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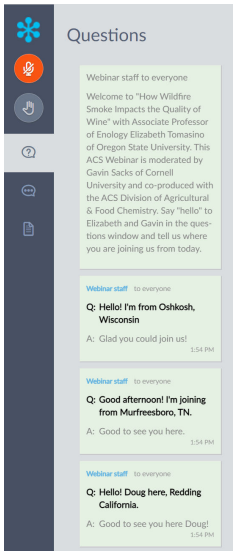

"Why am I muted?"
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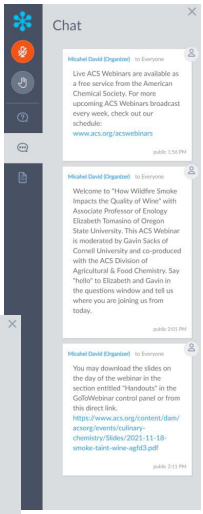
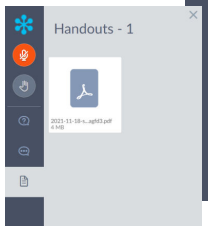
Announcements and hyperlinks from our team



Handouts

Download the PDF of today's slide deck



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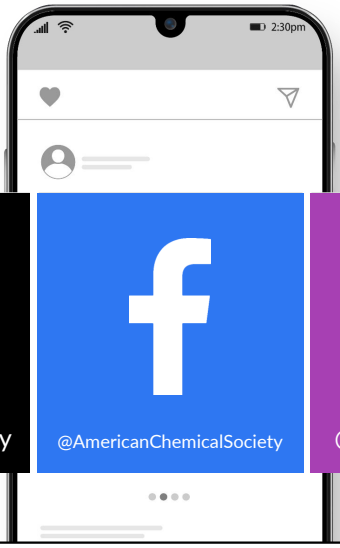

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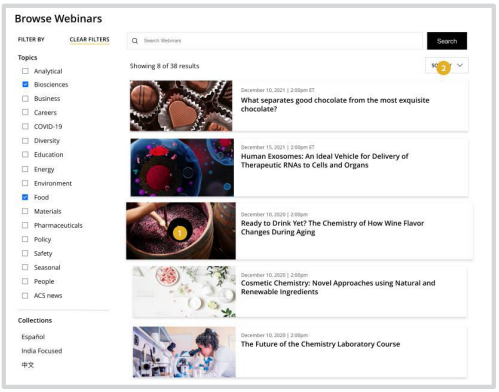

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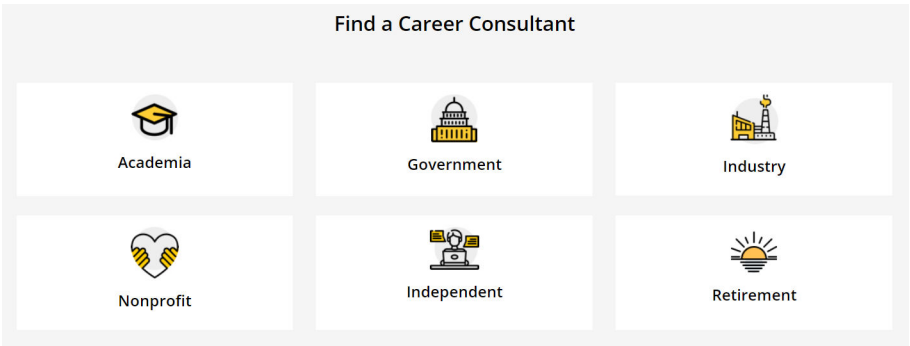

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Promote Successful program components

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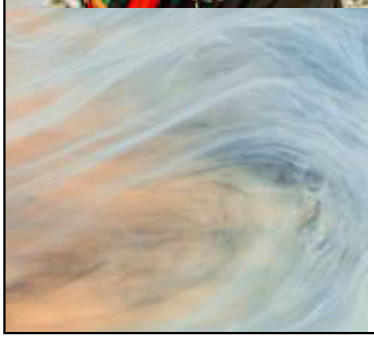
Group picture from 2022 CKS at ACS HQ in Washington, DC

Being a part of the ACS Bridge program has benefited me in several ways. I was able to pursue fully independent, fascinating research at a top institution, but even more importantly, I was exposed to a number of opportunities (such as conferences, career events, etc.) I never would have known about otherwise. The best thing about Bridge in my opinion, are the people at ACS who have worked to make it happen. Their dedication to helping me develop professionally and supporting me in good or bad times I will forever be grateful for.

Hanin Sarhan, Bridge Fellow at Indiana University



ACS Scholar Adunoluwa Obisesan

BS, Massachusetts Institute of Technology, June 2021
(Chemical-biological Engineering, Computer Science & Molecular Biology)

"The ACS Scholars Program provided me with monetary support as well as a valuable network of peers and mentors who have transformed my life and will help me in my future endeavors. The program enabled me to achieve more than I could have ever dreamed. Thank you so much!"

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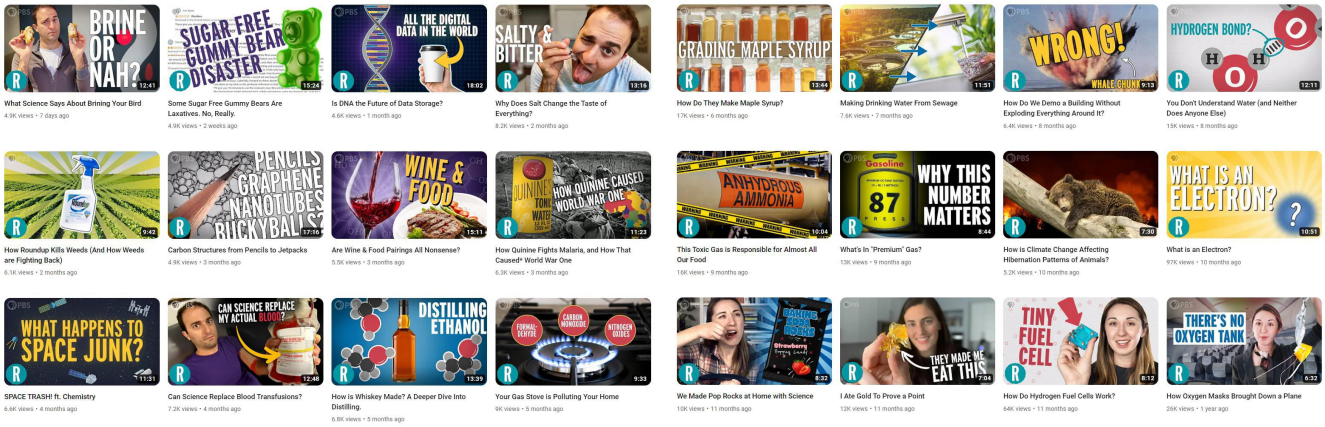

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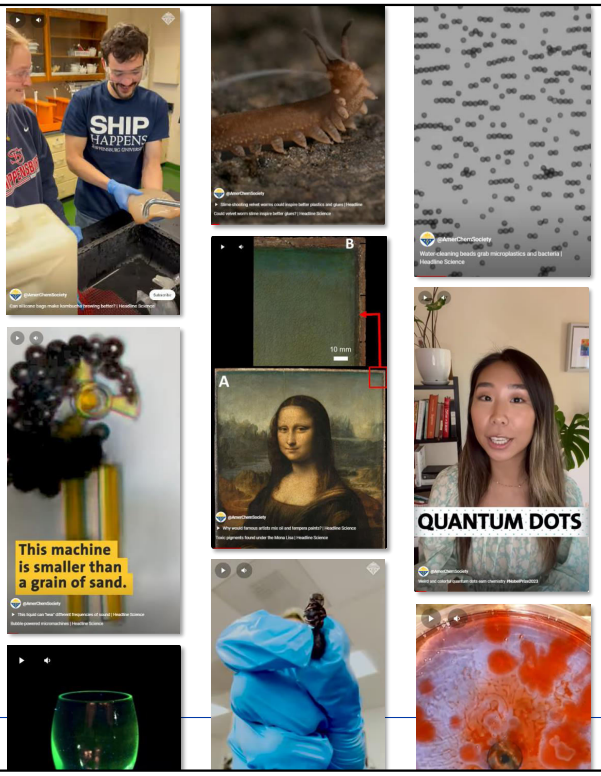

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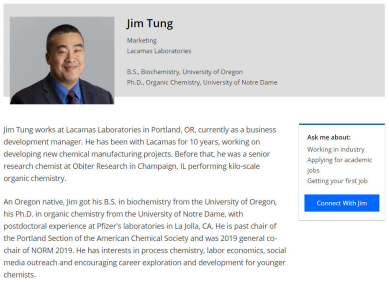

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Advancing ACS' Core Value of Diversity, Equity, Inclusion and Respect

Resources

Inclusivity Style Guide
Designed to help staff and members use language and images that respect diversity in all its forms.

ACS Webinars on Diversity
Covering diversity and inclusion at the workplace

ACS Publications DEIR Hub
See what ACS Publications is doing for fostering inclusivity in scholarly publishing

ACS Volunteer and ACS Meetings Code of Conduct
Fostering a positive and welcoming environment for attendees, volunteers and staff.

C&EN Trailblazers
C&EN highlights scientists from different backgrounds who are making an impact in chemistry.

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Download this educational guide for additional recommendations on videos, articles, books, podcasts, and more on diversity, inclusion, and related topics.

Quick Guide: Inclusion Moments
Learn more about what Inclusion Moments are and see ideas to host them during your meetings.

Quick Guide: How to host inclusive in-person events
Recommendations and best practices to ensure that your events can accommodate everyone.

Diversity, Equity, Inclusion, and Respect

**Adapted from definitions from the Ford Foundation Center for Social Justice:

Equity**
Seeks to ensure fair treatment, equality of opportunity, and fairness in access to information and resources for all. We believe this is only possible in an environment built on respect and dignity. Equity requires the identification and elimination of barriers that have prevented the full participation of some groups.

Diversity**
The representation of varied identities and differences (race, ethnicity, gender, disability, sexual orientation, gender identity, national origin, tribe, caste, socio-economic status, thinking and communication styles, etc.), collectively and as individuals. ACS seeks to proactively engage, understand, and draw on a variety of perspectives.

Inclusion**
Builds a culture of belonging by actively inviting the contribution and participation of all people. Every person's voice adds value, and ACS strives to create balance in the face of power differences. In addition, no one person can or should be called upon to represent an entire community.

Respect
Ensures that each person is treated with professionalism, integrity, and ethics underpinning all interpersonal interactions.

https://www.acs.org/diversity

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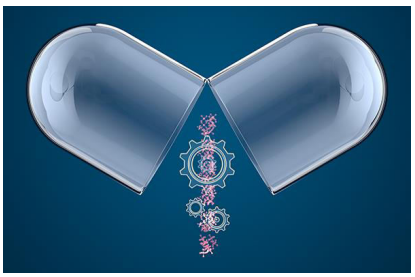

Wednesday, June 26, 2024| 2pm-3pm ET

Diseño Racional de Sistemas de Liberación de Fármacos

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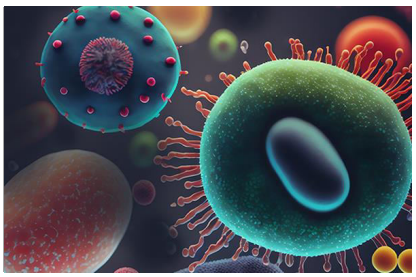

Thursday, June 27, 2024| 11am-12:30pm ET

Microbiome Mechanics: Building a Healthier Gut

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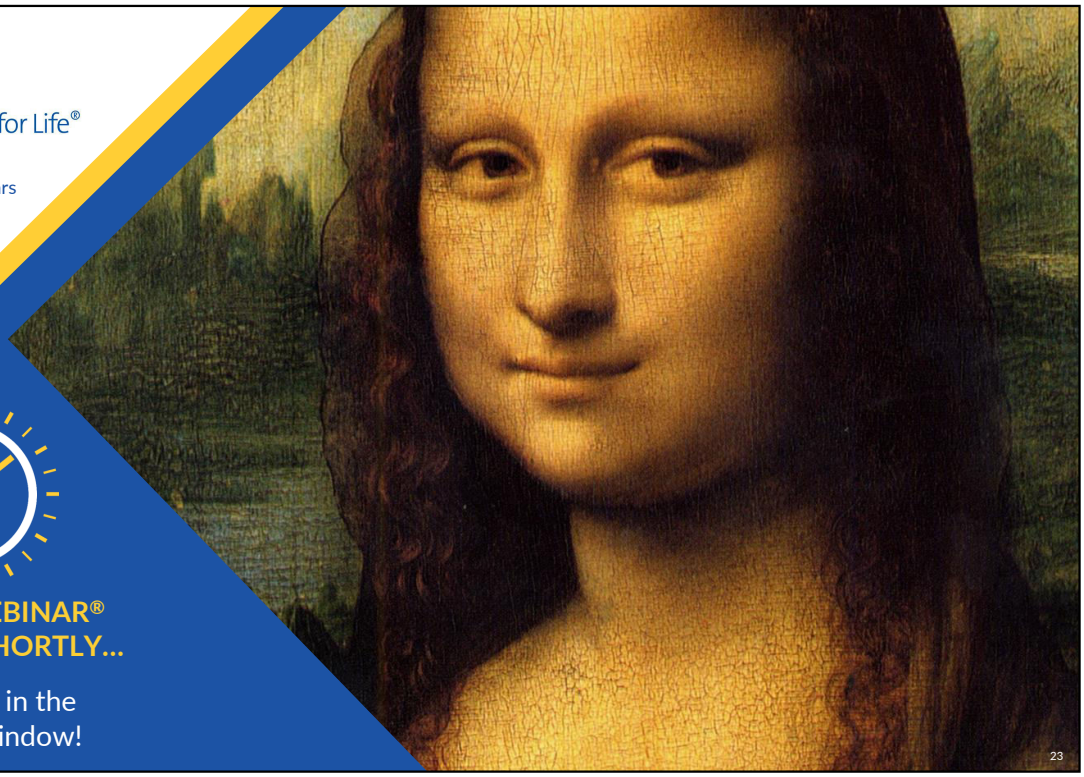

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Revealing Mona Lisa's Secrets Through Advanced Analytical Chemistry

VICTOR GONZALEZ, PhD

Researcher, French National Center for Scientific Research

PANČE NAUMOV, PhD

Full Professor of Chemistry, NYU Abu Dhabi and Global Network Professor of Chemistry, NYU

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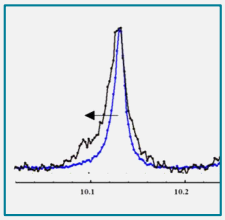
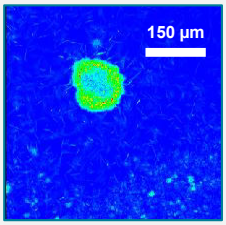
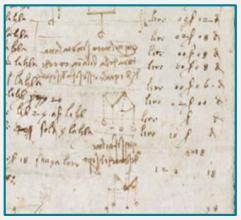
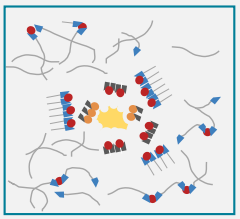
Revealing Mona Lisa's Secrets Through Advanced Analytical Chemistry

Victor Gonzalez
Université Paris-Saclay, ENS Paris-Saclay, CNRS, PPSM



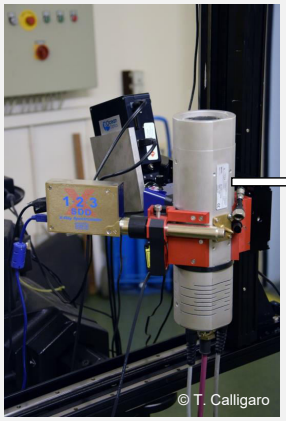


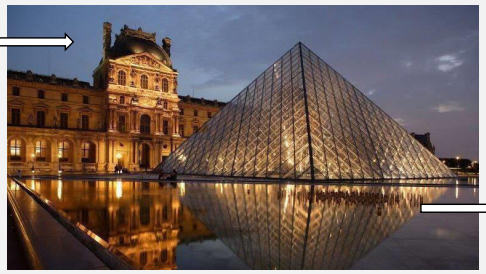


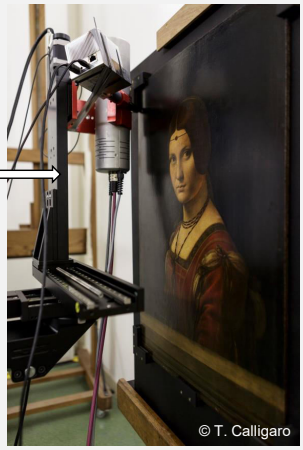


STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION | **STRUCTURE / OPTICAL PROPERTIES RELATIONSHIPS IN LEAD CARBONATES** | **PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI**

MACRO-SCALE ANALYSIS







- ✓ *in situ* analysis
- ✓ Versatility
- ✓ Chemical images at the scale of paintings

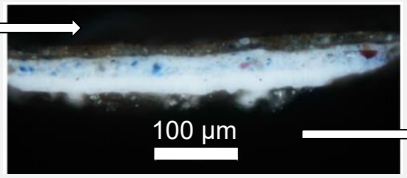
La Belle Ferronnière (**62×44 cm**)
c. 1495-1497, Musée du Louvre

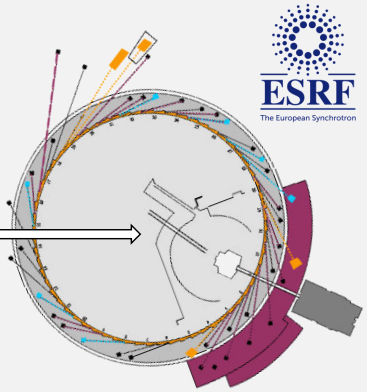
Macro X-ray Fluorescence (MA-XRF)
Lead (Pb) Kα map

168 cm

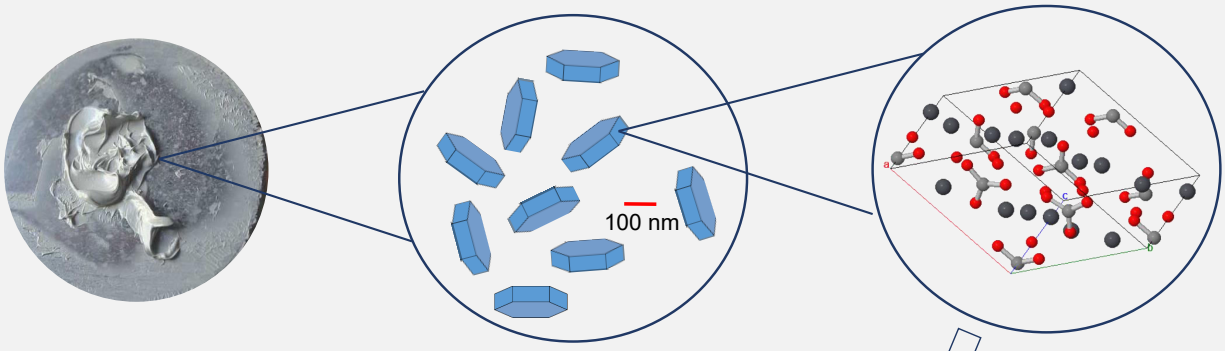
The Virgin and Child with Ste. Anne (**168×130 cm**)
c. 1503-1519, Musée du Louvre

MICRO-SCALE ANALYSIS





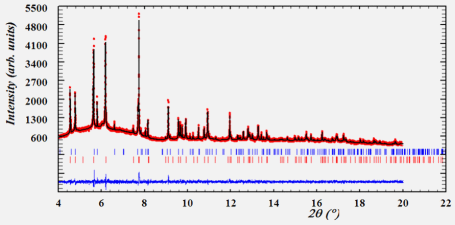
- ✓ **Speed**
- ✓ **Tunable beam size**
- ✓ **Sensitivity**
- ✓ **Signal / noise ratio**



X-ray diffraction

Crystalline phases
e.g Pb-carbonates :
Cerussite ($PbCO_3$) + Hydrocerussite ($Pb_3(CO_3)_2(OH)_2$)

Quantification **Distribution** **Crystallite sizes**

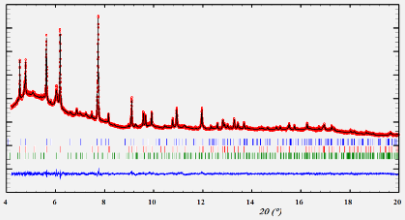


STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION
STRUCTURE / OPTICAL PROPERTIES RELATIONSHIPS IN LEAD CARBONATES
PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI

ID22 : High-angle resolution XRD (SR-HR-XRPD)

- Energy: ~35 keV
- Scan range 2θ: 3 – 20°
- Analysis time: ~20 min/powder;~2 h/historical sample
- Instrumental function 2θ (FWHM of (111)Si peak) ~ 0.0027°
- Samples in capillaries

→ Precise and sensitive identification of crystalline phases, quantification, and characterization of their microstructural and structural properties



ID13 : High-lateral resolution XRD (SR-µ-XRPD)

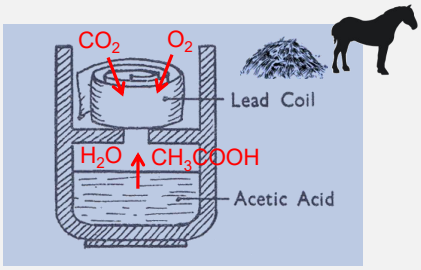
- Energy: ~13 keV
- Analysis time: ~10 min -2 h/map (15ms/ pixel)
- Beam size ~ 2×2µm²
- Samples as thin sections (preferable) or cross-sections

→ Stratigraphical distribution of crystaline phases at the micrometer scale

STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION

STRUCTURE / OPTICAL PROPERTIES RELATIONSHIPS IN LEAD CARBONATES

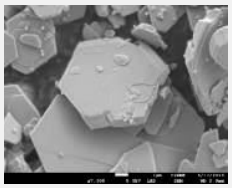
PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI



- Manufacture : *stack process* until the 19th c., then multiple synthesis ways
- Two main lead carbonates crystalline phases
- Mix with the organic binder by the artist

Lead white pigment

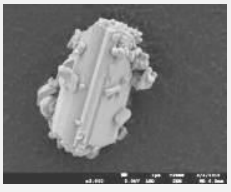
$Pb_3(CO_3)_2(OH)_2$
Hydrocerussite (HC)



rhombohedral R-3m
a = 5.2450 Å
c = 23.7009 Å

+

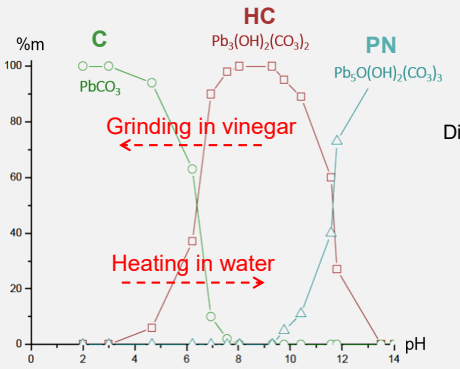
$PbCO_3$
Cerussite (C)



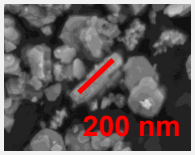
orthorhombic Pmcn
a = 5.1840 Å
b = 8.5001 Å
c = 6.1505 Å

V. Gonzalez, G. Wallez, T. Calligaro, D. Gourier, M. Menu, *Corr. Sci* (2019) **146**: 10

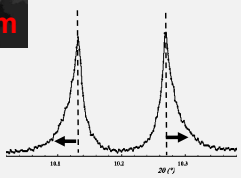
- Stability of Pb-carbonates is pH-dependant
- Influence of post-synthesis treatments on composition / micro-structure was examined in laboratory
- Fine micro-structural features (twinning) connected to post-synthesis



Washing / grinding in vinegar (AcOH)

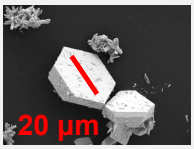


C > HC
Crystallite size ↓



Dissolution-cristallisation
Crystal twinning
Assymetrical Lorentzian broadening

Heating in water

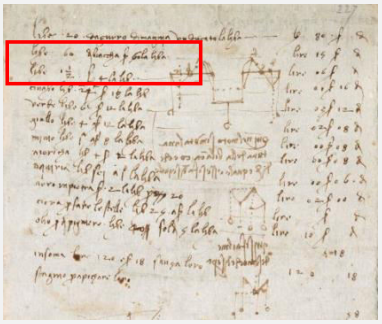


HC > C
Crystallite size ↑

V. Gonzalez, T. Calligaro, G. Wallez, M. Eveno, K. Toussaint, M. Menu, *Microchem. J.* (2016) **125**: 43

STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION
STRUCTURE / OPTICAL PROPERTIES RELATIONSHIPS IN LEAD CARBONATES
PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI

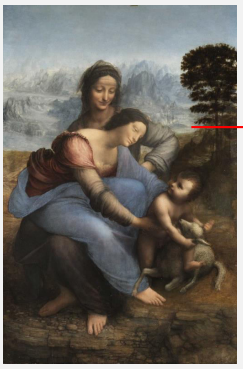
Arundel MS 263, f.227v – British Museum



- 5 pounds of lead white A at 3 lira / pound
- 1,5 pounds of lead white B at 4 lira / pound

Order by Leonardo of two distinct lead white subtypes

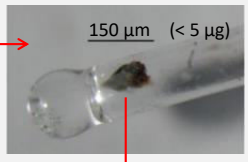
- Structural study on an extended artistic corpus, combined with Rietveld refinement
- Precise quantification of mineral phases and crystallites modeling at the nanometric scale

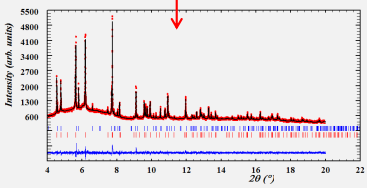


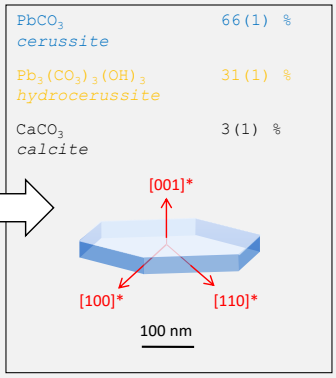
Leonardo da Vinci, *The Virgin and Child with Ste. Anne*, c.1503-1519

ESRF The European Synchrotron

High-angular resolution XRPD
ID22 beamline
λ = 0,3547 Å



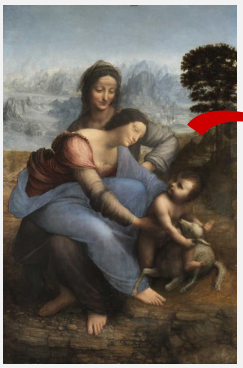




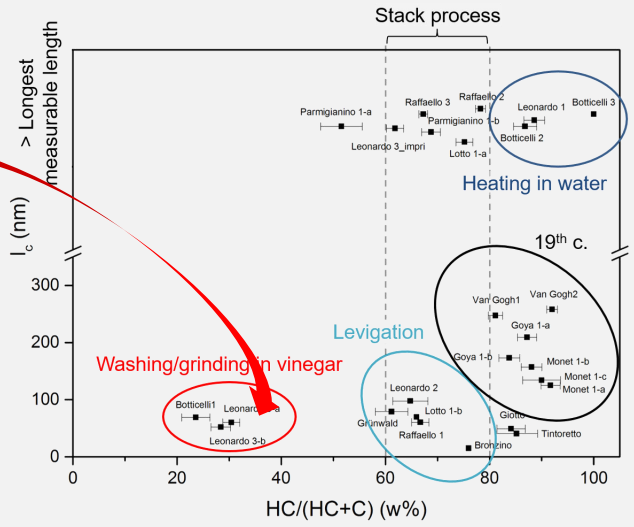
V. Gonzalez, G. Wallez, T. Calligaro, M. Cotte, W. De Nolf, M. Eveno, E. Ravaud, M. Menu, *Anal. Chem.* (2017) **89**(24): 13203

STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION
STRUCTURE / OPTICAL PROPERTIES RELATIONSHIPS IN LEAD CARBONATES
PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI

- Structural study on an extended artistic corpus, combined with Rietveld refinement
- Precise quantification of mineral phases and crystallites modeling at the nanometric scale
- Identification of post-synthesis processes in historical paintings



Leonardo da Vinci, *The Virgin and Child with Ste. Anne*, c.1503-1519



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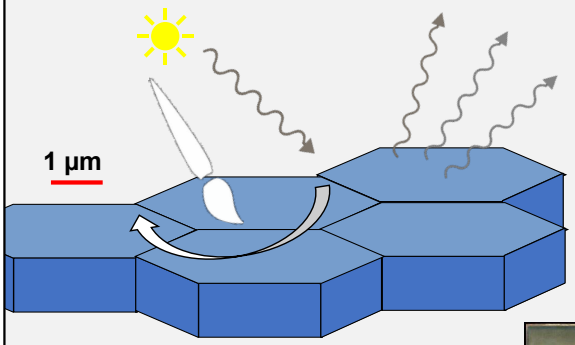
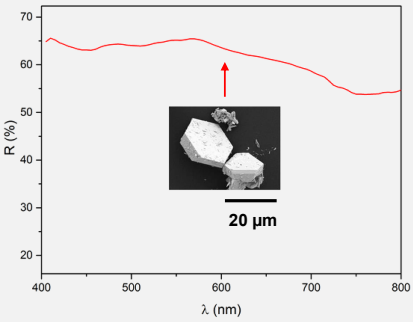
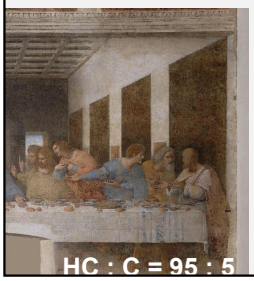


HC : C = 71 : 29





HC : C = 95 : 5

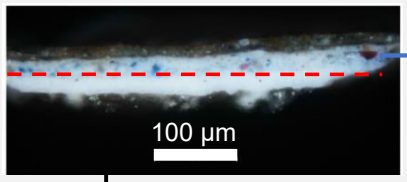
HC : C = 66 : 34

HC : C = 88 : 22

- Ground layers characterized by high content of hydrocerussite (HC : C > 60 : 40) ; large crystallites = optimal covering power
- Hypothesis : post-synthesis treatment: heating in aqueous environment?

STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION
STRUCTURE / OPTICAL PROPERTIES RELATIONSHIPS IN LEAD CARBONATES
PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI

Léonard de Vinci – *Sainte Anne, la Vierge et l'Enfant jouant avec un agneau,* (c. 1503-1519), Musée du Louvre

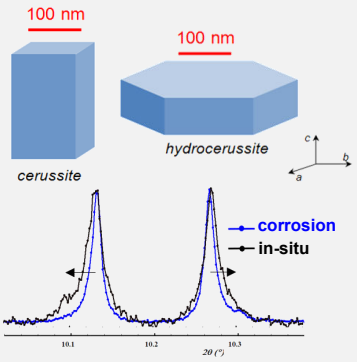


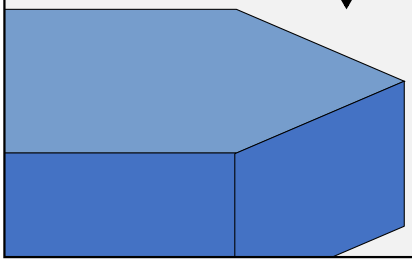
imprimatura

HC : C = 71 : 29

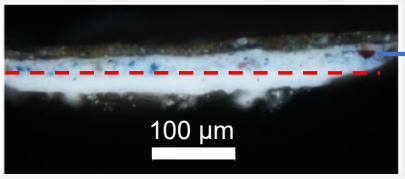
Top layer

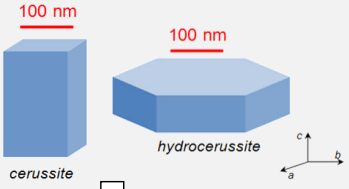
HC : C = 30 : 70





- Micro-structural features point to a process in acidic environment, inducing dissolution-recrystallization mechanisms
- Hypothesis : post-synthesis treatment: grinding/washing in acidic environment?



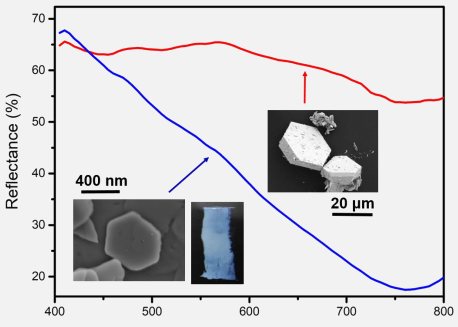


Top layer

HC : C = 30 : 70

« […] if he puts on it a very fine and transparent layer of ceruse, he will see that the white of this ceruse will actually be a beautiful blue, but it has to be very fine and well grinded »

Leonardo da Vinci, MS. LEIC. folio 4 (r)



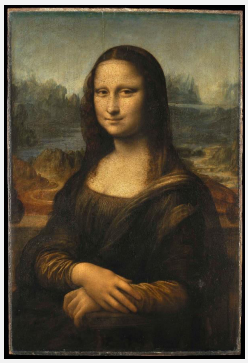
- Identification of pigment sub-types with specific optical properties, obtained following controlled processes
- Information of historical syntheses / material choices of Leonardo

V. Gonzalez, S. Hageraats, G. Wallez, M. Eveno, E. Ravaud, M. Thoury, M. Menu, D. Gourier, *Sci. Rep.* (2021) **10**:21715

STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION
STRUCTURE / OPTICAL PROPERTIES RELATIONSHIPS IN LEAD CARBONATES
PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI

- Study of Leonardo da Vinci's ground layers
- Choice of specific paint formulation for these grounds?

La Belle Ferronnière (**62×44 cm**)
c. 1495-1497, Musée du Louvre



Mona Lisa (**77×53 cm**)
c. 1503-1519, Musée du Louvre

The Virgin and Child with Saint Anne (**168×130 cm**)
c. 1503-1519, Musée du Louvre

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Article

X-ray and Infrared Microanalyses of *Mona Lisa*'s Ground Layer and Significance Regarding Leonardo da Vinci's Palette

Victor Gonzalez,* Gilles Wallez, Elisabeth Ravaud, Myriam Eveno, Ida Fazlic, Tiphaine Fabris, Austin Nevin, Thomas Calligaro, Michel Menu, Vincent Delieuvin, and Marine Cotte

Cite This: https://doi.org/10.1021/jacs.3c07000
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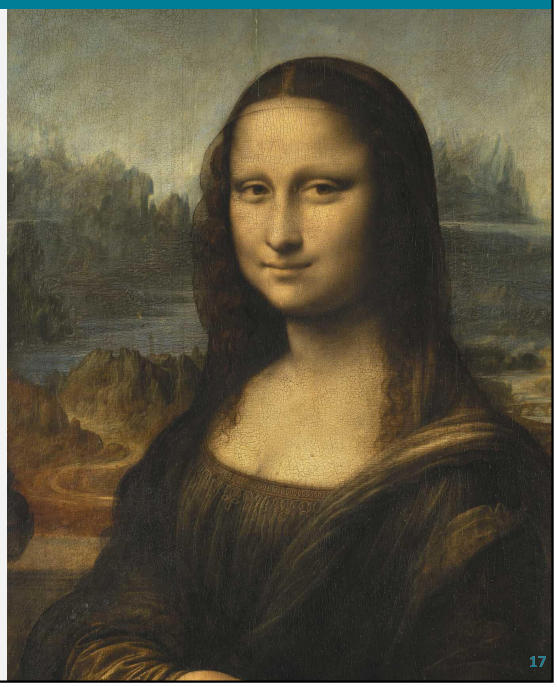
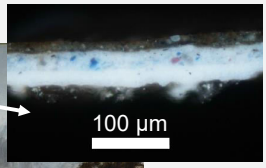


STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION
STRUCTURE / OPTICAL PROPERTIES RELATIONSHIPS IN LEAD CARBONATES
PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI

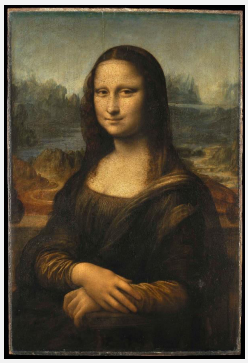
Oil-base ground
Lead white ($PbCO_3$ + $Pb_3(CO_3)_2(OH)_2$)
+ minium (Pb_3O_4)

Oil-based imprimitura
Lead white ($PbCO_3$ + $Pb_3(CO_3)_2(OH)_2$)
Glue-based ground
Gesso ($CaSO_4.2H_2O$)



?

La Belle Ferronnière (**62×44 cm**)
c. 1495-1497, Musée du Louvre

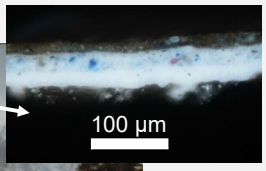


Mona Lisa (**77×53 cm**)
c. 1503-1519, Musée du Louvre

The Virgin and Child with Saint Anne (**168×130 cm**)
c. 1503-1519, Musée du Louvre

Oil-base ground
Lead white ($PbCO_3$ + $Pb_3(CO_3)_2(OH)_2$) + minium (Pb_3O_4)

Oil-based imprimitura
Lead white ($PbCO_3$ + $Pb_3(CO_3)_2(OH)_2$)
Glue-based ground
Gesso ($CaSO_4.2H_2O$)



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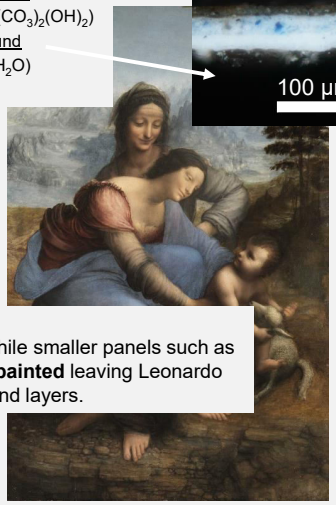

Hypothesis:
Large panels were already pre-prepared with *gesso* at the carpenter studio, while smaller panels such as the ones used in the *Belle Ferronnière* or the *Mona Lisa* were purchased **unpainted** leaving Leonardo more freedom to experiment mixtures of his own for the ground layers.

La Belle Ferronnière (**62×44 cm**)
c. 1495-1497, Musée du Louvre

Mona Lisa (**77×53 cm**)
c. 1503-1519, Musée du Louvre

The Virgin and Child with Saint Anne (**168×130 cm**)
c. 1503-1519, Musée du Louvre

STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION
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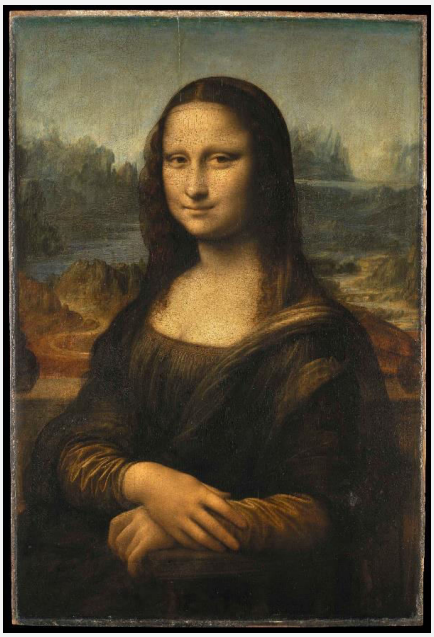
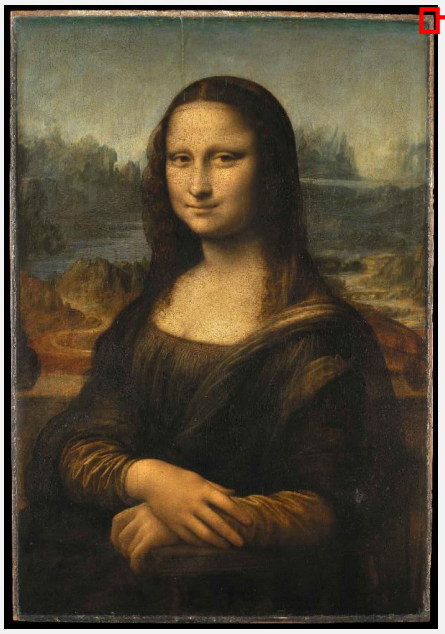
PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI

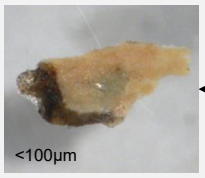


Pb-Lα MA-XRF map
© E. Laval, T. Calligaro – C2RMF

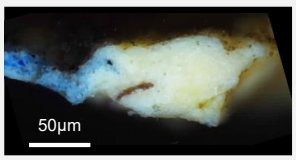
X-ray radiography
© E. Ravaud – C2RMF

The ground of the *Mona Lisa* contains **lead compounds.**



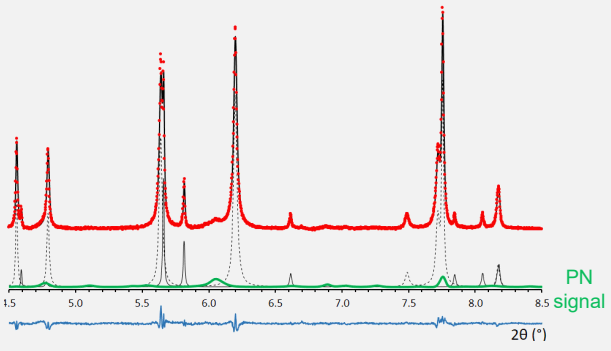


Unembedded part of the sample
(kept for SR-HR-XRPD analyses)

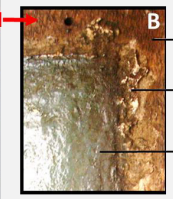


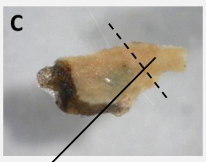
Part of the sample embedded in resin
(for SEM and µFTIR mapping)

- Detection of nano-crystalittes of plumbonacrite $Pb_5O(CO_3)_3(OH)_2$, meta-stable compound (pH > 10) in *Mona Lisa*'s ground layer

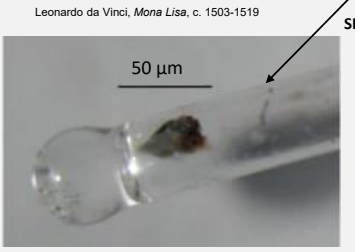


Leonardo da Vinci, *Mona Lisa*, c. 1503-1519





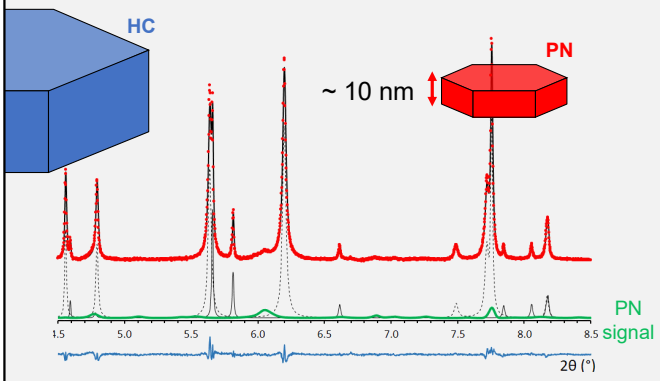
SR-HR-XRPD

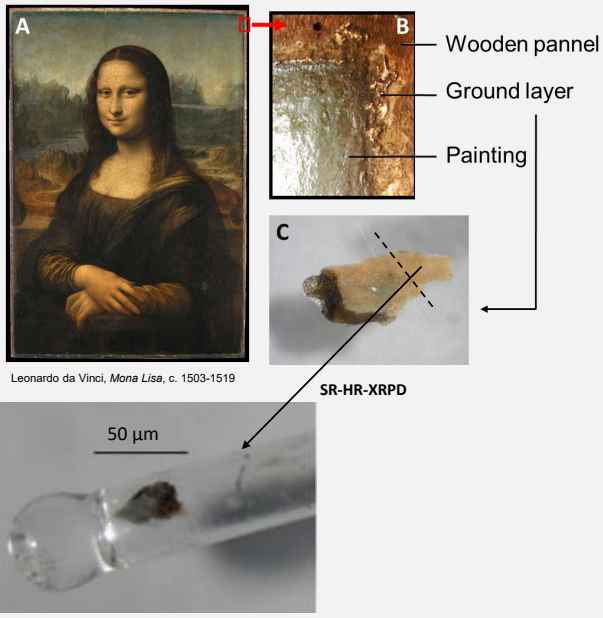


What created the chemical conditions for plumbonacrite formation?

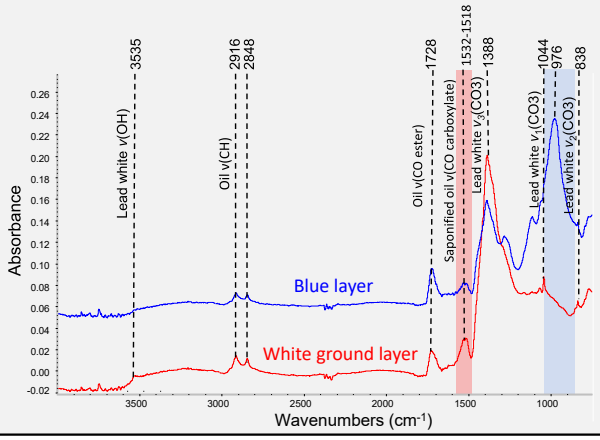
- A specific paint formulation used by da Vinci?
- An alteration phenomenon?
- A later restoration campaign?

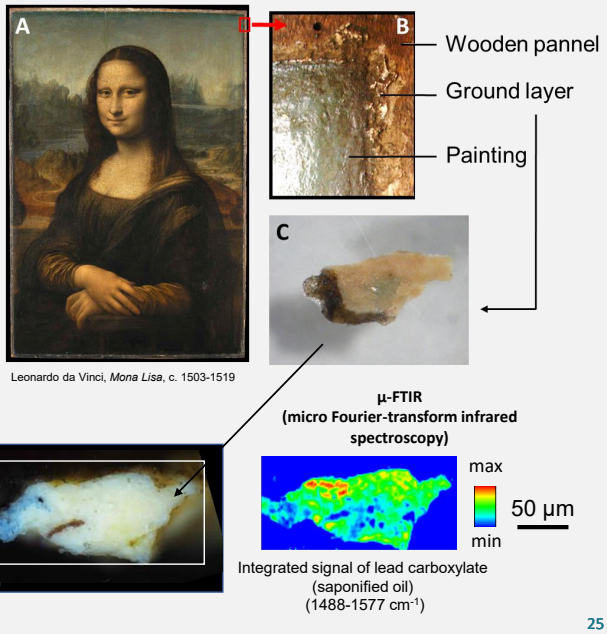
- Detection of nano-crystalittes of plumbonacrite $Pb_5O(CO_3)_3(OH)_2$, meta-stable compound (pH > 10) in *Mona Lisa*'s ground layer
- Microstructural features point to *in-situ* crystallization
- Choice of specific paint formulation resulting in alkaline environment?



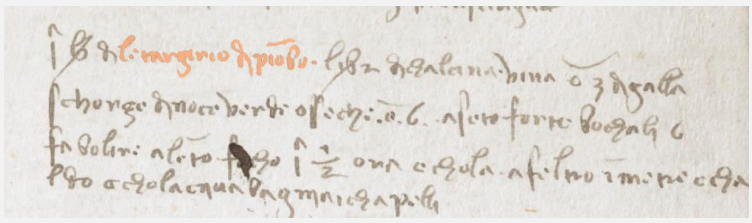


- Detection of nano-crystalittes of plumbonacrite $Pb_5O(CO_3)_3(OH)_2$, meta-stable compound (pH > 10) in *Mona Lisa*'s ground layer
- Choice of specific paint formulation resulting in alkaline environment?
- High saponification rate in lead white-based ground





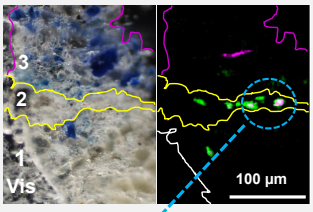
- Detection of nano-crystalittes of plumbonacrite $Pb_5O(CO_3)_3(OH)_2$, meta-stable compound (pH > 10) in *Mona Lisa*'s ground layer
- Identification of non-dissolved PbO-α grains in the priming layer of the *Last Supper*
- Examination of da Vinci's manuscripts to confirm that lead(II) oxide was part of the painter's palette

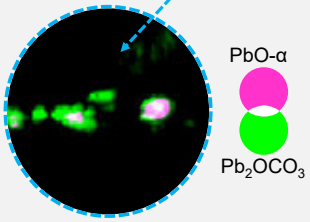


1 libra di letargirio di pionbo, libre 2 di calcina viva, once 3 di galla […]


Leonardo da Vinci, *The Last Supper* (detail), 1494-1498

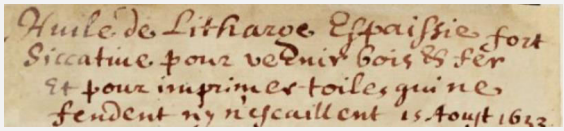
SR-μ-XRPD

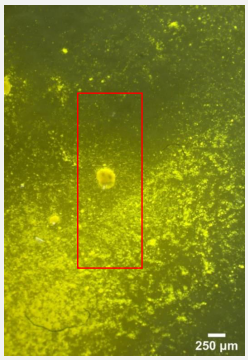


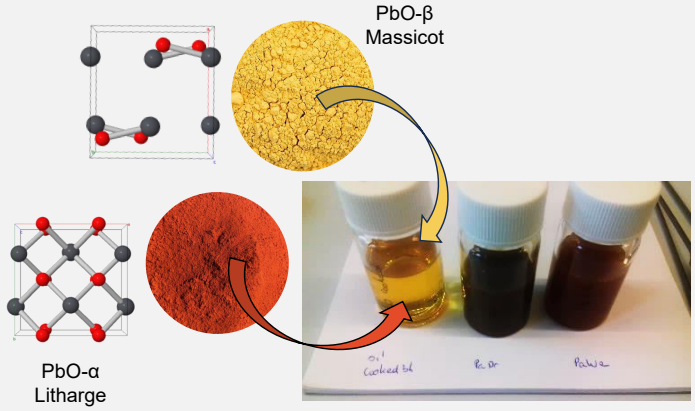


- Exploration of chemical pathways involving lead (II) oxide, used as a siccative agent in paint formulations
- Structural mapping at the scale of individual pigment particles
- Identification of in-situ formed crystalline compounds

Recipe for the preparation of *huile de litharge* by De Mayerne (1573-1655)



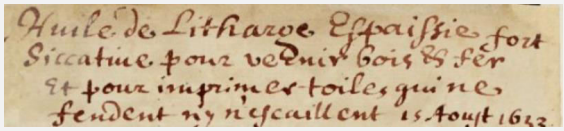


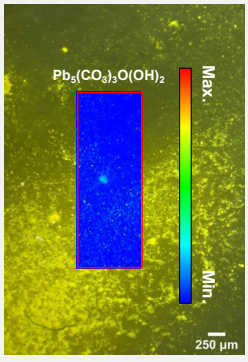


STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION
STRUCTURE / OPTICAL PROPERTIES RELATIONSHIPS IN LEAD CARBONATES
PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI

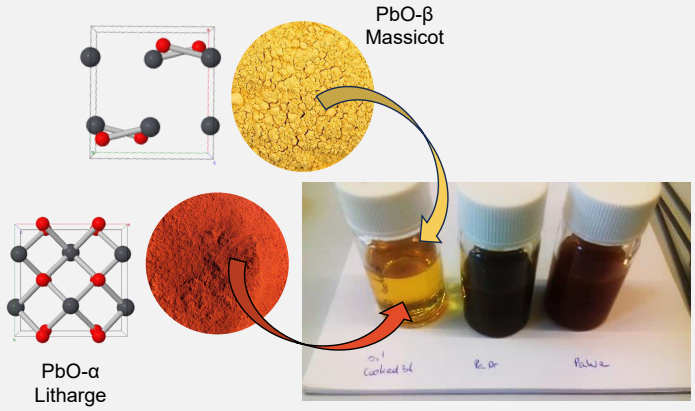
- Exploration of chemical pathways involving lead (II) oxide, used as a siccative agent in paint formulations
- Structural mapping at the scale of individual pigment particles
- Identification of in-situ formed crystalline compounds, **among them plumbonacrite**

Recipe for the preparation of *huile de litharge* by De Mayerne (1573-1655)





SR-µ-XRPD



- Exploration of chemical pathways involving lead (II) oxide, used as a siccative agent in paint formulations
- Competing mechanisms that can result in spatially heterogeneous cristallisation within paint layers
  - ➢ Carbonation of partially dissolved PbO particles

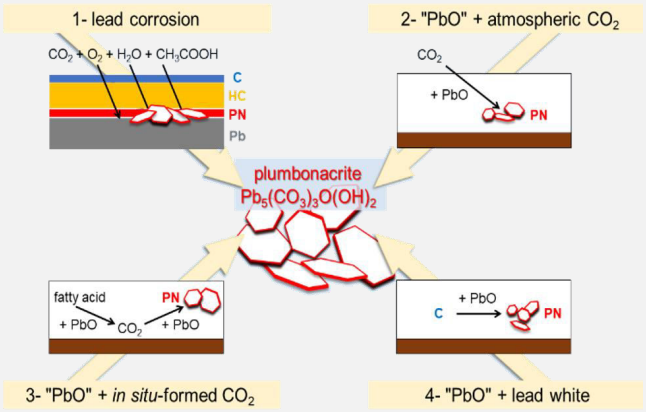
$$5\,PbO + 3\,CO_2 + H_2O \longrightarrow Pb_5O(CO_3)_3(OH)_2$$

  - ➢ Solid / solid reaction between lead-carbonate pigment and PbO

$$3\,PbCO_3 + 2\,PbO + H_2O \longrightarrow Pb_5O(CO_3)_3(OH)_2$$

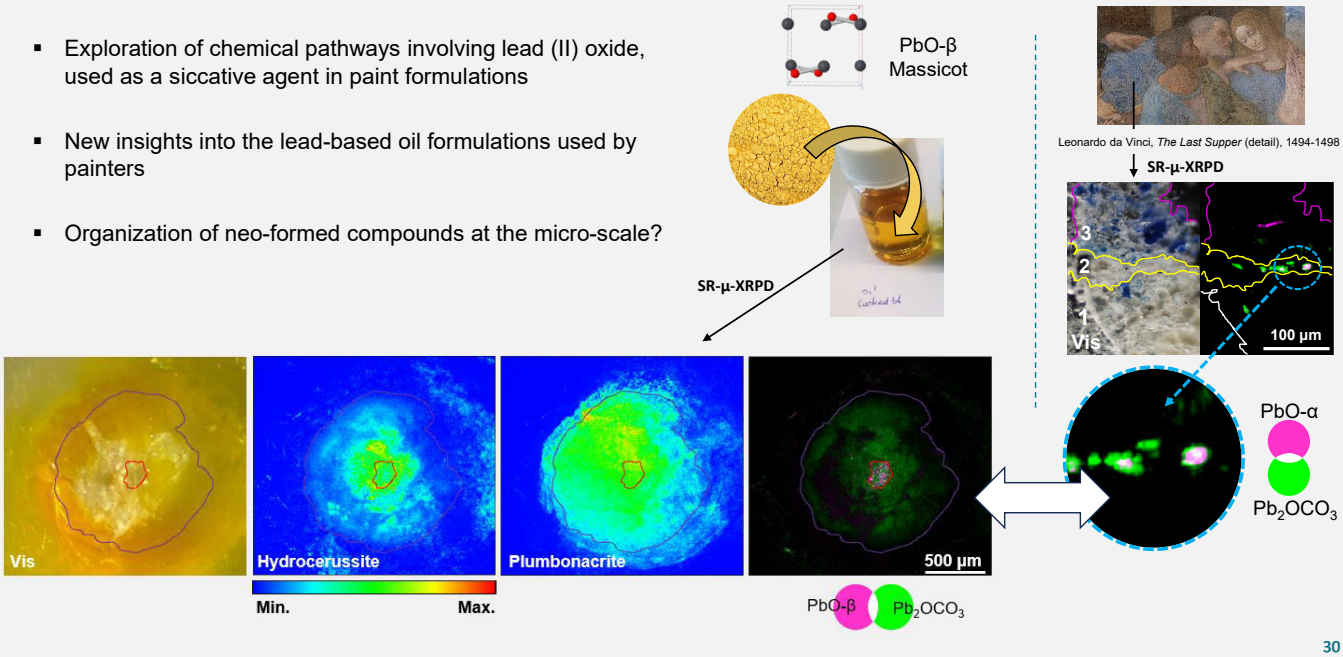
$$Pb_5(CO_3)_3O(OH)_2 + PbCO_3 + H_2O \longrightarrow 2\,Pb_3(CO_3)_2(OH)_2$$

- Plumbonacrite as a clue for a possible use of siccative medium



V. Gonzalez, M. Cotte, G. Wallez, A. van Loon, W. De Nolf, M. Eveno, K. Keune, P. Noble, J. Dik., *Angew. Chem. Int. Ed.* (2019) **58**(17): 5619

- Exploration of chemical pathways involving lead (II) oxide, used as a siccative agent in paint formulations
- New insights into the lead-based oil formulations used by painters
- Organization of neo-formed compounds at the micro-scale?



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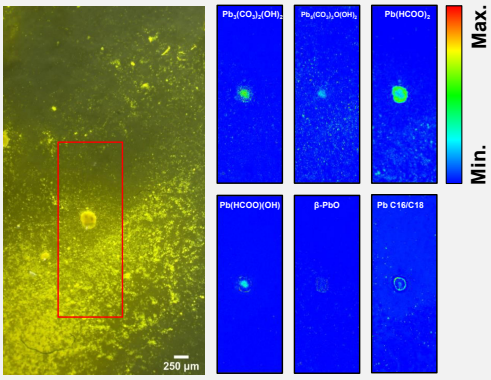
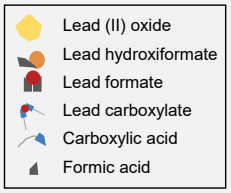
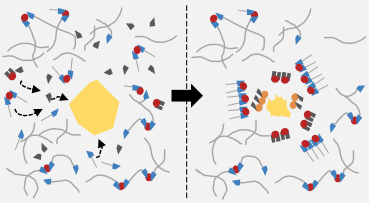
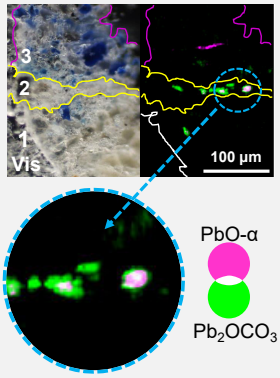






Leonardo da Vinci, *The Last Supper* (detail), 1494-1498

SR-µ-XRPD



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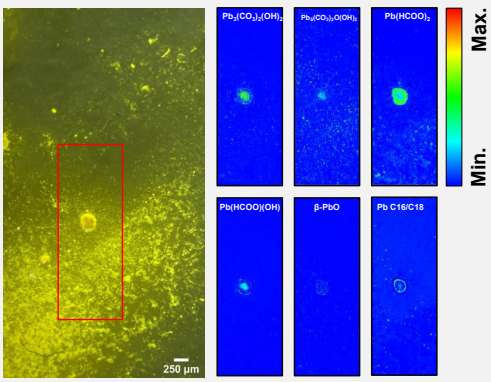
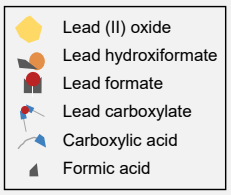
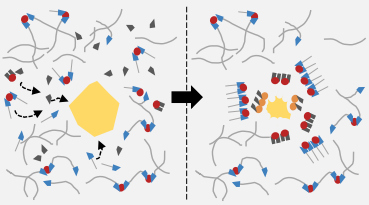
STRUCTURAL ANALYSIS VIA SYNCHROTRON RADIATION
STRUCTURE / OPTICAL PROPERTIES RELATIONSHIPS IN LEAD CARBONATES
PROBING THE PAINT FORMULATIONS OF LEONARDO DA VINCI

- Exploration of chemical pathways involving lead (II) oxide, used as a siccative agent in paint formulations
- Organization of neo-formed compounds at the micro-scale?
  → complex transport mechanisms at stake







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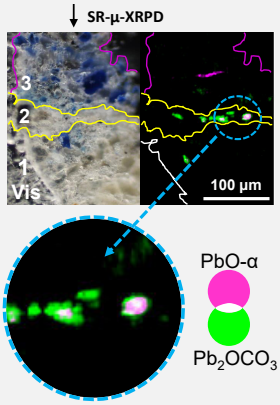

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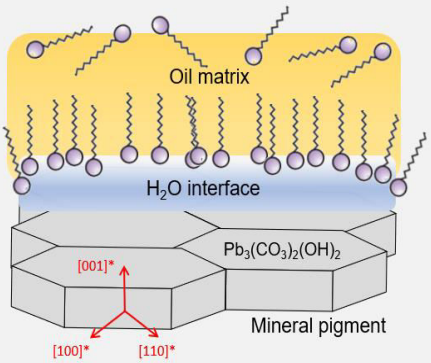

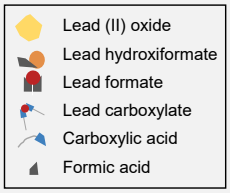

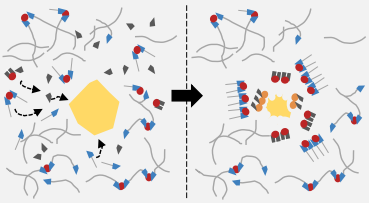

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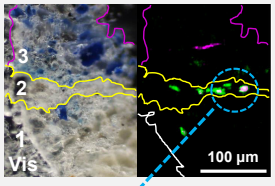

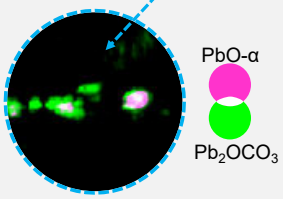

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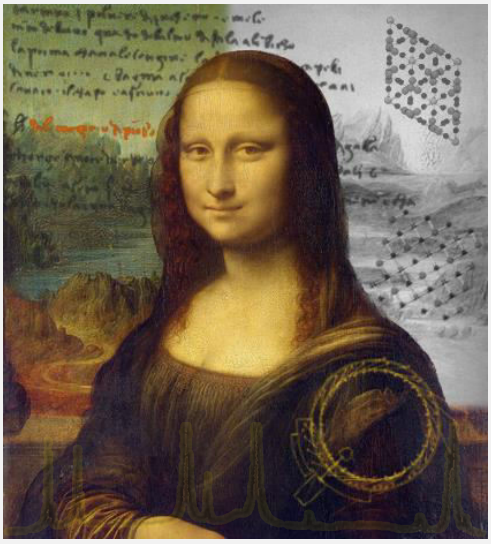
CONCLUSIONS

- **Hypothesis: to paint the ground layer of *Mona Lisa*, Leonardo relied on a siccative oil, prepared with lead (II) oxide**
- **Micro-scale organization of non-original inorganic compounds provide insights on the chemical mechanisms active in historical paint layers**



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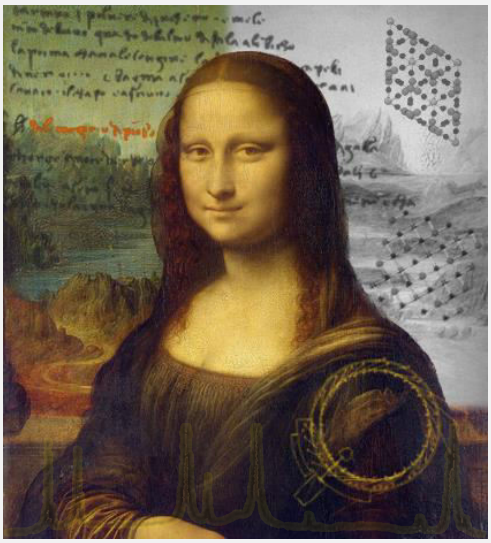
THANK YOU FOR YOUR ATTENTION!

CONCLUSIONS

- **Synchrotron-based structural analysis to probe the paint formulations used by Leonardo da Vinci (and other artists!)**
- **High complementarity of high-angular and high-lateral resolution synchrotron-XRPD**



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ACKNOWLEDGEMENTS

Horizon 2020 Programme

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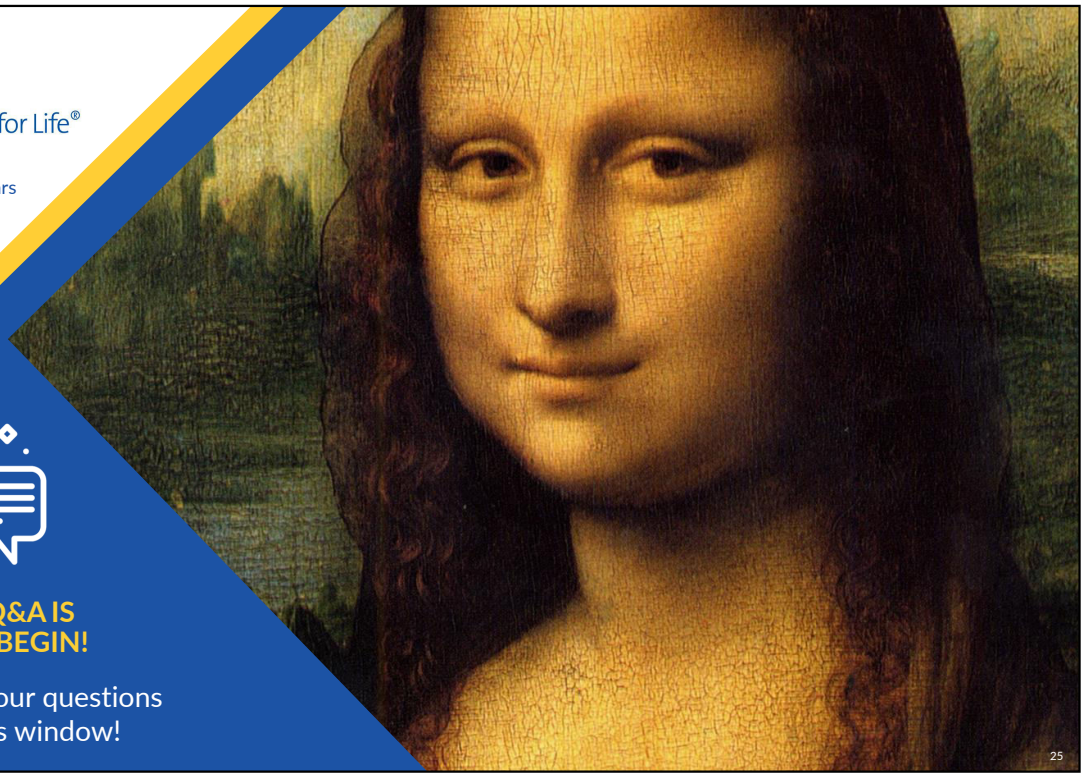

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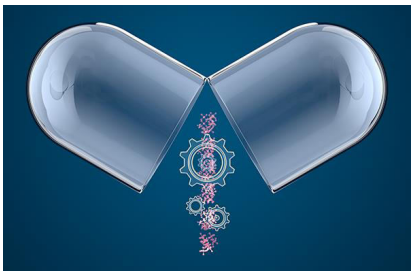

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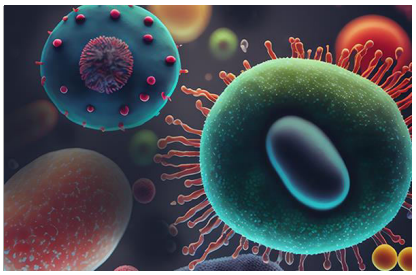

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