European Network of Fourier-Transform Ion-Cyclotron-Resonance Mass Spectrometry Centers

Grant Agreement n° 731077

Deliverable D2.12 – Final report of the different Short Courses – Location: Liege-Lille

Start date of the project: 1st January 2018
Duration: 60 months
Project Coordinator: Christian ROLANDO – CNRS-
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Document Classification

Title	Final report of Short Course 10 & 11
Authors	P1 CNRS – Christian Rolando P2 LIEG – Edwin DE PAUW
Work package	WP2 Training, education and networking activities
Dissemination	PU = Public
Nature	R: Document, report
Doc ID Code	20231103_EU_FT-ICR_MS_D2.12
Keywords	Short Courses on Mass Spectrometry Imaging & Cultural Heritage

Document History

Name	Date	Comment
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Document Validation

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Document Abstract

This deliverable is the final report of Short Course 10 & 11 on FT-ICR Mass Spectrometry Imaging & FT-ICR for Cultural Heritage Sciences, an event organized and held within WP2 – NA- Training, education and networking activities. Due to the relatively close proximity between Lille and Liege the two events were organized jointly in the same week.

The objective of WP2 is to promote education in the field of FT-ICR MS, sharing newly developed state-of-the-art procedures for experiments and data analysis and training qualified and highly skilled FT-ICR specialists. In addition, Short Course will include a number of social events creating a fruitful platform for beginning international collaborations.

This deliverable explains content and outcome of Short Course 10 & 11 (SC10, SC11) performed in the framework of the EU_FT-ICR_MS project.

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1. Introduction

Short Course 10, entitled on FT-ICR Mass Spectrometry Imaging and Short Course 11 FT-ICR for Cultural Heritage Sciences, were originally planned to be held at the University of Liege (Belgium) and University of Lille on M34 & 44. In view of the relatively close proximity between Lille and Liege the two events we decided to organize the 2 SC jointly the same week. Due to COVID19 pandemic restrictions, SC10 & 11 have been postponed to M59 (Nov 2022) to be held in presence.

The announcement flyer is presented below in Figures 1.

Figure 1. Flyer of SC10 & 11.

2. Final report of SC10

Uliege MSLab contributors for Mass Spectrometry Imaging:

Prof. Edwin DE PAUW (EU FT-ICR MS network supervisor in Liège) and Dr. Johann FAR (site manager)

- Pr. Edwin DE PAUW, Dr. Johann FAR, and Wendy MULLER (matrix assisted laser desorption ionization MALDI, surface enhanced laser desorption ionization SALDI, mass spectrometry hardware used for imaging, mass spectrometry imaging MSI and applications)
- Dr. Virginie BERTRAND, Dr. Johann FAR (histology and tissue section, brain MSI)
- Sophie RAPPE (thin layer chromatography with MSI detection)

- Pierre BURGUET (imaging of interacting bacteria co-culture in agar of metabolites)
- Dr. Kune CHRISTOPHER and Thomas TILMANT (data sciences with Python)

Uliege MSLab content:

The Mass Spectrometry Imaging (MSI) short course included a few of theoretical aspects of mass spectrometry imaging, especially matrix assisted laser desorption ionization mass spectrometry imaging, MALDI MSI, and a quick overview of other MSI techniques, instrumentation, and applications.

The practical sections of the training included tissue section of mouse brains, preparation of thin layer chromatography (TLC) of lipids, and handling of bacterial co-cultures chemically interacting in agar.

The short course also included the matrix deposition using an automated sprayer (SunChrom SunCollect), and the MALDI MSI of the various samples using the Bruker dual source ESI/MALDI FT-ICR 9,4T fitted with the paracell, the solariX xR.

Finally, a short introduction about the use of Python for data sciences was scheduled, exploring and extracting some data of interest from mass spectrometry mzML converted data. A short demo has been show what could be expected when applying such tools within an in-house software [1], allowing the reconstruction of MSI data using a Kendrick Mass Defect clustering strategy. Additionally, the basic use of SCiLS™ Lab software (Bruker) [2] for processing MSI data will be tackled during the short courses.
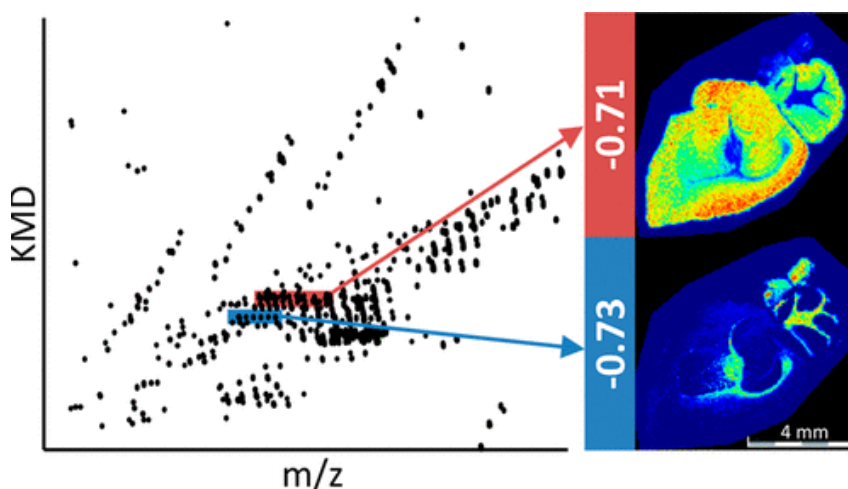

Figure 2. Example of the differentiation of 2 families of molecules by their Kendrick mass defects in an high resolution FTICR MALDI imaging of a tissue.

A social event was scheduled. A night out in a brasserie (restaurant) has been organized to eat typical food from Liege and drink Belgian beers.

[1] *Anal. Chem.* 2019, 91, 20, 13112–13118 ; https://doi.org/10.1021/acs.analchem.9b03333
[2] https://www.bruker.com/en/products-and-solutions/mass-spectrometry/ms-software/scils-lab.html

The course had 12 participants (with a M/F ratio of 8/4) all of them with experience in mass-spectrometry and some with experience in FT-ICR-MS. Participants came from 6 different European countries: Czech Republic, Italy, Lithuania, Portugal, Romania and UK.

All participants expressed their satisfaction and considered the event useful for their future.

3. Final report of SC11

ULille TopOmics contributors for Mass Spectrometry for Cultural Heritage Science:

Dr Christian ROLANDO, CNRS Senior scientist (EU FT-ICR MS network supervisor for Lille and global coordinator of the network) and Dr. Fabrice BRAY (site manager)

- Dr. Fabrice BRAY (Species identification forf paleolithic and archeological bones by proteomics [1]; analysis of lipids in bones and sherds [2])
- Stéphanie FLAMENT (identification of proteins in sherds and tempera painting [3])
- Dr Marie YAMMINE (analysis of watercolor and gouache [4])
- Dr. Christian ROLANDO and MSc Bayan ALMASRI (analysis of oil or alkyd artist paint)

ULille TopOmics training content:

The Mass Spectrometry Short course for Cultural Heritage was focused on the identification of natural products preserved over the centuries which are very often polymeric. The short course began with brief theoretical courses covering an overview of Mass Spectrometry applied to Cultural Heritage, the principle of Fourier Transform Ion Cyclotron Resonance Mass Spectrometry (FT-ICR MS), the concept of fine isotopic structure as well as the basis of proteomics, glycomics and lipidomics.

The practical sections of the training comprised examples from these various topics. Firstly, demonstration of sample preparation has been held: (i) collagen extraction and digestion with minimal induction of posttranslational modifications, quantification of deamidation; (ii) digestion of model gums, watercolor or gouache modern paint and (iii) and depolymerization of alkyd artist paint. Subsequently, the prepared samples have been analyzed with high resolution MALDI FT-ICR and nanoLC Orbitrap results. The obtained spectra have been further compared to their corresponding ones acquired through lower resolution instruments such

as MALDI TOF MS And finally, for proteomics, a comparison related to data treatment showed different outcomes from the same spectra depending on the database sources, with publicly available and in-house established data bases.

The short course has been carried our using our Bruker solariX xR ESI/MALDI FT-ICR 9. 4 Tesla fitted with a harmonized cell, our Q-Exactive+ Orbitrap fitted with a nanoLC U3000 RSLC and on our server running commercial and homemade software.
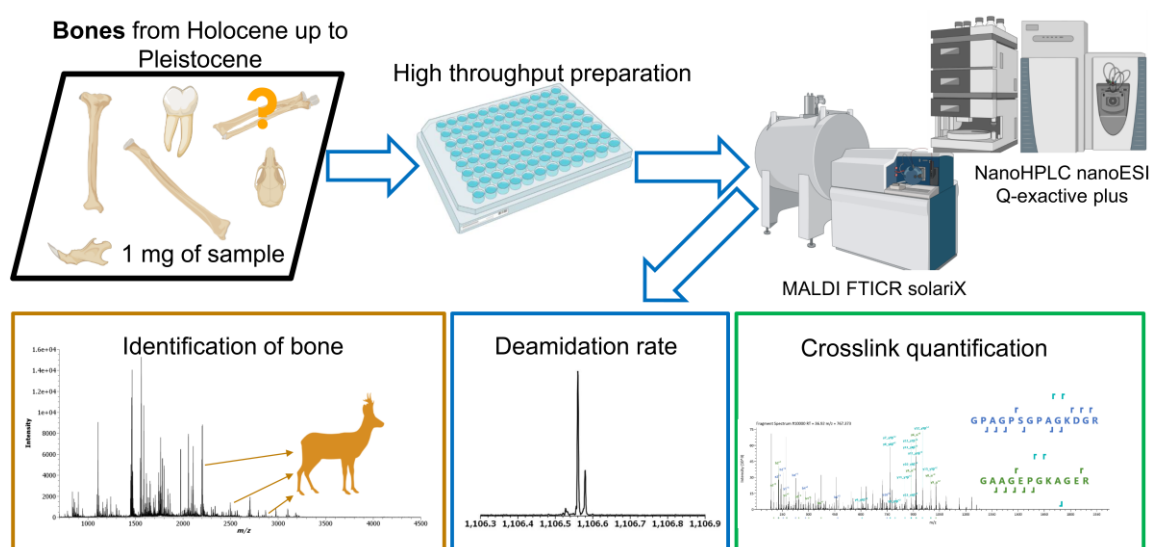

Figure 3. Workflow of high throughput paleoproteomics by MALDI FTICR.

!!!! Participants have been encouraged to bring their own samples if they are not too precious (100 mg of bones, 1 g of sherds, 100 µg of paints (watercolor or oil). It's a hundred times the minimum amount required for precious sample analysis. !!!!

A social event was scheduled. A competitive night out in a brasserie (restaurant) was arranged to compare Flemish specialties in Lille to their counterparts in Liège.

[1] *Archaeometry* (2022). https://doi.org/10.1111/arcm.12800.
[2] *Int. J. Mass Spectrom.* 284.1-3 (2009): 47-56. https://doi.org/10.1016/j.ijms.2009.03.003?
[3] *Anal. Chem.* 83.24 (2011): 9431-9437. https://doi.org/10.1021/ac201978j.
[4] *Scientific reports* 7.1 (2017): 1-15. https://doi.org/10.1038/srep44538.

The course had 18 participants (with a M/F ratio of 7/11) all of them with experience in mass-spectrometry and some with experience in FT-ICR-MS. Participants came from 8 different European countries: Czech Republic, France, Germany, Italy, Ireland, Portugal, Romania and UK.

All participants expressed their satisfaction and considered the event useful for their future.