SIGART 2014

Study of modern paint materials and their stability

Dubravka Jembrih-Simbürger[1],
Iva Bogdanovic Radovic[2],

[1]Institute of Science and Technology in Art, Academy of Fine Arts Vienna, Schillerplatz 3, A-1010 Vienna, Austria;
d.jembrih@akbild.ac.at

[2]Laboratory for Ion Beam Interactions, Rudjer Boskovic Institute, Bijenicka 54, HR□10000 Zagreb, Croatia; iva@irb.hr

UKF-founded project "Study of modern paint materials and their stability using MeV SIMS and other analytical techniques"

Cooperation between Rudjer Boskovic Insitute (RBI) and Academy of Fine Arts Vienna (AFA)

Co-workers:

AFA: Marta Anghelone, Rita Wiesinger,

RBI: Nikola Markovic, Zdravko Siketic, Tonci Tadic, Milko Jaksic

Why we need scientific research using advanced analytical methods?

- Determination of chemical composition of materials used for art objects combined with the expertise of art historians is very important tool for dating and authenticity of an art object

- This information is needed for understanding how the particular art work will behave through longer time periods and for the selection of appropriate conservation techniques to preserve it from degradation.

- Paintings and other objects that are part of our cultural heritage contain a wide variety of organic materials of natural as well as synthetic origin.

- …and they contain often more than only 1 material class and are composed of complex mixtures of different molecules having a wide range of physical and chemical properties

a variety of analytical methods has to be applied for their investigation

Focus of the Project
Part I

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⇒ on the synthetic organic materials in paints (commercial and self prepared mock-ups)

polymers

synthetic organic pigments (SOPs)

- Sample preparation
- Artificial ageing ⇒ UV light outdoor conditions!

- Analysis of unaged and aged samples using different analytical methods ⇒ identification of the pigments and binders, detection of the degradation products

Focus of the Project
Part II

⇒ mock-ups with selected polymers used for outdoor sculptures

↙ polyester ↓ epoxy ↘ PVC

- Sample preparation
- Weathering of the samples using different gases (SO_2, NO_x, relative humidity - RH, UV-light)
- Analysis of samples before and after weathering using different analytical methods ⇒ detection of the degradation products

Part I: Sample preparation

1)paint mock-ups using SOPs of different chemical classes:

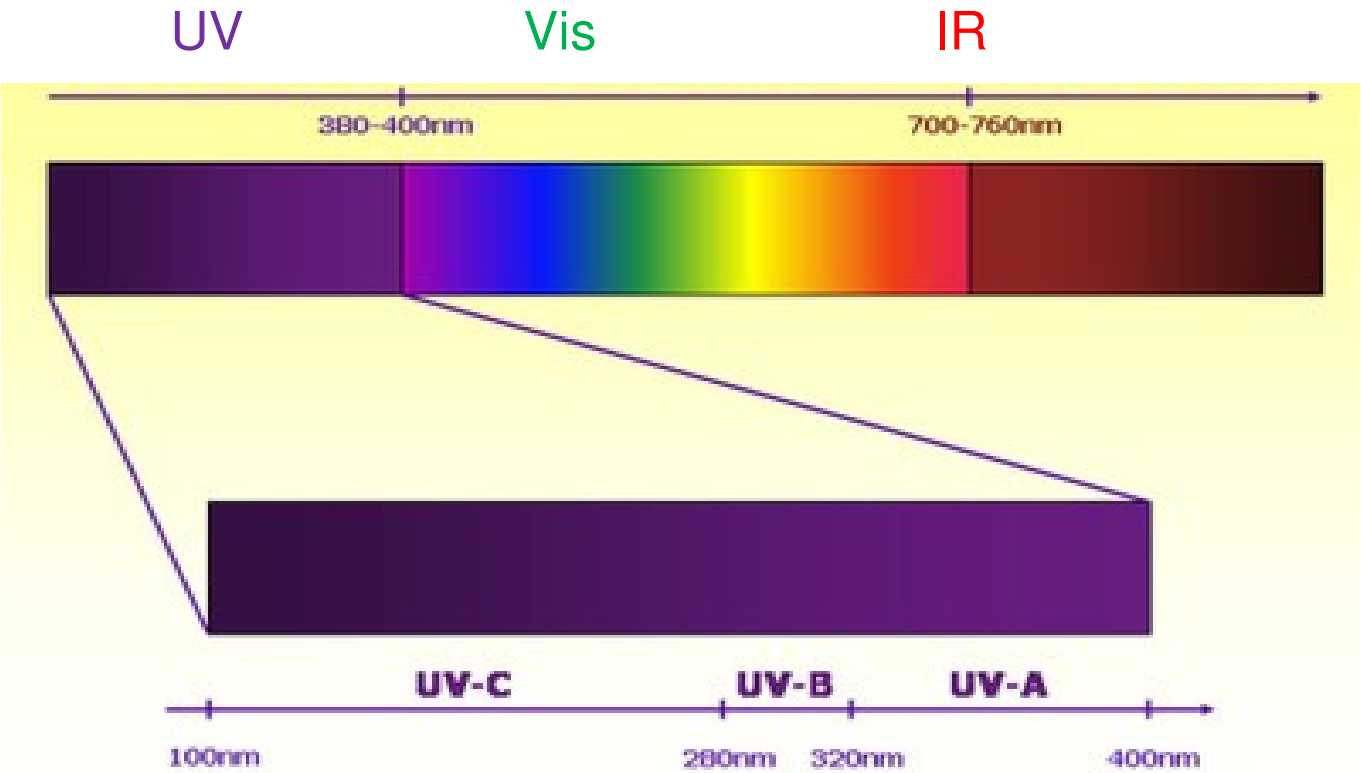
- ⇨ **blue** and **green phthalocyanines**
- ⇨ **yellow azo** pigments

mixed with modern binding media (**acrylics** and **alkyds**) **without additives**

2) mock-ups from **commercial tube paints** containing the same SOPs and acrylic or alkyd binder

Artificial Ageing

⇨ UVA and UVB are affecting artworks exposed to outdoor conditions, promoting photo-oxidative reactions causing discoloration, cracks and other damages



Artificial Ageing

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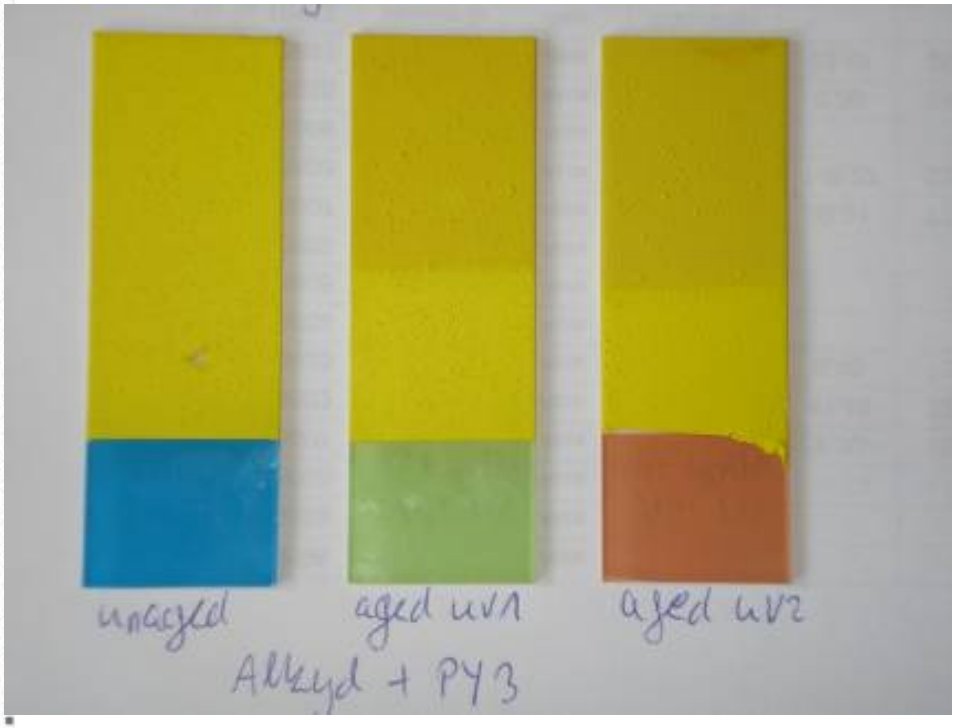
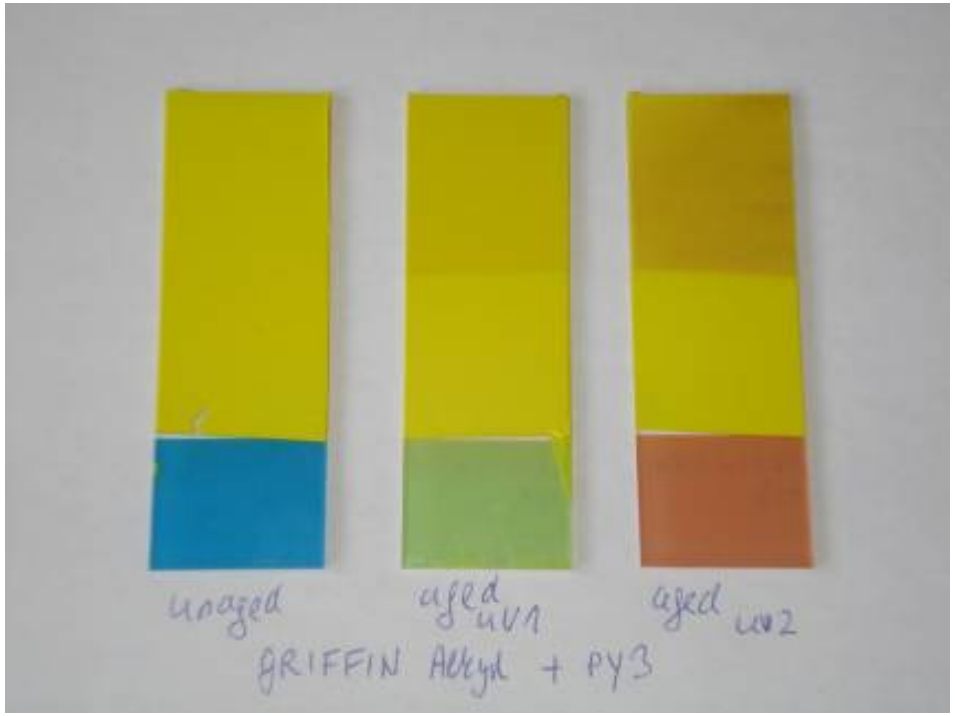
⇒ **2 and 4 months under outdoor conditions** (295 nm - 3000 nm) in sunlight simulator - Xenon Arc simulator - (Temp. approx. 50 °C, RH not controlled)

Unaged | 2 months aged | 4 months aged



self-made paints

Unaged | 2 months aged | 4 months aged



commercial paints

Analytical methods

Academy of Fine Arts Vienna:

- FTIR-ATR and Mikro-Ramanspectroscopy
- Py-GC/MS
- UV/Vis spectroscopy
- IRRAS and QCM
- Kelvin Probe

Rudjer Boskovic Institute, Zagreb:

- MeV SIMS
- PIXE

MeV SIMS (Secondary ion mass spectrometry using primary ion beam in MeV range)

- During the project the first application in the field of cultural heritage is tested

MeV SIMS applications until now:

- medicine (Japan) – " wet SIMS" - low pressure interface
- forensic science (England) – developing MeV SIMS in air

In 2012 MeV SIMS microprobe was installed at the RBI/Croatia - accelerator with highest energy of primary ions!

MeV SIMS

- **desorption of larger organic molecules from the surface** due to the electronic sputtering effect by heavy fast ions **causing less fragmentation of organic molecules** compared to keV SIMS
- **μ-beam** is available
- **chemical imaging in submicron level**
- can detect both, **organic pigments** (as well as inorganic) and **binding media** during one measurement

MeV SIMS

- secondary ion yield of larger organic molecules is increasing with the energy and charge of the primary ions

  MeV SIMS: 10^2 -10^3 higher yield compared to keV SIMS

- Surface sensitive! - **concerning material degradation - often changes in the uppermost layers** – not enough material for chromatographic methods (e.g. GC/MS)

MeV SIMS at Rudjer Boskovic Institute (RBI), Zagreb, Croatia

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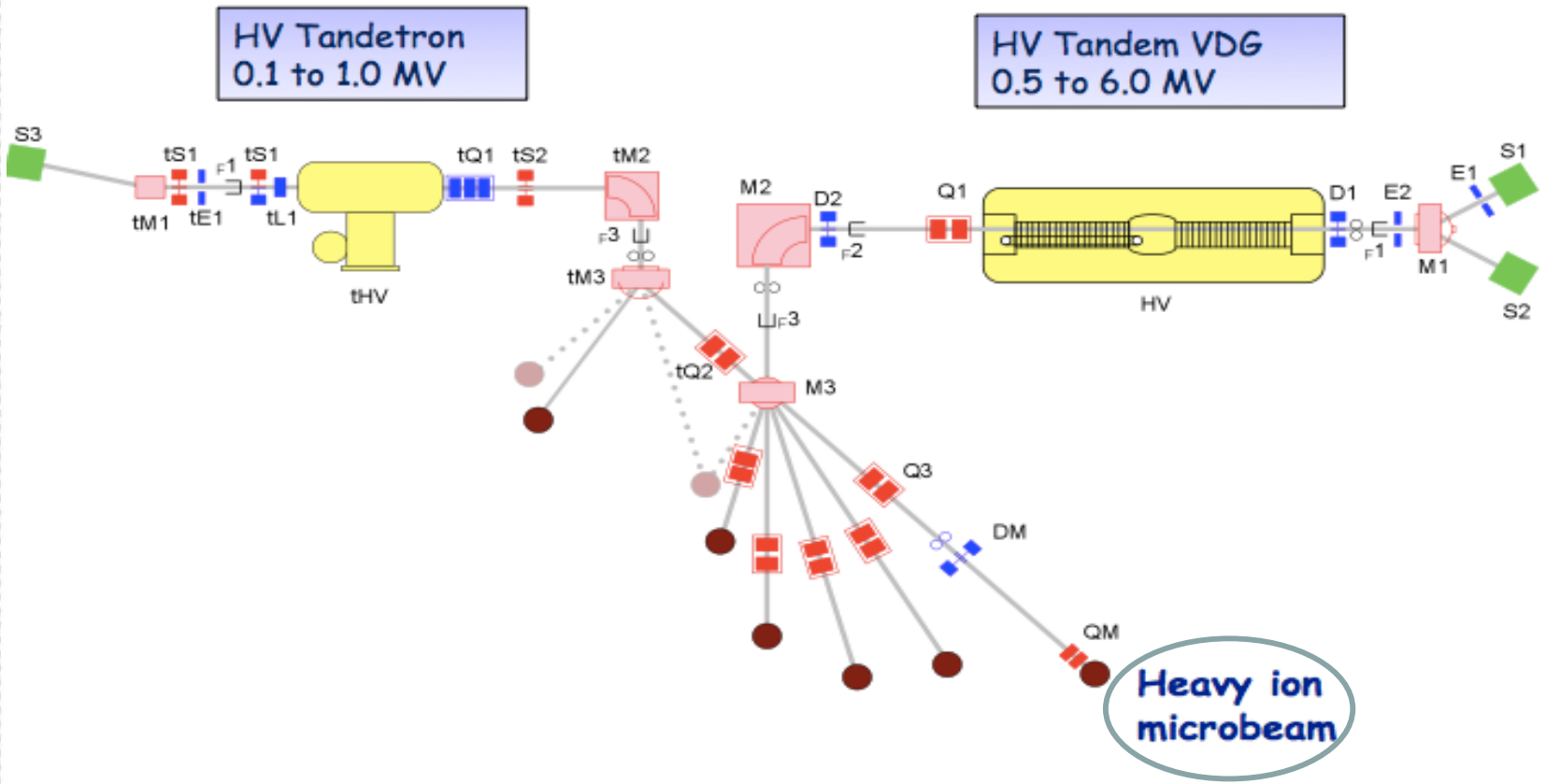
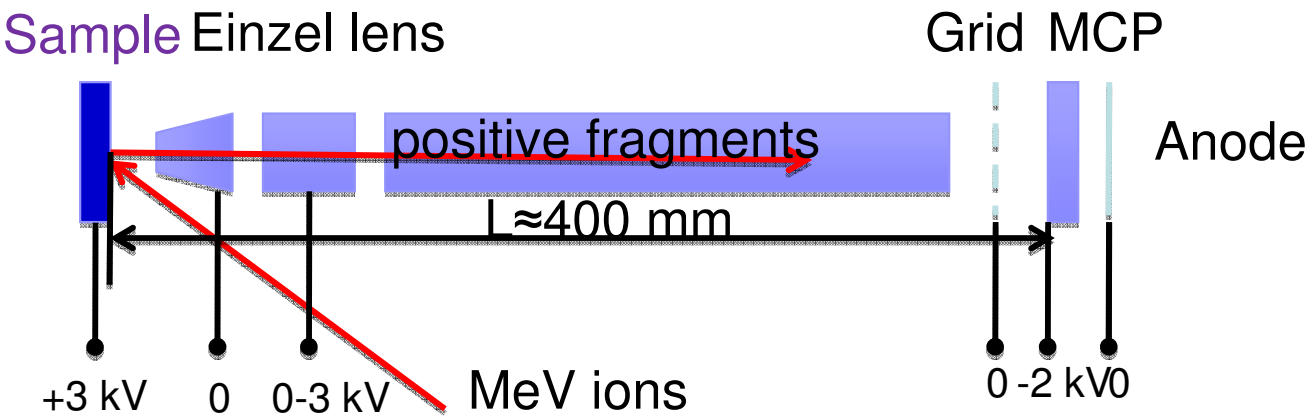
TOF MeV SIMS setup @ Heavy Ion Microbeam



MeV SIMS at Rudjer Boskovic Institute (RBI), Zagreb, Croatia

Sample chamber with TOF telescope



secondary molecular ions were extracted from the sample after collisions with primary ions using acceleration potential difference between the sample (+3 kV) and grounded extractor (0 kV)

MeV SIMS

First measurements on selected 2-component **mock-ups** with paint layers:

- binding media:

  alkyd and acrylic

- phthalocyanine blue pigments:

  PB 15:1, PB15:3, and PB 16

Questions:

1)Can we distinguish between different phthalocyanine blue pigments?

2) … and different binding media used in the paint layers applying MeV SIMS?

Identification of different blue phthalocyanine pigments in alkyd paints

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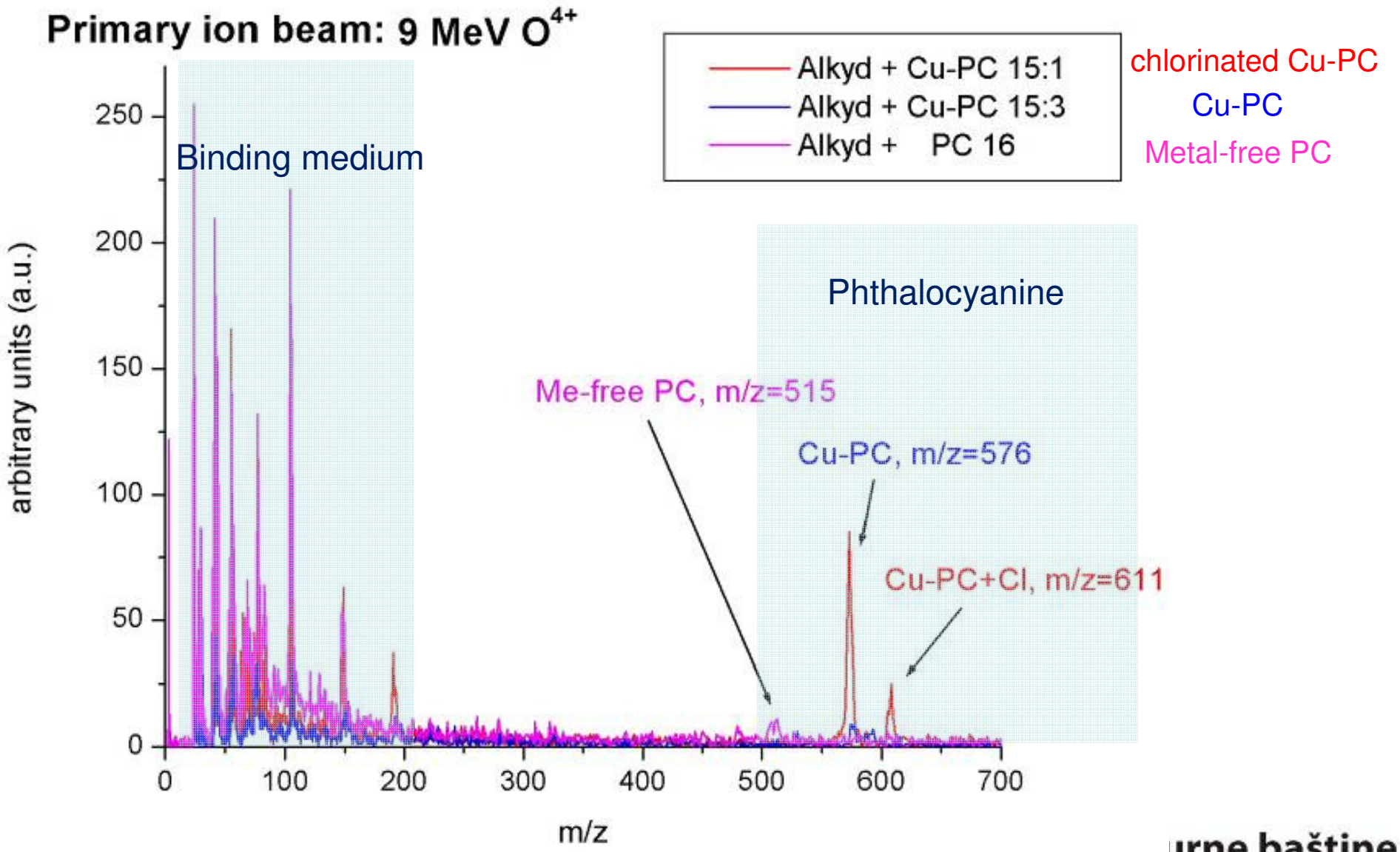
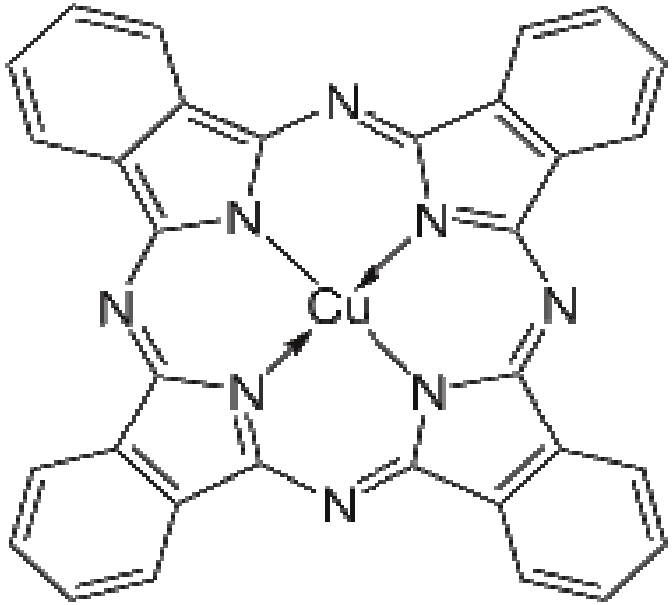
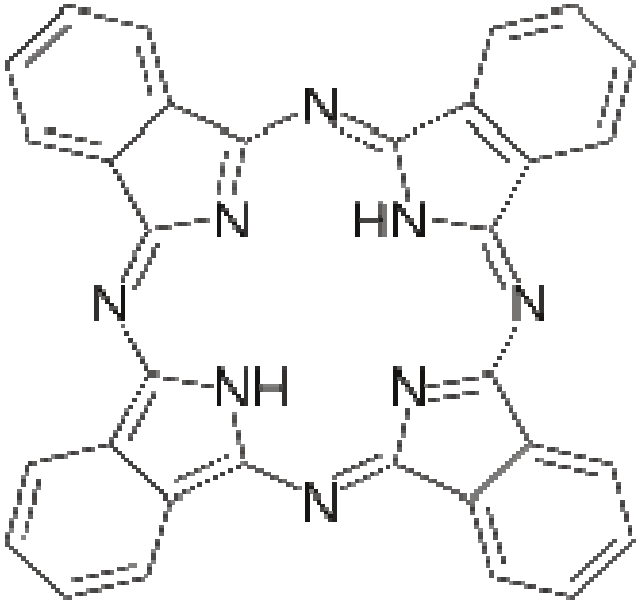


Blue phthalocyanine pigments



Copper-PC



Metal-free PC

Summary MeV SIMS

- MeV SIMS spectra are simpler than Py-GC/MS spectra, no oligomers are present (influence of measuring parameters) in polymer fragments, no fragmentation of the PC
- different polymorphs of blue PC-pigments can be easily identified/distinguished in the paint layer
- Detection of both, binding medium and PC-pigments in one spectrum (pigment and binding medium region in the spectrum is not overlapping)
- Data processing (e.g. chemometrics) not necessary
- Chemical imaging with submicron level lateral resolution - distribution of different components in the paint layer

Summary MeV SIMS

- But…

we need an accelerator for producing MeV primary ion beam

and we need small samples from the art works

Acknowledgement

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Thank you for your attention!