TRAINING CAMP Dalla diagnostica alla fruizione museale: le opere del Museo del Colle del Duomo di Viterbo

7 - 13 NOVEMBRE 2021

REGIONE LAZIO

Synchrotron Radiation and Cultural Heritage

Dott.ssa Antonella Balerna

DTC LAZIO DISTRETTO TECNOLOGICO BENI E ATTIVITÀ CULTURALI CENTRO DI ECCELLENZA
INFN CHNet Istituto Nazionale di Fisica Nucleare Cultural Heritage Network
UNIVERSITÀ DEGLI STUDI DELLA TUSCIA
POLO MONUMENTALE COLLE DEL DUOMO VITERBO
ARCHEOARES

Synchrotron Radiation and Cultural Heritage

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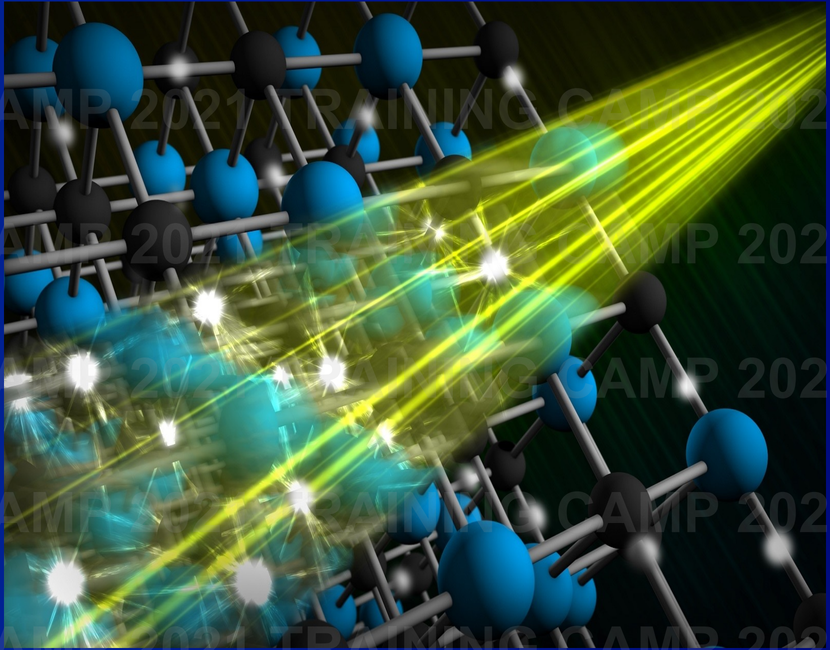
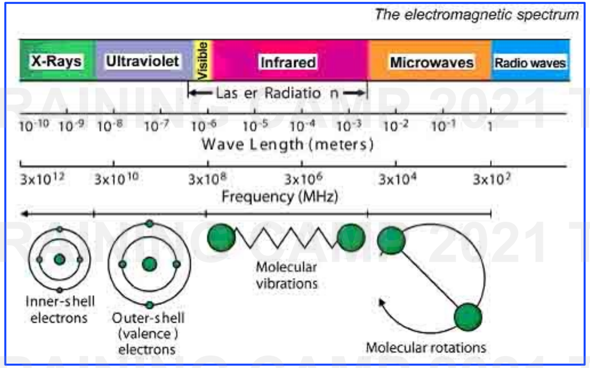
2. Synchrotron Radiation (SR)
3. SR and Cultural Heritage
4. Some applications
5. How to apply for beamtime
6. Beamtime and proposals
7. Case study
8. Conclusions

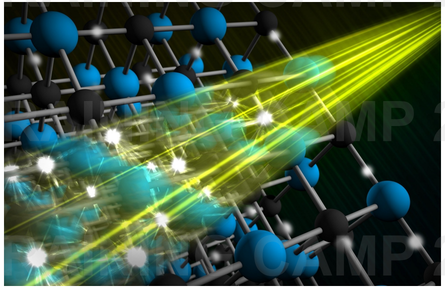


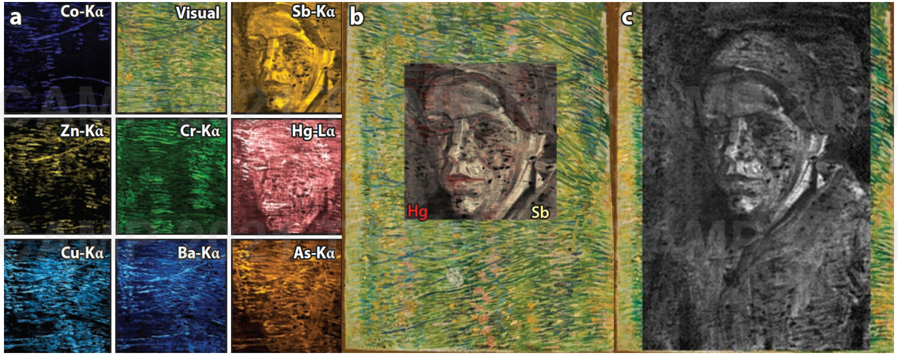
Short introduction

Light, material science and cultural heritage



Different wavelengths different spectroscopies





Making the invisible visible with X-rays

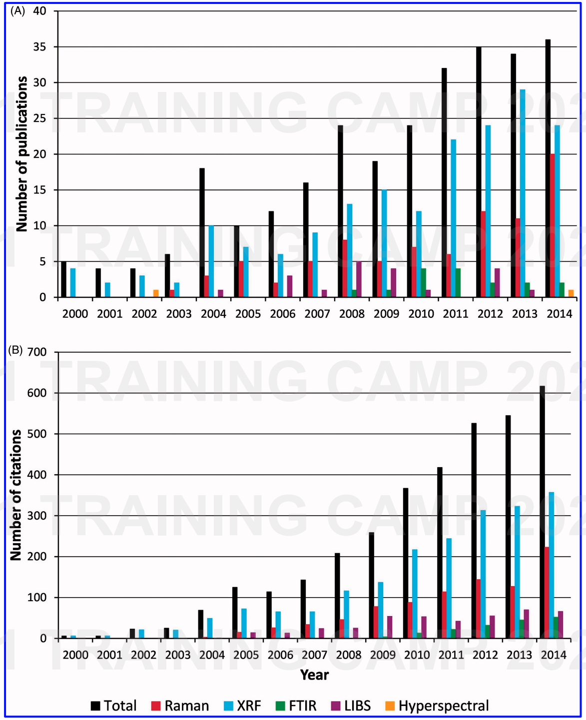
K. Janssens et al. *The Use of Synchrotron Radiation for the Characterization of Artists' Pigments and Paintings*, Annual Rev. Anal. Chem. 2013. 6:399–425
DOI: 10.1146/annurev-anchem-062012-092702

Portable Laboratory Instrumentation

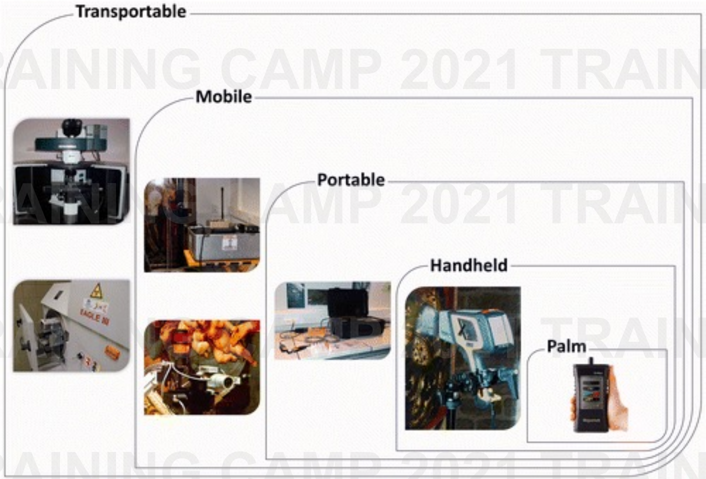
Laboratory instrumentation is of growing importance in Cultural Heritage field. Equipment is used **in the field or at museums**, thus **avoiding transportation or risk of damage to valuable artifacts**. Many spectroscopic techniques are **non-destructive and micro-destructive** in nature, which **preserves the cultural heritage objects themselves**.

Raman spectroscopy, **X-ray fluorescence spectrometry**, **Fourier Transform Infrared spectroscopy** and other techniques are the ones mainly used.

Review of over **160 references** pertaining to the use of **mobile spectroscopy** for archaeometry **up to 2014: P. Vandenabeele**, *Mobile Spectroscopic Instrumentation in Archaeometry Research* , **Applied Spectroscopy 2016**, Vol. 70(1) 27–41 - DOI: 10.1177/0003702815611063

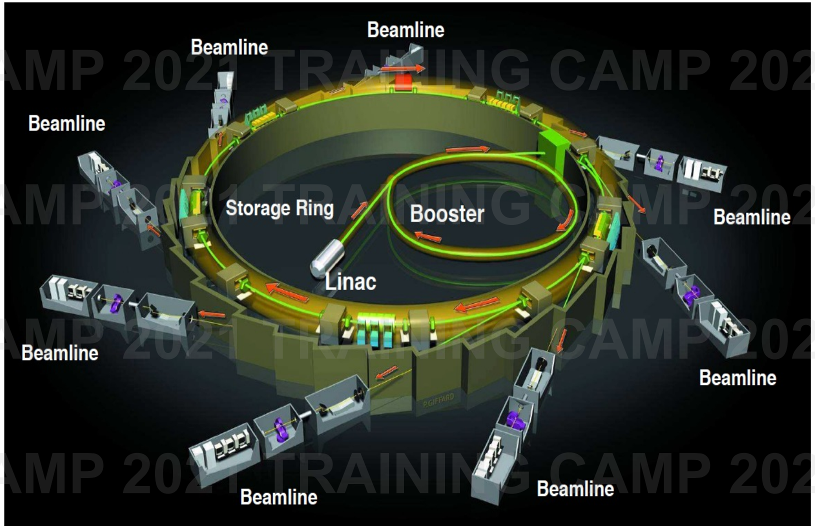


Moving from portable laboratory to SR-based instrumentation



Laboratory instrumentation

P. Vandenabeele, *Mobile Spectroscopic Instrumentation in Archaeometry Research* , **Applied Spectroscopy 2016**, Vol. 70(1) 27–41 - DOI: 10.1177/0003702815611063

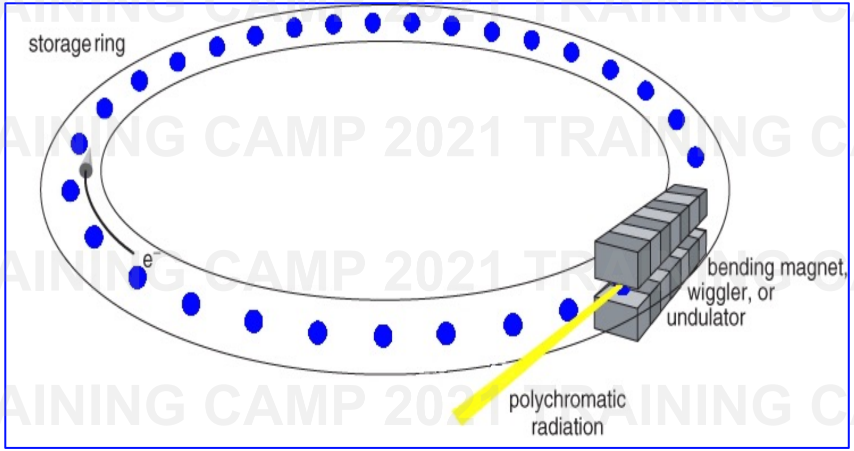


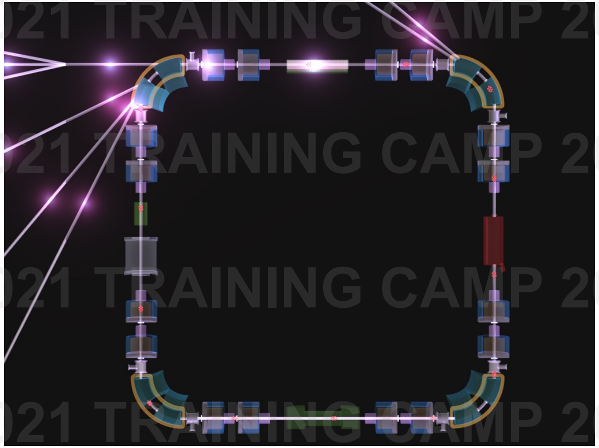
Particle accelerators as light sources for studies in different fields

Synchrotron Radiation

What is synchrotron radiation?

The term "**synchrotron radiation**" refers to the electromagnetic radiation – **x-rays, ultraviolet, visible and infrared** – emitted by **high-energy electrons circulating in a particle accelerator**. Specifically, the accelerators used in modern facilities are "storage rings" in which the electron, accelerated to **nearly the speed of light** and forced to move on **circular paths by bending magnets** emit Synchrotron Radiation – that is collected by several beamlines and used for different experiments.





https://www.isa.au.dk/animations/animations.asp

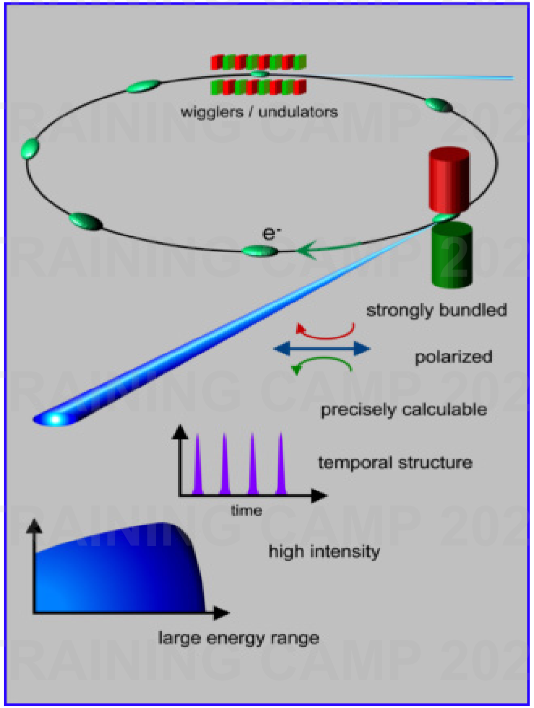
Synchrotron radiation properties

Synchrotron radiation has **important properties**:

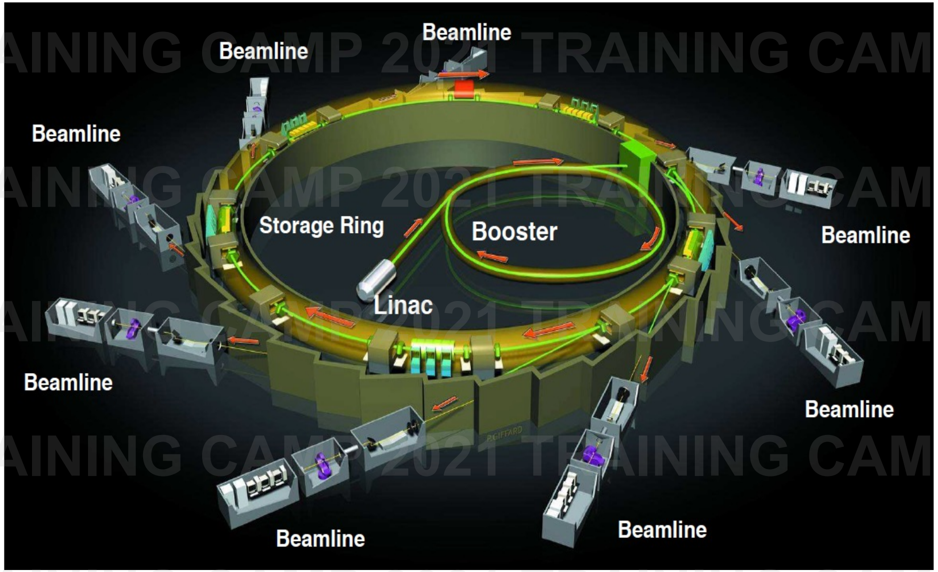
- a **bright beam**, which permits **fast acquisitions**, with **improved detection limits**, which translate into wider corpus, wider fields of view, **higher representativeness of the results** and **higher sensitivity**;

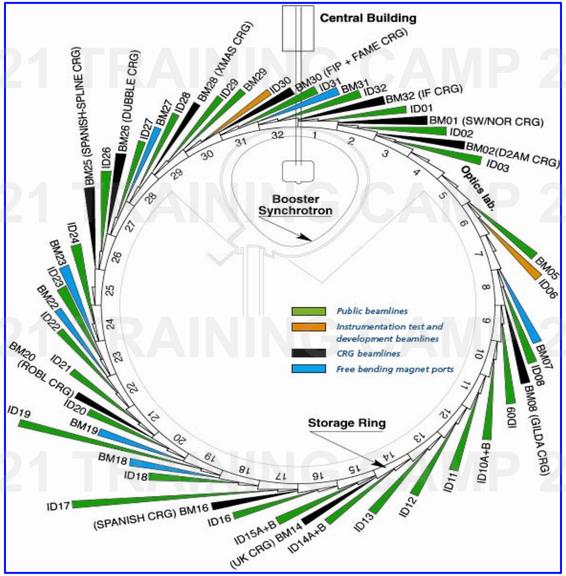
- a **collimated beam**, which permits to focus the beam to the **micron range**, and **even smaller** with **state of- the-art nano-probe beamlines**. This is particularly important considering the **heterogeneity of artistic materials**;

- an extended and almost **continuous energy range**, which gives access to **X-ray spectroscopy**. This technique is particularly relevant for the analysis of artistic materials since it **probes element speciation, which is usually altered during object manufacturing or degradation**. Information **about chemical reactions involved throughout the entire life of the works of art** can thus be obtained. Beside, synchrotron radiation also gives access to lower energies and spectroscopy performed in the **UV/Visible** and the **Infrared domains** can offer **complementary characterization tools**.



Synchrotron radiation facilities



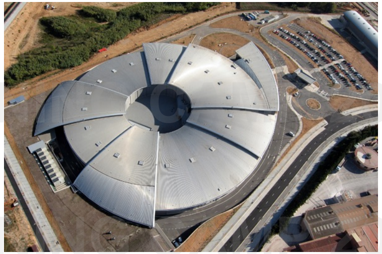


Schematic views: ESRF, Grenoble - France 6 GeV, C = 844 m opened to users in 1994

Synchrotron radiation facilities

ESRF - France

DIAMOND - UK



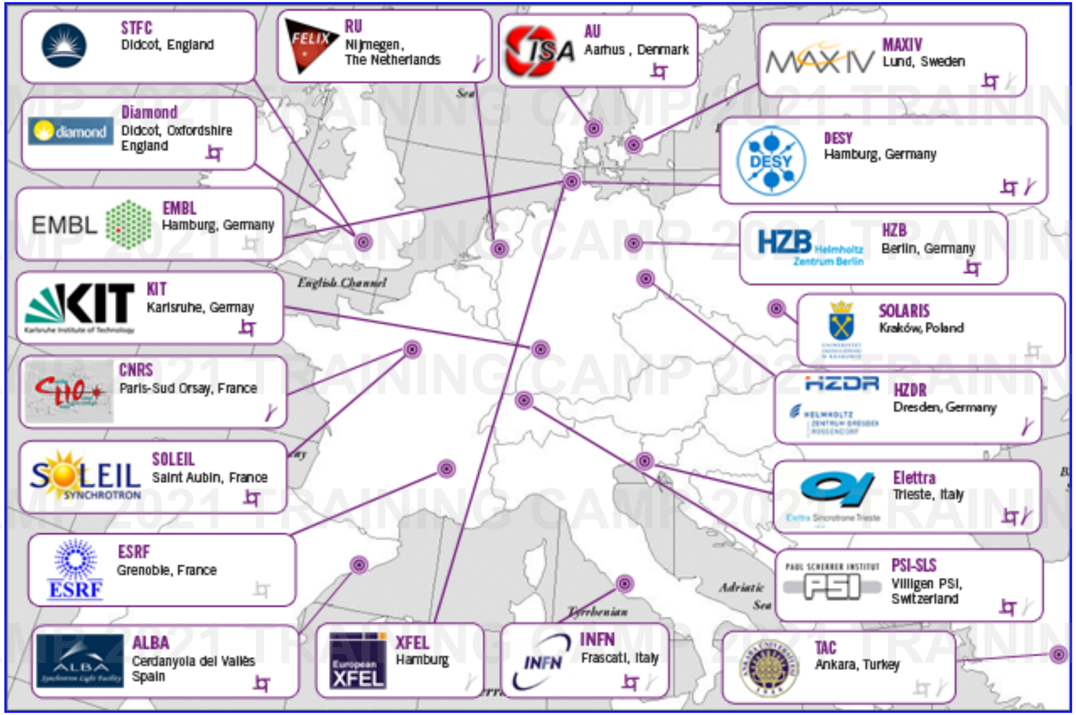
ALBA - Spain

Lund - Sweden

Sirius - Brazil

Shanghai -China

Synchrotron radiation facilities and Europe

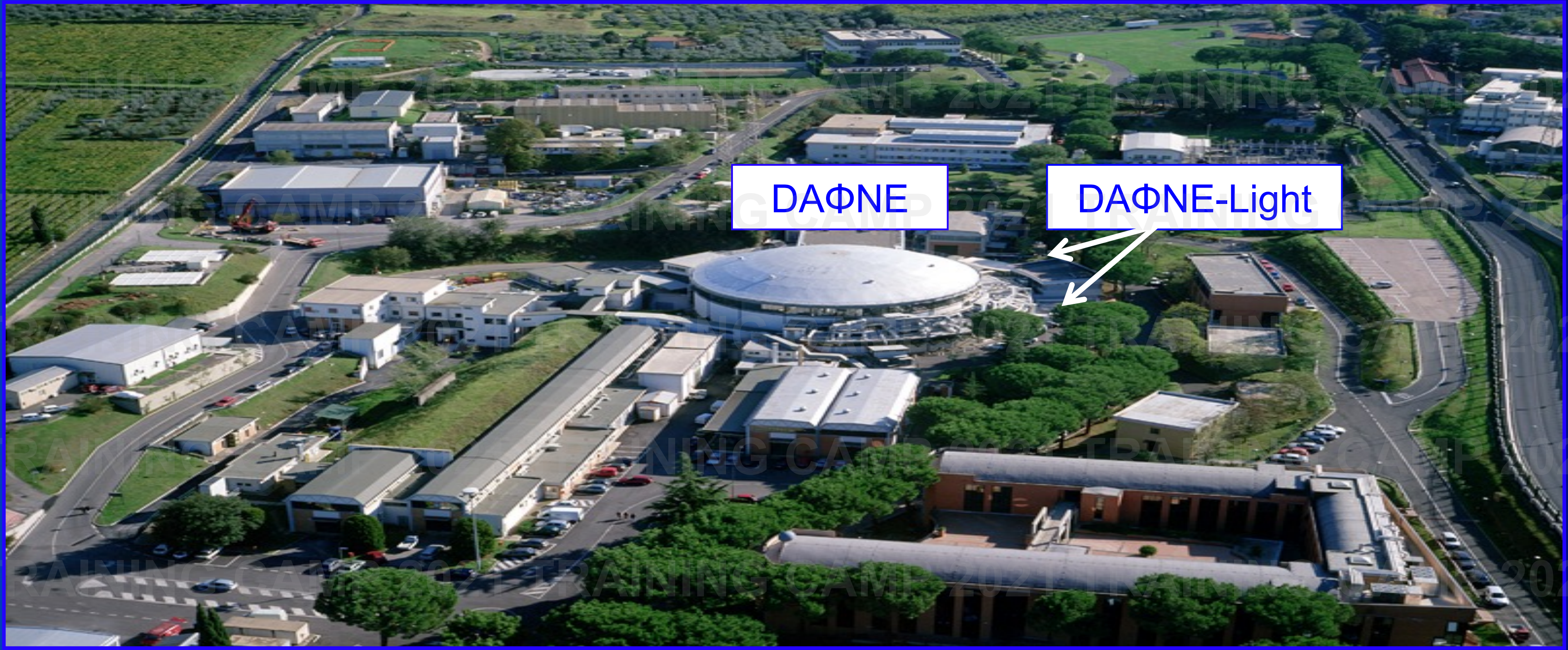


CALIPSOplus

http://www.calipsoplus.eu/

DAΦNE-LIGHT
INFN-LNF Synchrotron Radiation Facility

INFN Frascati National Laboratory



Beamlines @ DAΦNE-Light

OPEN to USERS

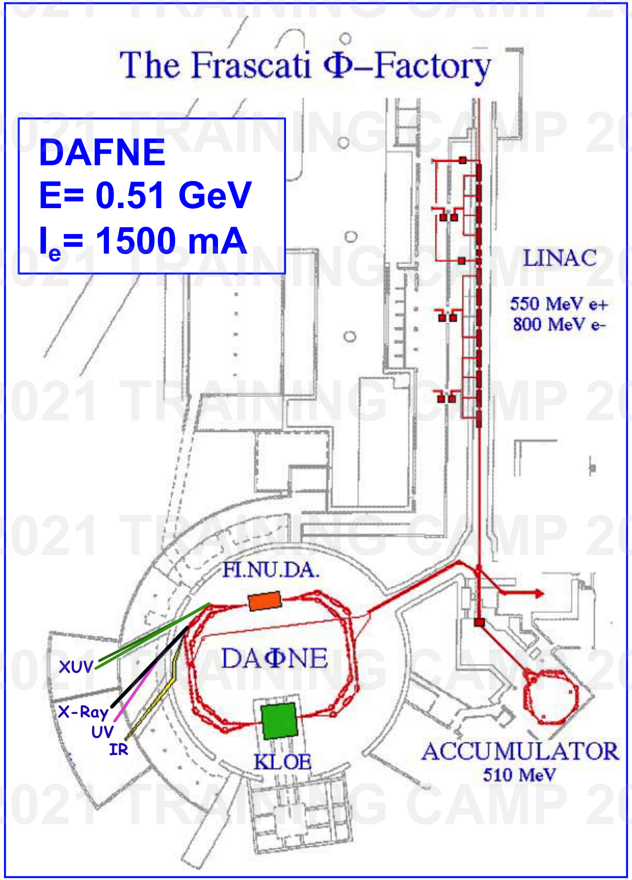
1) SINBAD - IR beamline (1.24 meV - 1.24 eV)
2) DXR1- Soft x-ray beamline (900-3000 eV)
3) DXR2 – UV-VIS beamline (2-10 eV)

XUV beamlines UNDER COMMISSIONING

4) XUV1 - Low Energy Beamline (30-200 eV)
5) XUV2 - High Energy Beamline (60-1000 eV)
6) New XUV2 Branch White Line -WINDY

Call for proposals (two/year) deadlines:

- 20 June
- 18 December



Techniques available at DAΦNE-Light

- **FTIR spectroscopy, IR microscopy and IR imaging - BS Mariangela Cestelli Guidi**
- **UV-VIS absorption spectroscopy - BS Marco Angelucci**
- **Photochemistry: UV irradiation and FTIR micro-spectroscopy and imaging.**
- **Soft x-ray spectroscopy: XANES (X-ray Absorption Near Edge Structure) light elements from Na to S - BS Antonella Balerna**
- **SEY (secondary electron yield) and XPS (X-ray photoelectron spectroscopy) – by electron and photon bombardment - BS Roberto Cimino**

http://dafne-light.lnf.infn.it/team/

Synchrotron radiation and Cultural Heritage

Synchrotron Radiation as a powerful tool

Questions often asked on artifacts:

1) what material is it made of ? or Composition

2) when was it realized? or Dating

3) where was it made? or Provenance

4) how was it made? or Art Technology

5) how can we avoid its damaging? or Conservation

Some investigations that can give answers using SR and X-rays:

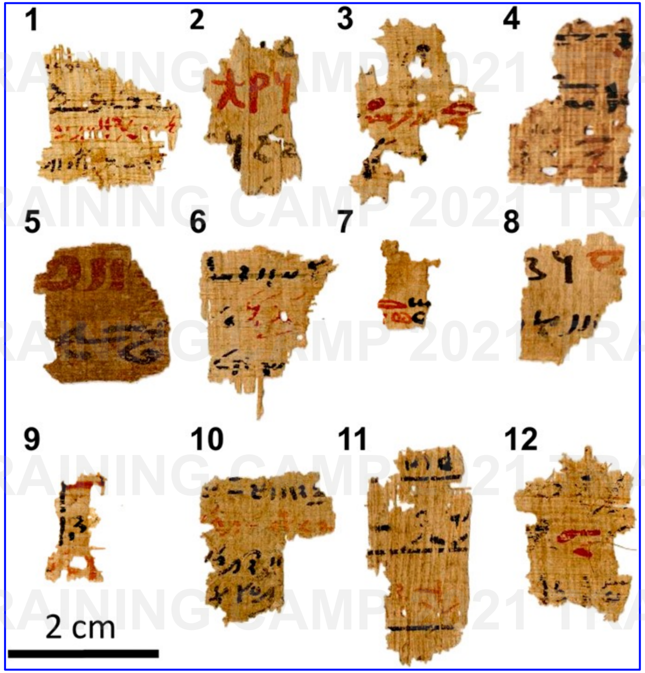
1) Elemental microanalysis down to the sub-ppm level using of X-ray fluorescence analysis (μ-XRF).

2) Local chemical state determinations of selected (trace) constituents using XAS and μ-XAS (X-ray absorption spectroscopy)

3) Imaging of entire objects using high energy SR to allow high quality radiographic or tomographic measurements, revealing the internal structure of these artifacts.

Some applications

Ancient Egyptian red and black inks on papyri and µ-XRF

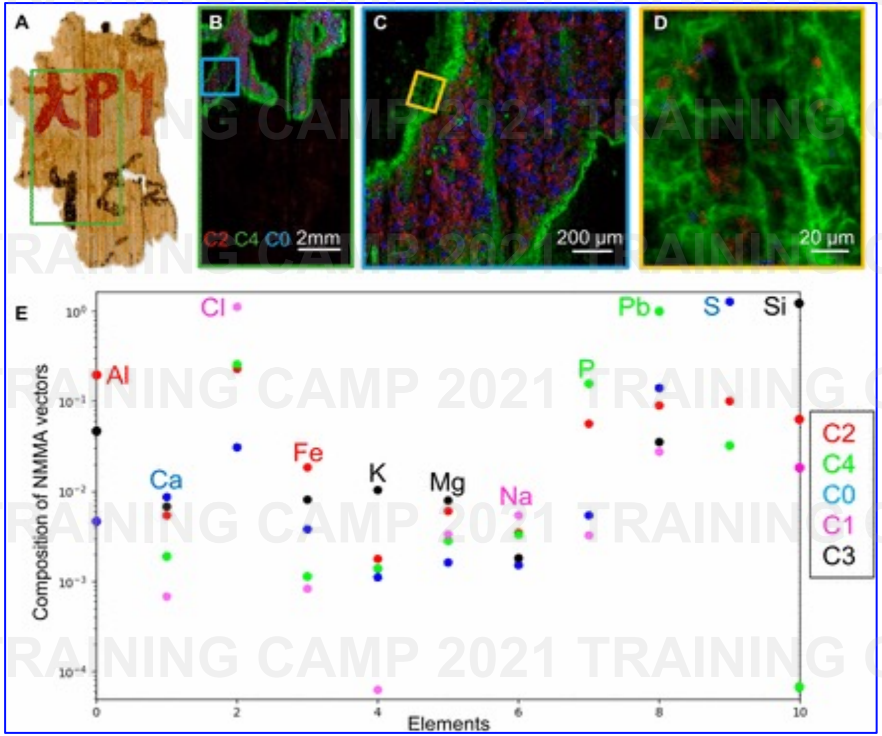


Ink, **invented in ancient Egypt circa 5,000 y ago**, is the established and time-honoured medium where with human kind commits words to writing. A comprehensive **synchrotron-based microanalysis of a considerable corpus of ancient Egyptian papyri from the Roman period**, inscribed with red and black inks, reveal a **hitherto undetected complex composition of inks**. Highlighted by the **presence of iron**, the **red colour can be attributed to the use of ochre**. Unexpectedly, **lead is regularly present in both the red and black inks** and is associated to phosphate, sulphate, chloride, and carboxylate ions. The analysis shows that **lead was probably used as a drier rather than as a pigment**, similar to its usage in 15th century Europe during the development of oil paintings.

µ-XRF

T. Christiansen et al. Proceedings of the National Academy of Sciences Nov 2020, 117 (45) 27825-27835; DOI: 10.1073/pnas.2004534117

Ancient Egyptian red and black inks on papyri and μ-XRF



(*D*) **μ-XRF** map (yellow rectangle in *C*). E = 2.9 and 8.7 keV. **Beam size: 0.54 μm horizontal × 0.75 μm vertical** . Area size: 140 μm horizontal × 140 μm vertical. Step size: 0.7 μm horizontal × 0.7 μm vertical.

Synchrotron-based microanalysis (μ-XRF) can give important information thanks to the beam properties!

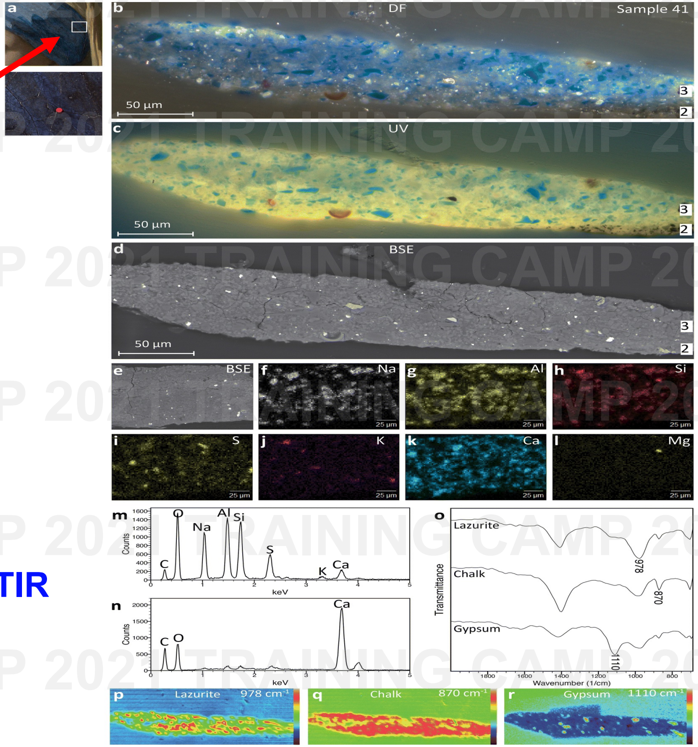
T. Christiansen et al. Proceedings of the National Academy of Sciences Nov 2020, 117 (45) 27825-27835; DOI: 10.1073/pnas.2004534117

Out of the blue: Vermeer's use of ultramarine and XAFS

By Johannes Vermeer *Girl with a Pearl Earring*
https://www.mauritshuis.nl/, Public Domain,
https://commons.wikimedia.org/w/index.php?curid=55017931

Micro-sampling

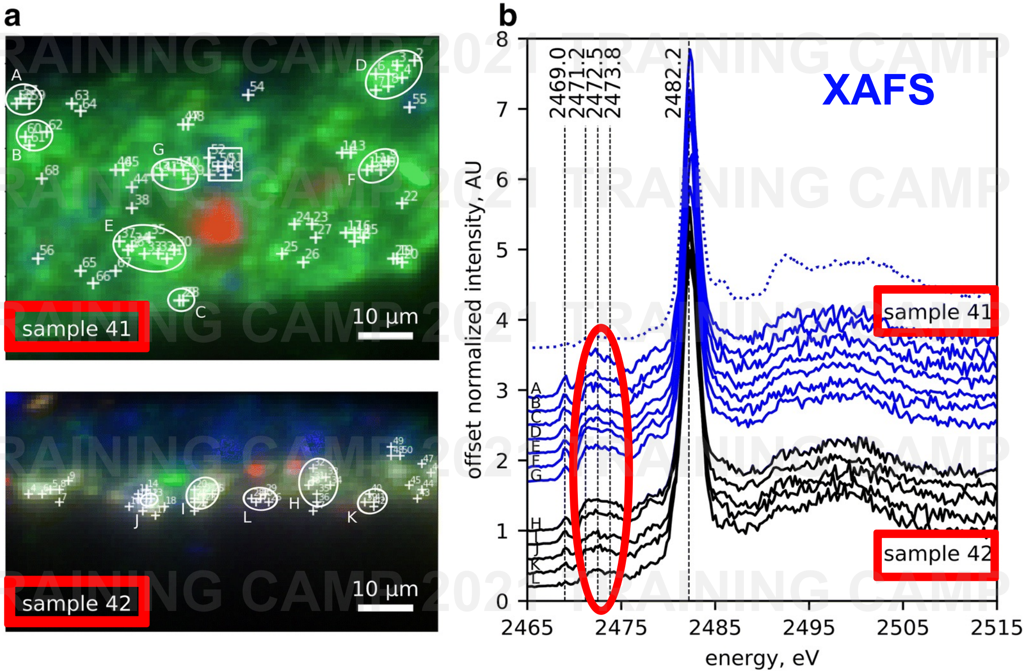
XRF and FTIR



A. van Loon et al. Herit Sci (2020) 8:25
https://doi.org/10.1186/s40494-020-00364-5

Out of the blue: Vermeer's use of ultramarine and XAFS

Sulphur K edge XANES or **X**-ray **A**bsorption **N**ear **E**dge **S**tructure



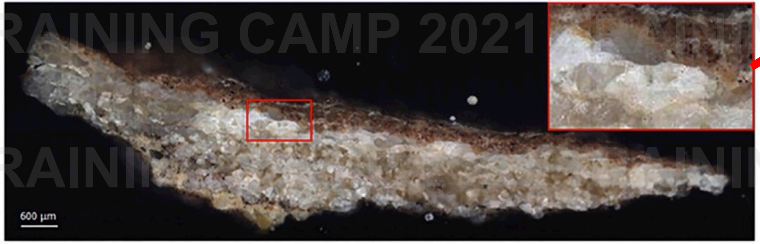
Analysis with synchrotron sulphur K-edge XANES suggested that **the ultramarine pigment was prepared- at least in part- from a heat-treated lapis lazuli rock**.

The relative ratio of peaks within the **'envelope' region- namely those at 2471.2, 2472.5, and 2473.8 eV - indicate the influence of heat-treatment** . It was recently found that for lazurite extracted from **lapis lazuli heated at 600 °C** the relative intensity of the peak at 2471.2 eV was greater than that at 2472.5 and 2473.8 eV

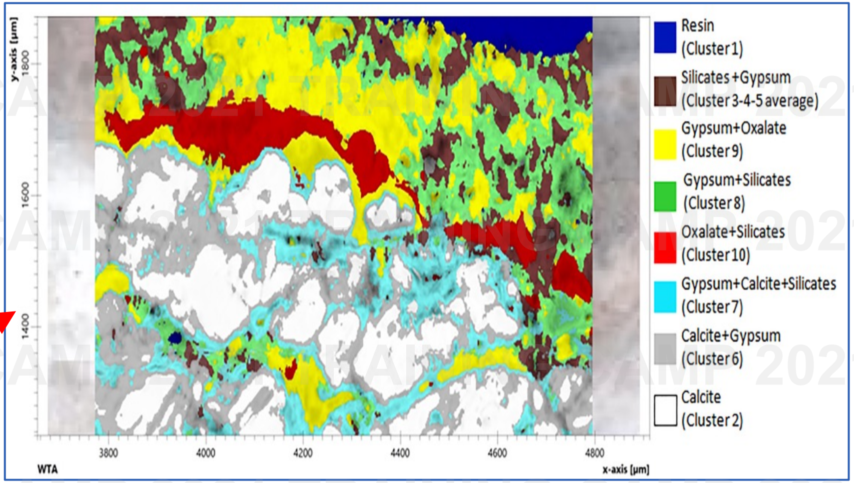
A. van Loon et al. Herit Sci (2020) 8:25
https://doi.org/10.1186/s40494-020-00364-5

Degradation products in artwork cross-section and FTIR

Among the different analytical techniques, FTIR imaging provides information on the Molecular Composition of the Material on a micrometric-scale in a **NOT DESTRUCTIVE** way. Establishing the distribution of materials and that of their degradation products in historical monuments/paintings is fundamental to **understand their CONSERVATION STATUS and give information for ART RESTORATION.**



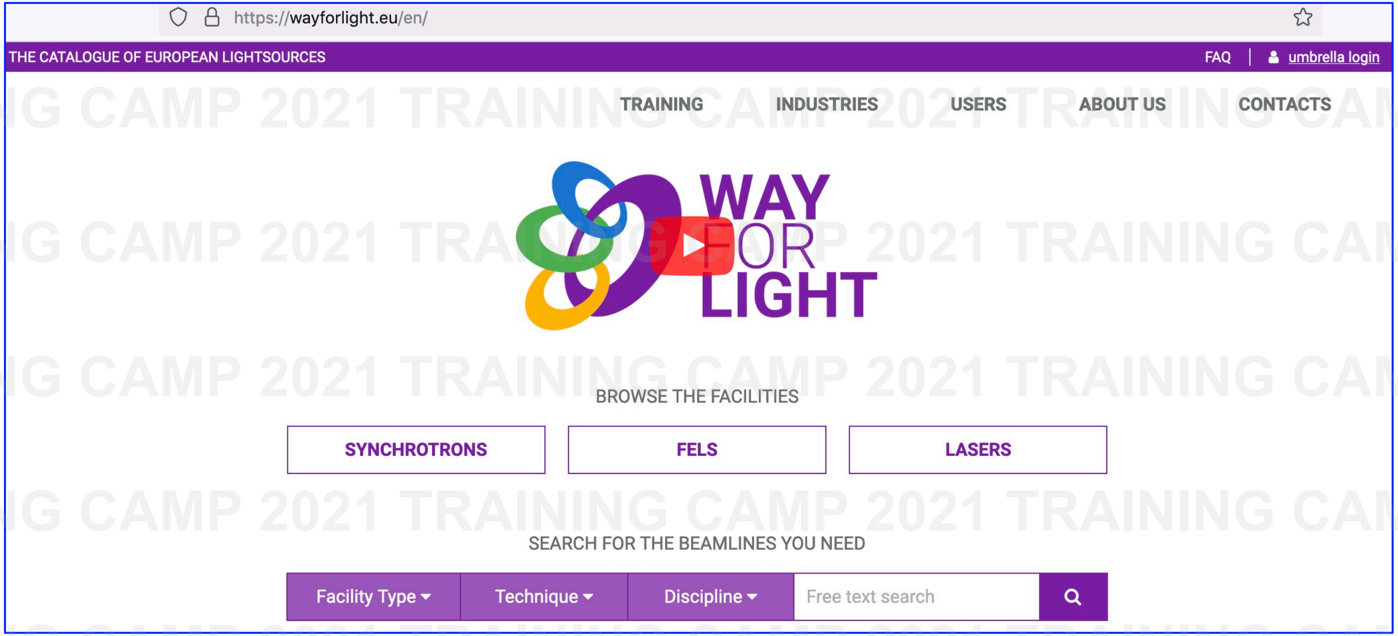
G. Capobianco et al. Microchemical Journal (2017) 132,69
https://doi.org/10.1016/j.microc.2017.01.007



Small fragment of Septimius Severus's Arch - ***Foro Romano (III AD)*** **new perspectives for FTIR imaging in Art Conservation for the study of the distribution of different components.**

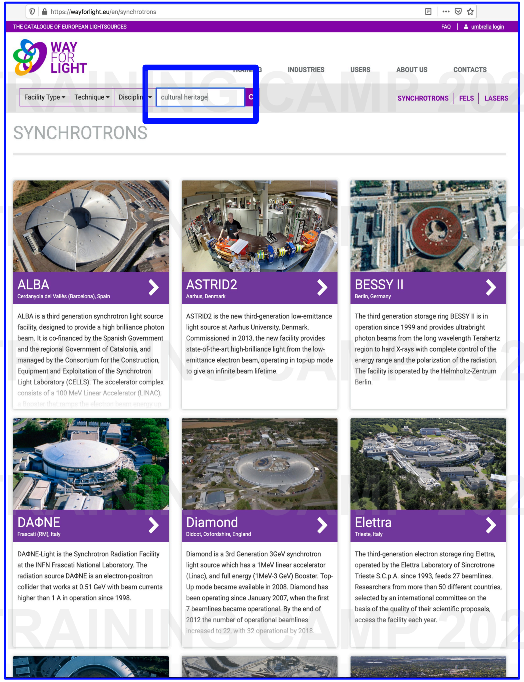
How to apply for beamtime

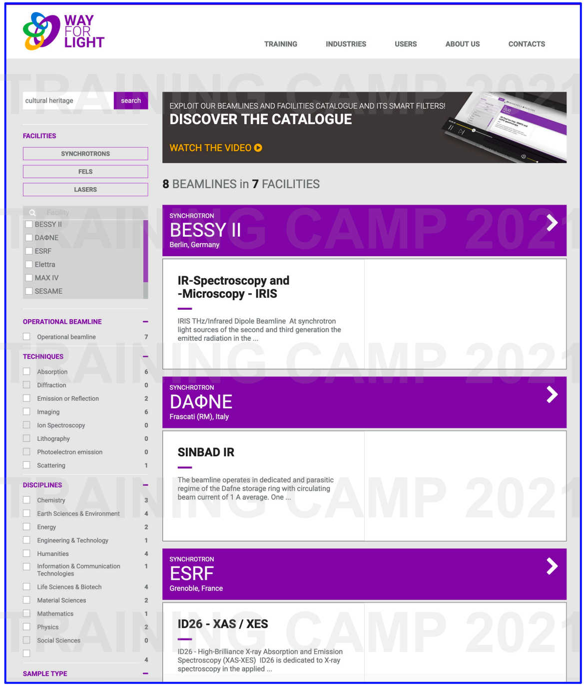
WAYFORLIGHT or Catalogue of the EU Light sources

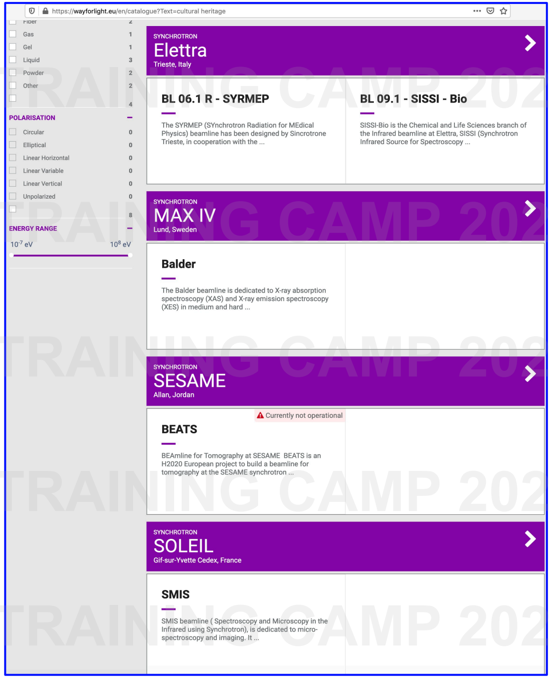


https://wayforlight.eu/en/

WAYFORLIGHT and Cultural Heritage







WAYFORLIGHT: Deadlines and calls

NEXT CALL DEADLINES

Elettra	15.09.2021
Diamond	29.09.2021
FLASH at DESY	01.10.2021
SOLARIS	01.10.2021
MAX IV	05.10.2021
DAΦN...	18.10.20...

view all the deadlines

USERS / NEXT CALL DEADLINES

SYNCHROTRON

15.09.2021 — **Elettra**

NEXT DEADLINE:

- **September 15th, 2021** for experiments to be scheduled from January 1st to June 30th, 2022
- Protein Crystallography: proposals can be submitted anytime, there is no deadline for the submission and evaluation is monthly.

SYNCHROTRON

29.09.2021 — **Diamond**

- First Wednesday in April (17:00) (for the period 1st October 31st March)
- First Wednesday in October (17:00) (for the period 1st April to 30th September).

Details for the next calls for Direct Access and Programme Mode are on the web at: http://www.diamond.ac.uk/Users .

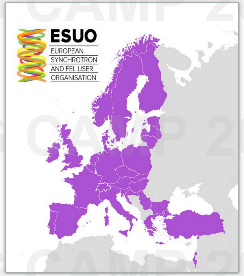
In addition to call deadlines specified above, there are also opportunities for rapid access to some beamlines. More details are given on the Diamond website by using the specified link.

FEL

01.10.2021 — **FLASH at DESY**

The deadline for the current call for proposals for FLASH is on 1 October 2021.

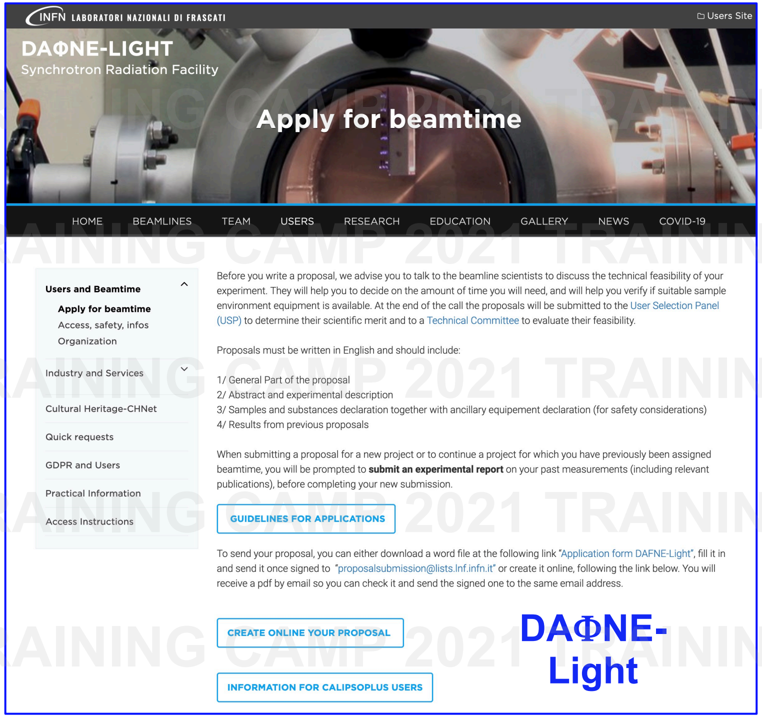
Due to the upcoming FLASH2020+ shutdown (from mid-November 2021 to mid-August 2022), this call relates to beamtime from November 2022 to June 2023. In order to facilitate a most efficient use of the FLASH beamtime it is expected that your own experimental setup, if provided, will be ready by November 2022.

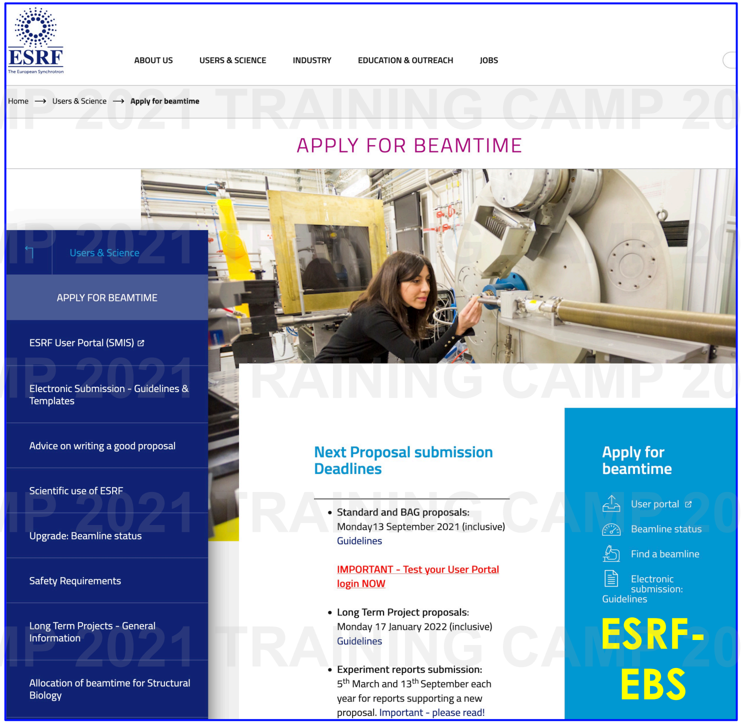


STANDARDISED PROPOSAL FORM

read more

Appling for beamtime





Beamtime and proposals

Proposals

- **Abstract or proposal summary**
- **Aims of the experiments and background (scientific background)**
- **Experimental Methods (measurement strategy)**
- **Beamlines and Beamtime requested**
- **Results Expected**
- **References**

- Main or Principal Proposer and Co-proposers

N.B. for any question or doubt concerning the experimental method or required shifts contact the Beamline Scientist

Abstract or proposal summary

- The summary will usually be the last thing to write, although it will appear at the beginning.
- Its purpose is the following: reviewers that are forced to read many proposals will not memorize all of them very well.
- You need to give them a very quick way to remember what was your point, in order to allow them to ‘defend’ your proposal in the review panel meeting, and grade it appropriately.

Aims of the experiments and background

- You need to explain in a rather compact manner (1/2 page or so) the status of our field, which are the open questions, and what is needed to answer the questions.
- You have to give an introduction in only few sentences, to make the point of what is still *problematic*, *unsolved*, *unknown* and it is also important to show that our sample has already been characterized by as many non-SR techniques as possible.
- Use literature references, but not too many and also refer to your own work on the topic, with or without SR, as well. Since at least a number of the panel members are experts of the field and know the literature, they will get an impression from the references: quoting one or two important references is therefore a good idea, since this indirectly shows our expertise.

Experimental Methods

- Here you need to explain how measurements will be performed, the instrumentation needed, number and kind of samples to be measured and how you are going to analyse the data.
- Beside the referees also the beamline scientist that will review our proposal to comment whether the proposed experiment is feasible at the chosen beamline.
- The referee needs to judge how experienced you are, how difficult and feasible the experiment is. If it is particularly challenging also this can be very attractive. Synchrotron facilities prefer to host the most forefront experiments.

Beamlines and Beamtime

- Here you must justify why you ask for a specific beamline, and how much beamtime is required for our experiment.
- The selection of the beamline is sometimes critical, but you already know that a certain beamline is well suited for the proposed experiment.
- The amount of beamtime is another question: while you want as much as possible, asking for unrealistically many "shifts" (typically 8 hours, so one day are three shifts) will rather be seen as a negative aspect of the proposal: reviewers will assume you know how synchrotrons operate and how beamtime is allocated. On the other hand it is clear that certain measurements take some time, you might need to measure at least three samples to get a trend of one parameter, and to ask for one shift extra for setup of the beamline is usually o.k.

Results Expected

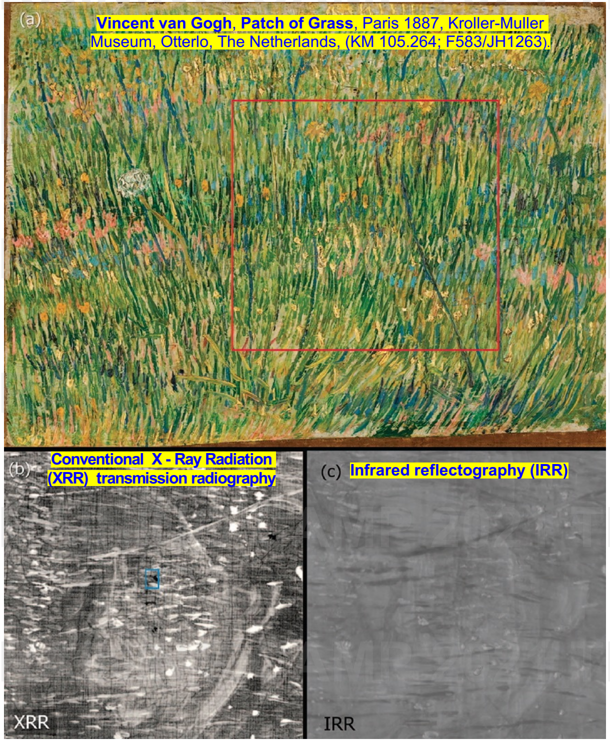
- You need to give a very compact view of what you hope to learn from the proposed experiment. It is better to be very clear and put well-defined, attainable results here. Making a list is not a bad idea, it makes it easier for the reviewers to orient themselves in the proposal.
- A particular problem is to find the balance between important enough results and not overdoing it. Having, in a realistic amount, the larger goal in mind, may help in cases, also to convince the panel with its diverse background that the scientific issue is topical and of a wider interest.

References

- As mentioned above, references do not serve the same purpose in a short proposal than in a publication. Of course you must refer to the original literature but since you do not have too much space, you can restrict yourselves to a few important papers of the field, high-impact ones and reviews, to show that you know the relevant literature.
- You must also refer to our own publications on the topic, with or without application of SR, to show that you are already experienced (to some extent) in the field, have done previous work (including characterization of the material by in-house, non-SR techniques), know the method etc. All this primarily serves the purpose of convincing the referee that you will manage to perform the proposed work.

Case study

Visualization of a Hidden Painting of van Gogh



Abstract or proposal summary

Vincent van Gogh (1853-1890), **one of the founding fathers of modern painting**, is best known for his vivid colours, his vibrant painting style, and his short but highly productive career.

His **productivity is even higher than generally realized, as many of his known paintings cover a previous composition**. This is thought to be the case in one-third of his early period paintings. **Van Gogh would often reuse the canvas of an abandoned painting and paint a new or modified composition on top**. These hidden paintings offer a unique and intimate insight into the genesis of his works. **Problem to solve**

Current museum-based imaging tools are unable to properly visualize many of these hidden images. Using **synchrotron radiation based X-ray fluorescence (XRF) mapping**, recording at a decimetre-scale XRF intensity maps of specific elements in the paint layers, will give the possibility to reveal the distribution of specific elements in the paint layers and bring to the **visualizing a woman's hidden face under the work Patch of Grass by Van Gogh with unprecedented detail**. **Purpose**

This reconstruction can be the missing link for the comparison of the hidden face with Van Gogh's known paintings and **this approach literally can open up new vistas in the non-destructive study of hidden paint layers**. **Defend your Proposal**

J. Dik et al., ***Visualization of a Lost Painting by Vincent van Gogh Using Synchrotron Radiation Based X-ray Fluorescence Elemental Mapping***, **Anal. Chem. 2008**, 80, 6436

Visualization of a Hidden Painting of van Gogh

Aims of the experiments and background

Vincent van Gogh is generally recognized as one of the founding fathers of modern painting [1]. **In recent decades his work has undergone extensive art historical and technical study**. One striking feature that emerged **is Van Gogh's frequent reuse of paintings in order to recycle the canvas** [2]. The artist would simply paint a new composition on top of an existing work. **This is usually attributed to the artist's lifelong economic hardship and the rapid, energetic evolution of his artistic ideas**. **Visualizing such hidden paintings is of interest to both specialists in the field of Van Gogh and the public alike**. Covered paintings in general provide an **insight into the making of artworks and the underlying conceptual changes**. In the case of Van Gogh, they also present a touchstone for comparison with preparatory drawings and the abundant literary record. Background

Non-destructive imaging of such hidden paint layers is usually realized by means of tube-based X-ray radiation transmission radiography (XRR). The absorption contrast in these images is mostly caused by **the heavy metal components of pigments employed**, such as lead in lead white or mercury in vermillion. **Conventional XRR, however, has a number of important limitations.** First of all, the observed X-ray absorbance is a summation of all element-specific absorbances. This implies that the contribution to the overall image contrast due to (low quantities of) weakly absorbing elements will frequently be obscured by heavier elements that are present in higher concentrations. Second, prior to the application of the paint layer, a canvas is usually primed with a homogeneous layer of lead white. This raises the overall background of the absorption image derived from the paint layers. Finally, **the polychromatic character of an X-ray tube further reduces the contrast in radiographic images**. As a result, **conventional XRR imaging of paintings frequently provides only a fragmentary view of their substructure, which can severely hamper the readability of hidden compositions** [3]. Background

The painting by Van Gogh from his 1886-87 Paris period, **Patch of Grass** (Kroller-Muller Museum, Otterlo, The Netherlands) was earlier examined with XRR and infrared reflectography (IRR) **vaguely revealing a head under the surface painting**. However, due to the limitations of XRR, the facial characteristics could not be clearly read, making the person portrayed far from identifiable. Instead of **using the absorption of primary X-rays as an imaging method, one can also record the intensity of secondary radiation, emitted by the atoms in the painting while a pencil beam of energetic X-rays is scanned over the surface**. This fluorescence technique has the added advantage that the emitted X-ray radiation is element specific. The covering surface layers will not significantly attenuate the high-energy fluorescence signals from heavy elements in the hidden layers; in this manner, the distribution of both minor and major components in the painting can be visualized. **The use of high intensity X-ray beams reduces the dwell time for data acquisition to such an extent that large, decimetre- sized areas can be scanned**. Aims of the experiment

Visualization of a Hidden Painting of van Gogh

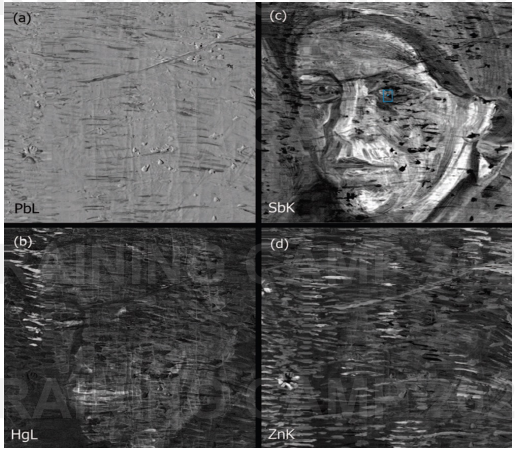
Experimental Methods

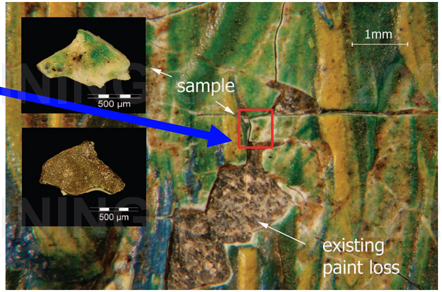
Safe transport of Patch of Grass to a micro-fluorescence synchrotron radiation beamline.

A **pencil beam** (0.5 x 0.5 mm^2) of **quasi monochromatic synchrotron radiation with an energy of 38.5 keV to be used for primary excitation**. With this beam a square of approximately **17.5 x17.5 cm^2**, corresponding to the position of the covered head must be scanned. With a **dwell time of 2 s per pixel**, so that the **total scan time was approximately 2 days**. Fluorescence spectra must be recorded for each pixel with a high-resolution energy dispersive Ge-detector.
X-ray Absorption Near Edge Structure (XANES) spectroscopy to learn about its chemical binding of the most relevant elements including standards.

A **microscopic sample must be taken from the painting** in order to study the **elemental composition of the upper and lower paint**. The paint sample will be embedded in epoxy resin to be measured with a SEM microscope with EDS.

As a complement to the high energy measurements performed on the painting itself, the same polished section employed for **SEM analysis** must be used for **characterization by means of low-energy synchrotron X-ray microbeam analysis**. The objective is to determine the elemental components and the correlation of their distribution within the paint layers. Under vacuum, **μ-XRF (1.1 x 0.3 μm^2 /horizontal - vertical) and μ-XANES measurements must be performed** at lower absorption edges for a better **identification of the pigments including standards**.





Beamlines --> WAYFORLIGHT Beamtime --> Beamline Scientists

J. Dik et al., *Visualization of a Lost Painting by Vincent van Gogh Using Synchrotron Radiation Based X-ray Fluorescence Elemental Mapping*, Anal. Chem. **2008**, 80, 6436

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Results Expected - XRF

1) **Elemental distribution images once reconstructed from the resulting sets of peak areas must be compared with features on the surface painting as well as the XRR and IRR images**.
Reconstruction of a significantly clearer and more detailed image of the hidden composition than the XRR and IRR images taken earlier. The brushstrokes and all physiognomic details, such as eyes, nose, mouth, and chin could be visualized. Understand if this reconstruction can prove to be the missing link for the comparison of the hidden face with Van Gogh's known paintings.

(a) **Tritonal color reconstruction of Sb (yellowish white) and Hg (red)** representing the flesh color of the hidden face. (b) Detail from Vincent van Gogh, Head of a Woman, Nuenen, winter 1884-85, oil on canvas, 42 cm °ø 33 cm, Kroller-Muller Museum, Otterlo (KM 105.591; F154/JH608). (c) Detail from Vincent van Gogh, Head of a Woman, Nuenen, winter 1884-85, oil on canvas, 42 cm °ø 34 cm, Van Gogh Museum, Amsterdam (F156/JH569).

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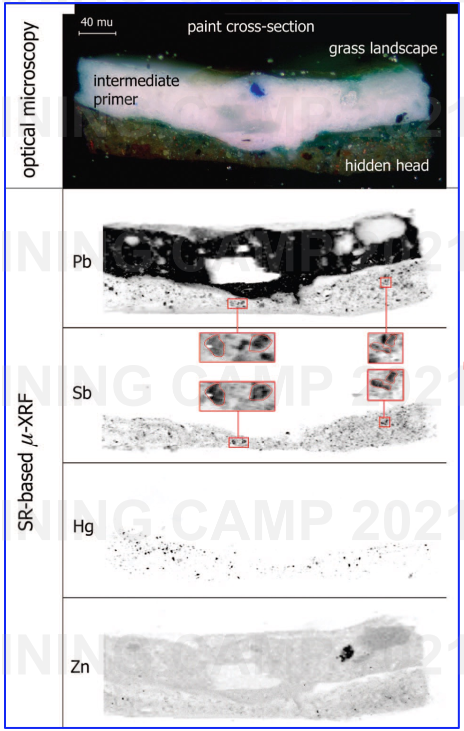
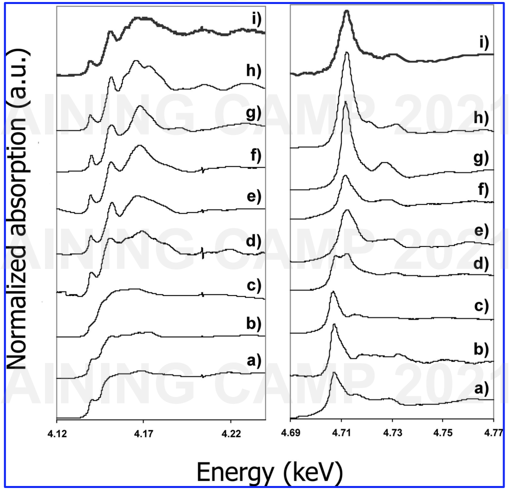
Results Expected XRF and XANES

2) Determination from the elemental distribution maps acquired by XRF of the most important traced element (probably antimony - Sb) of the hidden layer, measured on the most interesting points of the hidden layer and information using **X-ray Absorption Near Edge Structure (XANES) spectroscopy on its chemical binding to understand the pigment used**.

J. Dik et al., *Visualization of a Lost Painting by Vincent van Gogh Using Synchrotron Radiation Based X-ray Fluorescence Elemental Mapping*, Anal. Chem. 2008, 80, 6436





XANES spectra at the Sb-L_{III} edge (left spectrum) and at the Sb-L_I edge (right spectrum). Reference antimony compounds: Sb_2O_3 as (a) valentinite and as (b) senarmontite; (c) Sb_2S_2O, kermesite; (d) Sb_2O_4; (e) Sb_3O_6OH, stibiconite; (f) $KSbO_3 \cdot 3H_2O$; (g) $NaSbO_3OH \cdot 3H_2O$; **(h) Naples yellow**; and (i) **Sb pigment in the cross section of the Van Gogh painting**.

Visualization of a Hidden Painting of van Gogh

Why Synchrotron Radiation ?

- **Portable XRF allows for non-destructive elemental analysis** but has a **rather larger footprint on the painting**.
- It's acquisition **time per pixel is high** (200 s), so that scanning is fairly impossible. **Synchrotron sources have a high brightness and energy range compared to classical sources**.
- **SR-based XRF therefore allows one to quickly scan large, decimeter-scale areas with sub-millimeter spatial resolution, but the painting needs to be transported to an SR facility**.
- SEM has an easy access, has a higher lateral resolution, and probes a thinner superficial layer than SR µXRF. **Vice versa, µXRF has a higher sensitivity, notably for heavier elements. In addition, polychromaticity is essential for µXANES, which complements elemental identification with their chemical characteristics.**

References

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3) Krug, K., Dik, J., den Leeuw, M. *et al.* Visualization of pigment distributions in paintings using synchrotron *K*-edge imaging. *Appl. Phys. A* **83,** 247–251 (2006). https://doi.org/10.1007/s00339-006-3519-y

Conclusions

Conclusions

- Synchrotron Radiation as a **powerful tool** in the Cultural Heritage field.
- Compared to laboratory instrumentation it gives the possibility to **quickly scan large, decimetre-scale areas with sub-millimeter spatial resolution.**
- The **polychromaticity** of the synchrotron radiation source give s the possibility to **exploit also other spectroscopic techniques** that like XANES, which complements elemental identification giving **information on their chemical composition.**
- **The artworks must be transported to the facilities.**
- **You need to submit an experimental proposal than can seem complicated at the beginning but becomes simpler with time.**

Thank you for your attention

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