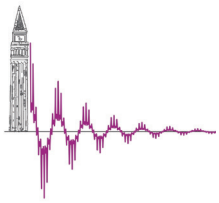
NEWSLETTER OF H2020-INFRAIA-02-2020 "PANACEA"

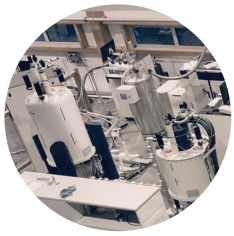
PANACEA news

-nmr.eu

A Pan-European Solid-State NMR Infrastructure for Chemistry-enabling Access

Issue 4, January 2024





PANACEA second Annual Users Meeting

The PANACEA second Annual Users Meeting in November kicked off with a deep dive into the world of solid-state NMR. The first day was filled with enlightening talks, showcasing compelling research of solid-state NMR for pharmaceuticals, inorganic and hybrid materials and examples of the latest developments in DNP and solid-state NMR.

The Florence venue presented a fantastic opportunity to unite PANACEA users with members of the PANACEA laboratories and further members of the research community with inspiring presentations and initiated discussion on topics as diverse as energy material and sustainability, single crystal diamond properties and solid-state NMR for biomaterials.

A round table discussion with representatives from solid-state NMR sites in European countries not currently part of the PANACEA access sites, from Bulgaria, Croatia, Czech Republic, Latvia, Poland and Romania, resulted in initiated discussions on the future perspectives and challenges for solid-state materials NMR.

The event gathered an engaged community of researchers, brough forward prominent reserach and fostered unique interactions among users and members of the PANACEA community.

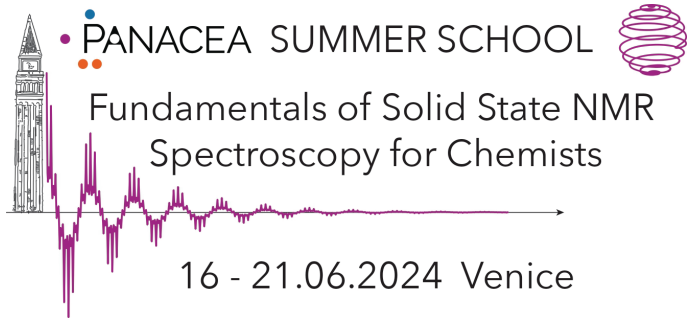
PANACEA key numbers

- *1650 access days*
- *27 spectrometers*
- *> 200 chemistry-related projects*
- *4 user meetings*
- *4 hands-on trainings*
- *1 NMR summer School*
- *3 industry-targeted workshops*
- *32 taster days for industrials*
- *8 summer fellowships for students*

PANACEA solid-state NMR school
Fundamentals of solid-state NMR spectroscopy for chemists

We are thrilled to announce PANACEA's upcoming event: *Fundamentals of Solid-State NMR Spectroscopy for Chemists* Summer School 2024. Taking place in the beautiful city of Venice, Italy, June 16 - 21, this unique educational opportunity is designed for academic and industrial researchers, graduate students, post-doctoral fellows, early-stage researchers and engineers working on chemistry and materials science related challenges.

The course will provide you with advanced theoretical and experimental knowledge in solid-state NMR, directly applicable to your professional work while offering a broader understanding of the field. This comprehensive training will encompass lectures, tutorials, discussions and guided problem-solving sessions, allowing you to delve into the possibilities and limitations of various solid-state NMR approaches for chemistry. The program is designed to cater to both novice and experienced users, covering fundamental concepts, instrumentation, and basic solid-state NMR correlation experiments, along with advanced topics such as anisotropic interactions, tensor description, magic angle spinning and more.



With a maximum capacity of 60 attendees, the summer school will offer an enriching learning experience. The venue for the event is San Servolo, a picturesque island in the Venetian Lagoon. Registration is open, subsidized by the PANACEA grant for all participants. Attendees will have the chance to showcase their research activities during flash oral presentations within thematic round tables.

NATIONAL HIGH MAGNETIC FIELD LABORATORY

PANACEA Fellow profile
local operator and scientific manager
Frederic Mentink-Vigier

Research Faculty, The United States National High Magnetic Field Laboratory, Florida, USA

Q: Why did you decide to pursue a PhD in Science?
The real story is very long, an abbreviated version would be; at the end of my Master's degree I wanted to challenge myself and do more physics. I had the opportunity to do a PhD thesis focused on material science combined with electron paramagnetic resonance for quantum computing applications.

Q: When did you choose a topic related to solid-state DNP NMR?
I started already during my Master's internship and the first year of my PhD with DNP using Overhauser. The decision to fully commit to DNP-NMR started in 2012 when I was working as a post doc at the Weizmann Institute of Science under the direction of Shimon Vega and Daniella Goldfarb.

Q: Do you have a science hero or mentor?
I have not one but several that I met throughout my education and I would give them credits here:

Olivier Douteau who made me love organic chemistry.
Olivier Guillot-Noel who made me want to do more physics while teaching us the density matrix formalism.
Didier Gourier and **Laurent Binet** who both advised me during my PhD studies.
Shimon Vega and **Daniella Goldfarb**, from whom I still draw inspiration from.

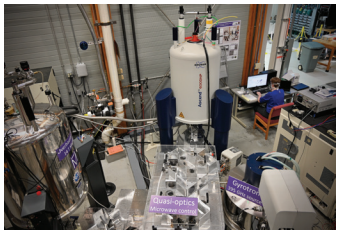
Q: As a child, what did you wish to become?
This is not your usual answer; I grew up on a farm and wanted to be like my father.

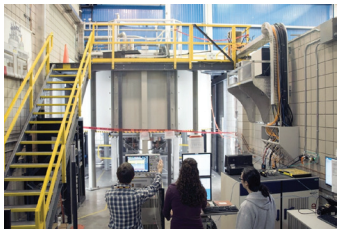
US National High Magnetic Field Laboratory

The United States National High Magnetic Field Laboratory (MagLab), comprised of seven user facilities across three sites - Florida State University, the University of Florida and Los Alamos National Laboratory - offers access to research tools in materials research, condensed matter physics, magnet technology, biophysics, chemistry and biology.

MagLab provides PANACEA user access to unique NMR spectrometers and probes, particularly the 1.5 GHz solid-state NMR instrument, the highest field strength in the world using the 36 T series-connected hybrid magnet and the 600 MHz DNP-NMR instrument, for NMR signal enhancement of solids, both open for access for materials and biosolids applications.

MagLab welcomes more than a thousand scientists from around the globe each year to use its unique magnets. MagLab offers a variety of tools and techniques for exploring physics, chemistry, biology and engineering in an interdisciplinary, collaborative environment.





Publication highlights

Leucopterin, the white pigment in butterfly wings: structural analysis by PDF fit, FIDEL fit, Rietveld refinement, solid-state NMR and DFT-D

In a recent published article, researchers Federica Bravetti, Lukas Tapmeyer, Kathrin Skorodumov, Edith Alig, Stefan Habermehl, Robert Hühn, Simone Bordignon, Angelo Gallo, Carlo Nervi, Michele R. Chierotti and Martin U. Schmidt presented a comprehensive study on leucopterin, the white pigment found in butterfly wings. Despite being a small and rigid molecule, uncovering the crystal structure and tautomeric state of leucopterin required an extensive experimental and analytical effort, employing around 20 different methods.

Pieris napi.
Photo: Robert Hühn, Frankfurt am Main, 23 June 2022, from the publication.

The research involved diverse techniques, such as syntheses, recrystallization attempts, thermal analysis, powder X-ray diffraction, synchrotron powder diffraction, solid-state NMR, and density functional theory with dispersion corrections (DFT-D). The team explored various tautomeric forms through lattice-energy optimizations and calculated ^{1}H, ^{13}C and ^{15}N chemical shifts in the solid state.

The key findings of the study reveal that leucopterin exhibits variable hydrate states, transitioning from hemihydrate to almost anhydrate. It crystallizes in a rare space group (*P2/c*) and adopts the 2-amino-3,5,8-H tautomeric form in the solid state. Moreover, the molecules demonstrate an exceptionally efficient packing, resulting in a high density of 1.909 kg/dm^3. This unique molecular arrangement may contribute to the observed light scattering and opacity in the wings of butterflies such as *Pieris brassicae*. The researchers underscore the significance of employing a multitude of methods, showcasing the complex nature of leucopterin and its role in the optical properties of butterfly wings.

Bravetti, F., Tapmeyer, L., Skorodumov, K., Alig, E., Habermehl, S., Hühn, R., Bordignon, S., Gallo, A., Nervi, C., Chierotti, M. R. & Schmidt, M. U. (2023). Leucopterin, the white pigment in butterfly wings: structural analysis by PDF fit, FIDEL fit, Rietveld refinement, solid-state NMR and DFT-D. *IUCrJ*, **10**, 448 - 463.

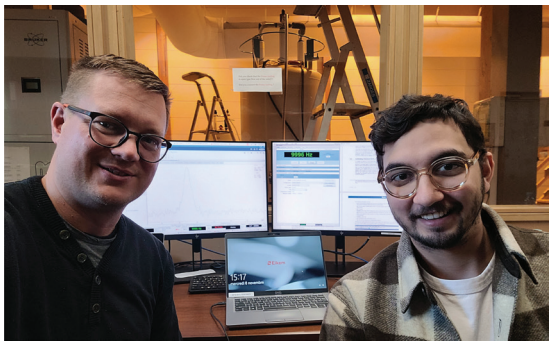
Consult the PANACEA website for more exciting PANACEA publications!

Industry Taster Day

Taster days are strategically organized to bring together individual scientists from industry, providing them access to the PANACEA sites. The aim is to inspire industry scientists by showcasing how experimental solid-state NMR can offer valuable insights into their material characterization challenges. In November, the CNRS Lyon organised a taster day where Tobias Schubeis invited Elkem's research engineer Arthur Lejeune.

During the PANACEA taster day, the CNRS Lyon together with Arthur Lejeune, conducted a comprehensive analysis of an Elkem Silicone using ^{29}Si solid-state NMR. Elkem is one of the world's leading providers of advanced silicon-based materials. Silicones, integral in various applications such as sealants, adhesives, lubricants, medical devices, cookware, and insulation, constitute Elkem's primary focus. The fundamental structure of silicone comprises polyorganosiloxanes, where silicon atoms form siloxane bonds with oxygen. A silicon atom can engage in 2-4 siloxane bonds (Q2, Q3, and Q4), with the remaining valences connected to organic groups like methyl, phenyl, vinyl or hydrogen. Despite Elkem's standardized megaton-scale production sites, daily quality control is imperative, and continuous efforts are made to advance procedures and formulations.

The successful quantification of different silicon sites in the polymer marks the potential commencement of a more extensive collaboration, indicating the possibility of regular solid-state NMR analyses on Elkem samples in the future.



Tobias Schubeis (left) and Arthur Lejeune (right).

Silicon is the second most abundant chemical element in Earth's crust, only second to oxygen. The stable and NMR active isotope ^{29}Si is 4.7 % naturally abundant.