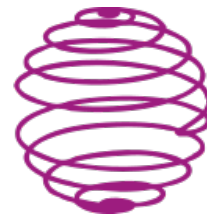


PANACEA *news*

-nmr.eu



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

Editor-in-chief: Maurane Vanderzwalme

Issue 1 - February 2023

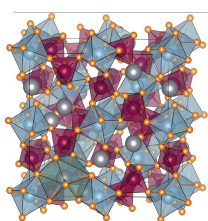
What is PANACEA?

A network providing access to solid-state NMR instruments
Page 1



Live from the PANACEA labs

NMR from catalysts to pharmaceuticals
Page 2



PANACEA fellows

Our new young NMR spectroscopists improving quality and quantity of access
Page 2



User visitor profiles

Benjamin Cheney & Daniel Duff - Jessica Wade

Zoom on a facility

Swedish NMR center, Göteborg

Page 3



Recent events

EUROMAR booth, Utrecht (NL) - Applied training, Lausanne (CH) - First user meeting and industrial day, Lyon (FR)
Page 4



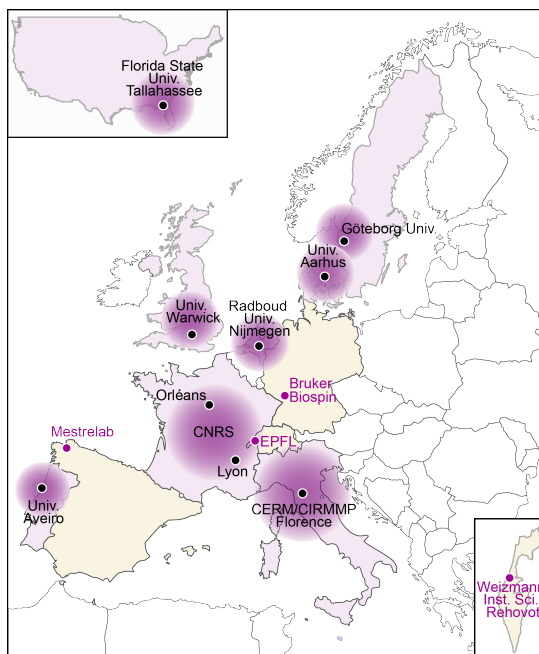
Upcoming events

Applied training, Aarhus (DK) - EUROMAR satellite, Glasgow (UK) - Second user meeting and industrial day, Florence (IT)
Page 4



What is PANACEA?

THE DEVELOPMENT OF MODERN CHEMISTRY relies on our capacity to investigate with atomic-level resolution increasingly complex solid substrates in frontier research areas crossing disciplines from catalysis and energy materials through polymers to pharmaceutical formulations and medical implants. Nuclear magnetic resonance (NMR) is in principle a technique of choice to analyse solid substrates with atomic resolution. However, state-of-the-art methods rely on the use of sophisticated and costly solid-state NMR equipment that is only available in a handful of national facilities. The rarity of the instrumentation and associated operational know-how has restricted the uptake of these enabling methods by the broader base. The PANACEA (A Pan-European solid-state NMR Infrastructure for Chemistry-Enabling Access) project aims to provide access to **cutting edge instrumentation and experiments** in solid-state NMR to European chemists, from both academia and industry. To achieve this goal, PANACEA brings together, and integrates on the European scale, seven national infrastructures across Europe (CNRS in France, Aarhus University in Denmark, CERM-CIRMMF in Italy, Radboud University in the Netherlands, University of Aveiro in Portugal, Göteborg University in Sweden and University of Warwick in the UK) as well as one infrastructure in the United States (at Florida State University) and opens them to all European researchers. Bruker Biospin and Mestrelab, as well as EPFL and the Weizmann Institute of Science are also involved in the consortium.



The project will provide users with **trans-national access** (TA) (1650 instrument days) to 27 unique NMR spectrometers ranging from 100 to 1500 MHz, fully equipped to cover the most advanced solid-state NMR techniques and applications. **Researchers with no prior expertise** in solid-state NMR are specifically encouraged to apply for access. A dedicated access portal has been designed: www.panace-nmr.eu. With the objective of promoting gender balance in scientific research, **women's participation** to on-site measurements is a priority criterion for the allocation of the measurement time.

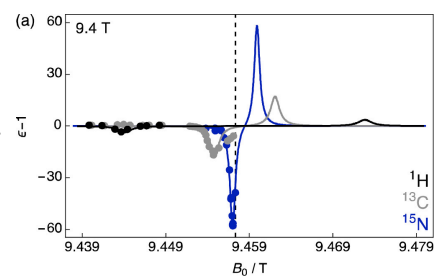
In parallel to access, the partners develop **joint research** (JRA) and **networking activities** (NA) such as staff exchange or joint data management, with the objective to improve the quality and quantity of the services provided by the infrastructures, and facilitate the use of modern solid-state NMR by non-expert users, widening the opportunities for novel application areas in chemistry. The PANACEA project will improve its impact through a number of actions, e.g. by holding diverse workshops (solid-state NMR schools for end-users as well as applied hands-on workshops), by training early-career (PhD, post-doctoral researchers) European chemists who are in high demand for jobs in industry and academia, and opening access for them to the different infrastructures through secondments and travel grants, or by organising workshops in collaboration with local or national NMR platforms of lower expertise.

- Key numbers :**
- 1650 TA days
 - 4 user meetings
 - 3 industry-targeted workshops
 - 27 spectrometers
 - 4 hands-on trainings
 - 32 taster days for industrials
 - > 200 chemistry-related projects
 - 1 NMR school for end-users
 - 8 summer fellowships for students

LIVE FROM THE PANACEA LABS

POLARISING AGENTS: Off-the-Shelf $Gd(NO_3)_3$ as an efficient electron source for DNP MAS NMR

Paramagnetic metal ions such as Gd^{3+} have recently shown promising results as polarizing agents (PAs) for DNP enhanced solid-state NMR spectroscopy. Prof. F. Blanc and co-workers recently demonstrated that the widely available and inexpensive

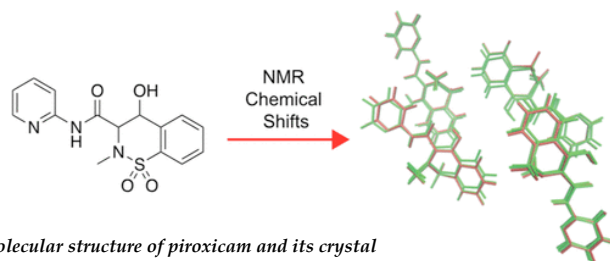


Experimental 1H , ^{13}C and ^{15}N DNP MAS NMR Zeeman field profiles of 1.5 M $[2-^{13}C,^{15}N]$ glycine doped with 20 mM $Gd(NO_3)_3 \cdot 6H_2O$ dissolved in $H_2O/D_2O/glycerol-d_8$ (1/3/6 v/v/v) as a function of the static magnetic field (B_0) acquired at 9.4 T.

chemical agent $Gd(NO_3)_3$ achieves sizeable enhancement factors for the ^{13}C and ^{15}N resonances of glycine at 9.4 T and ~ 105 K. Analysis of the field sweep profiles, jointly with with EPR data, reveals that the solid effect is the dominant polarisation transfer mechanism. This study paves the way to efficient DNP MAS NMR experiments without the need for challenging syntheses of Gd^{3+} polarising agents. The experiments at 9.4 T were carried out at CRMN Lyon in the framework of PANACEA.

PHARMACEUTICALS: solving crystal structures from machine learned chemical shifts

Determining the three-dimensional atomic-level structure of powdered solids, including drug molecules, is one the major challenges of modern chemistry. Solid-state NMR chemical shifts are structural probes of choice, but they are hampered by the high computational costs associated with crystal structure prediction methods and density functional theory (DFT) chemical shift calculations. Prof. L. Emsley and co-workers recently demonstrated that the crystal structures of ampicillin, piroxicam,



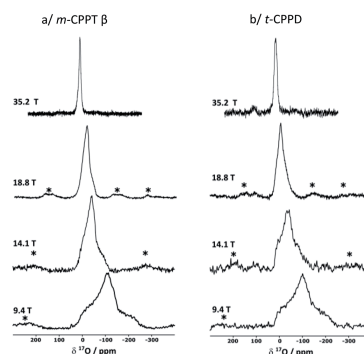
Molecular structure of piroxicam and its crystal structure determined by the machine learning approach. The red structures are the known structures, while the greens are the structures determined in this work that are less than 20 kJ/mol from the lowest energy determined structure and 0.5 ppm proton rmsd compared to the target shifts.

BALODIS et al, *J. Am. Chem. Soc.* 2022, 144, 7215–7223

^{17}O SOLID-STATE NMR: deciphering reaction mechanisms in mechanochemistry

Prof. D. Laurencin and collaborators, users of the PANACEA facility at Florida State University, investigated the crystal structure of two-phase calcium pyrophosphate, enriched in ^{17}O by mechanochemistry using ^{17}O -labelled water. ^{17}O NMR at various magnetic fields, including 35.2 T, jointly with DNP MAS experiments at 14.1 T, revealed the location of the water molecules within the crystalline network, shedding light into their role as reagents for ^{17}O -isotopic enrichment.

GOLDBERGA et al, *Farmday Discuss.* 2023, 241, 250–265.



^{17}O MAS NMR spectra of two hydrated phases of Ca-pyrophosphate at various magnetic fields, after ^{17}O -labelling using ball-milling.

PANACEA FELLOWS

PhD students and postdocs:
the next generation of NMR spectroscopists

WE ARE VERY PLEASED to welcome three new early stage-researchers involved in joint research and access activities of the PANACEA Network:

Thomas Robinson (PhD student, CNRS Lyon) - Armin Afrough (Post-doctoral researcher, Aarhus University) - Federico De Biasi (Post-doctoral researcher, EPFL)



Student profile: THOMAS ROBINSON

Q: Why did you decide to pursue a PhD in Science?

A: I started my studies by chemical engineering, but during my last year I also followed a Master's program in solid-state chemistry with a rich course on solid-state NMR. During my final Internship - that I performed at CRMN in Lyon - I definitely made up my mind about carrying on with a PhD.

Q: Why did you decide to become an NMR expert?

NMR is the only key to the understanding of numerous chemical systems that play an extensive role in tackling the challenges of our time. Hence becoming an "NMR expert" is my way to help chemists developing the game-changing compounds and materials of today and for tomorrow.

Q: Do you have a Science Hero or Mentor?

A: Antoine Lavoisier! He is the very first true chemist because he understood that he needed to develop a way of measuring precisely and quantifying the phenomena he observed before describing them. This talks to me in the context of NMR.

Q: What did you want to be when you were a child?

A: I actually wanted to be a chemist because I found fascinating to be able to understand phenomena occurring all over around, and to harness this knowledge to solve problems! I'm still very enthusiastic about that!

USER PROFILES

DANIEL & BENJAMIN
University of Liverpool (UK)

Q: Where are you from?

A: We are from Liverpool (UK) and we are working in Pr. F. Blanc's group.

Q: Which facility did you use for your experiments?

A: We went to the Centre de RMN à Très Hauts Champs de Lyon (CRMN) and we used their 400 MAS NMR spectrometer to conduct our experiments.

Q: Where have you heard of PANACEA?

A: We first heard about a project aiming to give access to national infrastructures regarding solid-state NMR, for academic and industrial researchers. When we saw a website was created to submit project, we did not hesitate.

Q: What did you do during your visit?

A: We wanted to help the development of new polarising agents (PA) for DNP, which can have

a large impact on many different fields of science. The sample used was a standard DNP preparation of glycine in frozen DNP juice solution, with and without $Gd(NO_3)_3$ as PA.

Q: Overall, did you enjoy this access opportunity?

A: We had a lot of help for our experiments, as they require a lot of patience and time. As the travel and accommodation was taken care by the PANACEA project, we did not have to worry about this aspect.

Q: Why would you recommend PANACEA?

A: One of the best feature of this European project is its free access for academic and industrial users!

We were glad our access facility (CRMN) did help us through the whole process with skilled experts, especially with some questions and challenges we had before and during the experiments. We highly recommend researchers which have no or little knowledge in solid-state NMR to submit projects, especially if they would like to have another point of view on their own research.



Daniel DUFF - Phd student & Benjamin CHENEY - Post doc from the University of Liverpool

Jessica WADE - Research Fellow at Imperial College, London

JESSICA
Imperial College (UK)

Q: Where are you from and what is your field of research?

A: I am from London; I work in materials science for the next-generation semiconductor devices.

Q: What was the purpose of your visit?

A: I came to CRMN Lyon to study the molecular structure of polymer – small molecule blends, and understand how chirality impacts the twist of the conjugated backbone.

Q: Are you familiar with solid-state NMR?

A: Before the trip, I only heard about solid-state NMR from papers. After the trip I feel I learnt a lot. I am inspired to learn more, and continue using it in our research.

Q: Overall, did you enjoy this access opportunity?

A: I loved it – the people, the centre, the magnets, the organisation. 10/10!



ZOOM ON A FACILITY

The Swedish NMR Centre
University of Göteborg

Göran KARLSSON (platform manager), Ulrika BRATH and Arthur PINON (local operator) are presenting their facility

Q: Describe your facility.

A: The Swedish NMR Centre at the University of Gothenburg is a national and international research infrastructure situated on the Swedish West Coast. We provide access to state-of-the-art NMR spectrometers in the areas of material science, biomolecules, metabolomics, organic chemistry and chemical biology. With magnetic fields ranging from 400 MHz to 900 MHz (including DNP) we offer open access and expert support to researchers and users from academia and industry.

Q: What are your main expertise fields?

A: NMR methods development is in focus at the Swedish NMR Centre, specifically techniques for non-uniform data acquisition,

and processing (NUS). The NUS methodology has been incorporated in the regular software for NMR spectrometer operation (TopSpin) provided by Bruker. The Swedish NMR Centre has recently entered a collaborative research project with AstraZeneca (PI Staffan Schantz) with a focus on MAS DNP-NMR applications in pharmaceutical science.

Q: In the PANACEA access activity, which spectrometers are open to access?

A: Users will have access to our 400 DNP-NMR spectrometer. The DNP-NMR system comprises a 400 MHz wide-bore magnet with a water cooled 263 GHz gyrotron and a 3.2 mm HXY-probe, for up to 15 kHz MAS. The probe can be tuned to accommodate the most widely used nuclear pair combinations for bio- and materials science, i.e. $^{13}C/^{15}N$, $^{31}P/^{13}C$, $^{27}Al/^{29}Si$, $^{11}B/^{29}Si$ and $^{29}Si/^{15}N$.



PANACEA EVENTS in 2022

EUROMAR, Utrecht - July 2022

PANACEA was present at the EUROMAR Conference, the largest European conference in NMR spectroscopy, gathering ~700-1000 scientists from all Europe across disciplines. PANACEA consortium members held a booth where they advertised the project and had fruitful interactions with future users. This allowed a very efficient promotion of the network within the European community of NMR spectroscopists.



PANACEA first annual user meeting and industrial day, Lyon - October 2022

The first annual users meeting and industrial day of PANACEA was held in Lyon. This two-day European event, entitled « Solid-state NMR for the gifted chemist » gathered 100 participants onsite and more than 50 participants online. The meeting led to extensive and high-level discussions between academic and industrial users, and consortium partners, on both the latest developments in solid-state NMR spectroscopy and their application in modern chemistry and materials research. Academic and industrial users

had the opportunity to participate to six different mini-symposia, each on a different theme: Solid-state NMR for glasses, inorganic and lightning materials; Solid-state NMR for pharmaceutical research; Solid-state NMR for polymers, biopolymers and biomaterials; Solid-state NMR for energy materials; Solid-state NMR for catalysis; Solid-state NMR instrumentation, software and remote access. In each of them, one presentation was given by an industrial user. The event ended up with a cello concert and a banquet on a cruise along the Saône river.

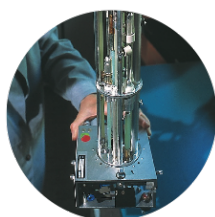


First PANACEA hands-on workshop, Lausanne - August 2022

The first PANACEA hands-on workshop entitled « Solid-state NMR methods for pharmaceutical formulations » was held at the Ecole Polytechnique Fédérale de Lausanne (Switzerland). This 3-day training event gathered 15 PhD students, post-docs and industrial scientists. The workshop was focussed on both theoretical and practical aspects of solid-state NMR for pharmaceutical research. The participants had the opportunity to implement experiments on 4 different instruments, under the supervision of experienced scientists. The workshop also provided excellent opportunities for early-stage researchers to exchange with international experts, including during the many coffee breaks and the banquet around a fondue and other Swiss specialities.

Second PANACEA hands-on workshop, Nijmegen online 27 May 2023 & on-site 12-16 June 2023

Radboud University (RU) jointly with Aarhus University and CNRS Orleans will organise an hands-on experimental workshop entitled « Quadrupolar NMR, from advanced experiments to data interpretation and numerical simulation » in the spring 2023. This event targets researchers already experienced in solid-state NMR, who would like to expand their knowledge and skills on this specific topic. This 3-day event will include practical training at the NMR spectrometer, on both model and challenging systems. Participants will be encouraged to bring their own samples. More information is coming on our website.



EUROMAR, Glasgow - 9-13 July 2023

PANACEA will be present at the 2023 EUROMAR Conference, which will be held in Glasgow, Scotland. A lunchtime workshop will be organised to promote the capabilities of the network, as well as to attract new users.

EUROMAR2023
The 19th European Magnetic Resonance Congress
9-13 July 2023
Scottish Exhibition Campus (SEC)
Glasgow, Scotland, UK

UPCOMING EVENTS

Satellite workshop with the Dutch Polymer Institute, Utrecht - November 2023

A PANACEA satellite event will be organised in the fall during the annual Dutch Polymer Institute (DPI) meeting. DPI is an industry-driven international collaboration platform for research in the field of polymers. This event targets a highly international community with industrial and academic research from all over Europe, but also from China, Canada, Brasil... This will represent a unique opportunity to advertise PANACEA to the polymer community. More information is coming on our website.

PANACEA 2nd annual user meeting and industrial day, Florence - November 2023

The second annual users meeting and industrial day of PANACEA will be held in Florence (IT). This will be a two-day event, with a similar format as the one organised in 2022. More information is coming on our website.

