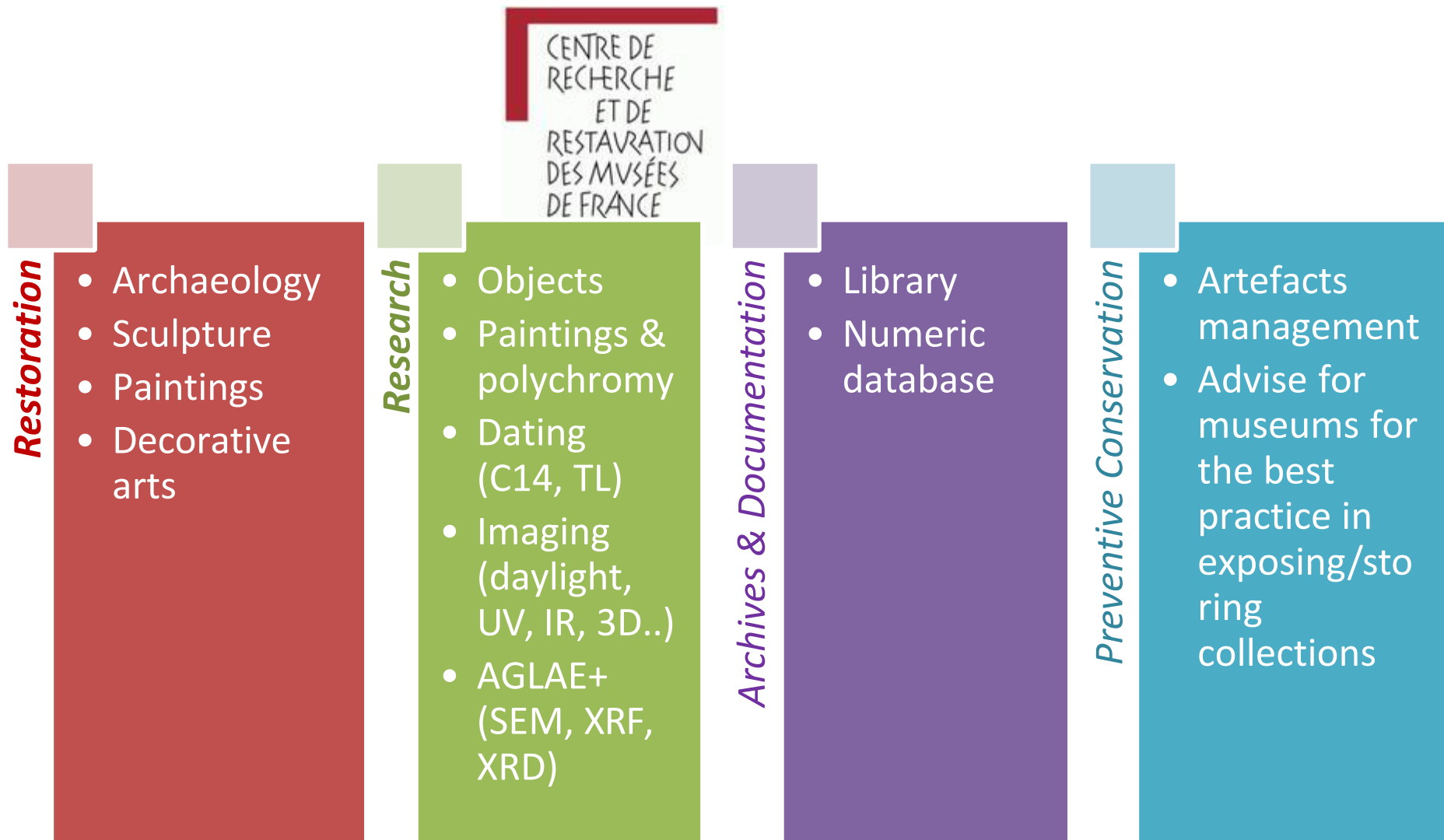
1931 : inauguration of
the Louvre laboratory



1988: AGLAE is settled in Le Louvre premises

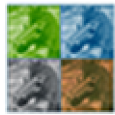


1995: Research Lab of French Museums



AGLAE beamtime:

- 1/ service : working for more than 1200 French museums
- 2/ Research projects: local (C2RMF), national, international
- 3/ instrument maintenance and development



IPERION CH

For more information, visit the website: www.iperionch.eu
Or write to claire.pacheco@culture.gouv.fr

Integrated trans-national access platforms. IPERION CH makes world-class scientific tools and knowledge available to Heritage scientists through three integrated Trans-National Access platforms

ARCHLAB

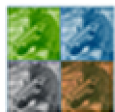
access to **ARCHIVES** (technical images, analytical data and conservation documentation) in datasets largely unpublished from **10 large archives** of prestigious European museums, galleries and research institutions.

MOLAB

access to **MOBILE INSTRUMENTS** for **NON-INVASIVE IN-SITU** measurements on precious, fragile or immovable objects, archaeological sites and historical monuments.

FIXLAB

access to **LARGE SCALE FACILITIES** (synchrotron, neutron sources, ion accelerators)



IPERION CH

For more information, visit the website: www.iperionch.eu
Or write to claire.pacheco@culture.gouv.fr



BNC-WIGNER (Budapest, Hungary) with access to various neutron-based instruments to investigate elemental and structural composition

contact:
kasztovszky.zsolt@energia.mta.hu



synchrotron SOLEIL (Gif-sur-Yvette, France) 26 beamlines covering Fr IR to Hard X-ray range; analysis of μ -samples; imaging over all regions of the EM spectrum; structural analysis
contact:
frederique.fraissard@synchrotron-soleil.fr



**MTA-ATOMKI,
DEBRECEN**

MTA-ATOMKI nuclear microprobe (Debrecen, Hungary) for high spatial resolution measurements with a focussed ion beam in vacuum and with external beam

contact:
szikszai.zita@atomki.mta.hu

WP 4 FIXLAB



AGLAE ion beam accelerator at **C2RMF** (Paris, France), providing elemental analysis with an external beam for whole art objects

contact:
claire.pacheco@culture.gouv.fr

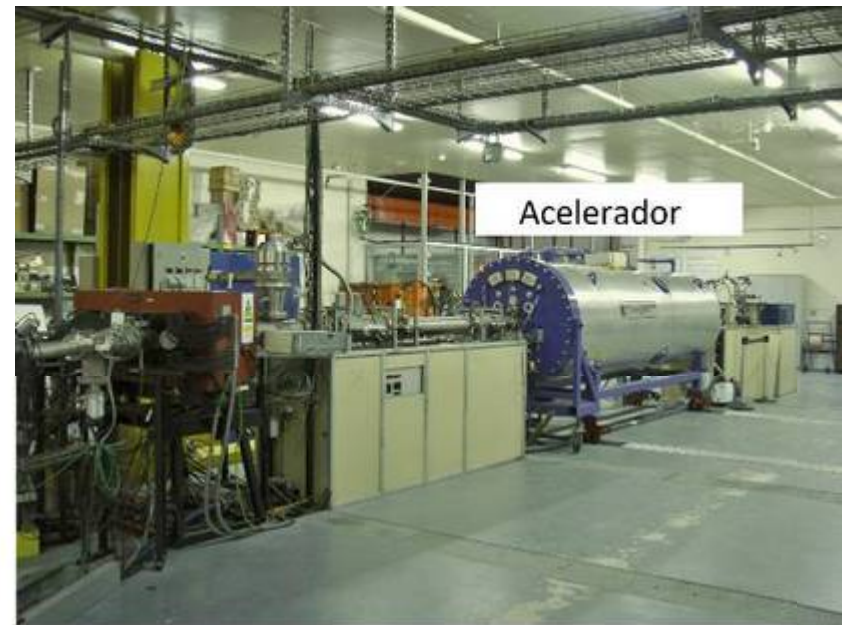


Accelerator

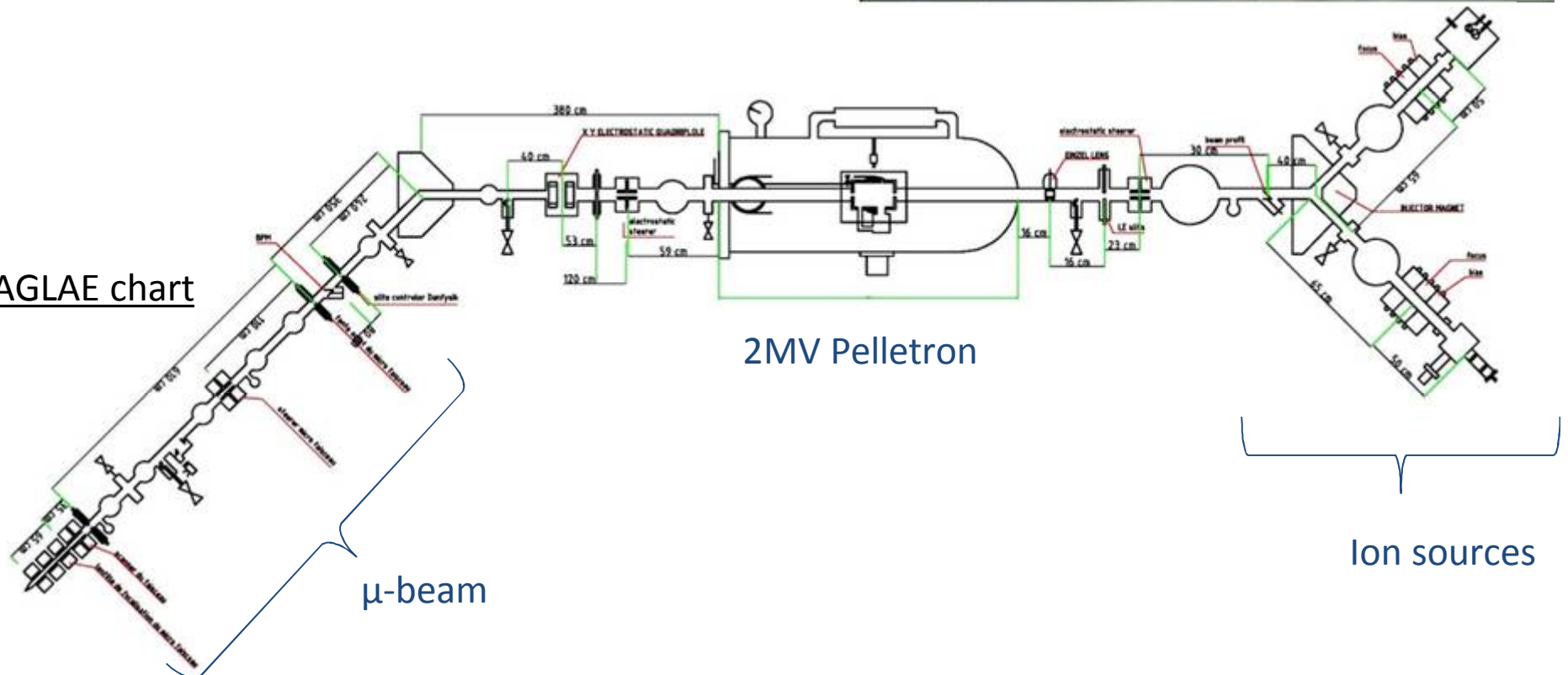
AGLAE: Accelerator Grand Louvre for Elemental Analysis

Unique for combining:

- Strategic localization
- 100% dedicated to the study of Cultural Heritage (CH)
- Data compiling from decades
- Beamline development for questions specific to CH questions and constraints of museum objects

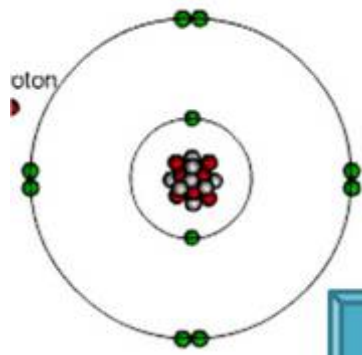


AGLAE chart



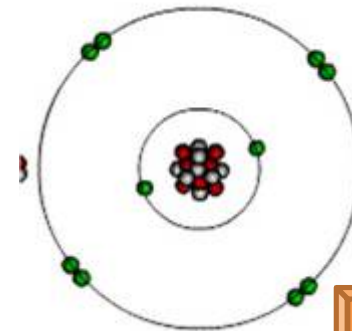
Ion Beam Analysis (IBA) in a nutshell...

PIXE: Particle Induced
X-ray Emission



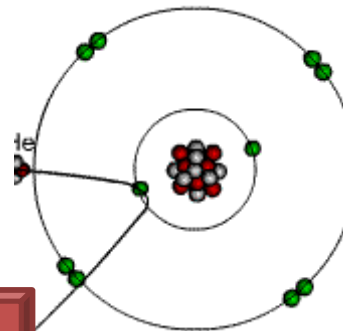
X-ray detector

PIGE: Particle Induced
 γ -ray Emission



γ -ray detector

(R)BS: (Rutherford) Backscattering Spectroscopy



particle detector



Which questions can answer AGLAE ?

- IBA on CH objects give knowledge on the studied material, which is part of the answer of a more general human science questioning
- Human science: a better understanding of societies and of their interactions in well defined geo-chronological contexts
 - How old is it ? → *production centres, workshops,...*
 - How was it made ? → *making process and technique history*
 - Where does it come from ? → *provenance, trade routes,...*

Case of provenance study



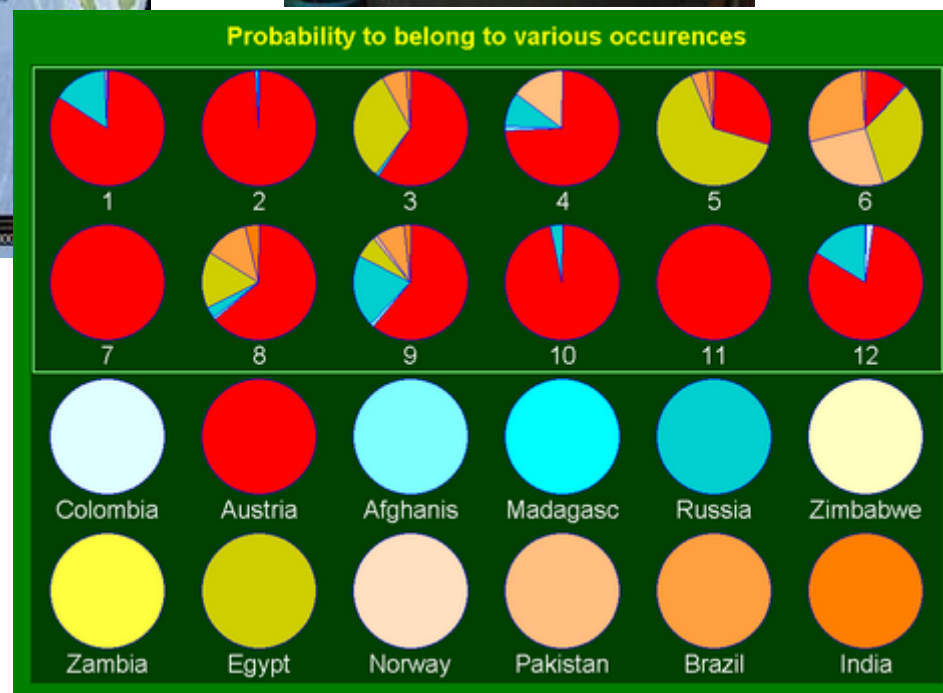
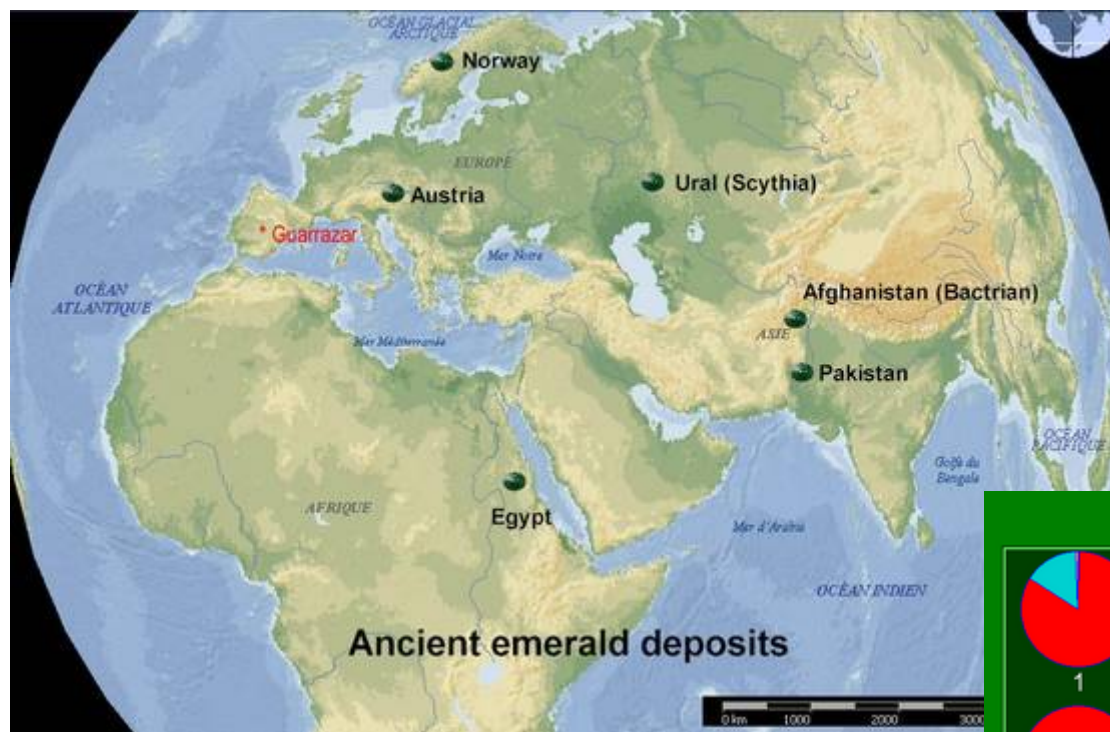
Guarrazar treasure

Spain, 7th c.

*Gold, gems, pearls, rock
crystals and glass jewellery*

Musée National du Moyen Age, Paris

Discovered in 1859 next to Toledo: 26 [votive crowns](#) and gold [crosses](#) that had originally been offered to the [Roman Catholic Church](#) by the [Kings of the Visigoths](#) in the seventh century in [Hispania](#), as a gesture of the orthodoxy of their faith and their submission to the ecclesiastical hierarchy.



Results:

The $\text{Be}_3\text{Al}_2(\text{SiO}_3)_6$ structure of emeralds relies trace elements: Na, Mg, Ca, Ti, Rb, Ni, Cu, Zn, Cs and Li, F.

Most of the emeralds are likely to come from the Alps, a deposit that was known to be exploited only since 13th c. from the texts.

Th. Calligaro et al., NIMB 161-163 (2000) 769

Which questions can answer AGLAE ?

- IBA on CH objects give knowledge on the studied material, which is part of the answer of a more general human science questioning
- Human science: a better understanding of societies and of their interactions in well defined geo-chronological contexts
 - How old is it ? → *production centres, workshops,...*
 - How was it made ? → *making process and technique history*
 - Where does it come from ? → *provenance, trade routes,...*
- Conservation science
 - Is it a fake ? → *authentication*
 - Why is it degraded ? → *degradation mechanism*
 - Will a restoration product be worse than doing nothing ?

Case study of authentication



PIXE: presence of F probably due the use of HF to depolish the glass and artificially age it.

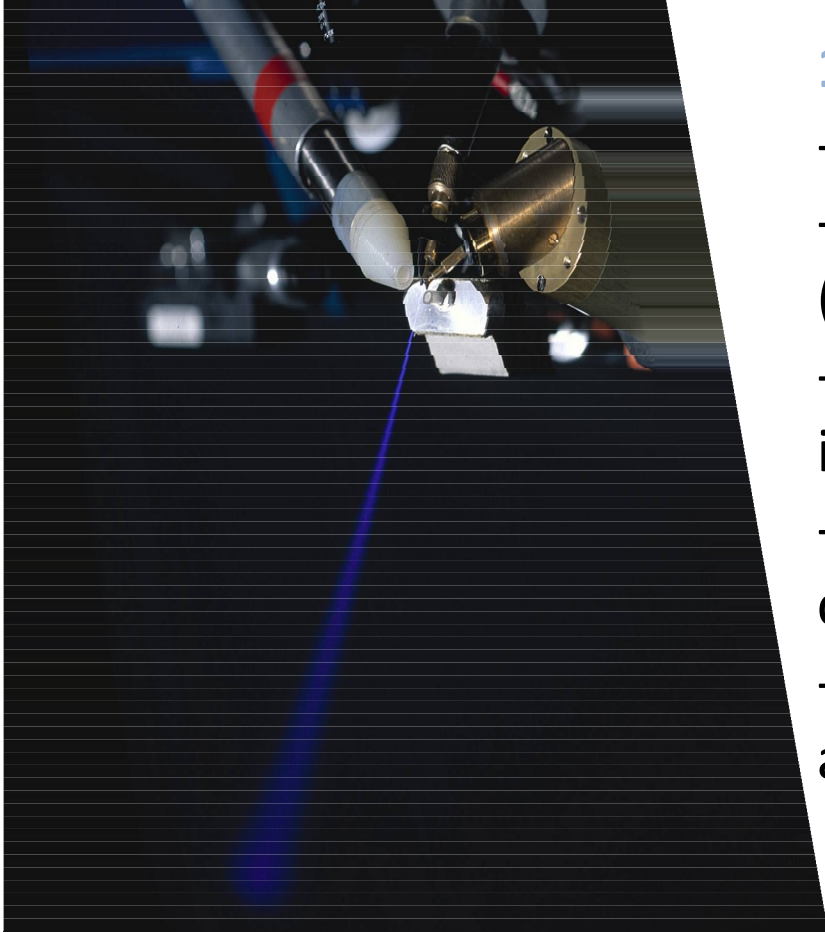
Référence : Geneviève Pierrat-Bonnefois et Isabelle Biron, La tête égyptienne en verre bleu : la conclusion d'une enquête, *La Revue du Louvre et des Musées de France*, n°3, 2003, 27-37.

Égypte ancienne

%	Bleu foncé	Turquoise	XVIII ^e dynastie
SiO₂	48 - 77	44 - 76	Sable
Al ₂ O ₃	0.8 - 5.2	0.9 - 3.4	
FeO	0.39 - 1.48	0.33 - 0.99	
Na ₂ O	6 - 20	6 - 19	Source Na
MgO	2.3 - 4.9	0.7 - 6.1	végétale
K ₂ O	0.9 - 2.6	0.9 - 3.3	cendre de plante
P ₂ O ₅	< 0.55	< 1	marine - salicorne
Cl, SO ₃	< 3	< 3	
CoO	0.1 - 0.5	0	Source cobalt Égypte CoMnNiZn
CuO	0 - 0.7	0.8 - 3.2	
Sb₂O₃	5.5 - 9.7	2.6 - 9.5	opacifiant
As203	0 - 2.4	0.5 - 5.7	antimoniante de Ca

XIX^e siècle

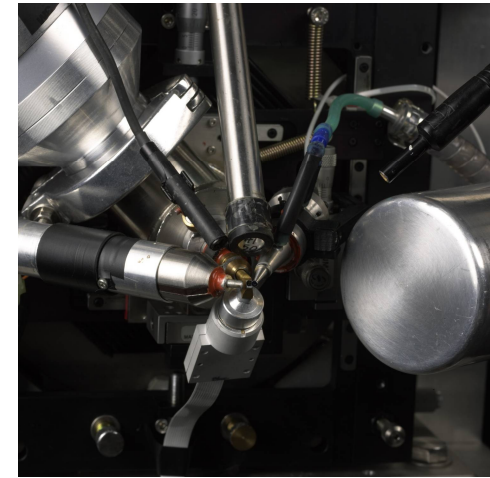
%	Bleu ciel	Bleu foncé	Tête bleue Louvre
SiO₂	47	40	Sable
Al ₂ O ₃	1.4	2.4	
FeO	0.4	0.63	
CaO	1.9	2.1	
Na ₂ O	12	11.9	Source Na
MgO	2.1	1.9	non végétale
K ₂ O	2.4	3.4	
P ₂ O ₅	0	0	
Cl, SO ₃	0 - 0.23	0 - 0.48	
CoO	0.16	0.4	Source cobalt
CuO	0.08	0	CoAlNi
Sb₂O₃	0.1	0.3	opacifiant
PbO	25	28	arséniate de Pb
As203	5.4	4	



1/ External beamline applied to CH:

- analysis of items of any size and shape
- no degradation due to vacuum (dehydration, outgassing,...)
- reduces the risk of heating and beam-induced damage
- no sample preparation (no deposit of conductive coating)
- simplifies sample changing and positioning

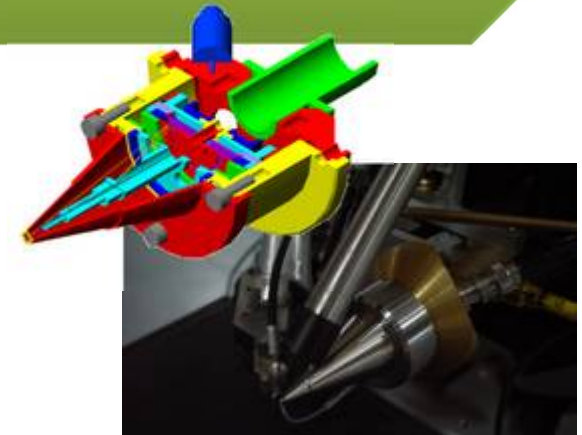
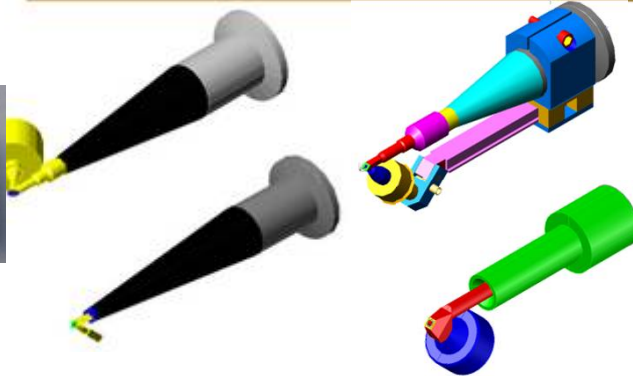




1989

1994-2003

2004-2009



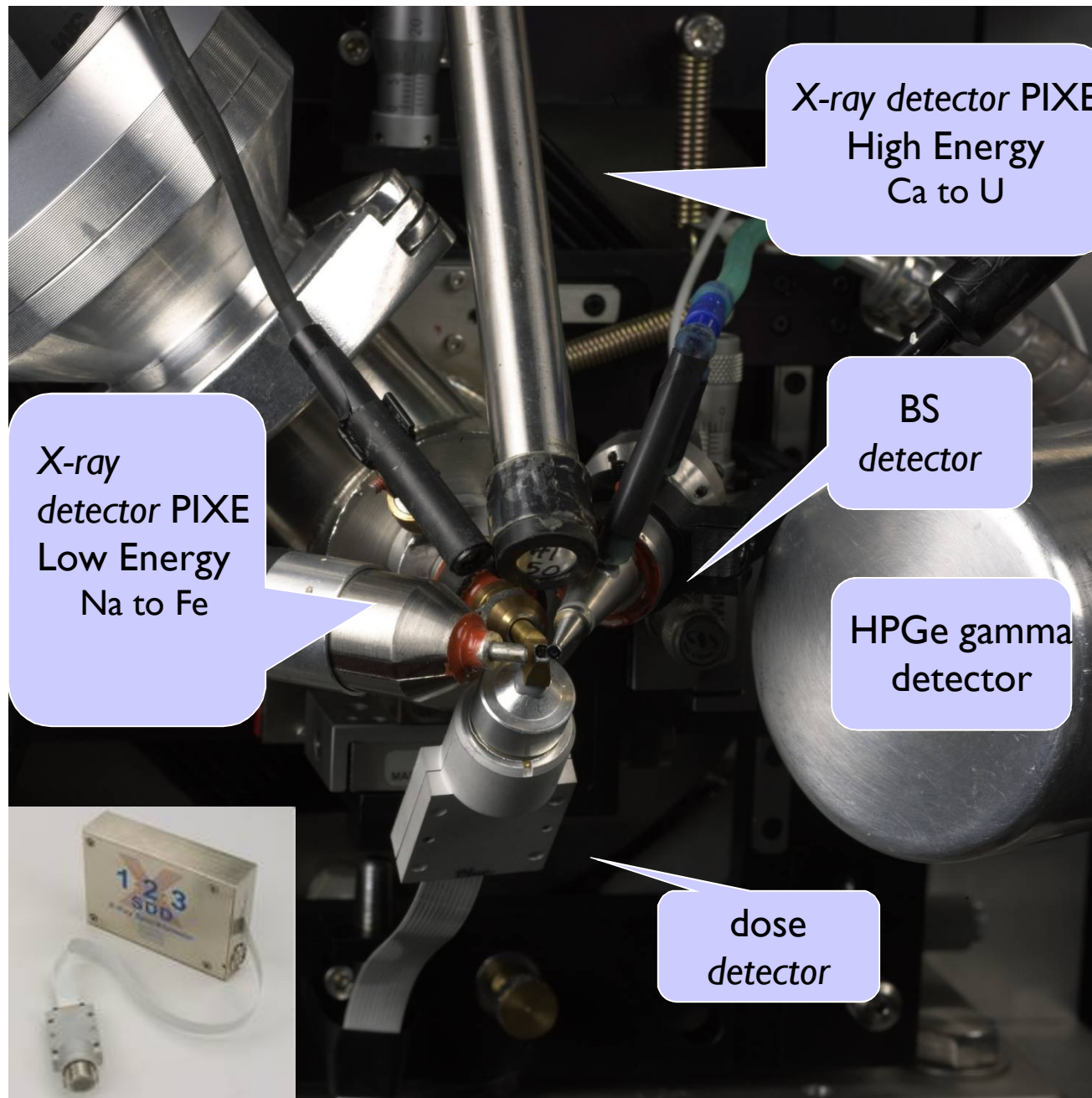
Evolution of the extraction nozzle

Annular RBS

Main contributors : J. Salomon, J.-C. Dran, Th. Calligaro, B. Moignard, L. Pichon, T. Guillou, F. Mathis

2/ Extending the range of measured elements in PIXE-PIGE

- at 1st, PIXE determined medium to high-Z elements at trace levels with a 3-MeV H⁺ irradiation; main components measured by XRF, SEM-EDS...
- to extend to $Z < 20$, process based on 2 successive runs at low and high beam energies
e.g. 1 run at 1 MeV (no X-ray absorber) and 1 run at 3 MeV with an appropriate absorber
- Introduction of a dual-detector system with a single run at 3 MeV developed by the Florence group McArthur et al., NIMB 45 (1990) 315



X-ray detector PIXE
High Energy
Ca to U

BS
detector

HPGe gamma
detector

dose
detector

X-ray
detector PIXE
Low Energy
Na to Fe





NewAGLAE



1/ Multi-detector

2/ Systematic imaging

3/ Automation of the beamline



• Upgrades

Increase of detection solid angles
- better sensitivity for trace elements

- decrease of particle flux

→ Study of fragile materials

▶ PIGE :1 → 4 HPGe or CdTe

▶ PIXE - EDS:

2 Si(Li) (1 BE 10 mm² + 1 HE 30 mm²)

→ 5 SDD 50 mm²

▶ RBS : annular detector and/or external detector

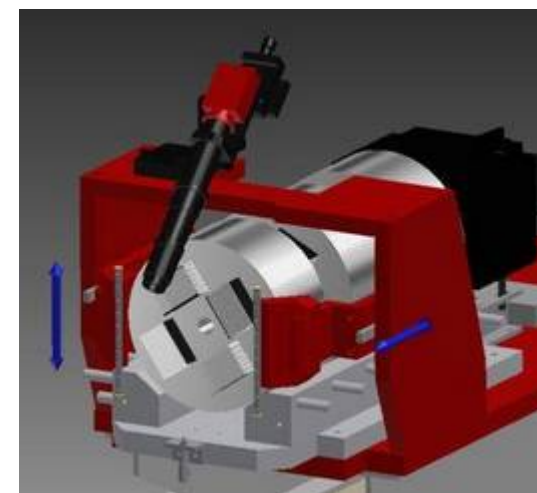
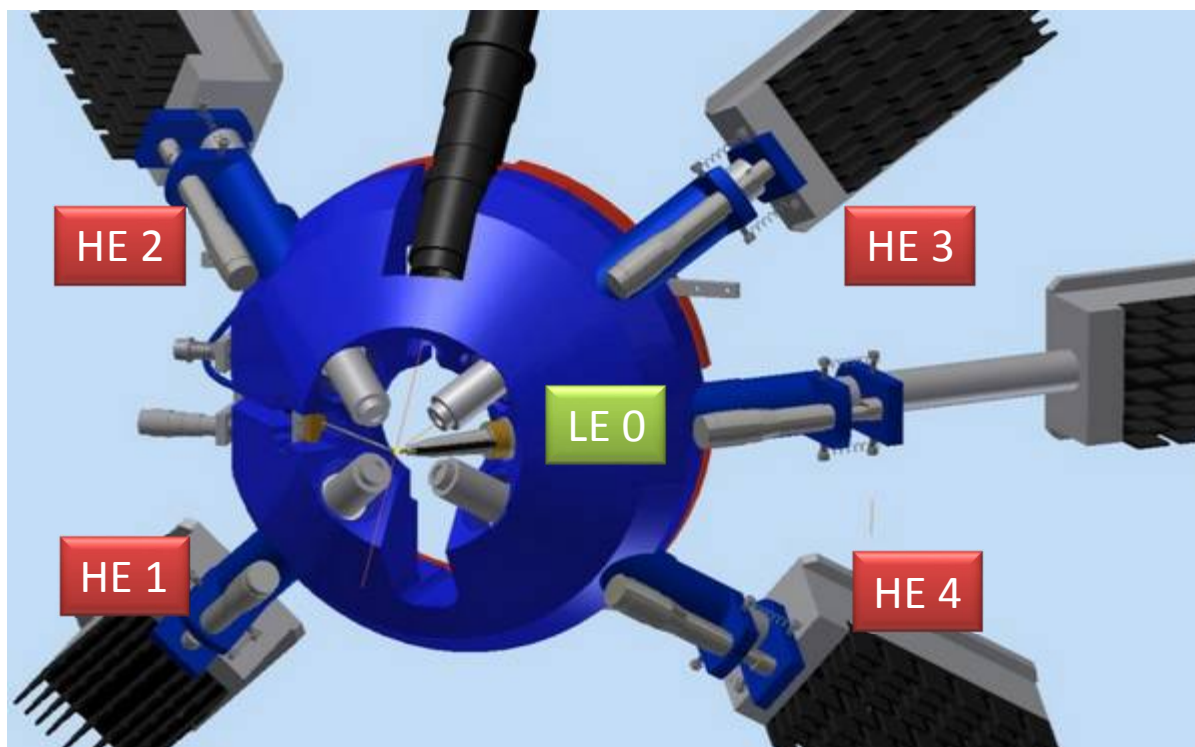
▶ Dose detector

• Novelties

• PIXE – WDS

• Iono-luminescence

• Systematic imaging



Support

- Design of aluminum support
- HE 1-4, 50° relative to beam axis , Distance: 20mm to 45mm
- LE0, 45° relative to beam axis

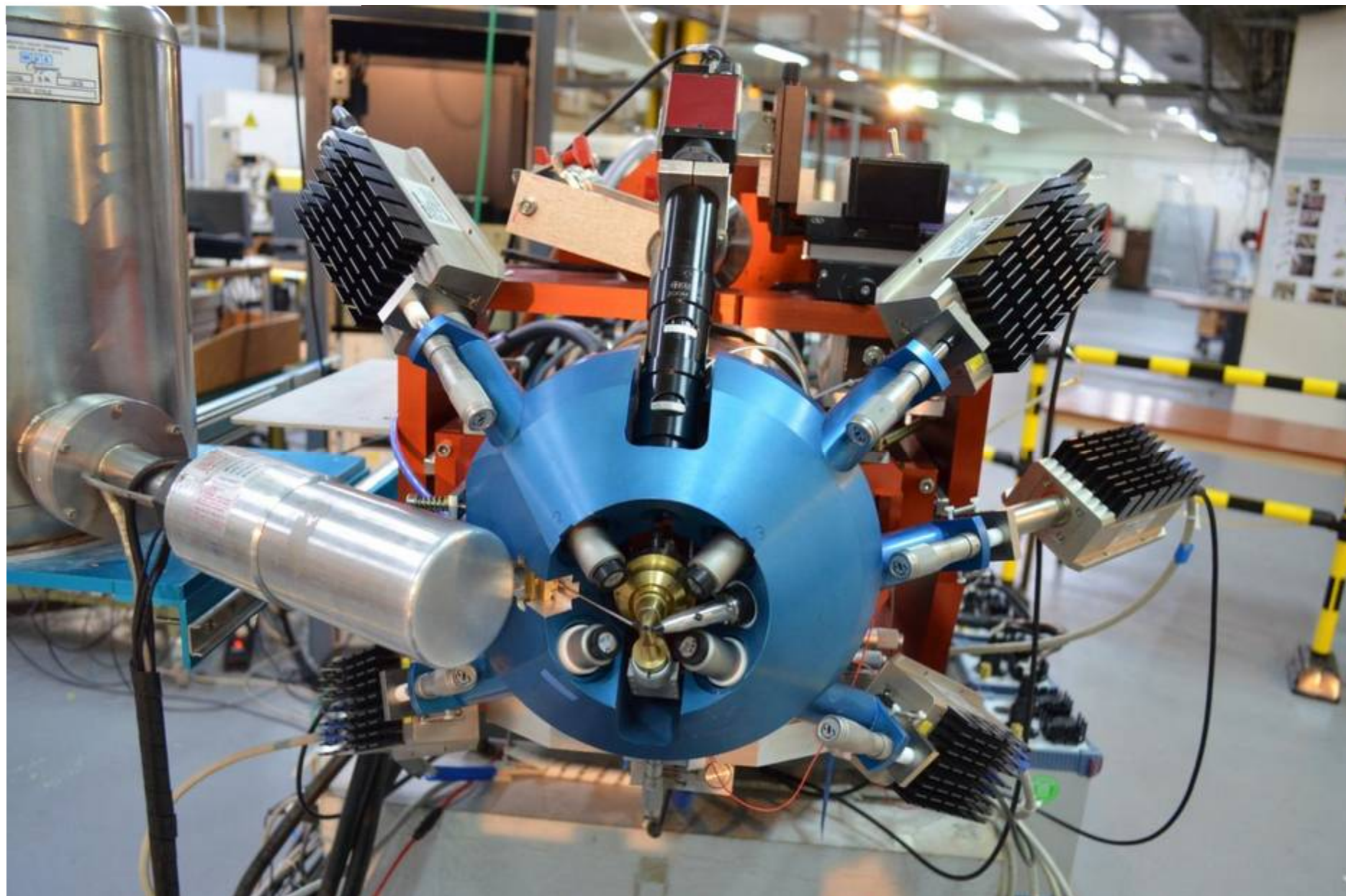


NewAGLAE

1/ Multi-detector

2/ Systematic
imaging

3/ Automation of
the beamline





• Upgrades

Increase of detection solid angles
- better sensitivity for trace
elements - decrease of particle
flux

→ Study of fragile materials

▶ PIGE : 1 → *Study in progress*
Ge or CdTe

▶ PIXE - EDS:

2 Si(Li) (1 BE 10 mm² → *Operational* HE 30 mm²)

→ 5 SDD 50 mm²

▶ RBS : annular detector and/or
external detector *Operational*

▶ Dose detector

• Novelties

• PIXE – WDS

Study in progress

• Iono-luminescence

Operational

• Systematic imaging



NewAGLAE

1/ Multi-detector

2/ Systematic
imaging

3/ Automation of
the beamline

5 SDD detectors
(Ketek)

2 x 4-channel DSP
Mercury-4 (XIA)

Listmode data MPA-3 acquisition
system (FastComtec)



- Customize firmware of the Mercury-4 DXP to provide standard ADC interface (Data and protocol)
- Digital processor allows high output count rate
30k cps per channel (30% DT) → 150k cps total
- HE1-4 : 145 eV resolution
- LE0 : 135 eV resolution

Nine channels :

- 5 - SDD
- 1 - RBS
- 1 - Gamma
- 1 - X positions
- 1 - Y positions



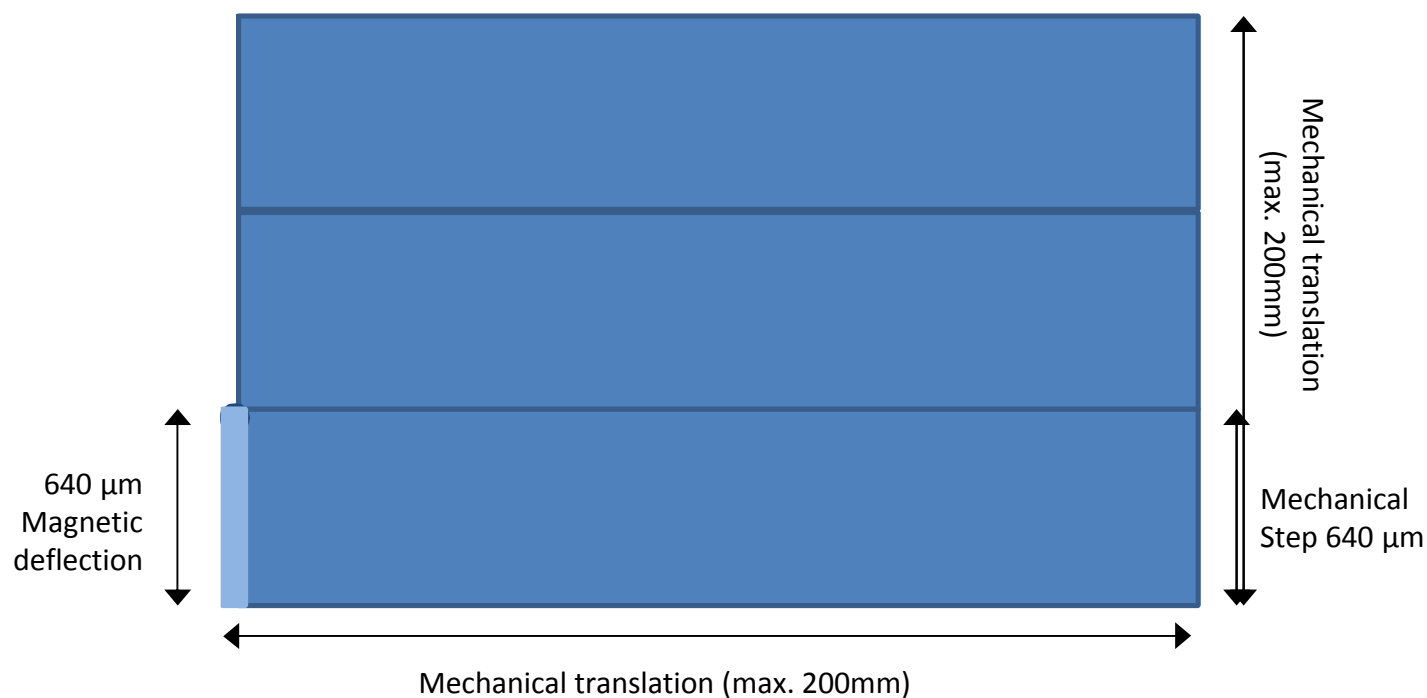
NewAGLAE

1/ Multi-detector

2/ Systematic
imaging

3/ Automation of
the beamline

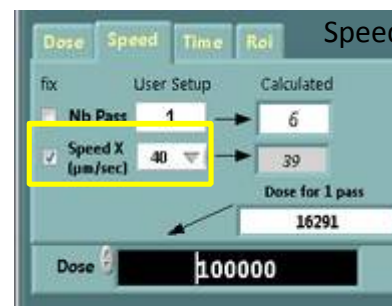
Vertical / Horizontal scanning



Two ways for mapping



- Dose monitoring by column
- Homogeneous dose/pixel



- No dose monitoring
- Fast map
(max. 500μm /sec)

The case study of ivories from Arslan Tash site, Syria, 8th c. BC

I. Reiche¹, M. Albéric¹, E. Apchain¹, K. Müller¹,
C. Heckel², R. White², H. Floss³, E. Fontan⁴,
L. Pichon⁵, Q. Lemasson⁵, B. Moignard⁵, C. Pacheco⁵

¹ LAMS UMR 8220 CNRS UPMC Paris VI, Paris, France

² Center for the Study of Human Origins, Department of
Anthropology, New York University, USA

³ Institut für Ur- und Frühgeschichte, University of Tübingen,

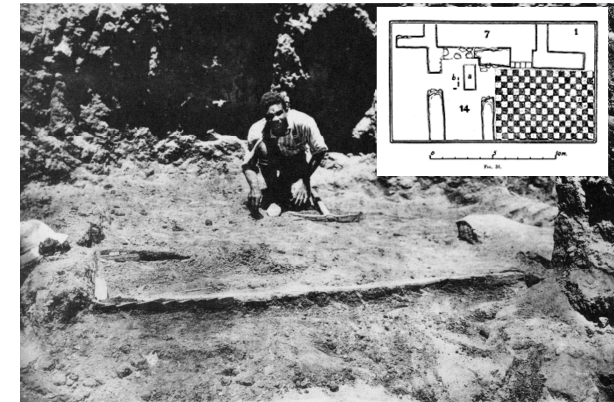
⁴ Département des Antiquités Orientales, Musée du Louvre,
Paris, France

⁵ C2RMF, Paris, France and Fédération de Recherche New
AGLAE (FR3506 CNRS)

With additional contributions of T. Borel,
D. Bagault, S. Pagès-Camagna, D. Large (C2RMF)
A. Caubet, S. Cecchini, F. Poplin, M.G. Amadasi

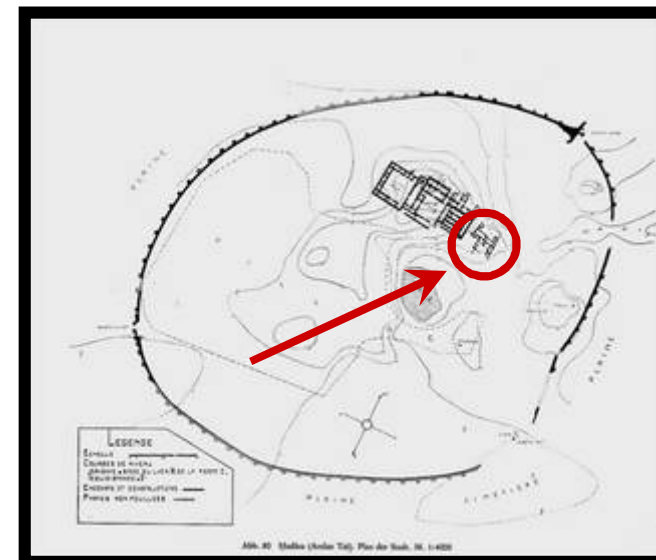


The archaeological site of Arslan Tash, Syria



Aim of the study

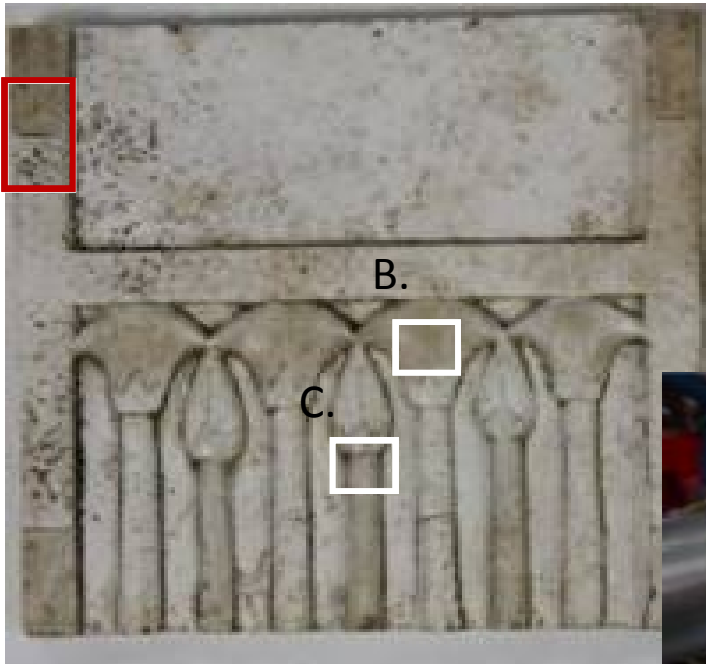
- ➔ Reconstructing the original appearance of the object
- ➔ Defining attribution criteria to the Arslan Tash site



PIXE chemical imaging

(enhancement factor)

A.

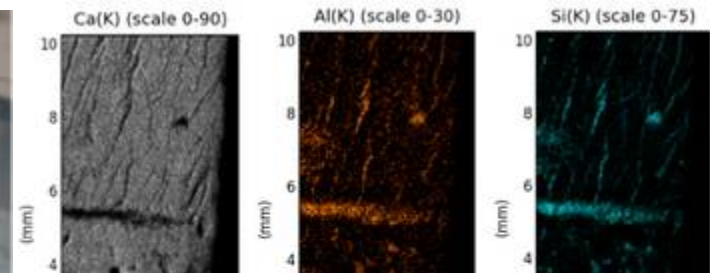


B.

C.



A.: analyzed surface: $4.8 \times 10.2 \text{ mm}^2$
step size: $20 \times 20 \mu\text{m}^2$



The papyri AO 11479 – Louv21

Ca distribution: indicative of surface stat
Al, Si, Fe: indicative of sediment traces
Cu and Pb: ancient polychromy

Position	CaO	Fe ₂ O ₃	CuO	ZnO	PbO	PbO/Cu
zone A	512906	7260	1750	966	383	0,22
zone B	546188	2238	2964	532	1133	0,38
zone C	546265	937	2513	394	805	0,32

Paints: different ratios of lead white
and a Cu pigment like Egyptian blue

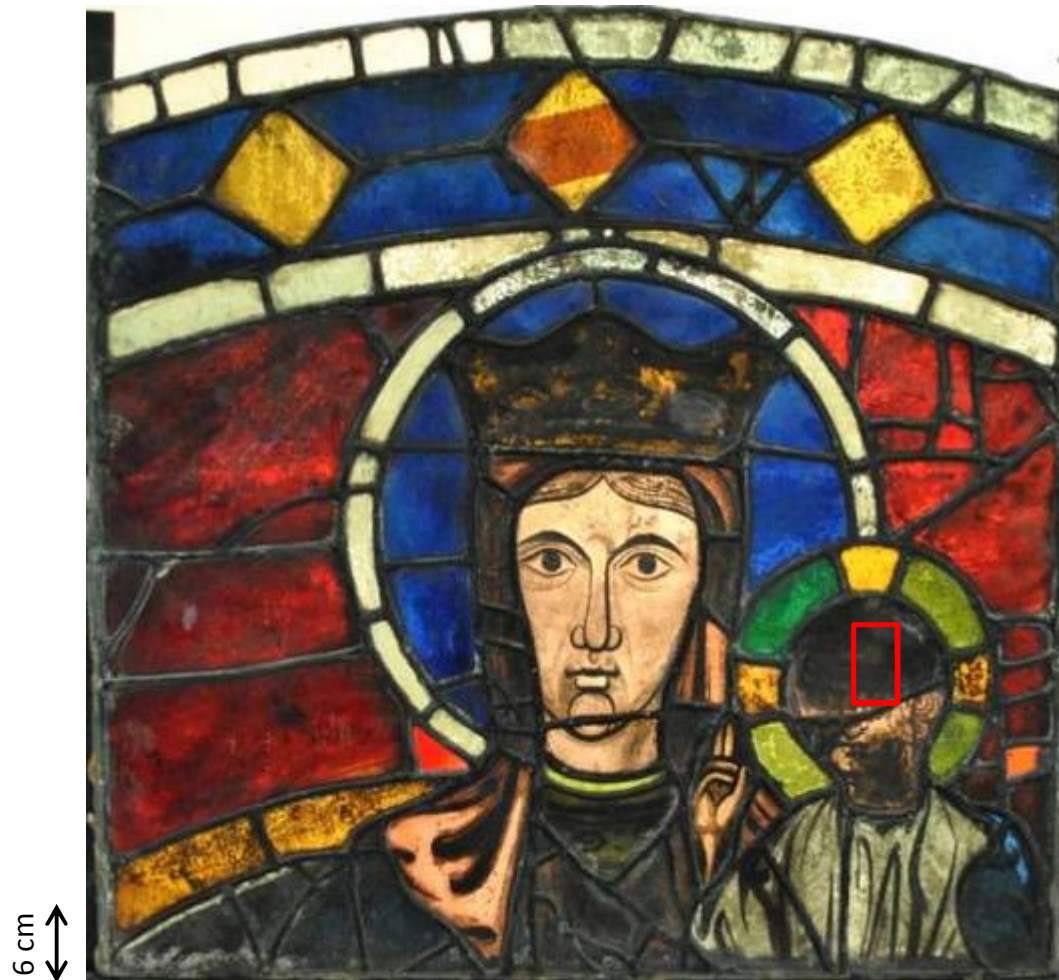
Albéric *et al.*, 2015, Talanta 137, 100-108

Fontan *et al.*, 2011, Archeosciences 35, 283-295

Possible reconstruction



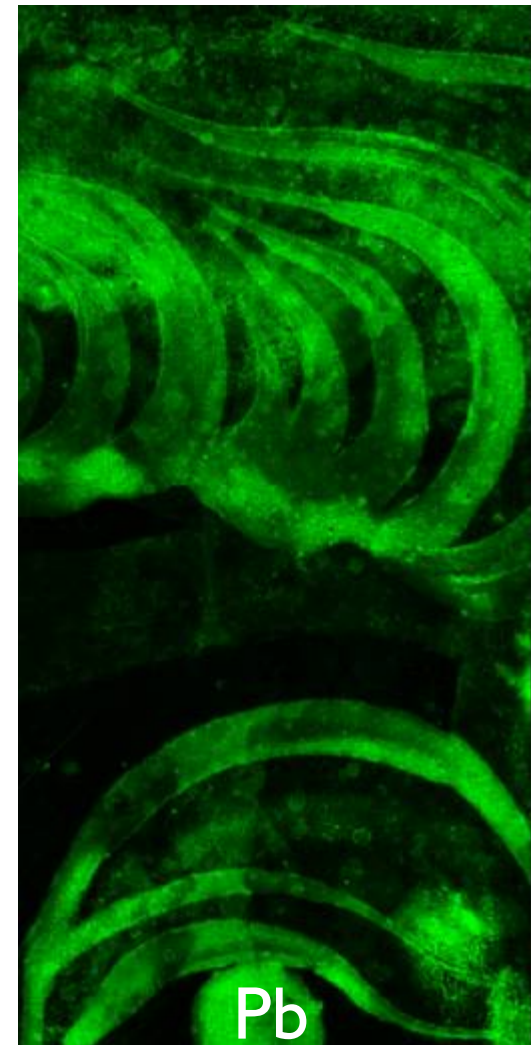
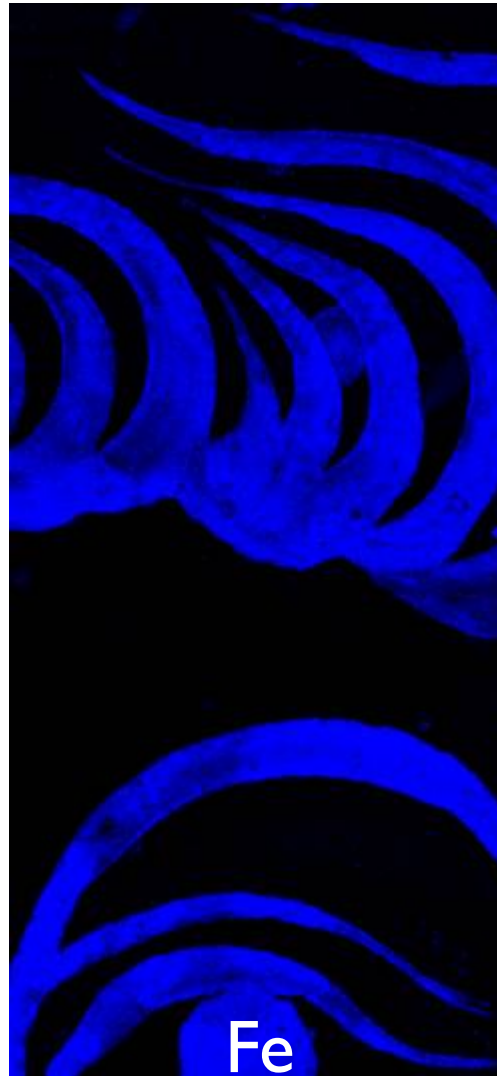
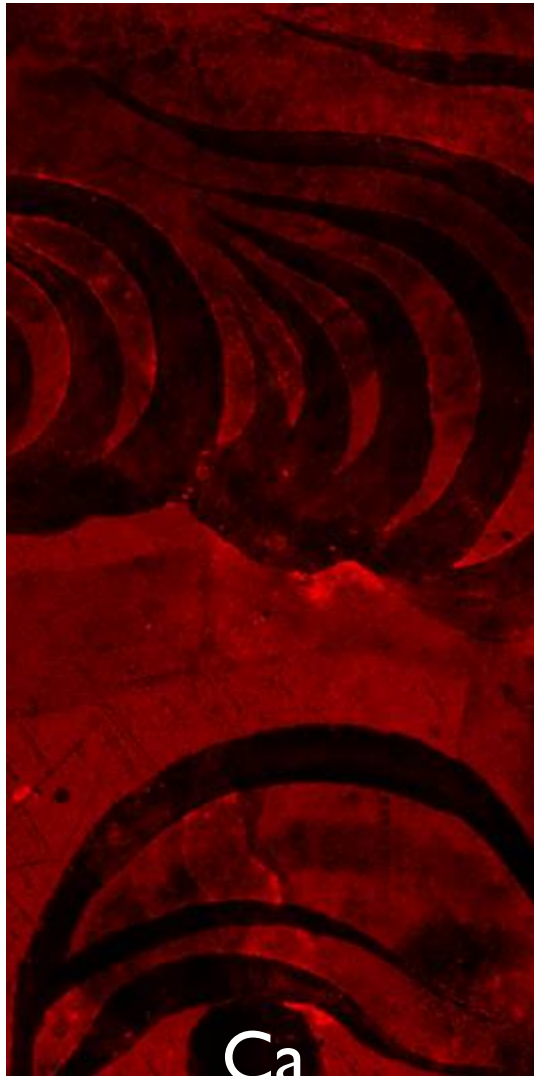
Stained glass window - Chartres Cathedral – 12th - 13th c.



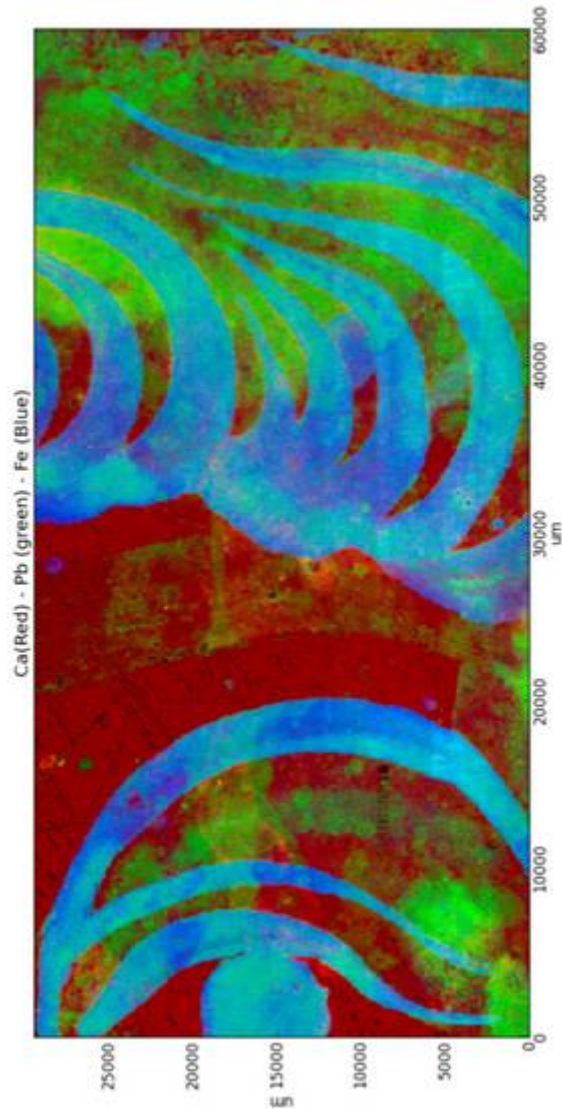
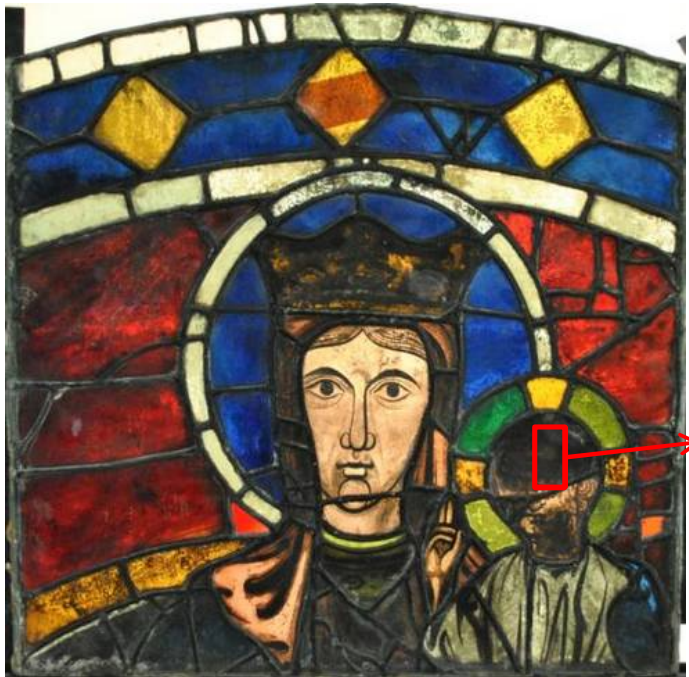
Grisaille: lead silicate +
metallic oxides/salts + firing

Lavis: diluted grisaille or
only lead silicate ?

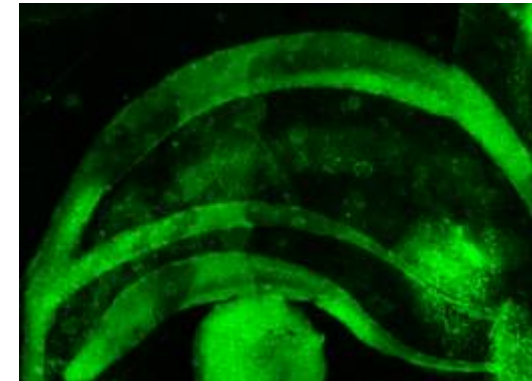
Stained glass window - Chartres Cathedral – 12th - 13thc.



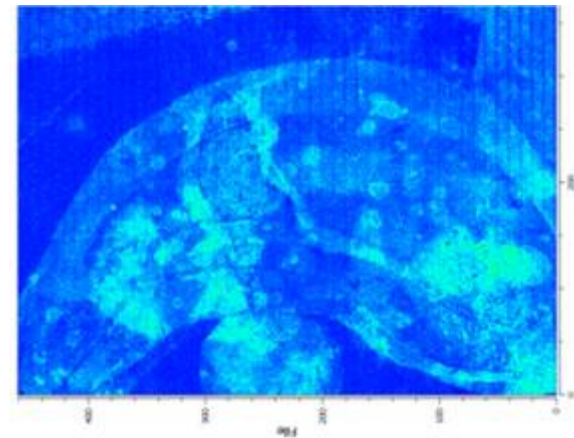
Stained glass window - Chartres Cathedral – 12th - 13thc.



Pb PIXE



RBS





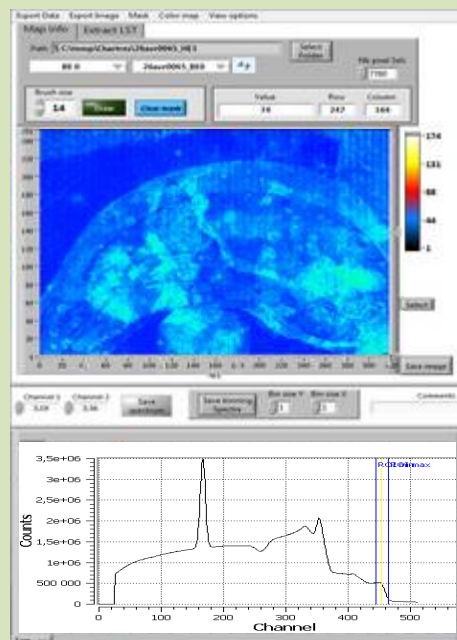
NewAGLAE

1/ Multi-detector

2/ Systematic
imaging

3/ Automation of
the beamline

Methodology for (R)BS Mapping @ AGLAE



RBS
Mapping
Tool

NDF
engine

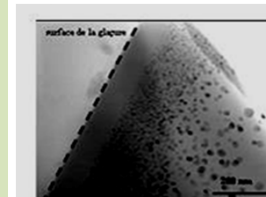
- Batch mode
- PIXE – PIGE
- roughness models

Every RBS
spectrum/pixel
fitted

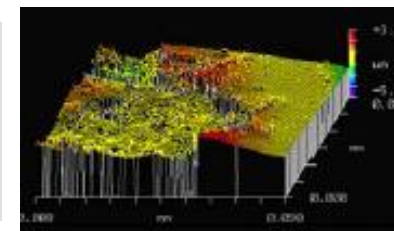
DatImaging

Imaging reconstruction with data
extracted from RBS fitted spectra

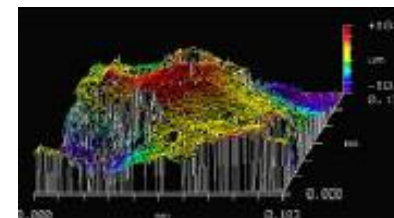
nm scale



μm scale



... and above





NewAGLAE

1/ Multi-detector

2/ Systematic
imaging

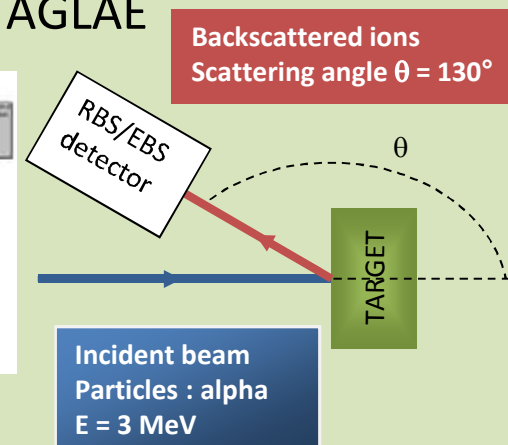
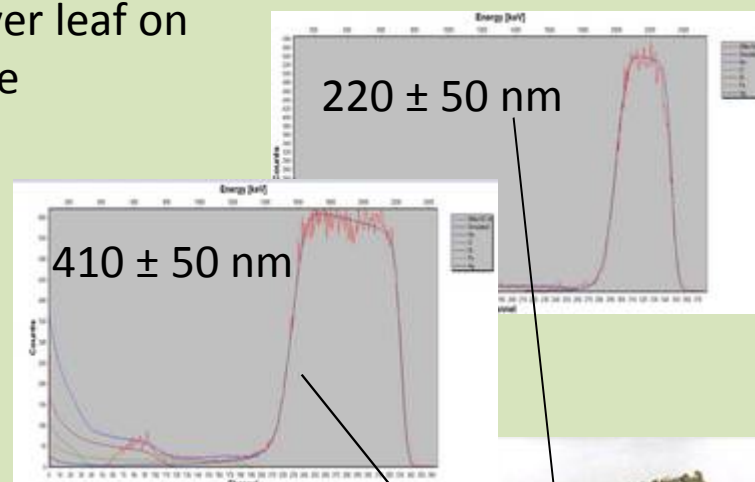
3/ Automation of
the beamline

Simple model to test the feasibility of the methodology for (R)BS Mapping @ AGLAE

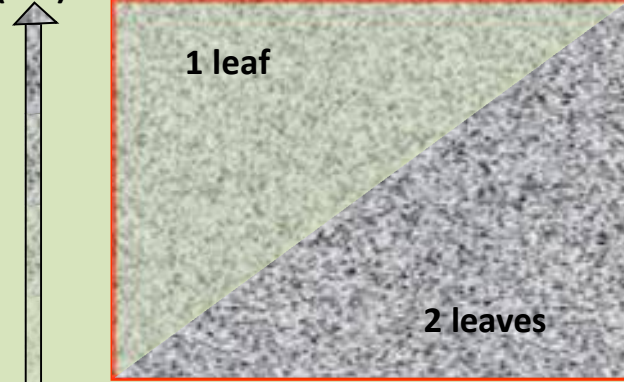
Simple model = folded silver leaf on
a microscope slide



Berta Battiloro - Venice



Thickness
(nm)



Projection of the expected
thickness mapping



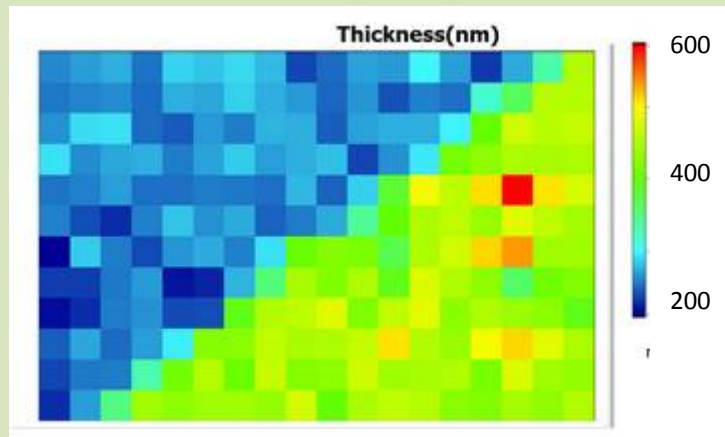
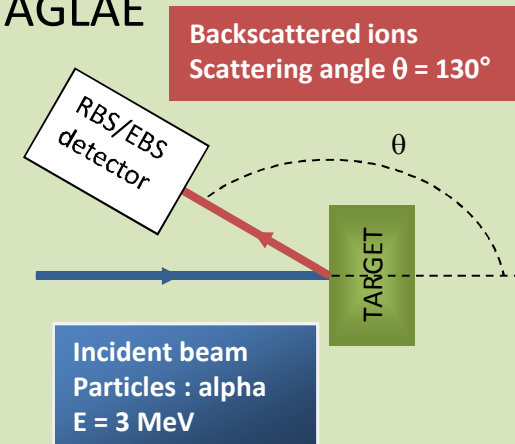
2 x 3 cm²





Simple model to test the feasibility
of the methodology for (R)BS Mapping @ AGLAE

- Construction of NDF batch :
each pixel imported as a sample
- 1 sample is fitted by annealing
- fit OK \rightarrow local search for the whole batch
- reconstruction of the image from
fitted concentration depth profile



2 x 3 cm²





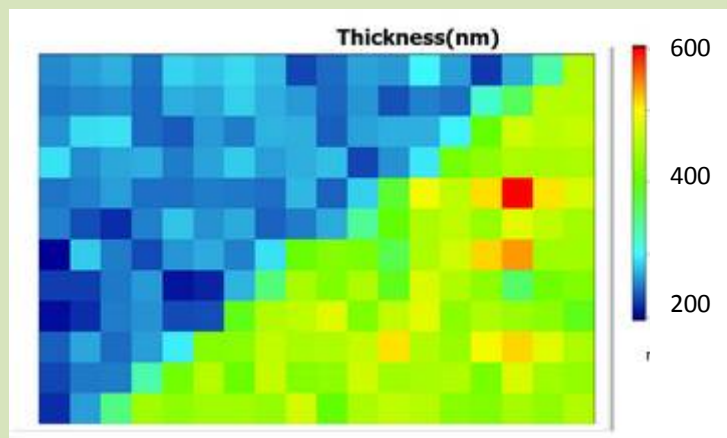
Simple model to test the feasibility
of the methodology for (R)BS Mapping @ AGLAE

- Construction of NDF batch :
each pixel imported as a sample
- 1 sample is fitted by annealing
- fit OK → local search for the whole batch
- reconstruction of the image from
fitted concentration depth profile

Batch mode: limited
number of pixels

TrauNDF

NDF is simultaneously
launched on several
processors





NewAGLAE

1/ Multi-detector

2/ Systematic
imaging

3/ Automation of
the beamline

A new philosophy...

... that must adapt to long-term projects !



Exchange networks of variscite during Neolithics

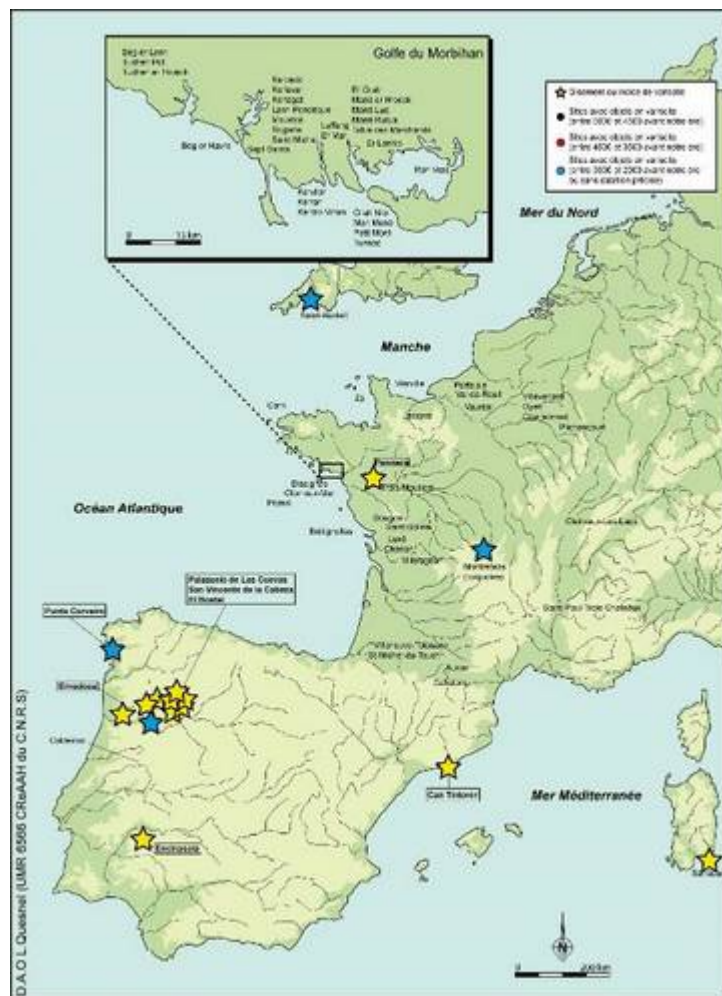
G. Querré¹, Th. Calligaro², S. Cassen³, S. Domínguez-Bella⁴

¹ CREEAAH, Université de Rennes, France

² C2RMF, Paris, France

³ CREEAAH, Université de Nantes, France

⁴ Universidad de Cadiz, España



Various ores and deposits of variscite and turquoise are known in occidental Europe.



Exchange networks of variscite during Neolithics

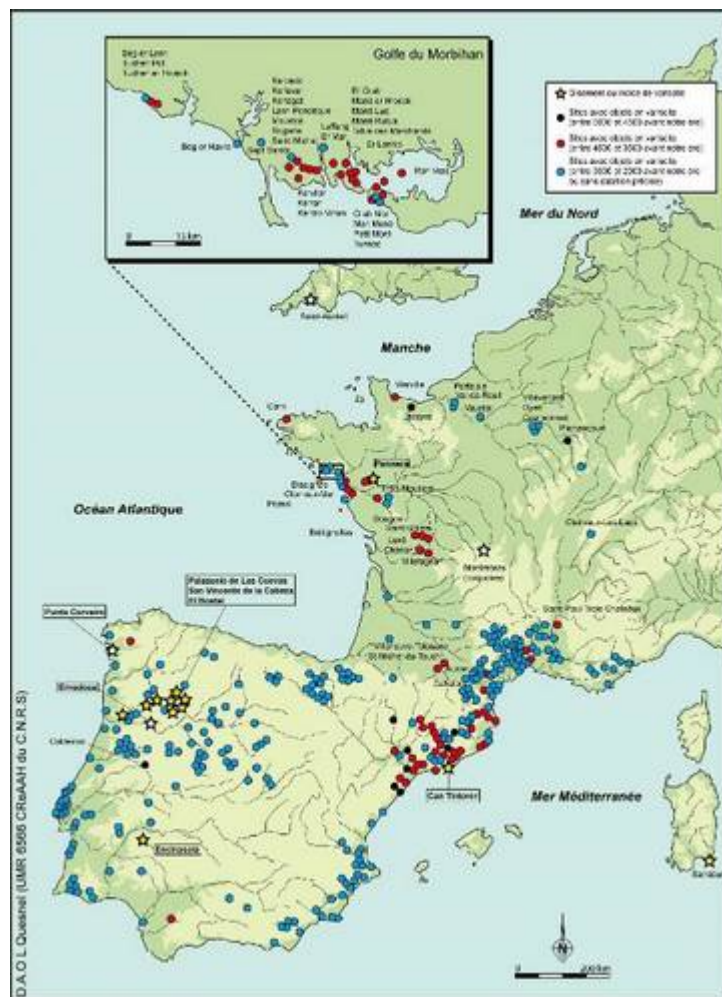
G. Querré¹, Th. Calligaro², S. Cassen³, S. Domínguez-Bella⁴

¹ CREEAAH, UMR 6566 CReAAH, France

² C2RMF, Paris, France

³ CREEAAH, Université de Nantes, France

⁴ Universidad de Cadiz, España



Prehistoric sites with jewels made from variscite.



Exchange networks of variscite during Neolithics

VARISCITE

Chemical composition: $\text{Al PO}_4 \cdot 2\text{H}_2\text{O}$
 system: orthorhómbic grup of phosphate, arsenite,
 vanadate $(\text{XO}_4) \cdot 2\text{H}_2\text{O}$ A: $\text{Fe}^{3+} + \text{o Al}$; X: P o ACE



Punta Conveiro



Palazuelo



Encinasola



Pannecé

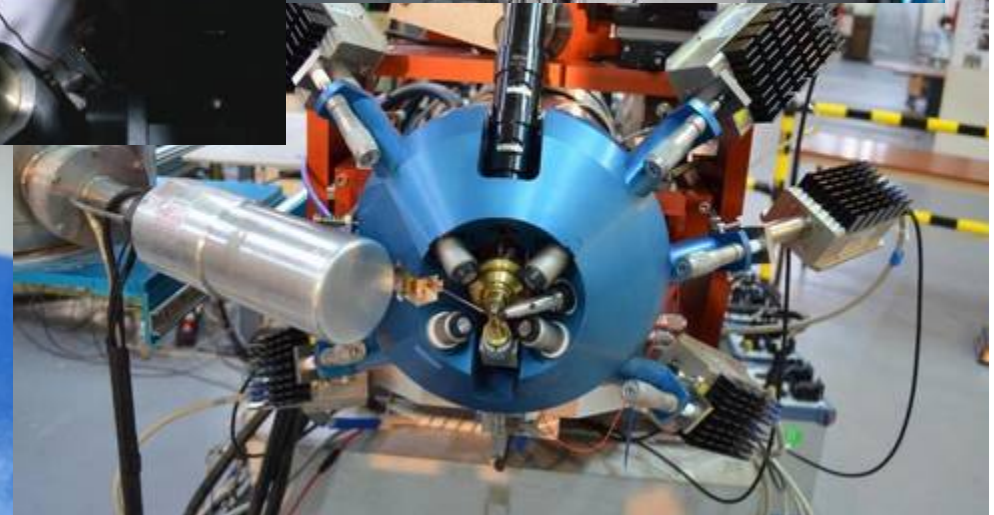
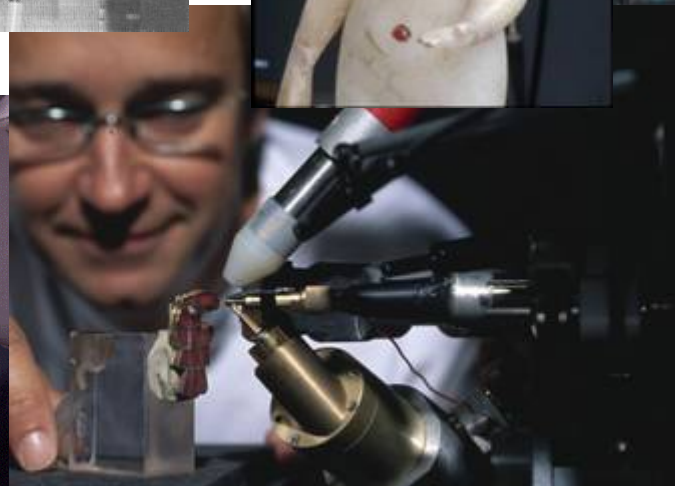
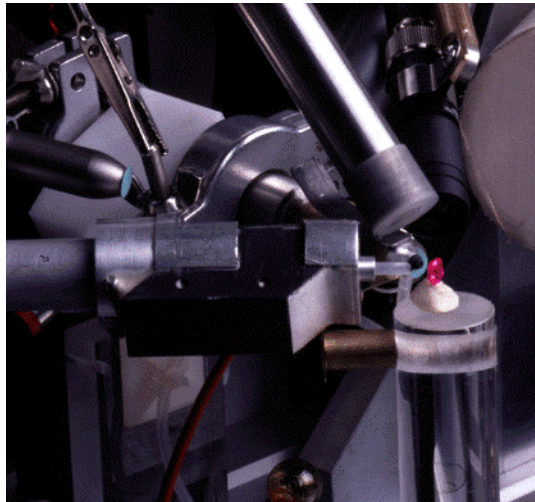
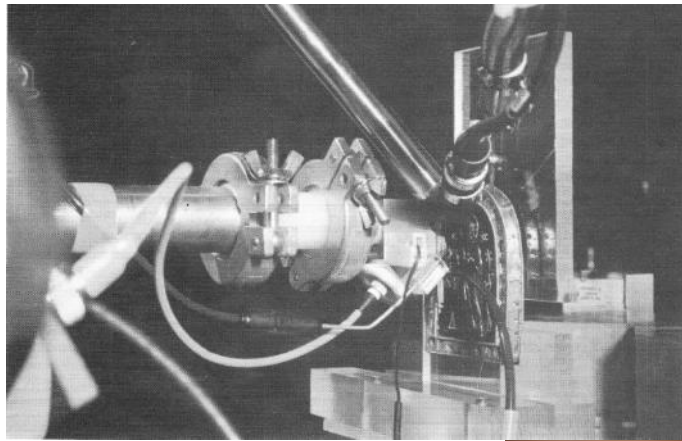


Ervedosa

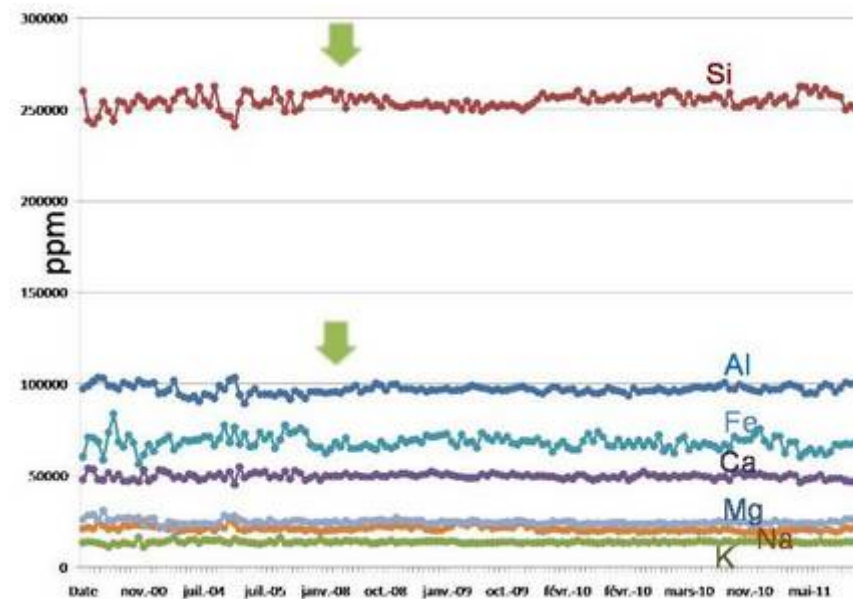


Gavà

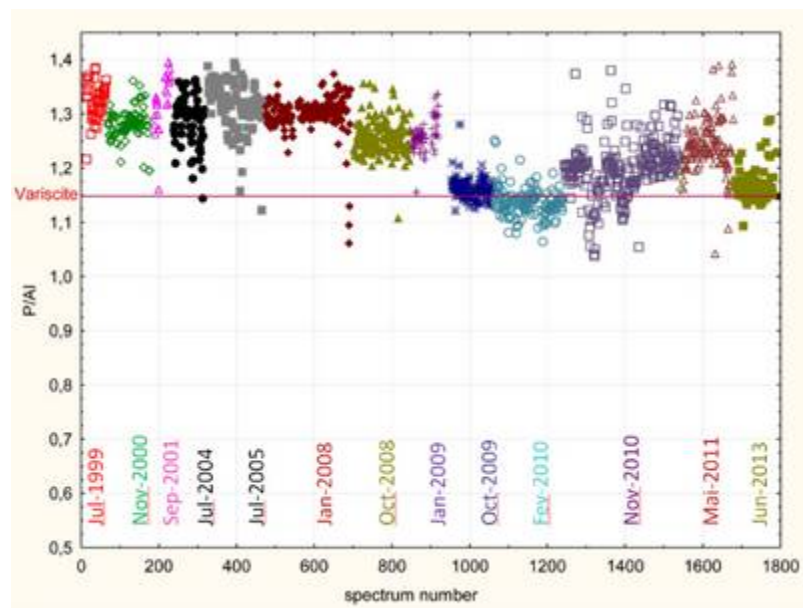
Variscite has been studied at the AGLAE for 15 years...



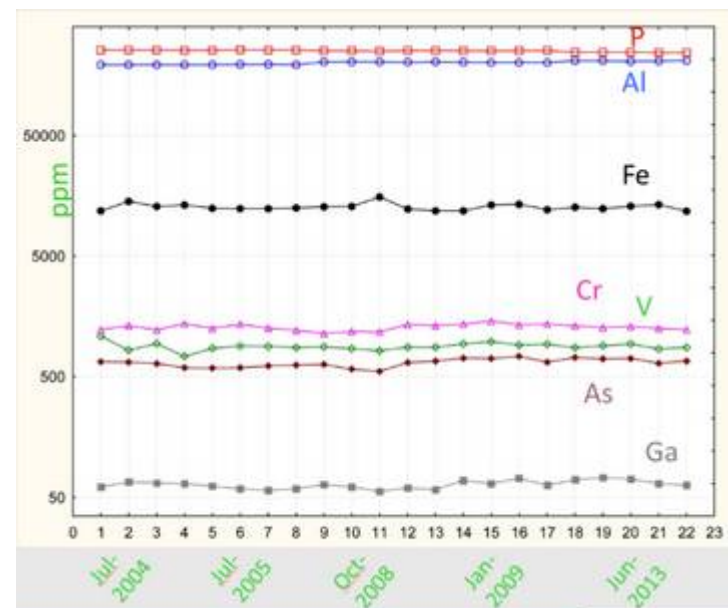
Evolution of PIXE results through time...



International standard Dr-N



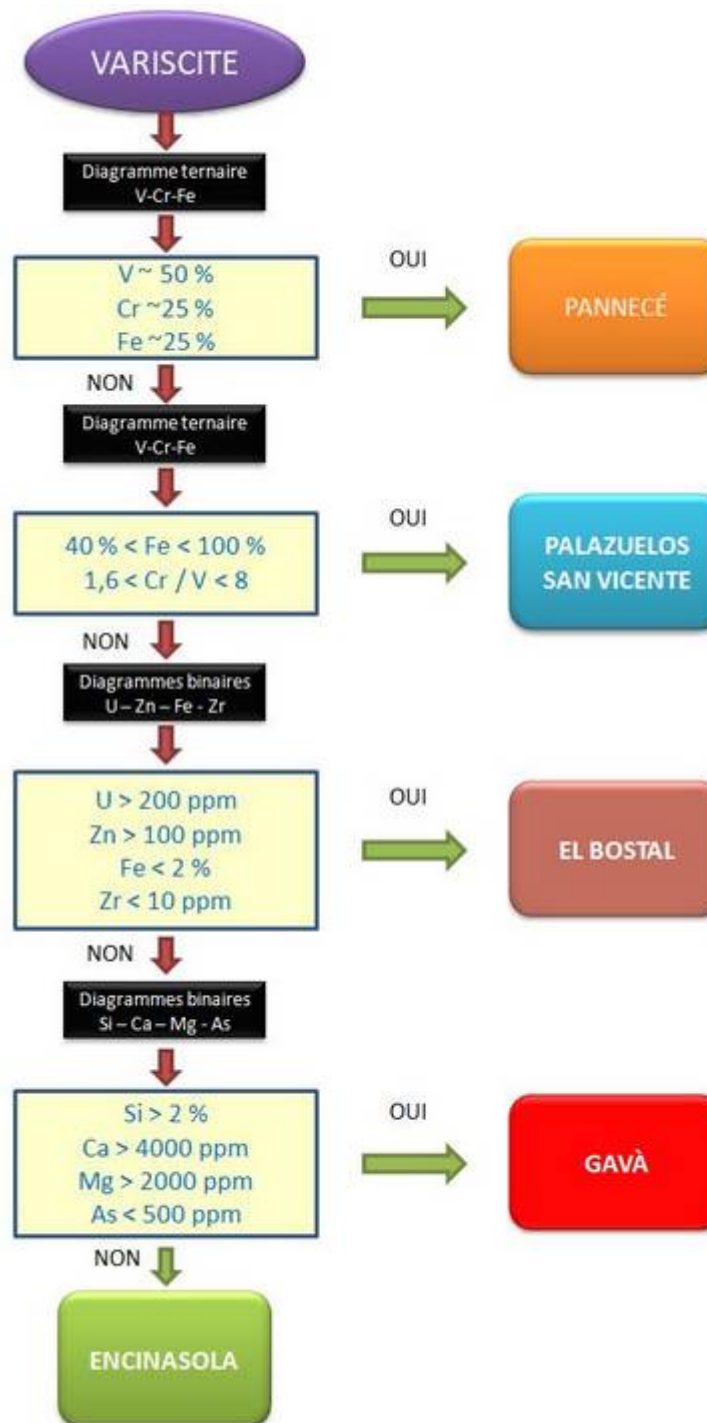
Variscite samples



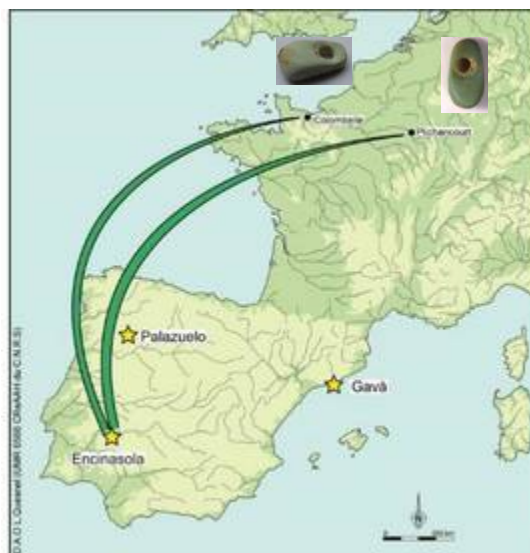
Variscite standard MeH

Development of a chemiometric model for provenance

Determination of the geochemical characteristics of each variscite ore



Evolution of the provenance of variscite found in France during Neolithic period



5.000 – 4.800 av. J.C.



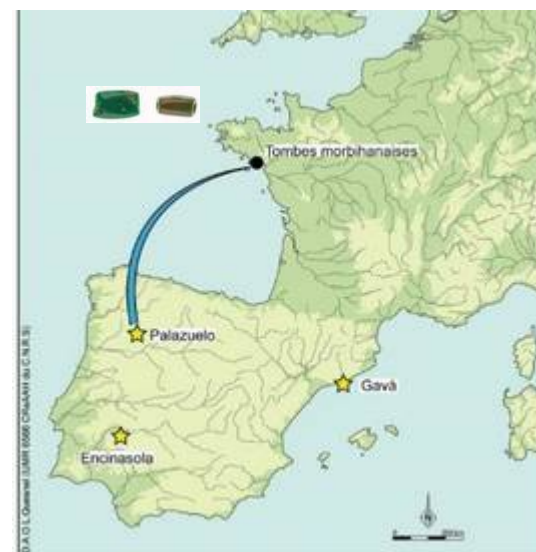
4.700 – 4.300 BC.



4.300 – 4.000 BC



4.000 – 3.800 BC



3.500 – 3.300 BC

Evolution of the provenance of variscite found in France during Neolithic period



4.000 – 3.800 BC



4.700 – 4.300 BC.



4.300 – 4.000 BC



3.500 – 3.300 BC



NewAGLAE

1/ Multi-detector

2/ Systematic
imaging

3/ Automation of
the beamline

- Purposes:

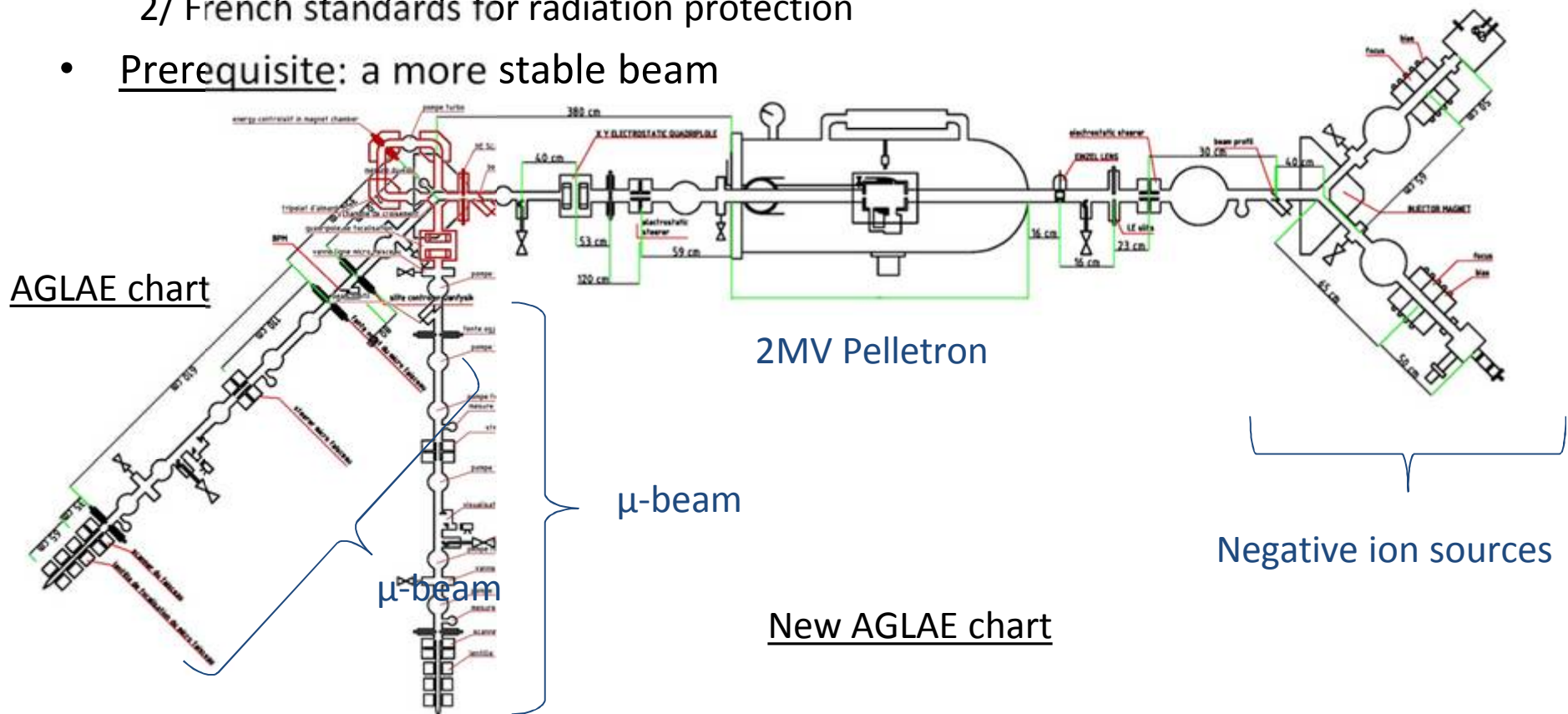
- 1/ More proposals → increase of beamtime

- By day: objects from museum collections

- By night: samples series, long mapping acquisition, etc. → remote command and control system

- 2/ French standards for radiation protection

- Prerequisite: a more stable beam





NewAGLAE

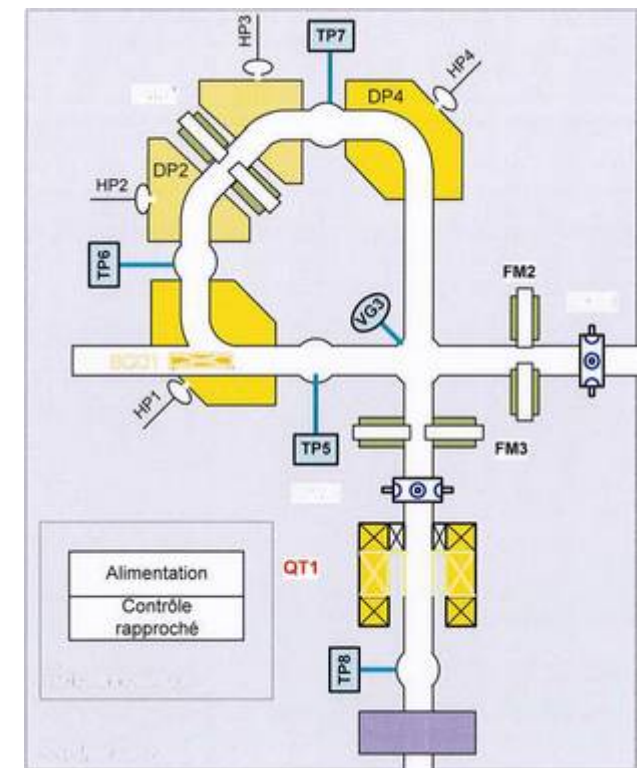
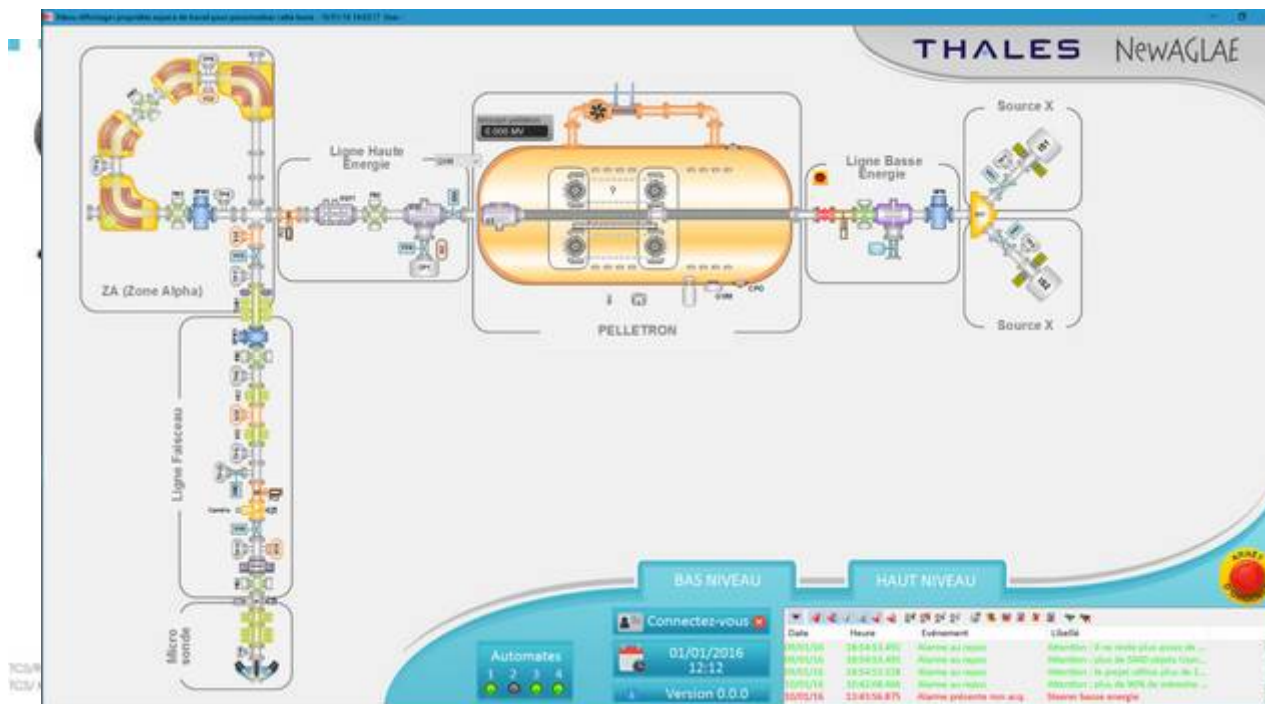
1/ Multi-detector

2/ Systematic
imaging

3/ Automation of
the beamline

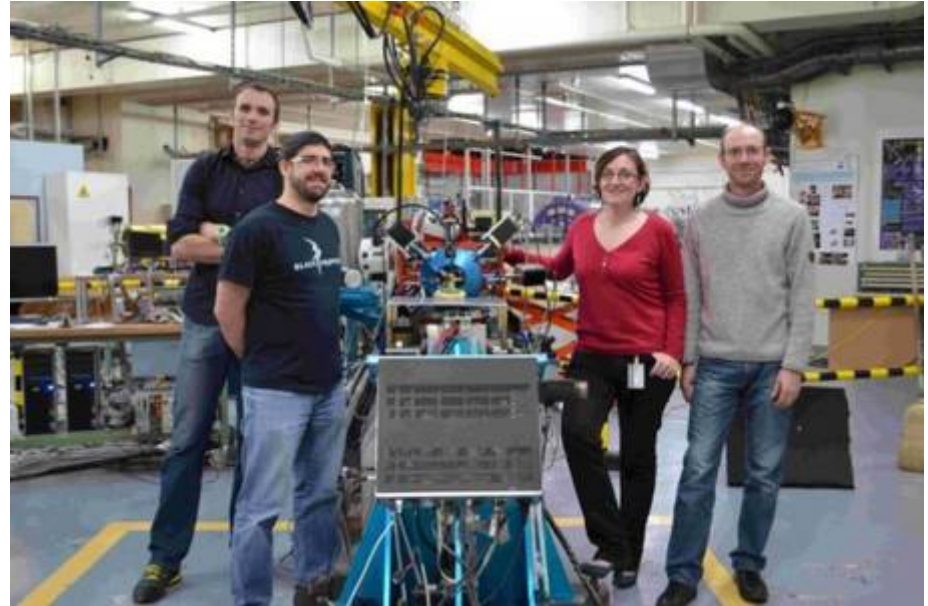
Thalès company won the bid with the following proposal:

- The Terminal Voltage is controlled through a digital system directly integrated to industrial automated machine
- New devices for the alpha-zone:
 - Two 90° magnets
 - Two 45° magnets
 - A magnetic quadrupole triplet
- A custom-made HMI



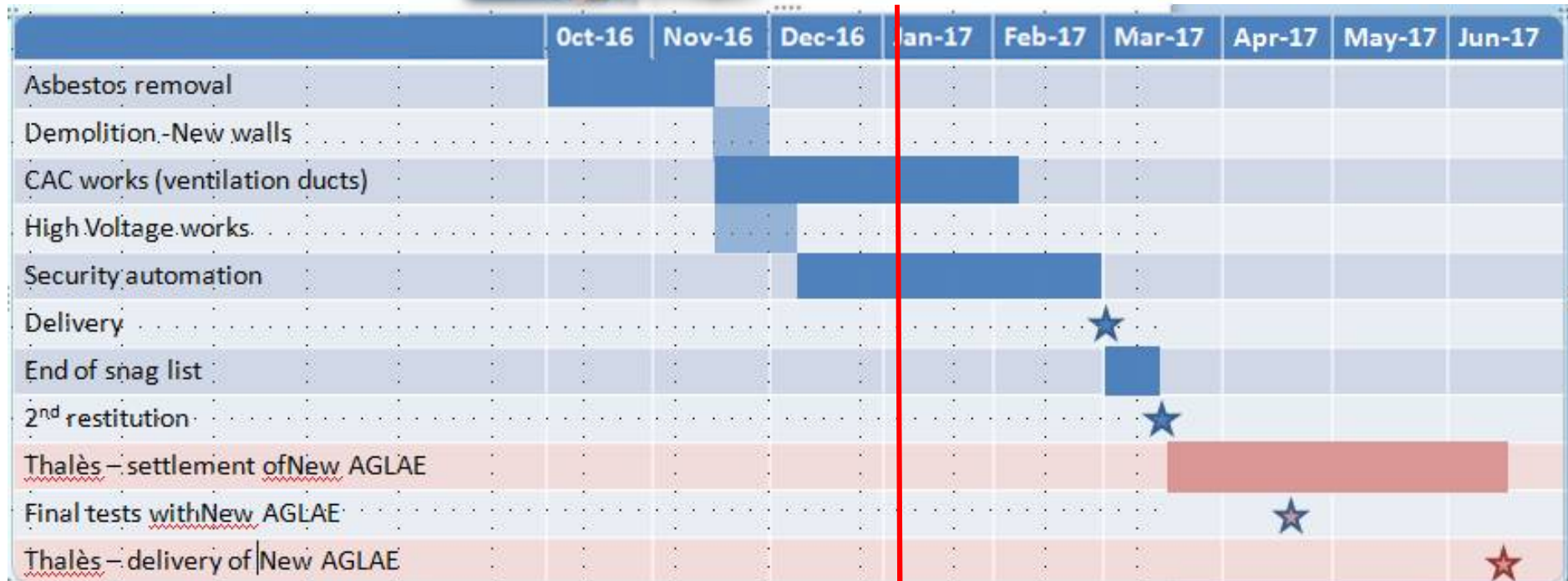


NewAGLAE





NewAGLAE



New AGLAE Opening to users



Acknowledgement

- The AGLAE team: Quentin Lemasson, Brice Moignard & Laurent Pichon
- Thomas Calligaro, Isabelle Biron, Rolland Février, Marie Radepont, Sandrine Pagès, Brigitte Bourgeois
- Ina Reiche
- Laurianne Robinet
- Claudine Loisel
- Julien Colaux
- Guirec Querré

Funding

- ANR-10-EQPX-22
- Ville de Paris
- Rennes Metropole

Thank you for your attention

Every science begins as philosophy and ends as art...

Will Durant (1885-1981)

