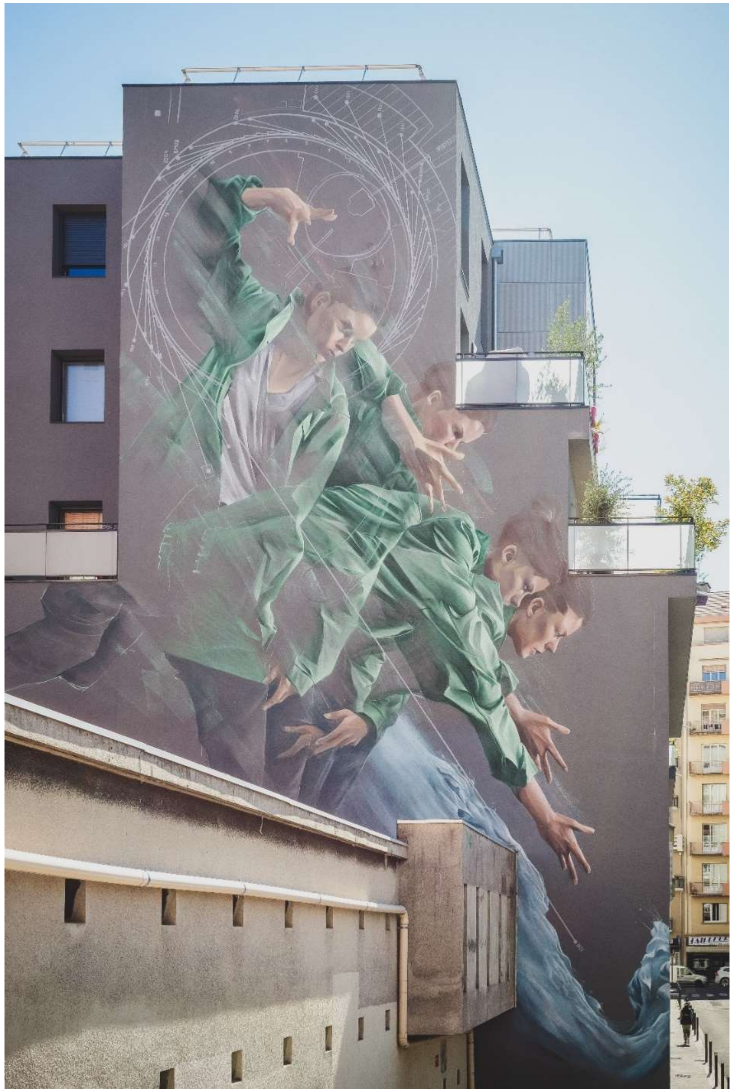


Li-Hill, 2019, Grenoble

Cultural Heritage at the ESRF:
from the discovery of masters' secrets to the conservation of artworks

M. Cotte[1,2]

1- ID21, European Synchrotron Radiation Facility, 71 av. des martyrs 38000 Grenoble, France
2- Sorbonne Universités, UPMC Univ Paris 06, CNRS, UMR 8220, Laboratoire d'archéologie moléculaire et structurale (LAMS), 4 place Jussieu 75005 Paris, France

Acknowledgments: all staff involved in maintenance and development of instruments, and our users

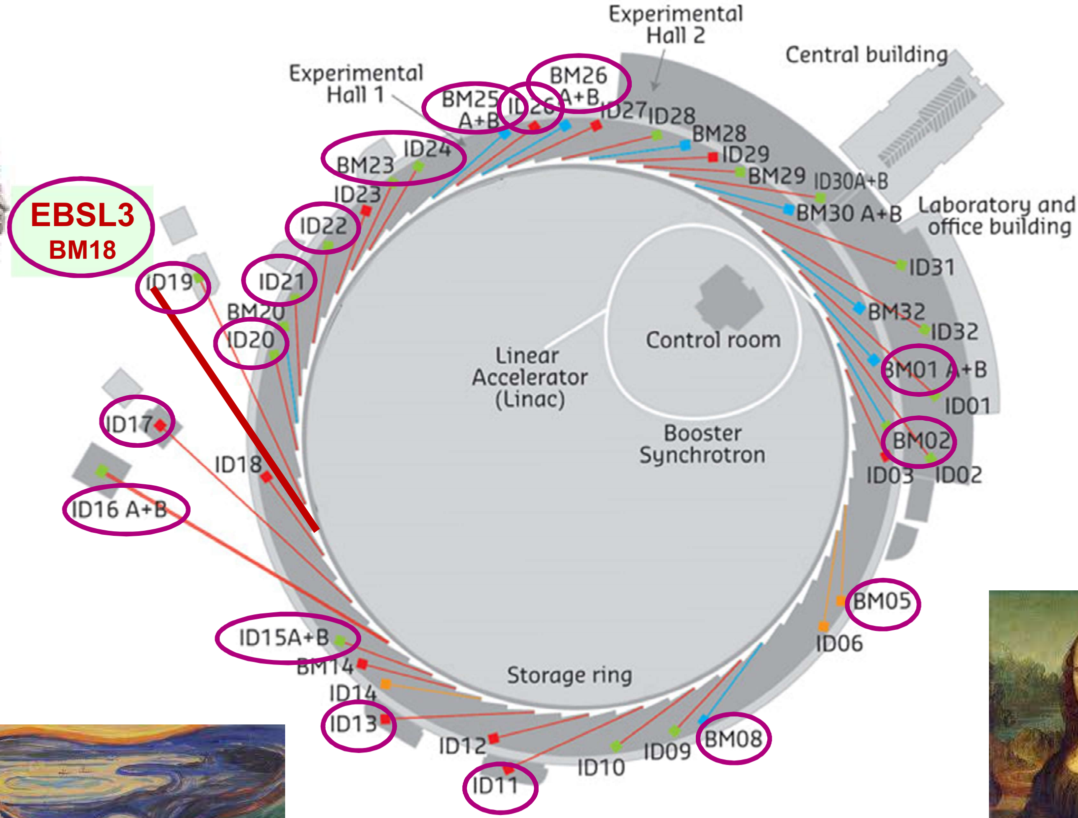
STREAMLINE

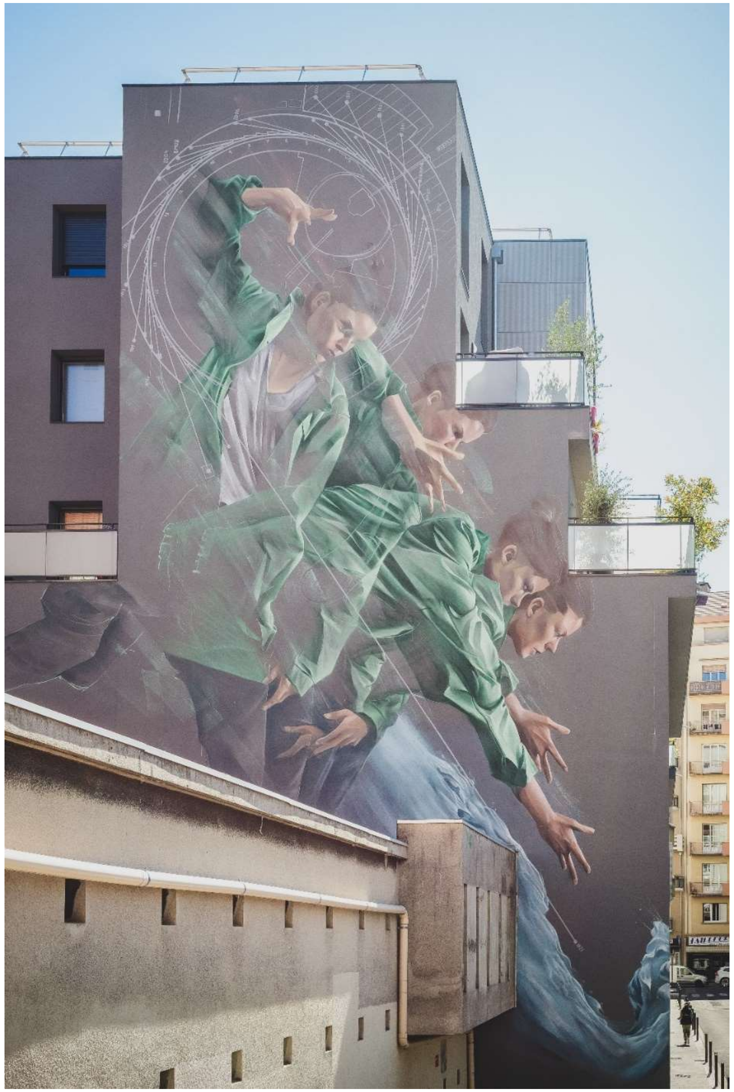
The European Synchrotron | ESRF

HERITAGE AT THE ESRF

Heritage studies via user program

X-ray diffraction
X-ray absorption spectroscopy
X-ray fluorescence
Phase contrast tomography





Li-Hill, 2019, Grenoble

Cultural Heritage at the ESRF:

~~from the discovery of masters' secrets~~

~~to the conservation of artworks~~

Applications to artefacts from Middle East

M. Cotte[1,2]

1- ID21, European Synchrotron Radiation Facility, 71 av. des martyrs 38000 Grenoble, France
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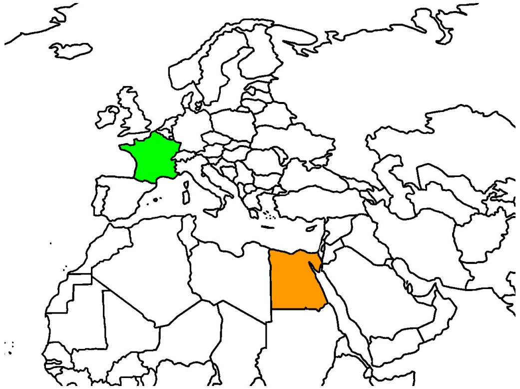
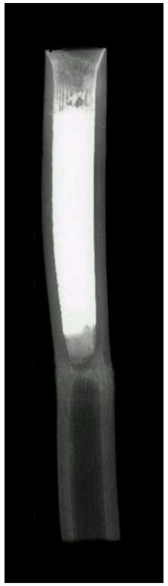
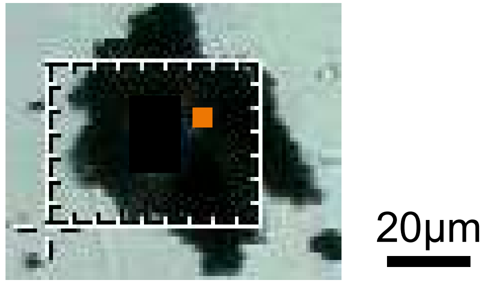
STREAMLINE

The European Synchrotron | ESRF

1- EARLY APPLICATIONS OF ESRF TO HERITAGE: COSMETICS IN ANCIENT EGYPT







WORKS LED BY

Philippe Walter, Center of Research and Restoration of French Museums, Paris

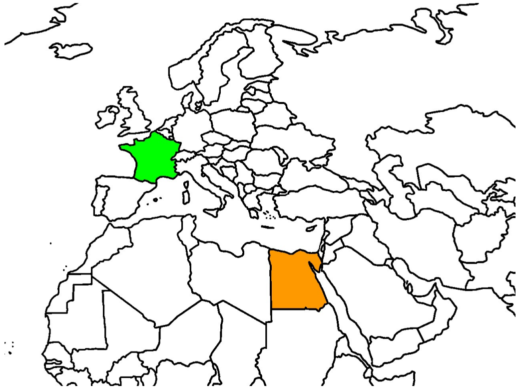
SAMPLES

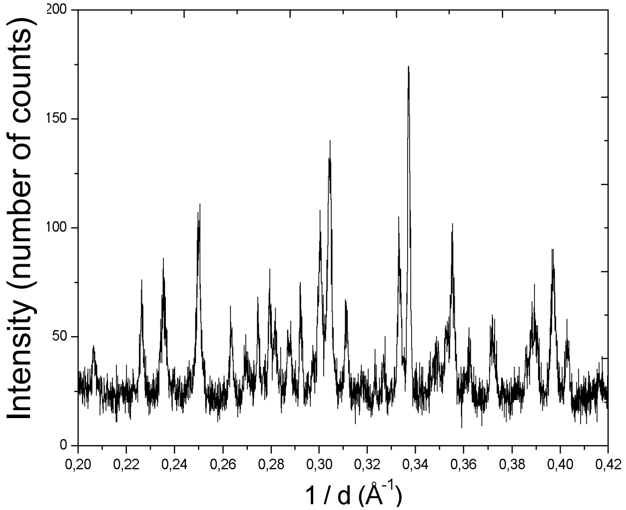
Fragments of cosmetics products, sampled in reeds and vials from the Louvre museum, 18th Dynasty, New Empire (13th C. BC)

QUESTIONS

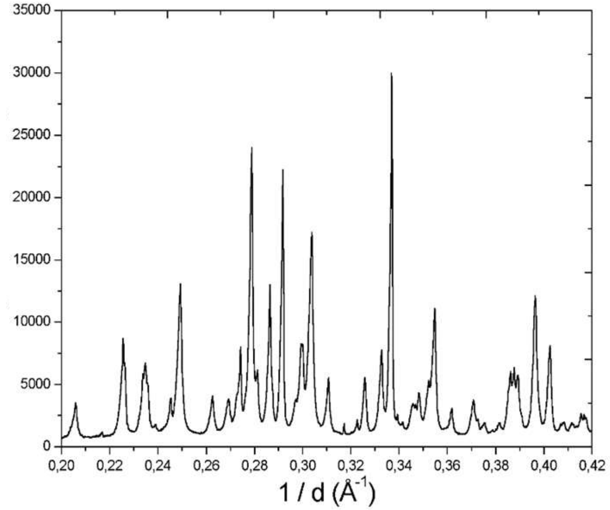
Can the chemical composition of the cosmetic powders reveal pharmaceutical practices and chemical skills in Ancient Egypt?

1- EARLY APPLICATIONS OF ESRF TO HERITAGE: COSMETICS IN ANCIENT EGYPT





63 h of acquisition in laboratory



4 h of acquisition at BM 16

TECHNIQUES

Powder X-ray diffraction (former BM16)
Fourier Transform Infrared microscopy (former LURE)

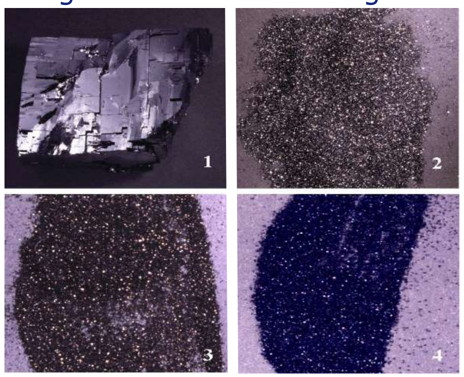
ADVANTAGES OF SYNCHROTRON RADIATION

High data quality even on low quantity
High resolution for mapping of components in mixtures

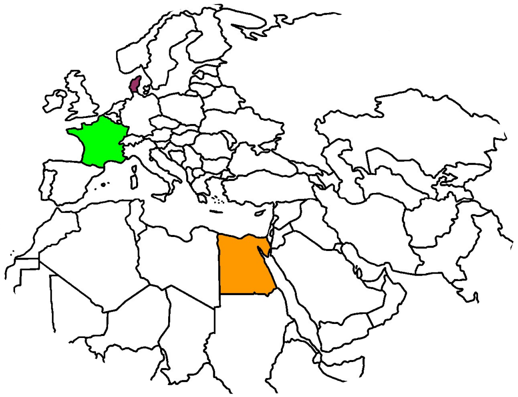
RESULTS

Signature of heat treatment of galena
Identification of lead compounds (laurionite, phosgenite), demonstrating the use of “soft” chemistry (in aqueous solution)
Preparation of hybrid products by reaction of minerals on organic compounds

galena | crushed galena



300°C 1h | 300°C 3h



WORKS LED BY

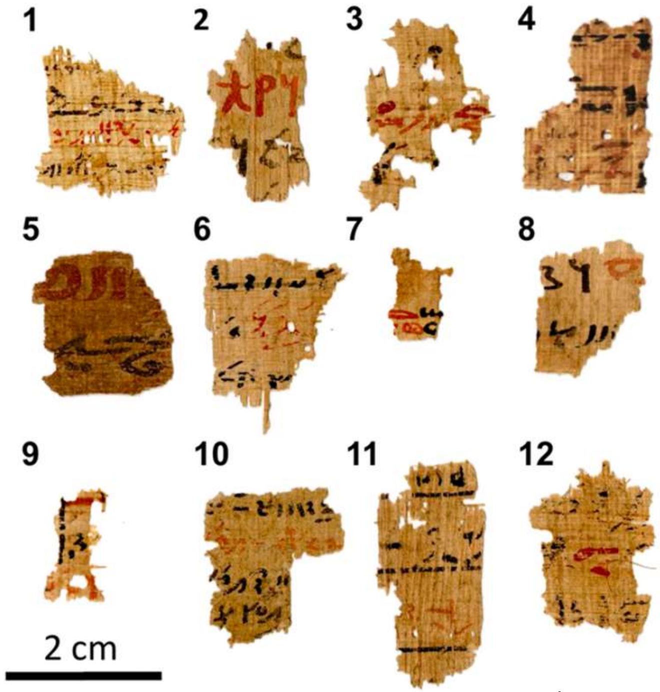
Thomas Christiansen, Sine Larsen, University of Copenhagen

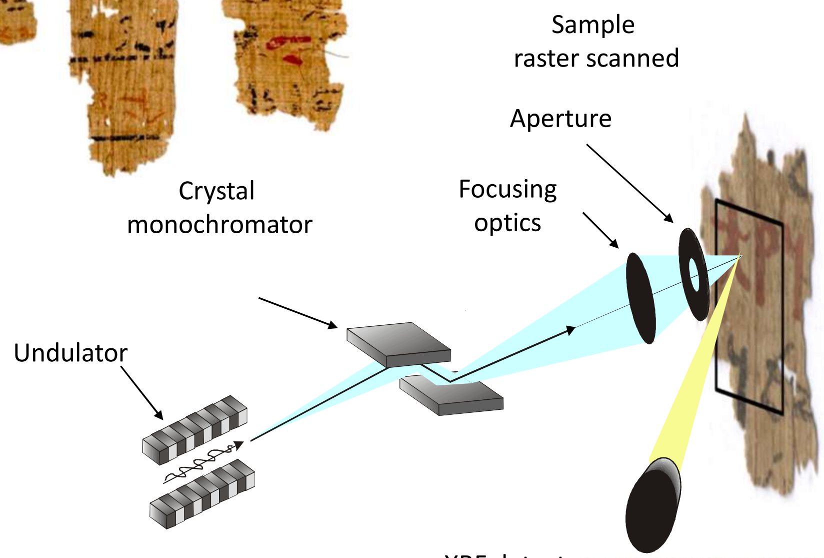
SAMPLES

12 fragments of papyri from the Carlsberg Collection (Tebtunis temple library, 1st-3rd C. CE), with both red and black inks

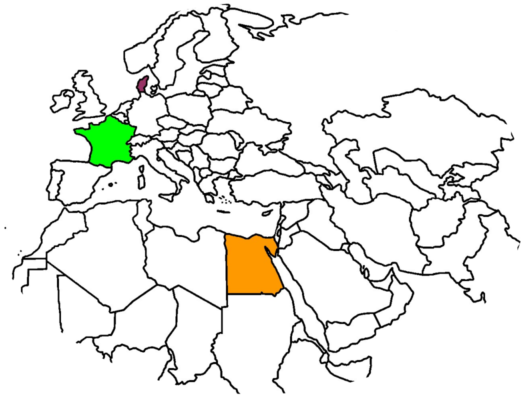
QUESTIONS

Which are the components present in inks? A unique recipe? Iron and lead identified with laboratory techniques. Which compounds?





2- INK COMPOSITION IN EGYPTIAN PAPYRI



TECHNIQUES

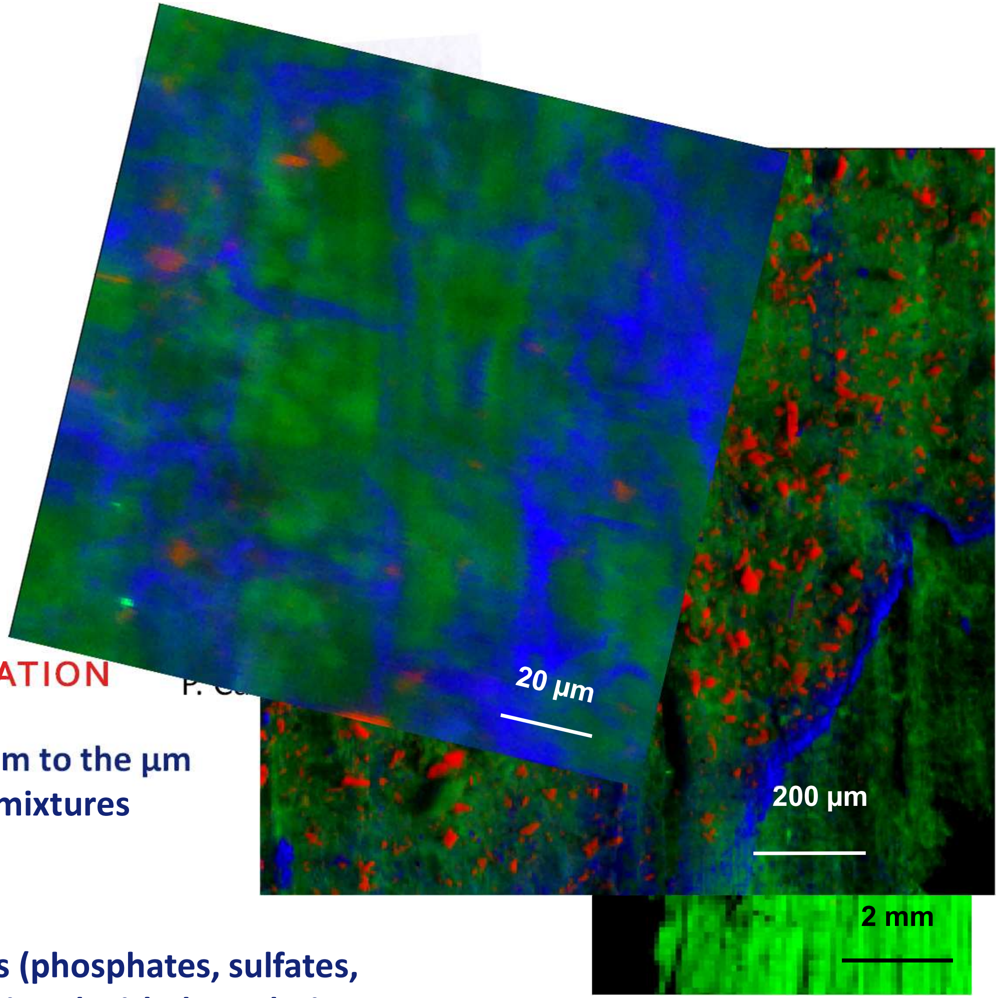
Macro and micro X-ray fluorescence (ID21)
Micro X-ray diffraction (ID21)
Fourier Transform Infrared microscopy (ID21)

ADVANTAGES OF SYNCHROTRON RADIATION

Possibility to explore the fragments from the mm to the µm
High resolution for mapping of components in mixtures

RESULTS

Identification of various lead-based compounds (phosphates, sulfates, chlorides, carboxylates). Some are clearly associated with degradation (grey crust); others have an undetermined origin. Composition and distribution suggests the use of lead-based driers



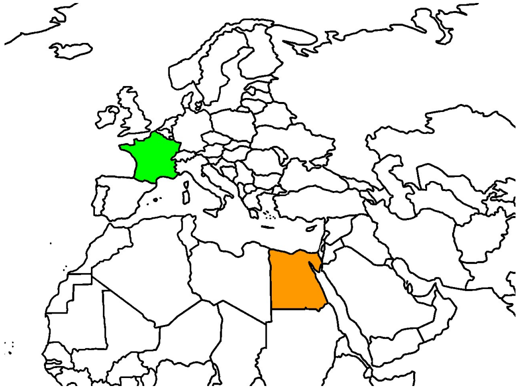
Beam size 0.3×0.7µm²
Pixel size 0.5×0.5µm²

Beam size 0.3×0.7µm²
Pixel size 4×4µm²

Beam size ∅ 100µm
Pixel size 100×100µm²

The European Synchrotron | ESRF

3- REVEALING THE MANUFACTURING PROCESSES OF OPAQUE GLASSES



WORKS LED BY

Sophia Lahlil, Isabelle Biron, Center of Research and Restoration of French Museums, Paris

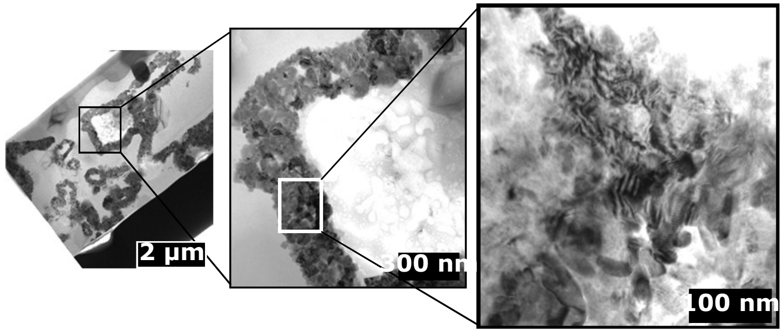
SAMPLES

Fragments of opaque glasses from Egyptian, Roman, French artefacts

QUESTIONS

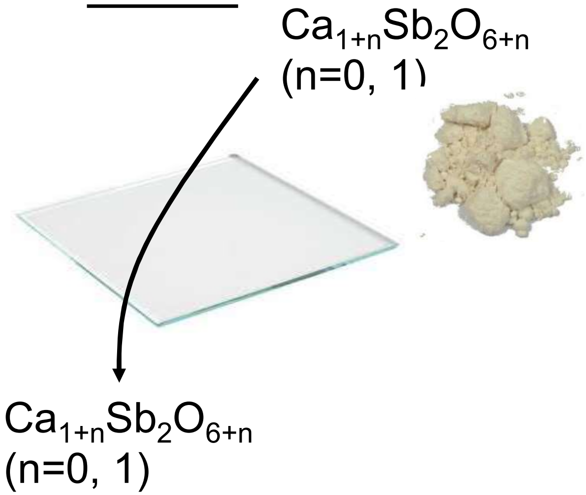
Can the chemical composition of glass reveal how these glasses have been manufactured?

18th Dynasty, (1570-1292 B.C.).

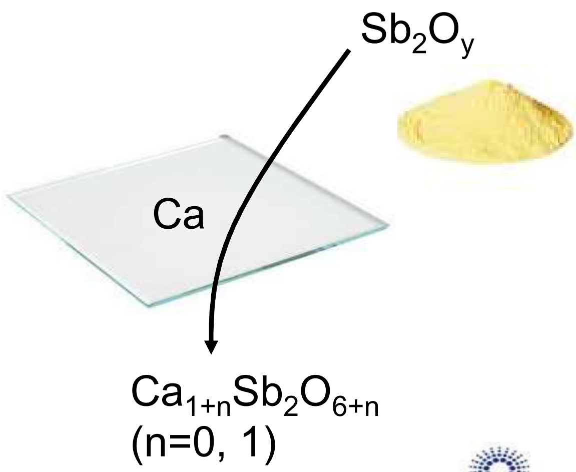


Nano-crystals of calcium antimonate

Ex-situ

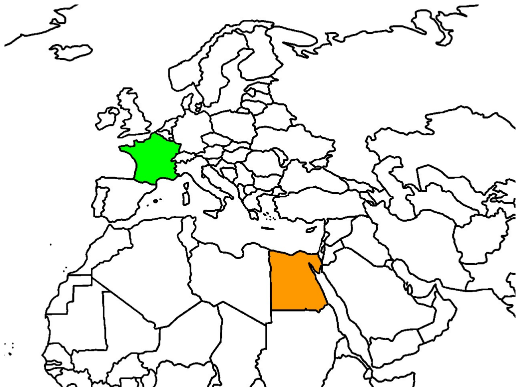


In-situ

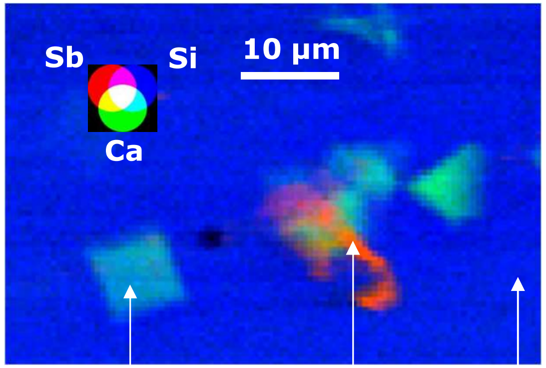


The European Synchrotron | ESRF

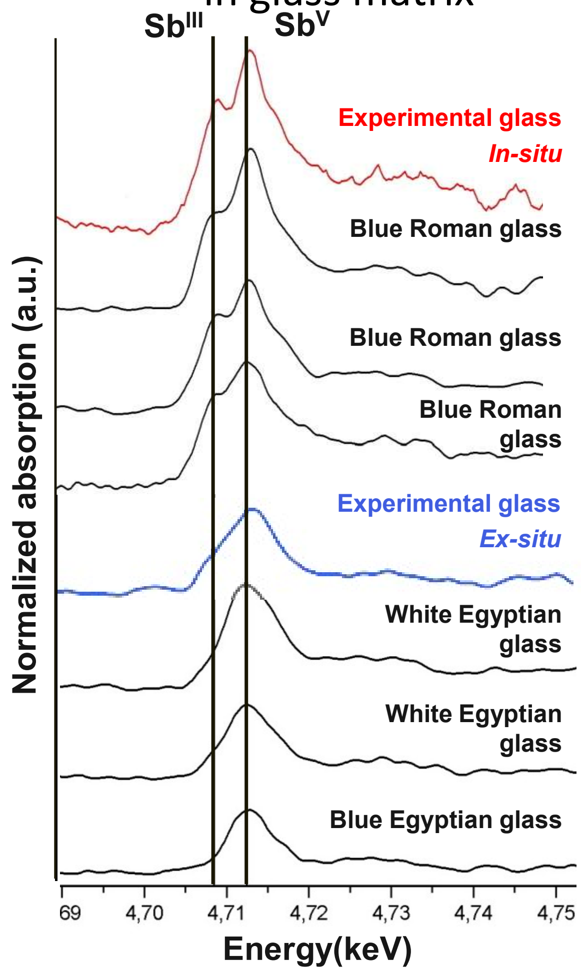
3- REVEALING THE MANUFACTURING PROCESSES OF OPAQUE GLASSES



Location of crystals and glass matrix



Signature of antimony chemical state in glass matrix



TECHNIQUES

Micro X-ray fluorescence map (ID21)
Micro X-ray absorption spectroscopy, Sb L_1-edge (ID21)

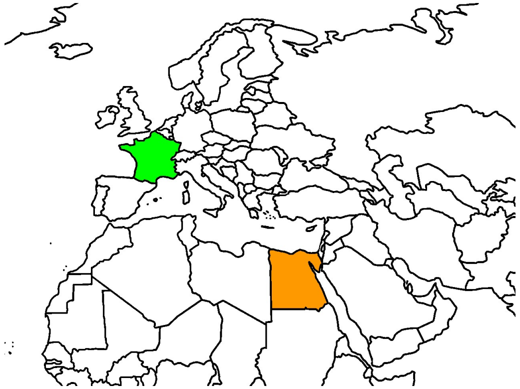
ADVANTAGES OF SYNCHROTRON RADIATION

X-ray absorption spectroscopy provides unique information.
This technique requires a tunable X-ray energy.
+ µm resolution to probe glassy matrix without crystals

RESULTS

Egyptians used the *ex-situ* synthesis, while Romans used the *in-situ* one, probably using Sb_2O_4 as a reactant.

4- OPENING VIRTUALLY A BIRD MUMMY



WORKS LED BY

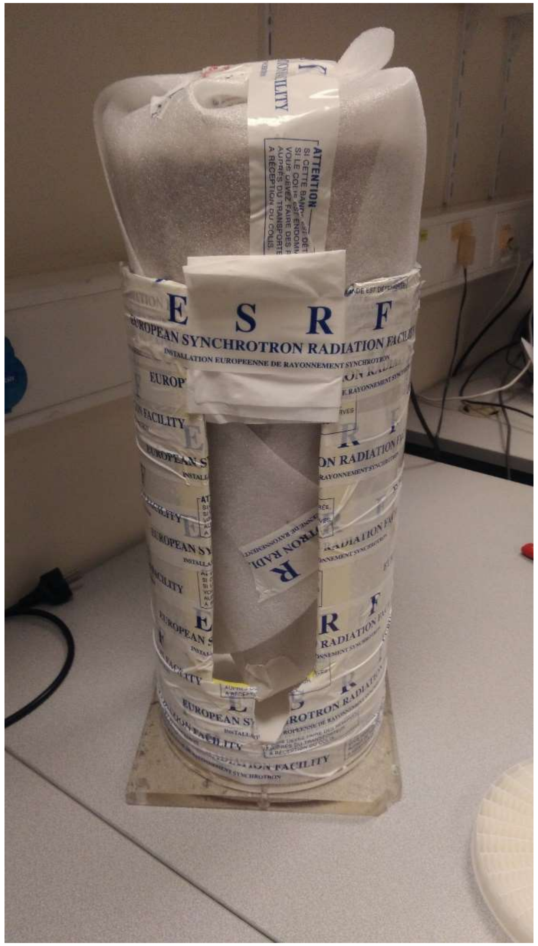
Paul Tafforeau, Camille Berruyer, ESRF

SAMPLE

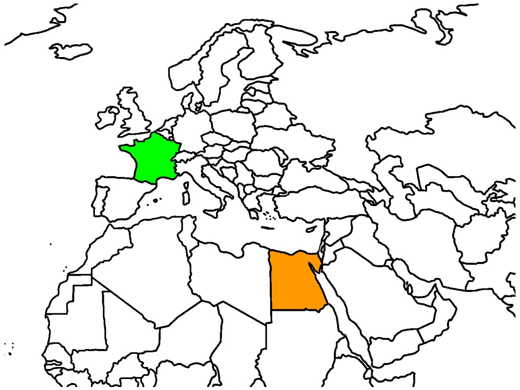
Egyptian Ibis mummy in its sealed jar curated at the Musée de Grenoble

QUESTIONS

What can we know about the mummification processes, from the bird to the jar?



4- OPENING VIRTUALLY A BIRD MUMMY



TECHNIQUES

Phase contrast X-ray tomography (BM05)

ADVANTAGES OF SYNCHROTRON RADIATION

Possibility to increase the contrast and to reveal subtle structures

Possibility to work at increasing resolution (hierarchical imaging)

RESULTS

Possibility to distinguish wild / farm animals

Possibility to identify pieces of different animals in a unique mummy

Information about mummification processes

5- PAINTING TECHNIQUE IN BAMIYAN WALL PAINTINGS

WORKS LED BY

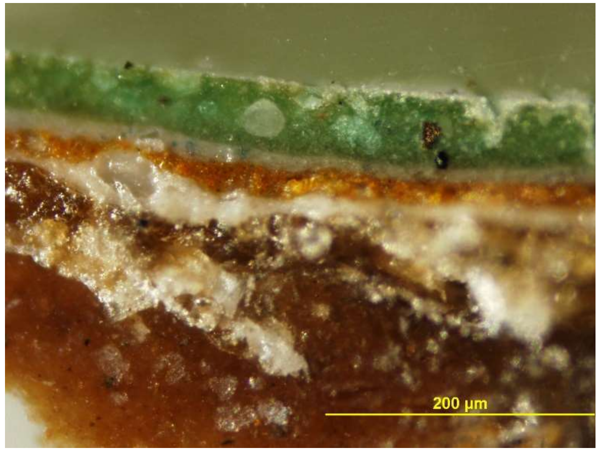
Yoko Taniguchi, Japan Center for International Cooperation in Conservation- National Research Institute for Cultural Properties, Tokyo

SAMPLES

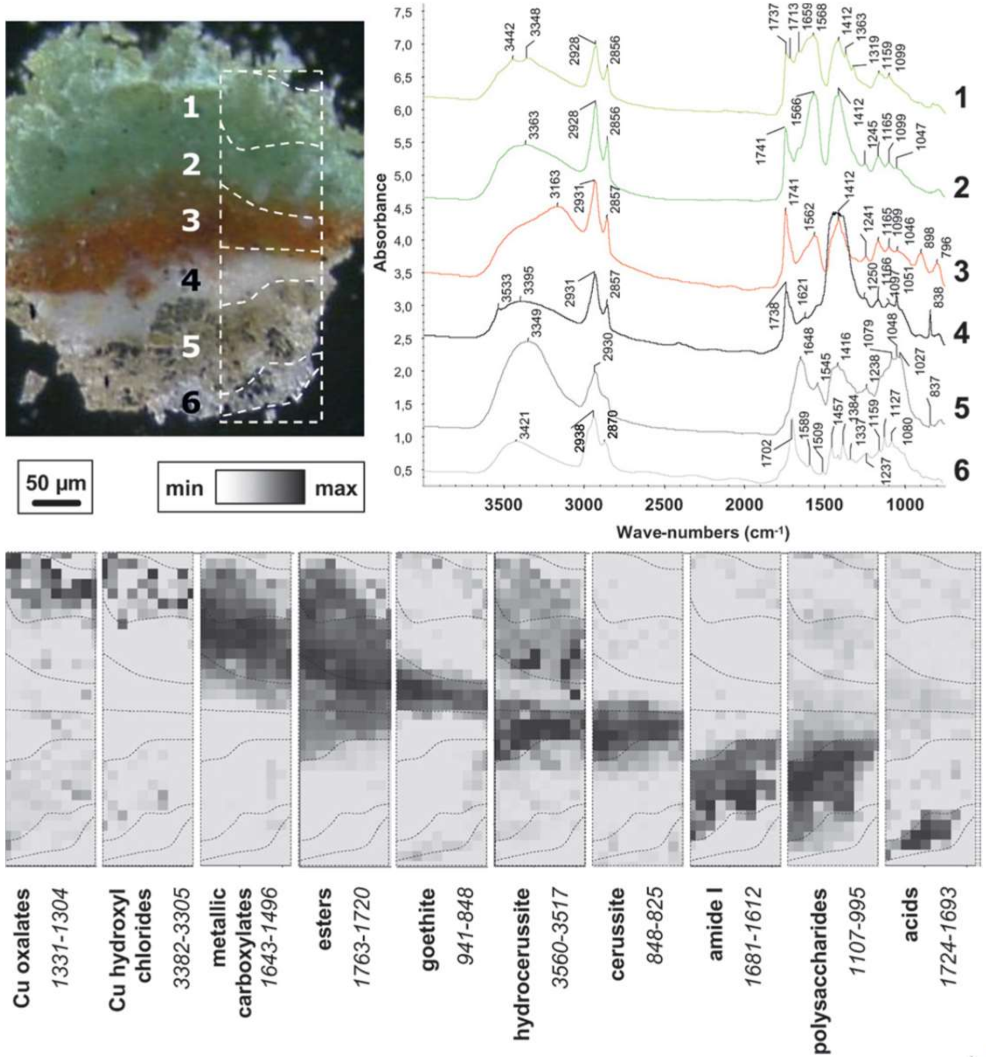
Fragments of paintings from Bamiyan Buddhist wall paintings, 6th-9th C.

QUESTIONS

Which were the painting techniques? Pigments? Binders?
Connection with other practices along the silk road?
Degradation state?



5- PAINTING TECHNIQUE IN BAMIYAN WALL PAINTINGS



TECHNIQUES

Micro X-ray fluorescence map (ID21, former ID18F)
Micro X-ray diffraction (former ID18F)
Fourier Transform Infrared microscopy (ID21)

ADVANTAGES OF SYNCHROTRON RADIATION

High resolution and mapping capabilities = possibility to detect and locate many components
Different complementary X-ray and infrared-based techiques

RESULTS

A very early use of oil painting, presence of metal carboxylates
Identification of many pigments, with various compositions
Identification of degradation compounds on the surface (sulfates, chlorides)...

CULTURAL HERITAGE : THE MAIN SCIENTIFIC QUESTIONS

Scientific objectives and challenges

Past: reveal the manufacturing secrets

Future: preserve our heritage

Formulation?

Components → Artwork

Evolution?

Artwork → Degradation

Reactants —*Synthesis*→ Products —*Slow reactions*→ Secondary products

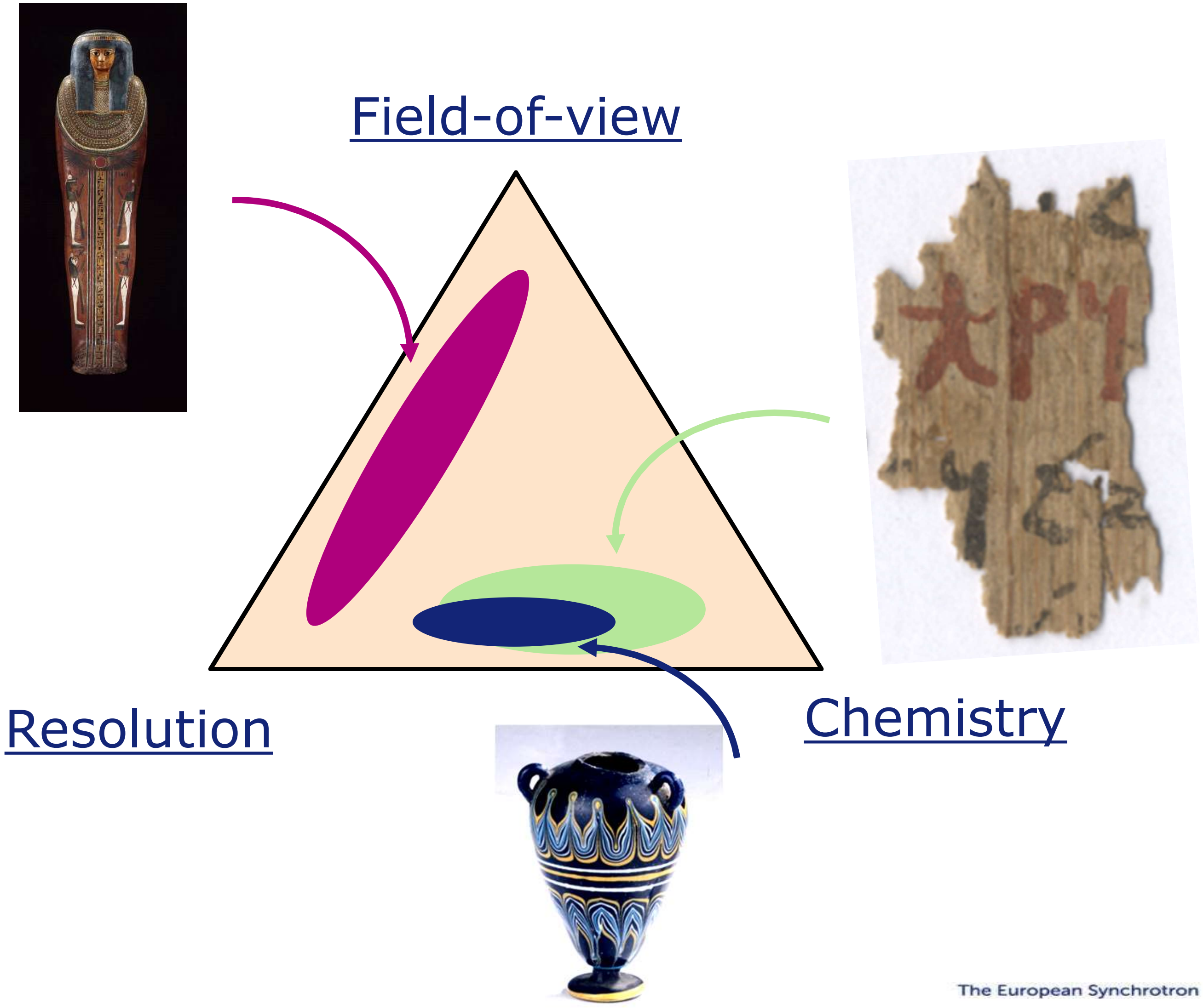
External reactants

Artist intention — Degradation

Authentication

THE ADVANTAGES OF SYNCHROTRON RADIATION

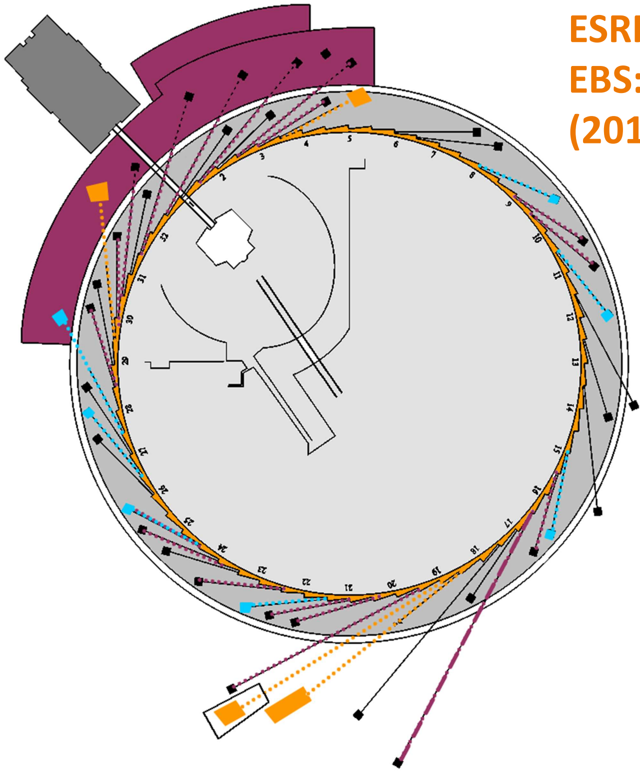
- Many techniques = many contrasts
- High brightness = small beam, intense beam (speed)
- Tunable energy, access to high energies
- High coherence



THE ESRF UPGRADE PROGRAMME (2009-2022)

ESRF UPGRADE PHASE I (2009-2015) - 180 M€ :

- 19 new beamlines, many specialised on nano-science
- Upgrade and renewal of facilities and support labs
- *Study for a new high brilliance high energy X-ray storage ring*



ESRF UPGRADE PHASE II EBS: Extremely Brilliant Source (2015-2022) - 150 M€

- A new generation of synchrotron storage ring
- 4 new flagship beamlines and 7 refurbished beamlines
- Detectors and instrumentation
- IT data strategy

Purple Book
January 2008

The

Orange Book
January 2015

BM18: A NEW BEAMLINE DEDICATED TO X-RAY PHASE CONTRAST TOMOGRAPHY

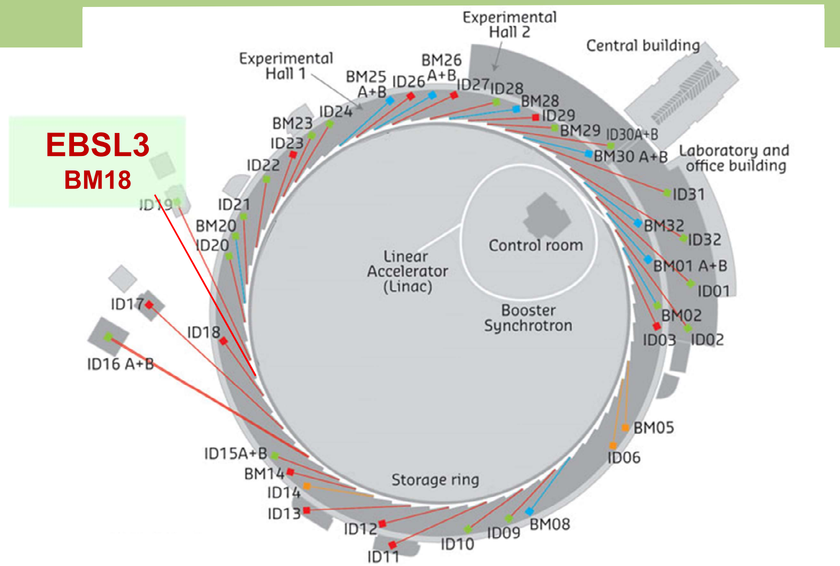
BM 18 specifications

- 220 m long beamline
- Smallest possible source in the ESRF-EBS new lattice
- 30 cm wide beam in polychromatic mode
- 45 m long experimental hutch
- Fully designed to ensure highest possible coherence level
- Up to 38m of propagation distance for phase contrast imaging

Energy range: 35-350 keV
Sample size: max 300 kg, 2.5m in height
Multi-detectors is for multi-resolution.

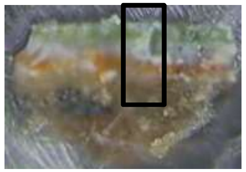
- 0.7 µm for the smallest
- 1-27 µm on a continuous range,
- 47 µm
- 200 µm

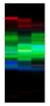
Only one sample stage => it will be possible to scan any part in high resolution by selecting it from a scan at lower resolution (a single 3D repository)



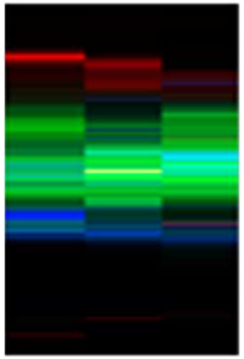
EBS FOR MICRO-PROBE BEAMLINES

Dec 2006
former ID18F beamline
map size: 150 × 60 µm²
pixel size: 1 × 20 µm²
15s / pixel (5s acq. + 10s lead time)
Total of 1h52





100µm

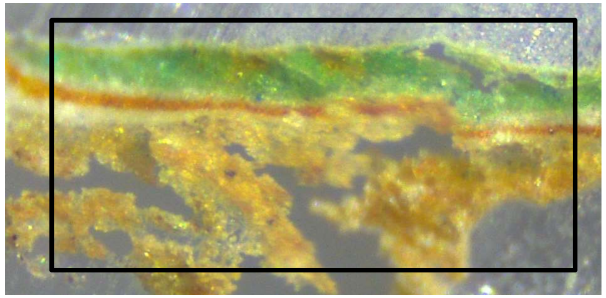


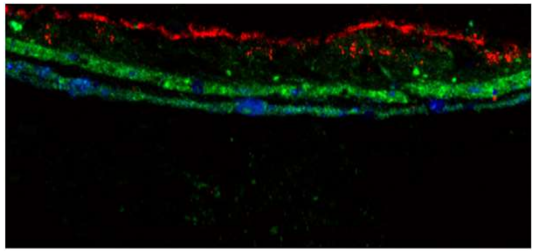
palmierite

hydrocerussite

cerussite

Nov 2021
ID13 beamline
map size: 800 × 370 µm²
pixel size: 1 × 1 µm²
0.016s / pixel (10ms acq. + 6 ms lead time)
Total of 1h18





BEYOND DATA COLLECTION

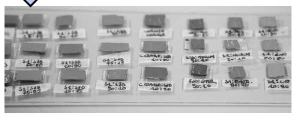
- New access models
- New communities
- User outreach

- New sample management tools
- New sample environments
- Automation

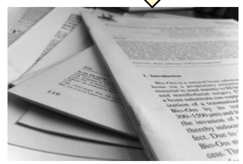
- New source (EBS)
- New/ refurbished beamlines
- New techniques

- New tools for data collection, on-line analysis, machine learning routines









Users

↗ More users
↗ New users
↗ More diverse users

Samples

↗ More samples
↗ More diverse samples

Data

↗ Quantity
↗ Quality
↗ Complexity

Results

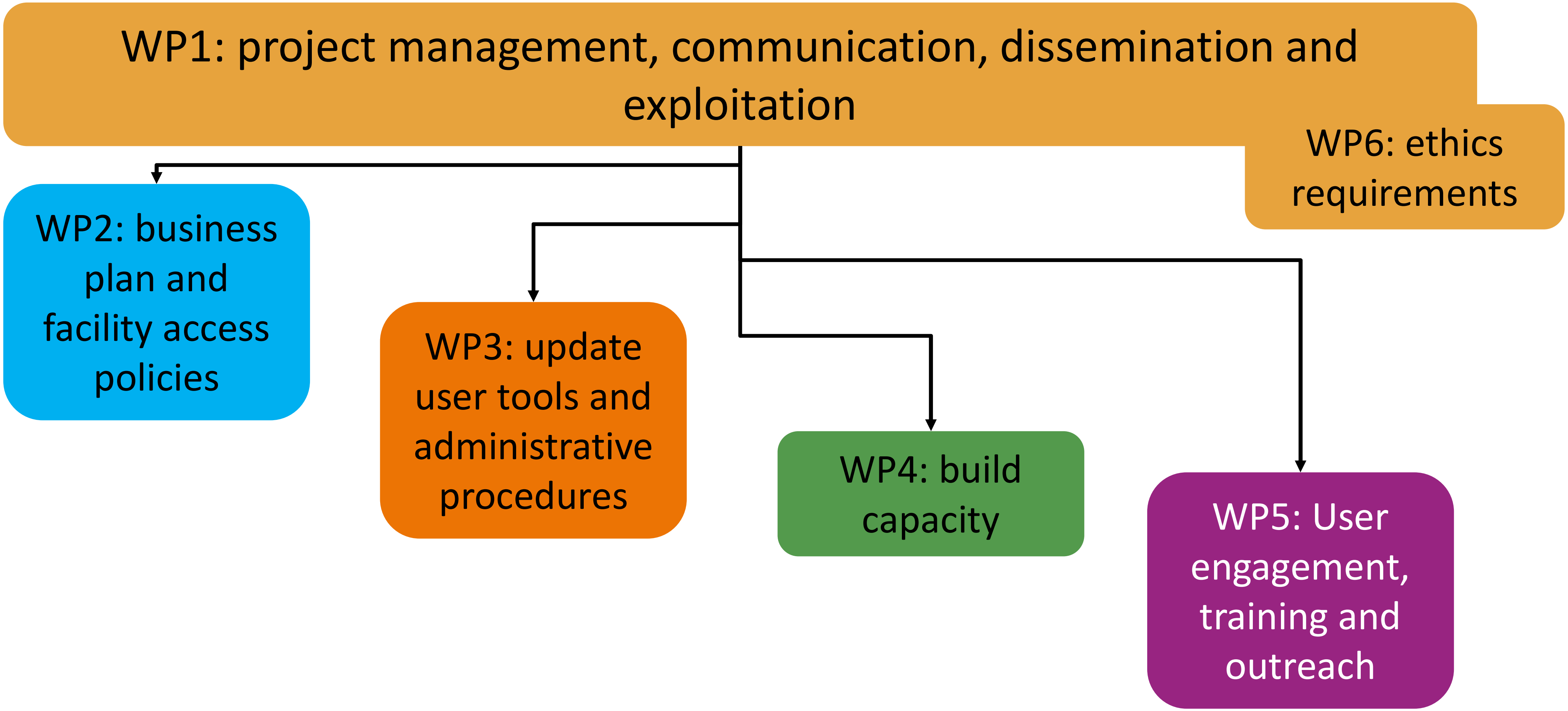
↗ Quantity
↗ Quality
↗ FAIR

The Eu

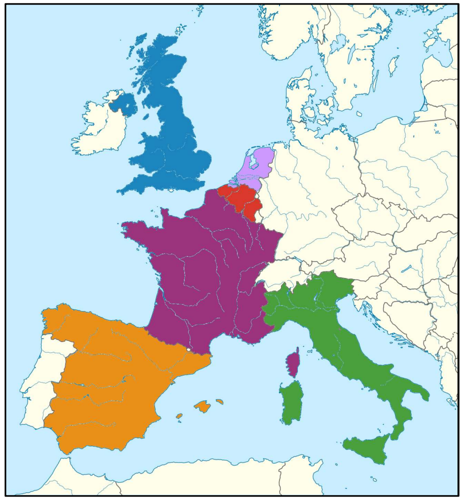
The STREAMLINE project

Sustainable research at micro and nano X-ray beamlines

THE STREAMLINE PROJECT



THE “HISTORICAL MATERIALS” BAG



11 European teams

- ENS Paris-Saclay: V. Gonzalez
- CNR-SCITEC: L. Monico
- Courtauld Institute of Art: A. Nevin, A. Burnstock
- Politecnico di Milano: D. Comelli
- Rijksmuseum: K. Keune
- IRCP/C2RMF: I. Reiche
- Universitat Politècnica de Catalunya: N. Jiménez
- ESRF: M. Cotte
- IRCP: G. Wallez
- University of Antwerp: K. Janssens
- TU Delft: M. Alfeld

Access to ID22 (6 shifts, HR-XRPD) and ID13 (12 shifts, µXRD/µXRF) every 6 months for 2 years

https://www.esrf.fr/BAG/HG172

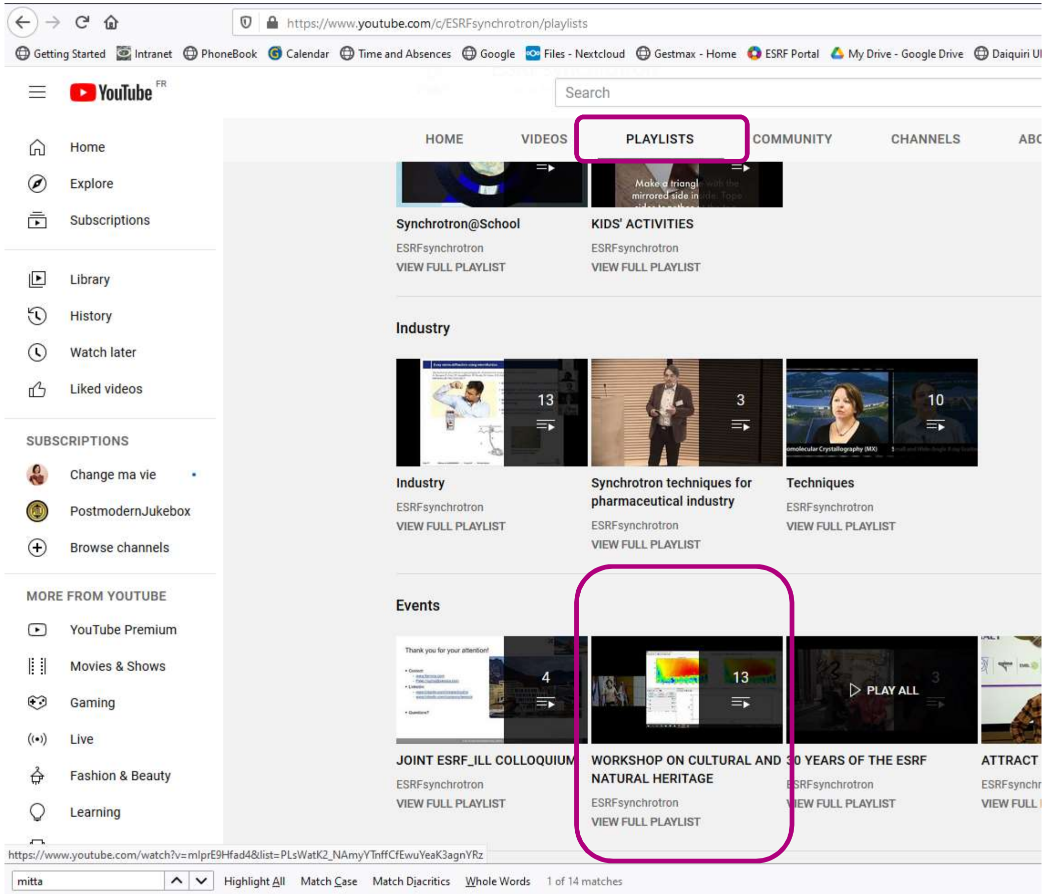
WANT TO KNOW MORE?

STREAMLINE

Cultural and Natural Heritage
ESRF-EBS workshop

ESRF – Grenoble - France
22-24 January 2020

ESRF YouTube channel
Playlists
Workshop on cultural and natural heritage



https://www.youtube.com/playlist?list=PLsWatK2_NAmyyA0n03OMJMAKobVIvow2D

THANK YOU FOR YOUR ATTENTION
Thanks to users and colleagues
involved in this research!

PIONEERING SYNCHROTRON SCIENCE

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Do not hesitate to contact me: cotte@esrf.fr