FACULTY OF SCIENCES

DEPARTMENT OF ORGANIC AND MACROMOLECULAR CHEMISTRY

NMR EXPERTISE CENTER

Est. August 2017

GHENT UNIVERSITY

NMR Expertise Centre

NMR & structural analysis unit

SOME HISTORY

A long long time ago…

> 68 years of NMR history and counting

Expo '58 - First commercial NMR spectrometer by Varian, installed at UGent
1958

60MHz
1969

100MHz
1966

1st 300MHz NMR spectrometer in EU is installed at UGent
1972

360MHz
1979

500MHz
1987

300MHz
2002

700MHz - Interuniversity collaboration for high-field equipment with a value of 2.019.502€
2005

500MHz
2011

400MHz
2015

500MHz
2026

1958 | 1968 | 1978 | 1988 | 1998 | 2008 | 2018 | **2027**

2002
Launch NMRSTR Research Group by Prof. J.C. Martins

NMR & structural analysis unit

2018
Start of NMR EC - funded by BOF UGent

2023
Transtion to Core Facility - funded by BOF UGent

NMR Expertise Centre

GHENT UNIVERSITY

NMR SPECTROSCOPY

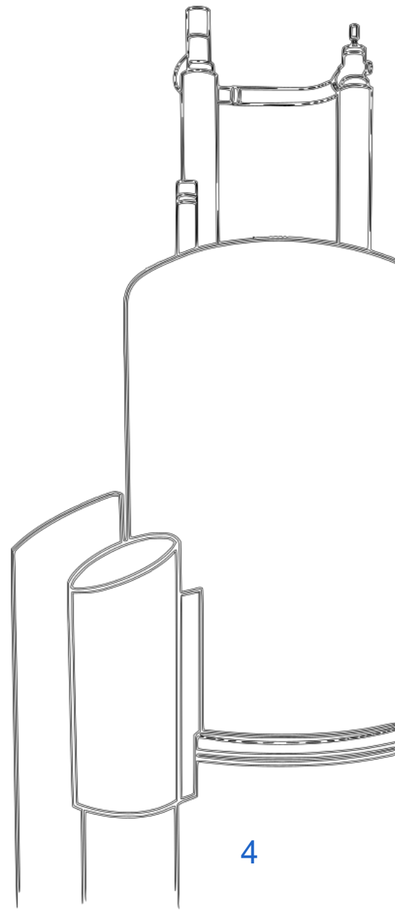
NMR is a non-destructive spectroscopic technique that can provide detailed information on the **structure**, **dynamics, reaction state** and **chemical environment** of molecules

Advantages

- Multi-element capabilities
  In principle any nuclei possessing spin can be studied
- Provides unique, well-resolved, analytically tractable and often highly predictable spectra for small molecules
- High-throughput method with high degree of automatization
- Non-destructive method
  Samples can be recuperated afterwards

Disadvantages

- Lower sensitivity
  Counteracted by the use of high-field & specialized probes
  Higher sample amounts can be used and recuperated afterwards



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EXPERTISE

Application areas

Life sciences

- Metabolome profiling of hypertensive mammals during early pregnancy
- Study of biopolymer-based wound dressings
- API impurity structure elucidation

Chemistry

- 2D & 3D structure of molecular conformations
- Membrane interactions of cyclic lipodepsipeptides
- Surface chemistry of metal oxide nanocrystals

Food Science

- Fingerprinting of Gouda cheese ripening
- Purity of foodstuff intermediates

Agriculture

- Study of emulsifier agents in diary industry
- Identification of false positives in antibiotic residue screening of farm milk
- Honeybee wax screening for adulteration

Engineering

Determine cross-linking efficiency in superabsorbent polymers for self-healing mortar applications

Technology

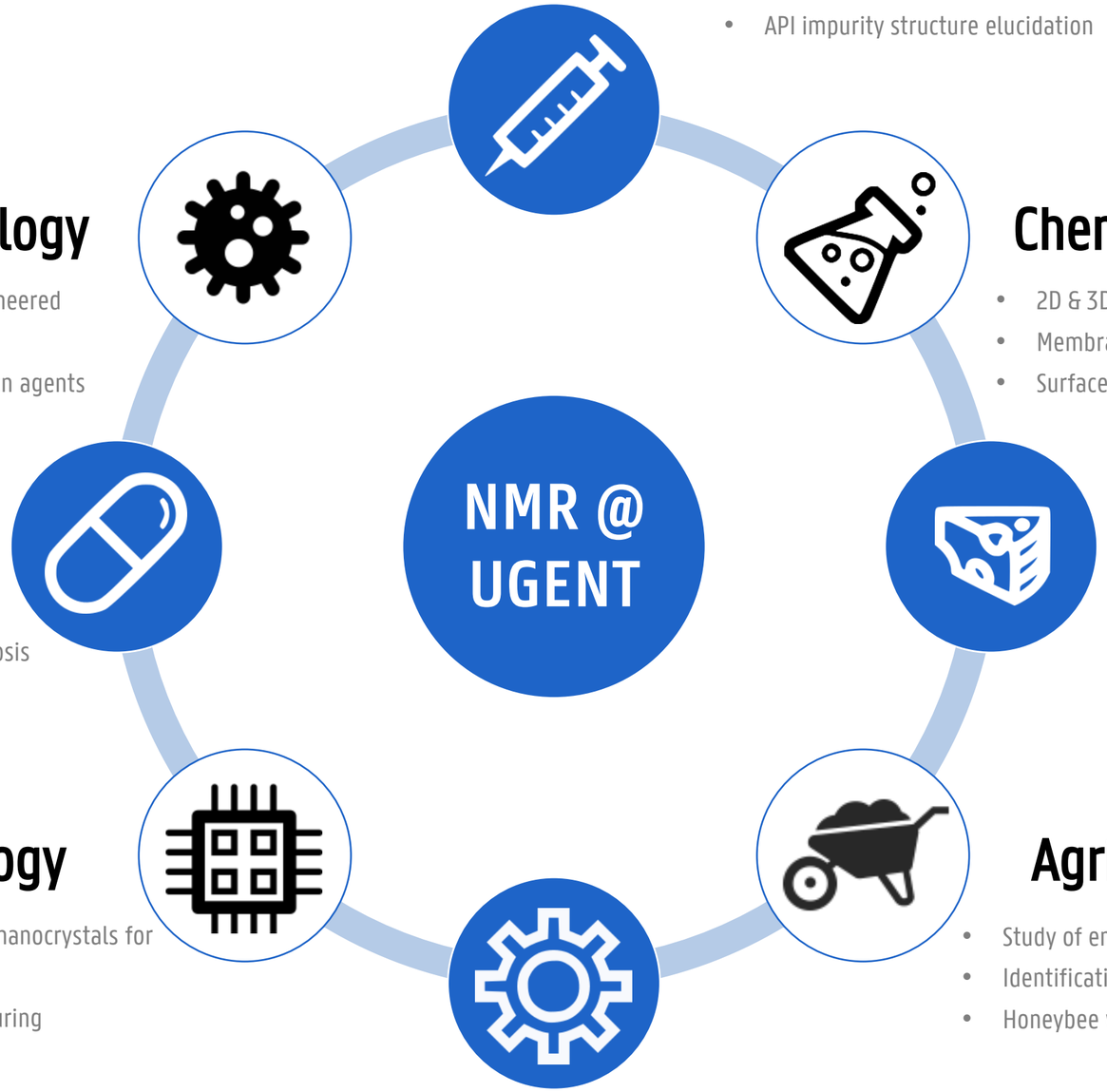
- Study of colloidal nanoparticles and nanocrystals for solar cell applications
- $GeCl_4$ purity study for chip manufacturing

Medicine

- Ibuprofen complexation studies
- Characterization of new API's for cystic fibrosis and cancer treatment

Biology

- Structure elucidation of genetically engineered plant metabolites
- Study of cyclic peptides as crop protection agents



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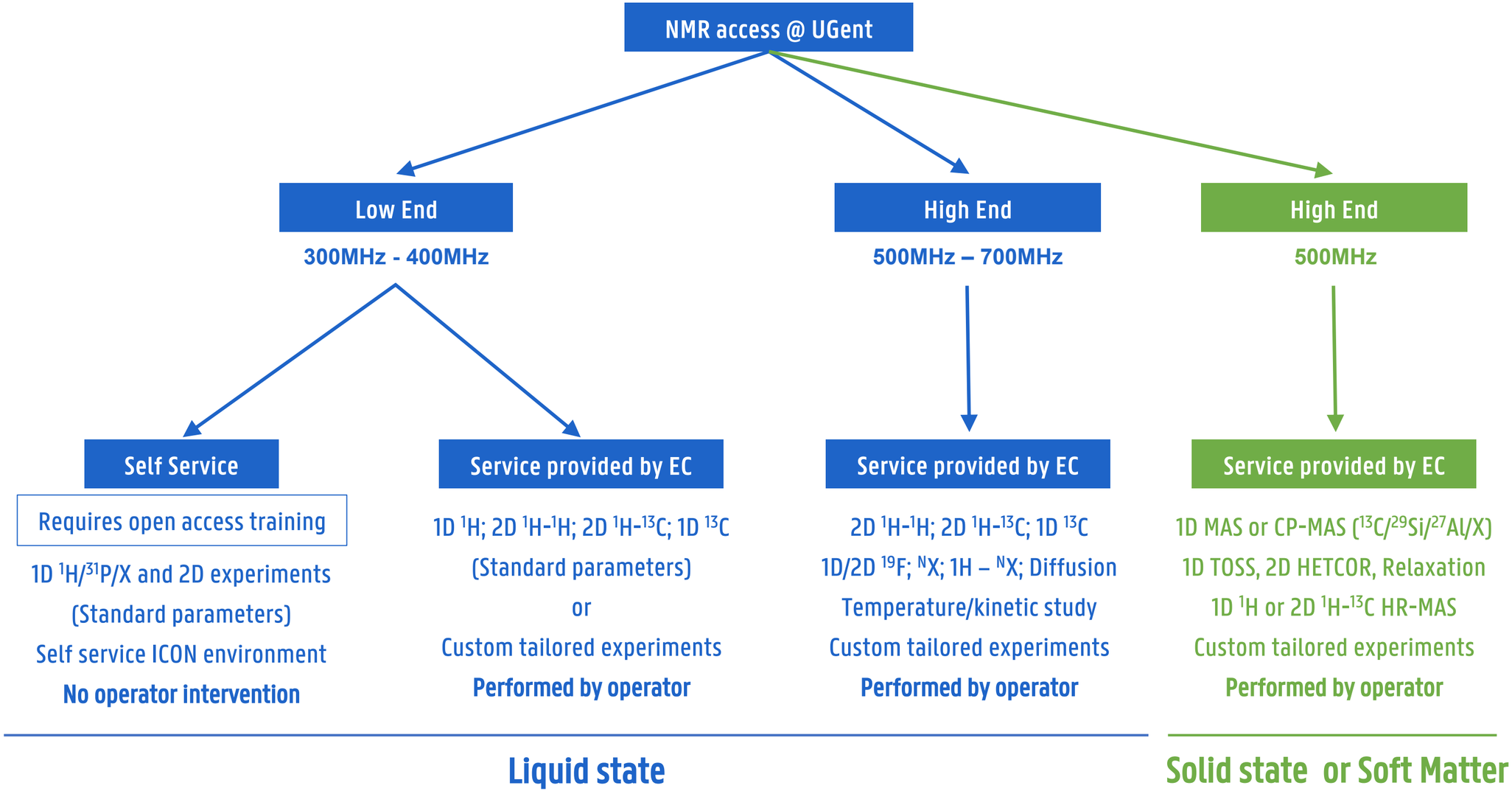
ACCESS



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SERVICES FOR INDUSTRY & ACADEMIA

What

Any soluble material that has at least one NMR active isotope
(spin > 0 and preferably = 1/2)
and
at least **50µmol** of sample material

Our offer

- Expert team with > 20 years of experience
- State-of-the-art infrastructure
- High sample throughput in routine applications
- Standardized or highly customized measurements
- NMR support from low to high-end applications
- Training for basic to advanced spectral interpretations (1D – 2D techniques)
- Hands-on access for externals

Pricing

Please contact us to receive a customized quote

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ANALYSIS REQUEST FLOW

1. Client defines the problem or questions to be solved

2. During a meeting or call the requirements for the measurements or analysis are discussed

3. Based on the requirements, a custom agreement is made

4. The client formally agrees with the offer and the samples are made available

5. NMR analysis is performed and a report is drafted

6. Following the report, the results can be discussed in detail and feedback is given

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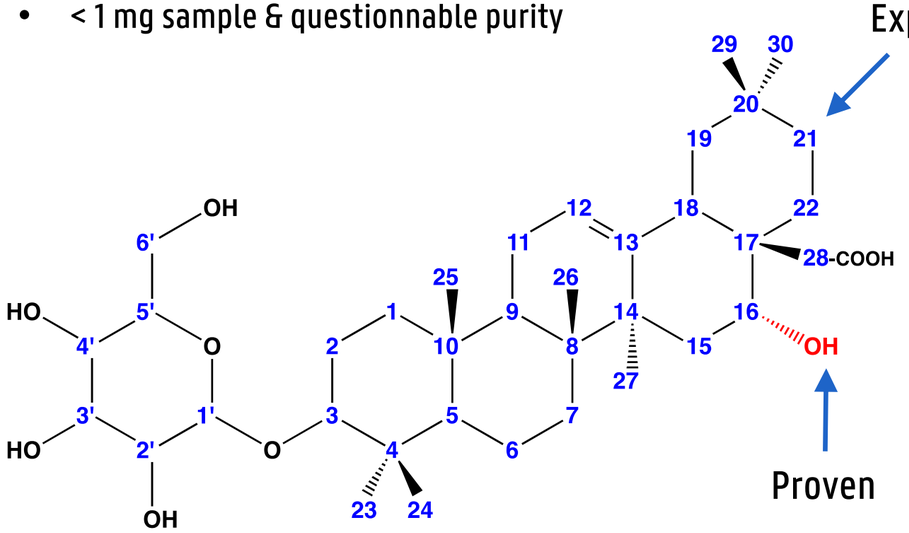
EXPERTISE

Structure elucidation and confirmation of small to medium organic compounds

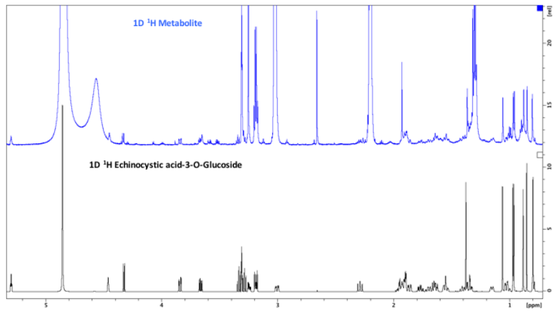
A. Goossens

Towards synthetic biology platforms for the production of bioactive triterpene sapo(ge)nins.

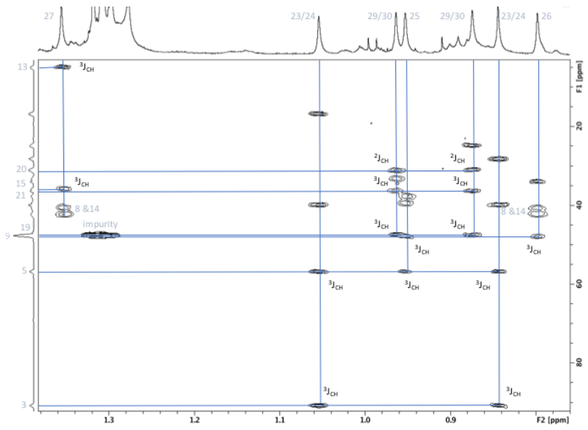
- Position of hydroxylation in 3-O-Glc-echinocystic acid?
  - -OH @ C_{16}, C_{21}, C_{28} or C_{30} position?
  - Relative stereochemistry?
- Structure intact?
- < 1 mg sample & questionnable purity



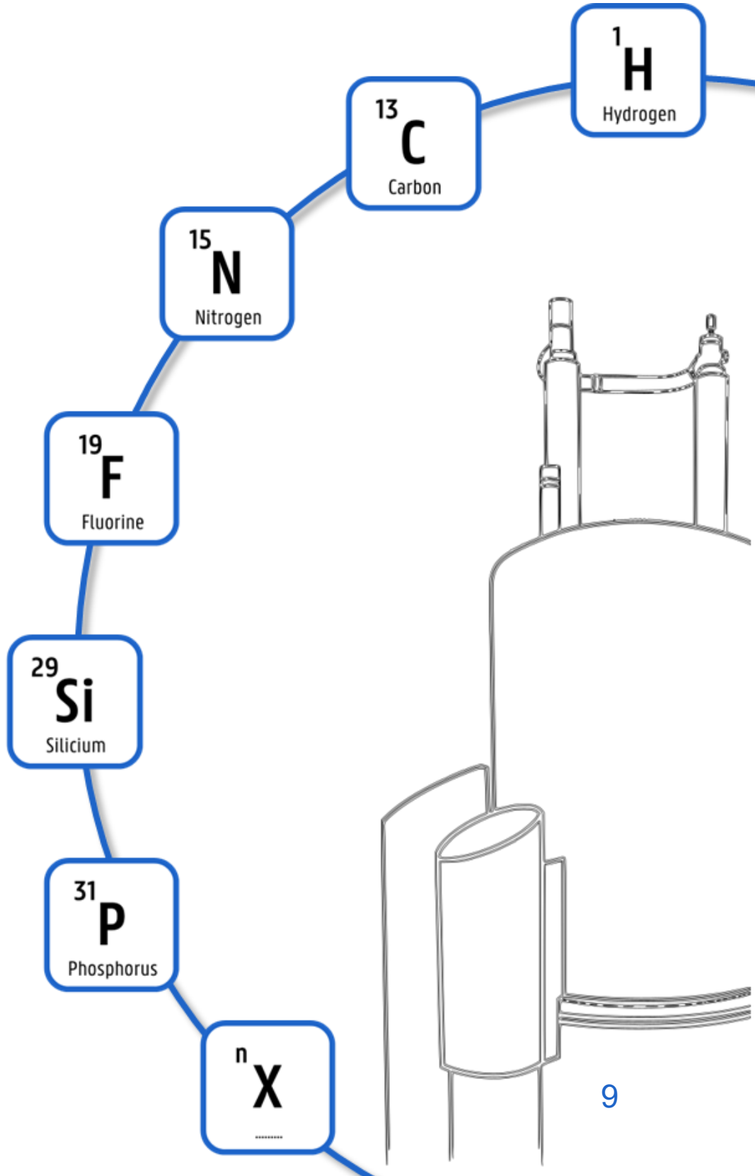
3-O-Glc-echinocystic acid



1D ^{1}H & ^{13}C spectra for comparison with know standard



2D ^{1}H –^{1}H, ^{1}H-^{13}C spectra for structural integrity and -OH localization



Moses, T., et al., *Proceedings of the National Academy of Sciences*, **2014**.
DOI 10.1073/pnas.1323369111

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VIB

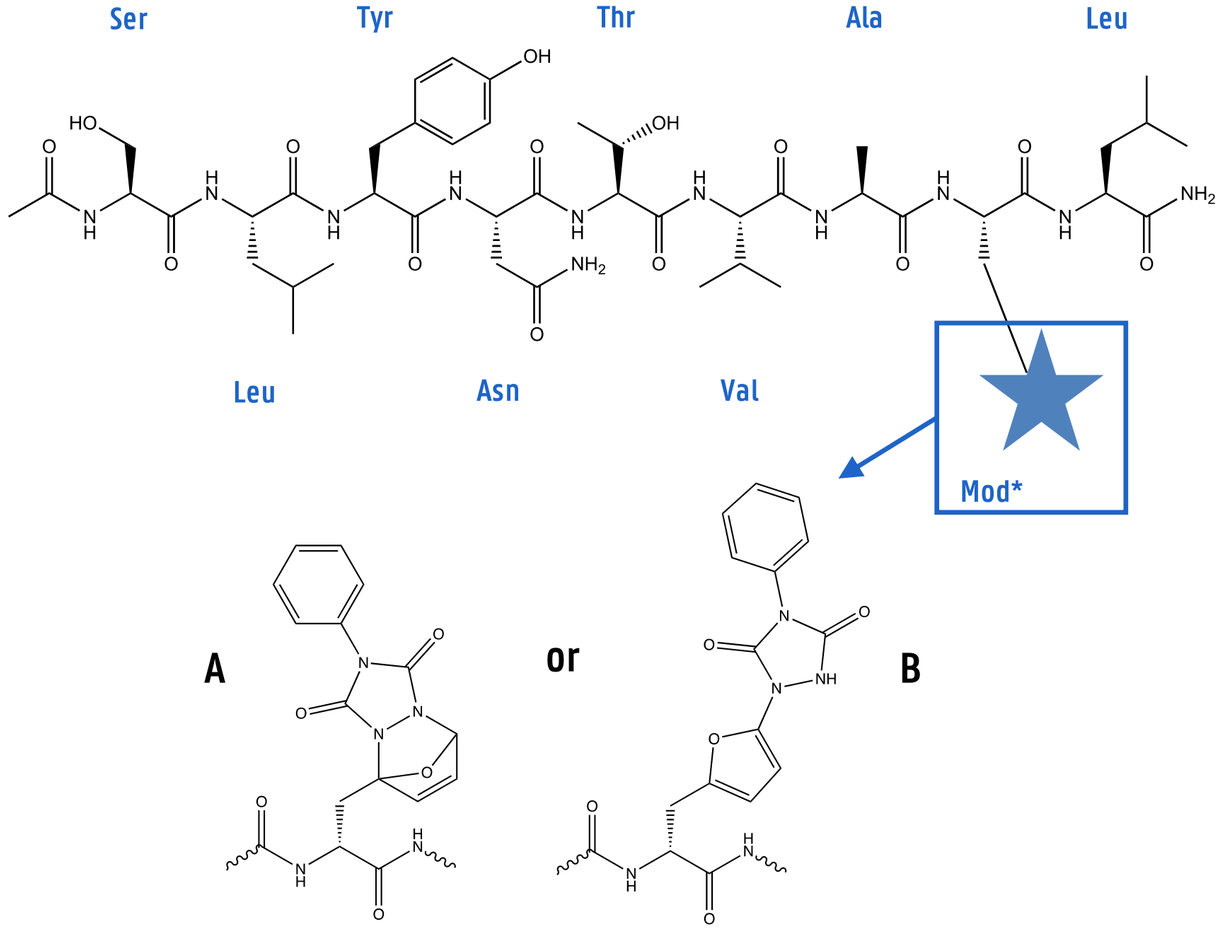
EXPERTISE

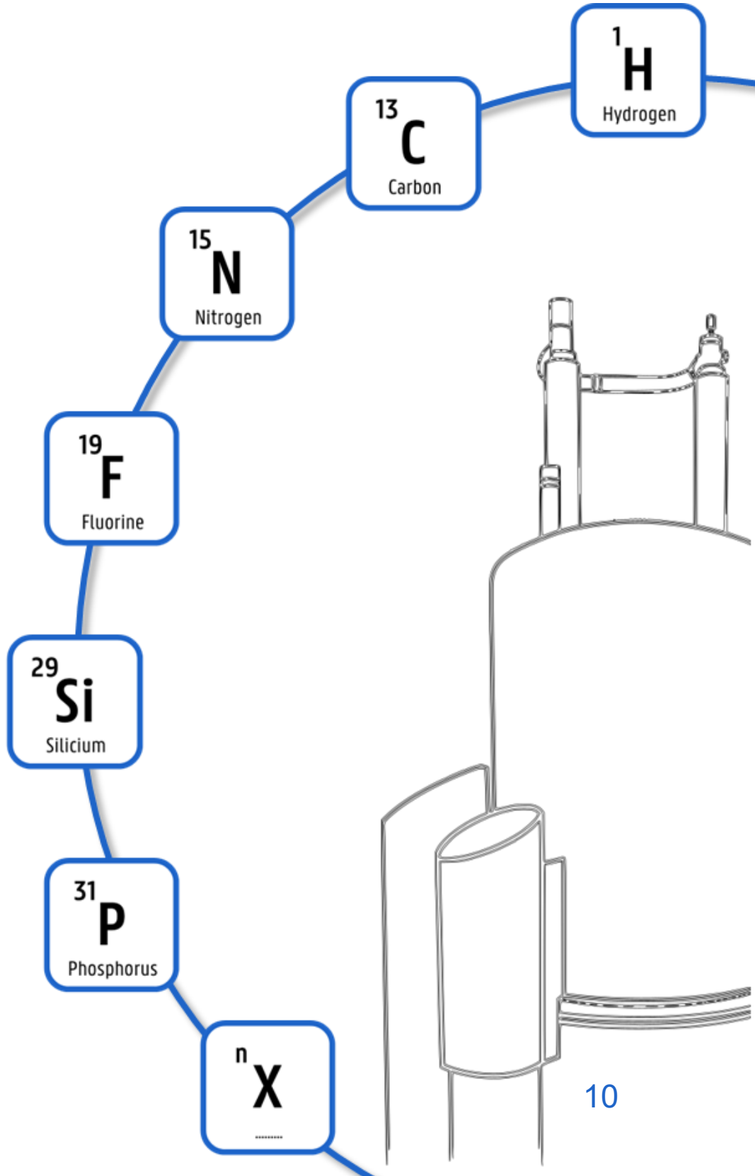
Structure elucidation and confirmation of peptides

K. Hoogewijs and A. Madder

Furan chemistry in reversible and irreversible orthogonal peptide labeling

- Amino acids and overall peptide sequence intact?
- Structure of the furan labeled peptide (Mod*)?





Hoogewijs, K., et al., *Chemical Communications*, **2013**.
DOI 10.1039/C3CC40588E

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EXPERTISE

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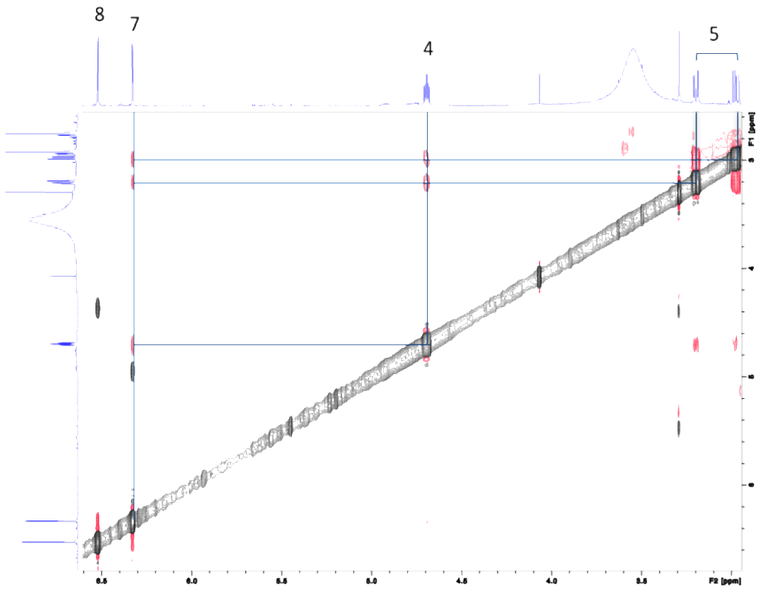
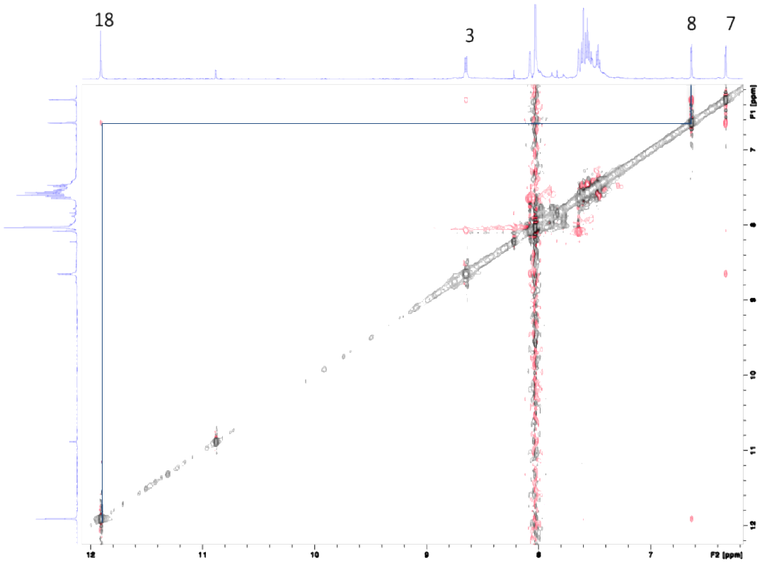
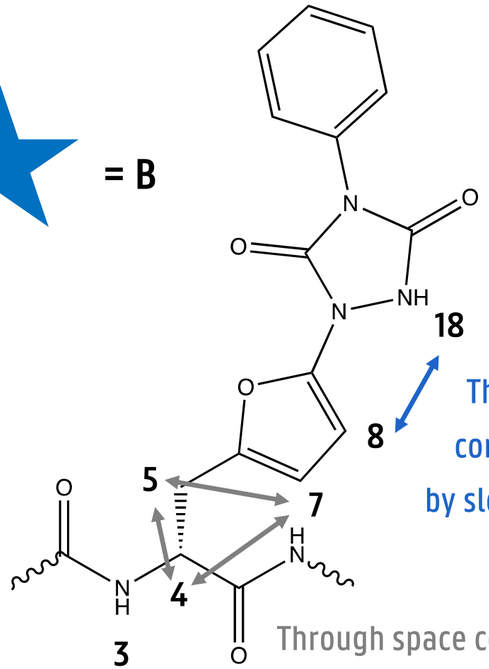


2D ROESY 500MHz, 150ms, 25°C



2D ROESY 500MHz, 150ms, -50°C

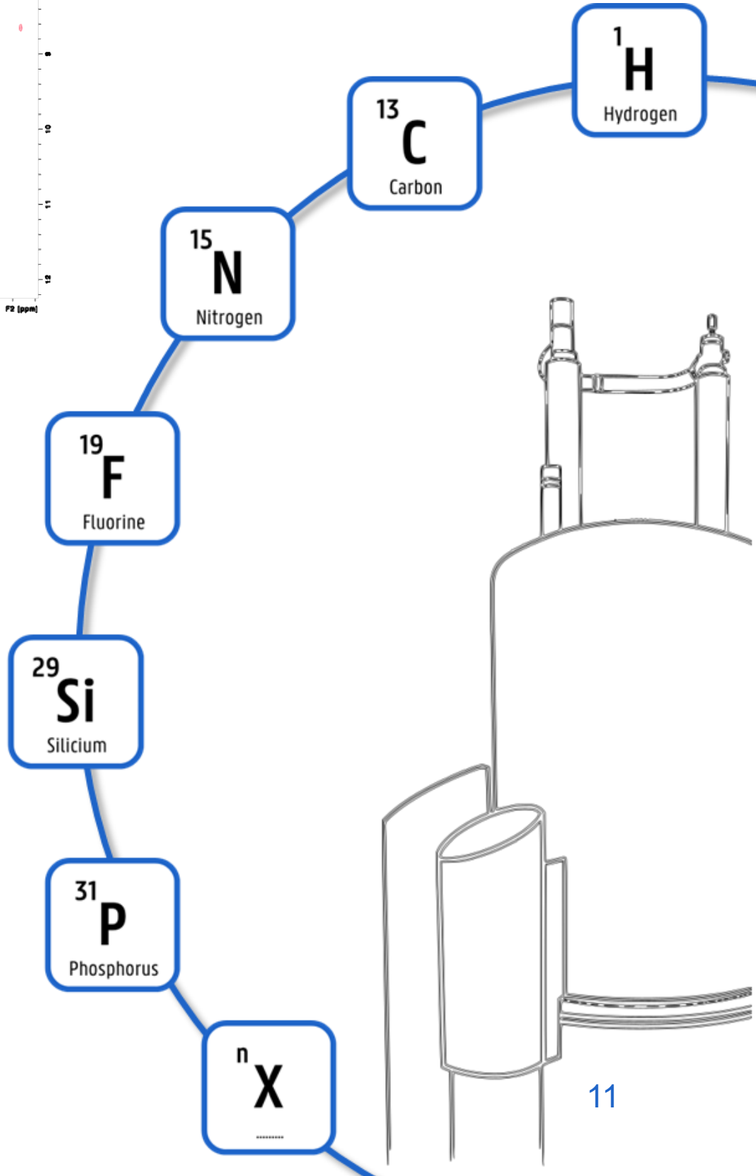
= B



Through space contact at **-50°C** by slowing exchange

Through space contacts at **25°C**

Hoogewijs, K., et al., *Chemical Communications*, **2013**.
DOI 10.1039/C3CC40588E



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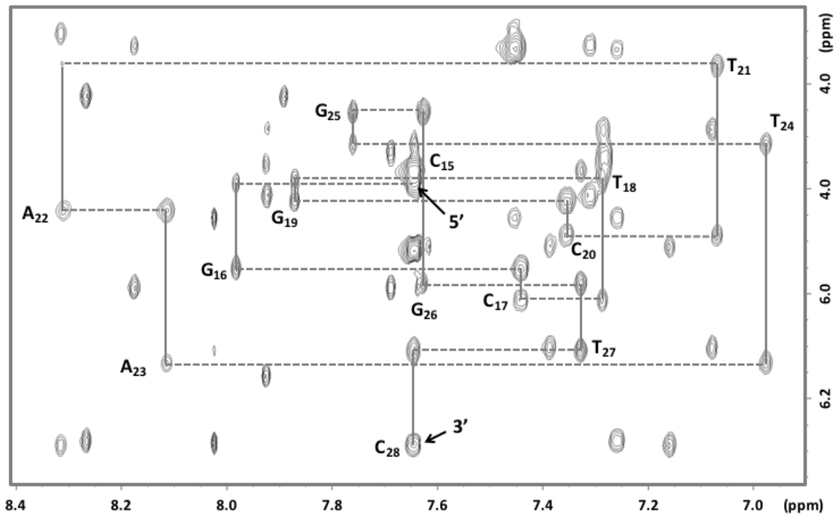
EXPERTISE

Structure elucidation and 3D structure determination of biomacromolecules

A. Madder and J.C. Martins

Cyclic lipodepsipeptide & chemically modified aptamer research topics



2D ^{1}H –^{1}H, ^{1}H-^{13}C spectra

&

Computer assisted model building

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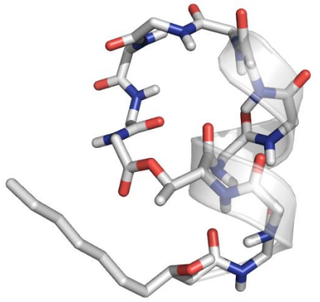
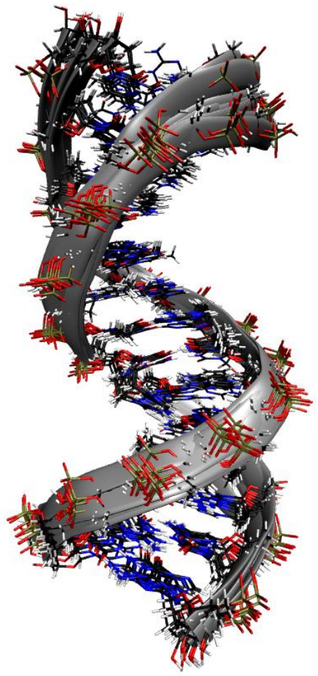
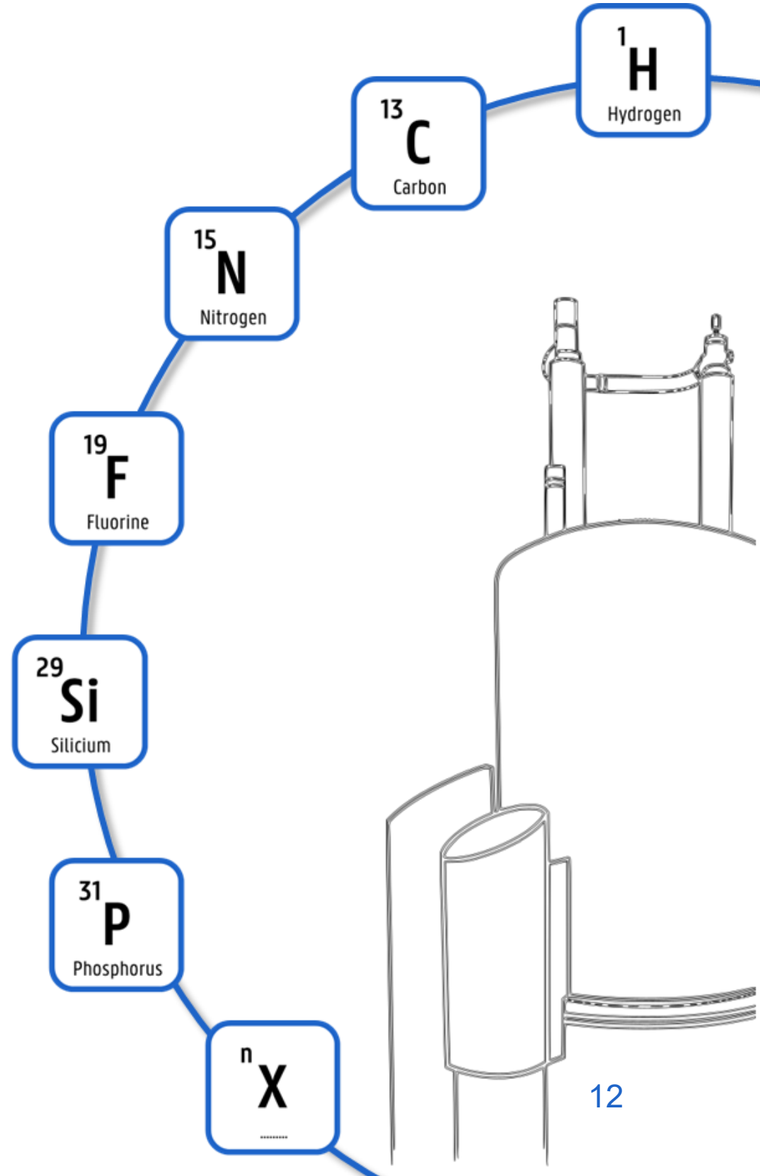


Peptides



Nucleic acids



- Buyst, D.; et al.; *Nucleic Acids Research* **2015**, 43. DOI 10.1093/nar/gku1306

- Geudens, N.; et al.; *Chembiochem* **2014**, 15. DOI 10.1002/cbic.201402389

EXPERTISE

hr-MAS as a tool to study polymer networks

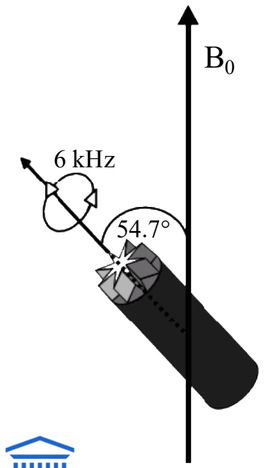
F. Du Prez & B. Fritzinger

hr-MAS NMR to monitor and quantify chemical modifications of cryogels

High resolution NMR
> Samples in solution

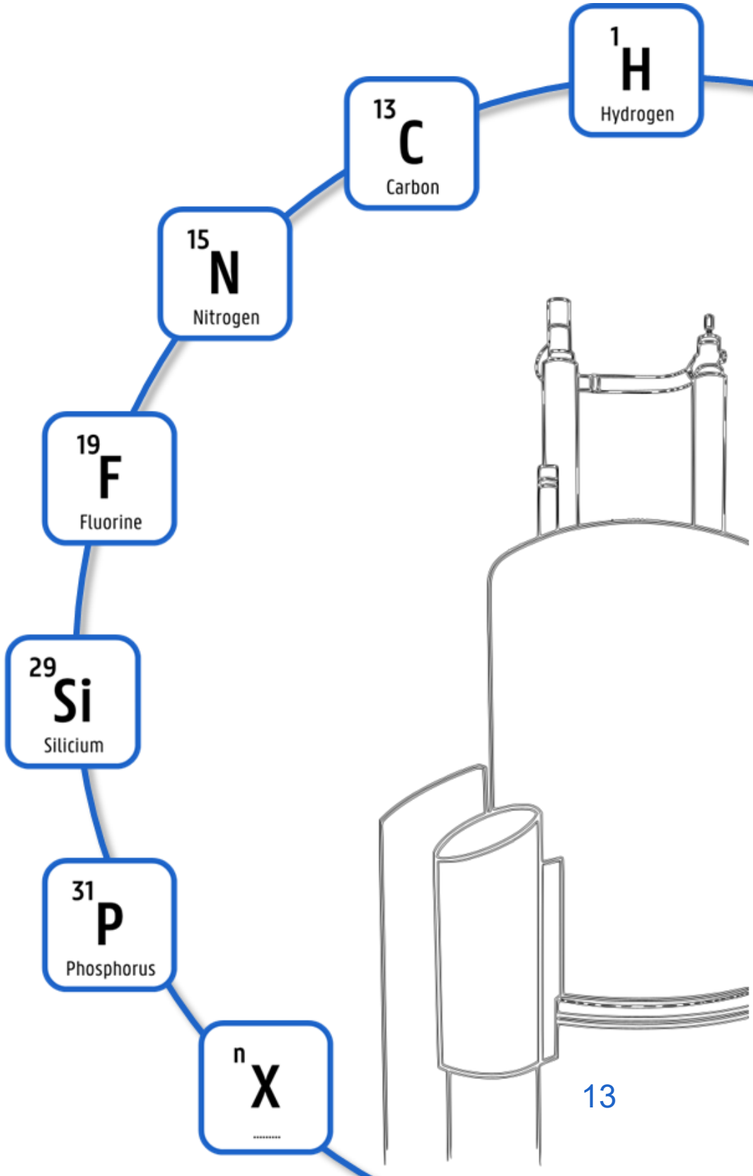
Magic Angle Spinning (MAS) NMR
> Solid materials as powder

High resolution MAS (hr-MAS) NMR
> Materials at the interface of solution and solid state



Sample in rotor is spun at high frequency under the magic angle

This allows the application of most high-resolution techniques to characterize molecules grafted onto insoluble supports
= interfaces, networks, matrices



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EXPERTISE

Van Camp, W.; et al.; *Macromol. Rapid Commun.* **2009**, 30.
DOI 10.1002/marc.200900087

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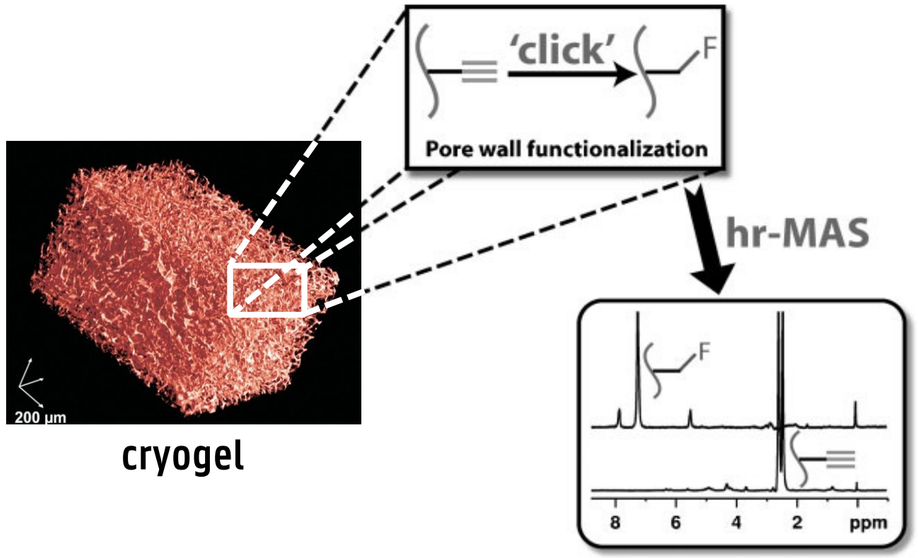
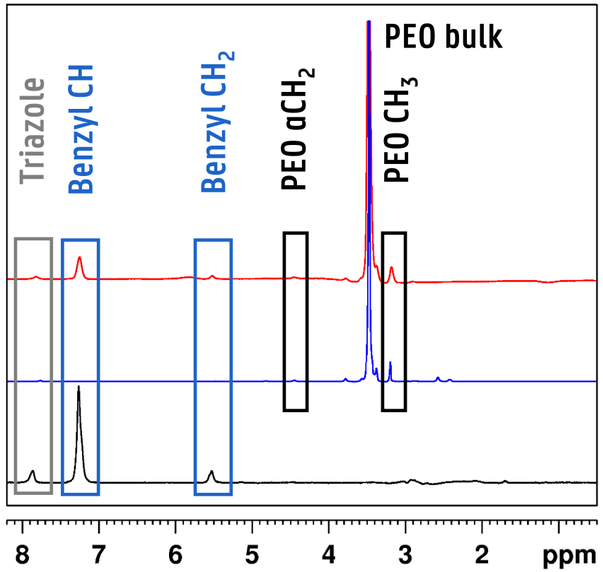
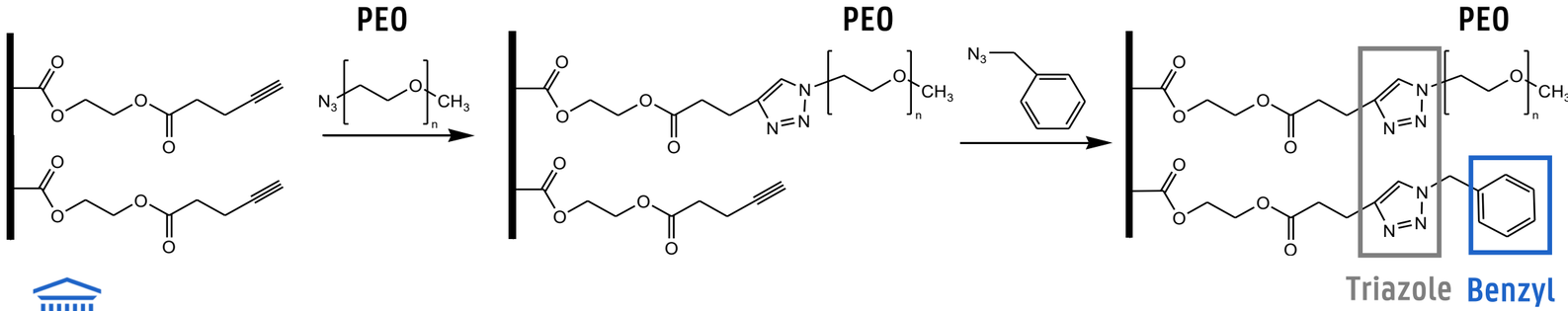
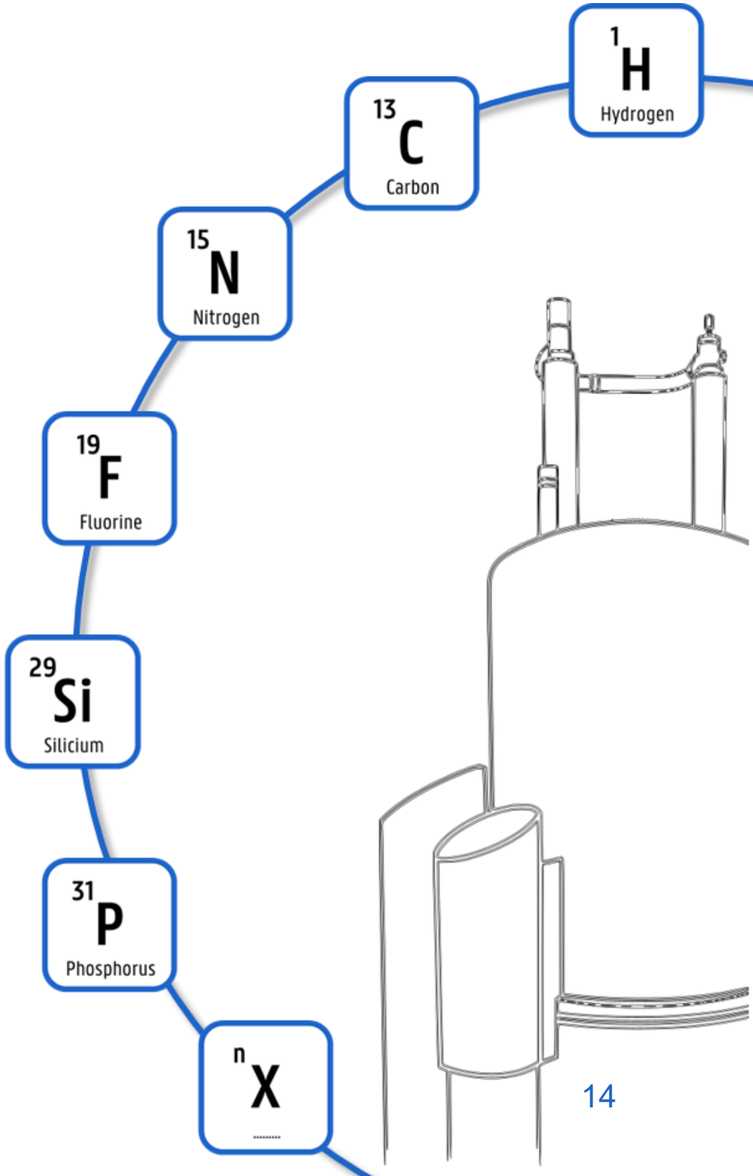


cryogel





Quantifying multistep reactions in 'solid-like' materials



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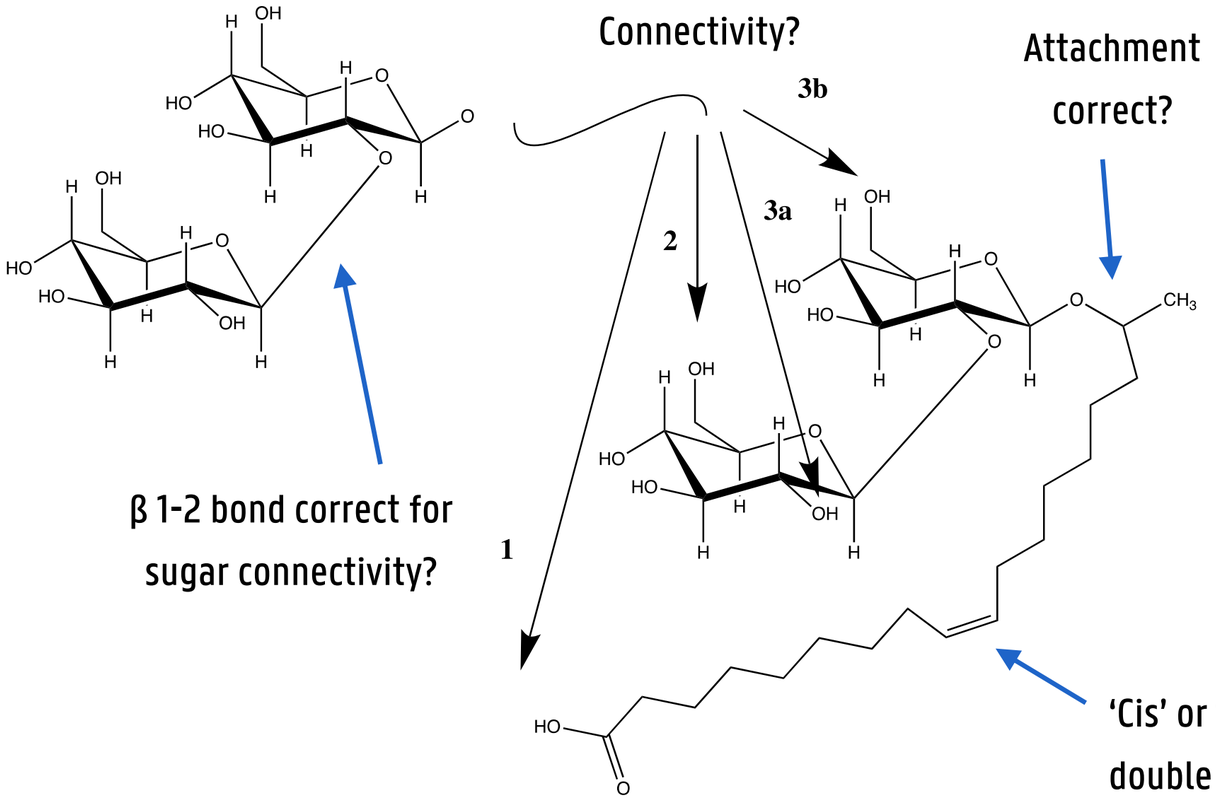
EXPERTISE

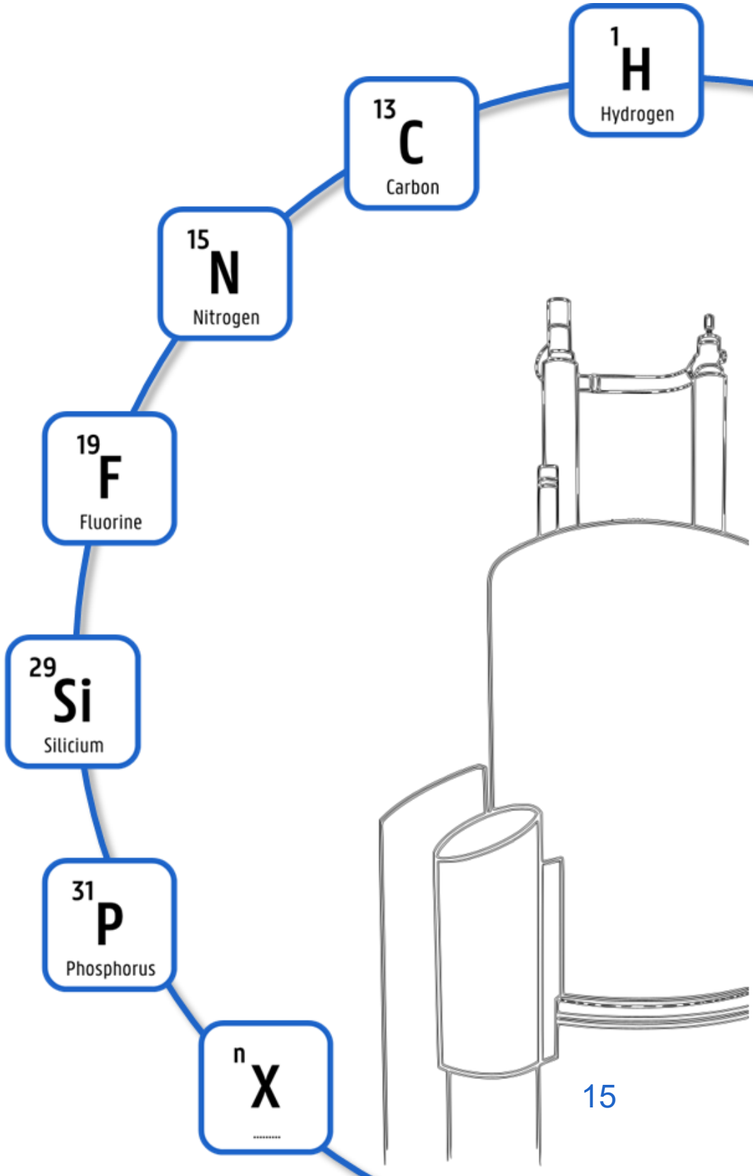
Structure elucidation of surfactants

I. Van Bogaert

Synthesis of bolaform surfactants by an engineered Starmerelle bobicola yest

- How is the aliphatic tail attached to the sugar entity 'A,B' ?
- How is sugar entity 'C,D' attached to the main part of the molecule and what is their mutual connectivity?
- What is the configuration of the aliphatic tail double bond?
- What are the anomeric configurations of the four sugar rings?





Van Bogaert, I.; et al.; *Biotechnology and Bioengineering* **2016**, 49.
DOI 10.1002/bit.26032

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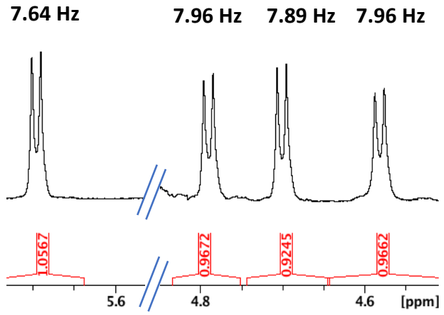


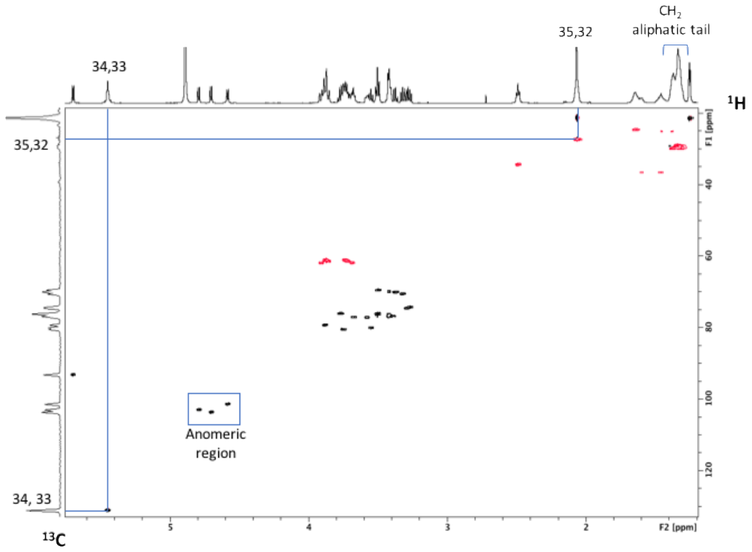
Anomeric ^{1}H scalar couplings prove overall β configuration of all sugars

Correct!

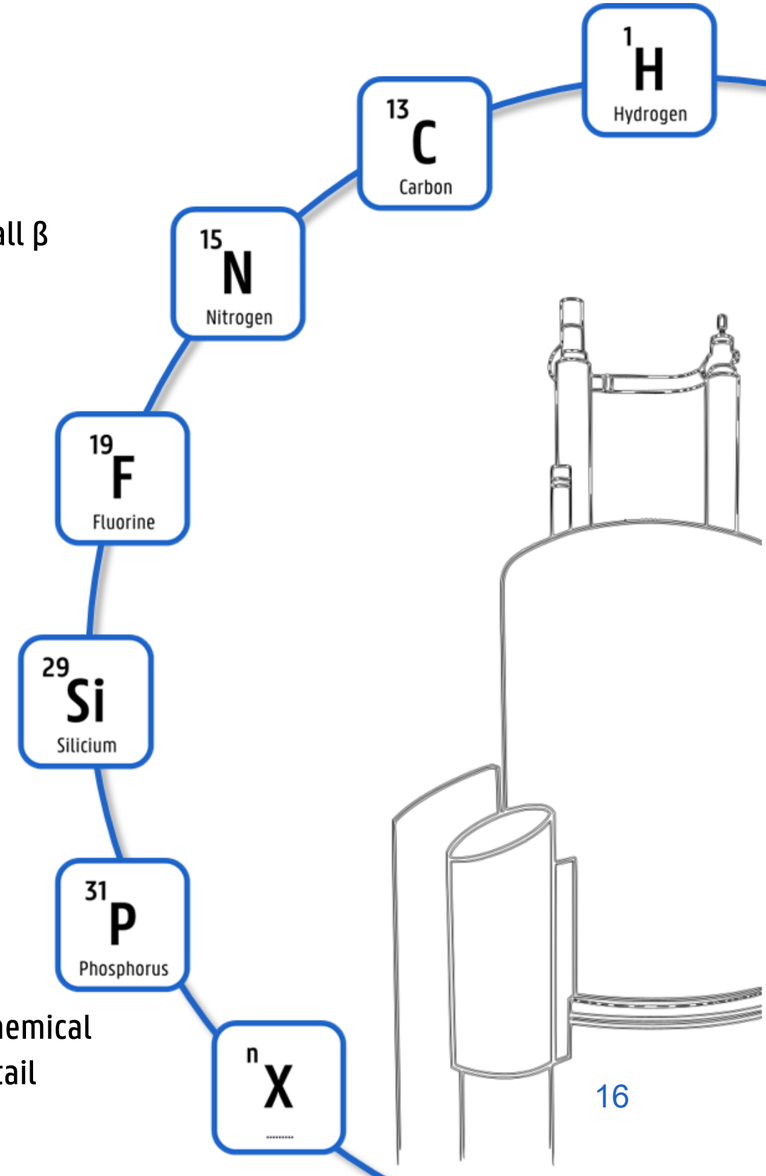
Connectivity proven!

'Cis' !

β 1-2 bond correct!



^{1}H-^{13}C assignment shows connectivities of different sugars & ^{13}C chemical shift analysis shows orientation double bond in the aliphatic tail



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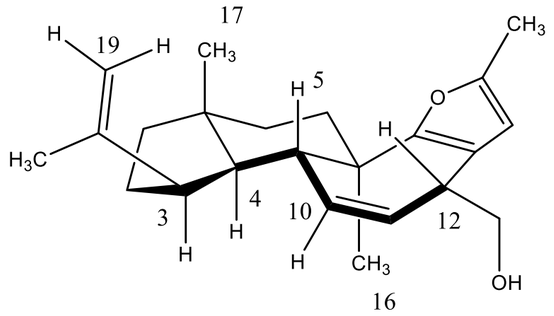
EXPERTISE

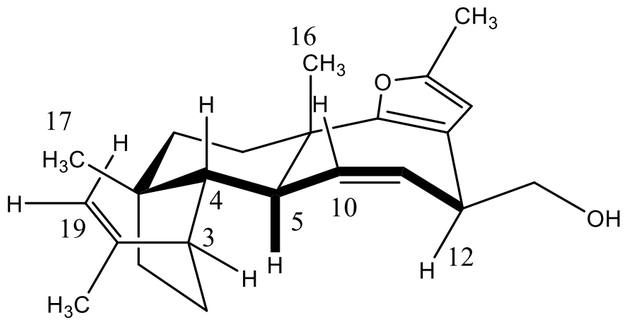
Extracting coupling constants for conformational discrimination

J. Winne

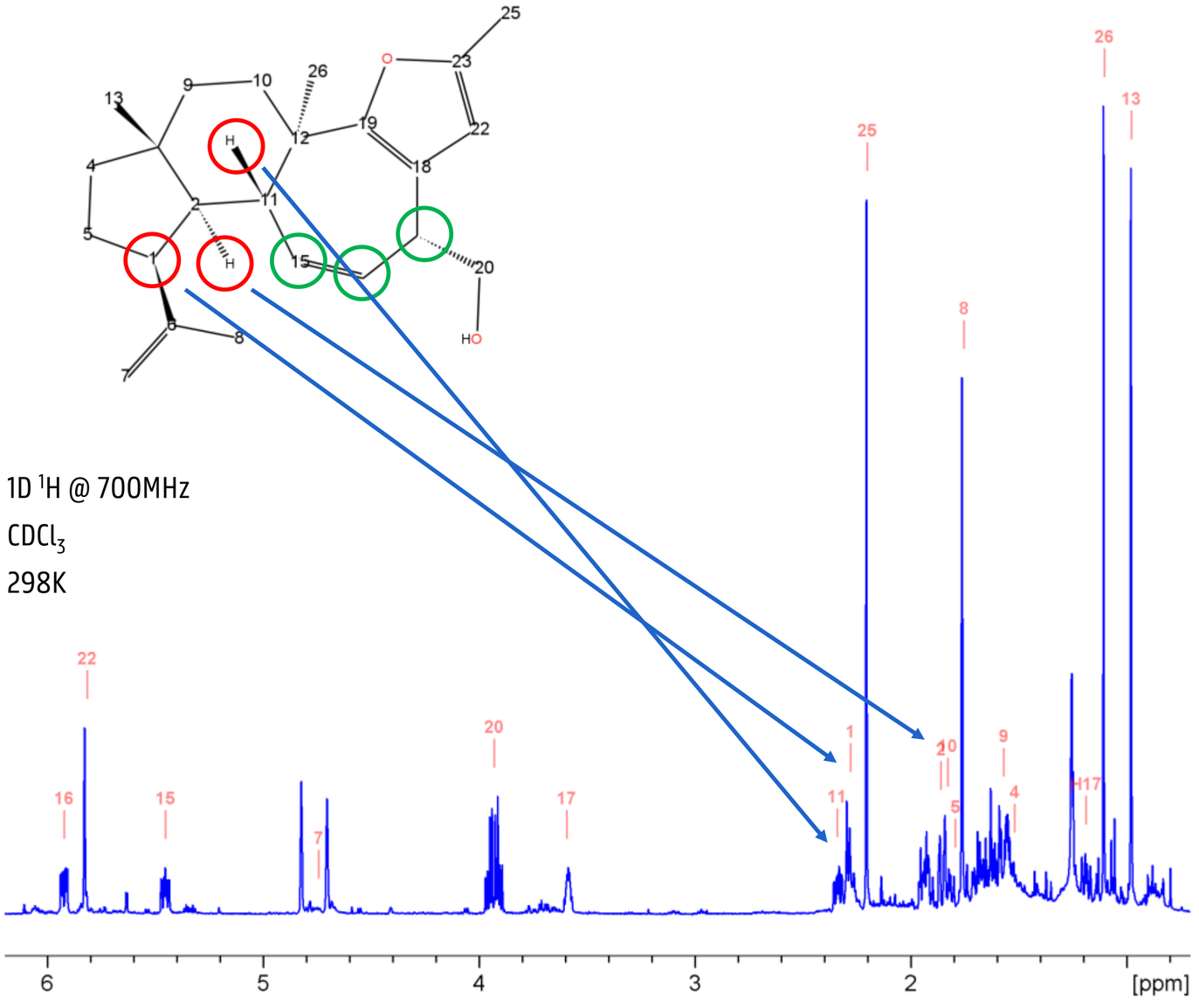
From 2D to 3D NMR structure elucidation in terpenes



Conformation A or B ?



Step 1 – identify key signals



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Sinnaeve, D.; et al; *Angewandte Chemie* **2016**, 55 .
DOI 10.1002/anie.201508691

EXPERTISE

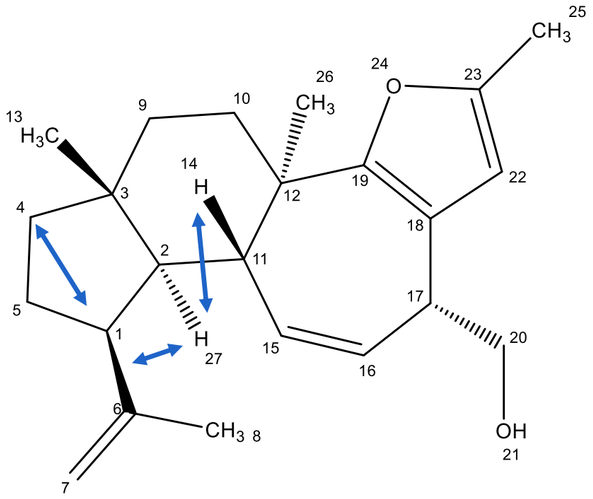
Extracting coupling constants for conformational discrimination

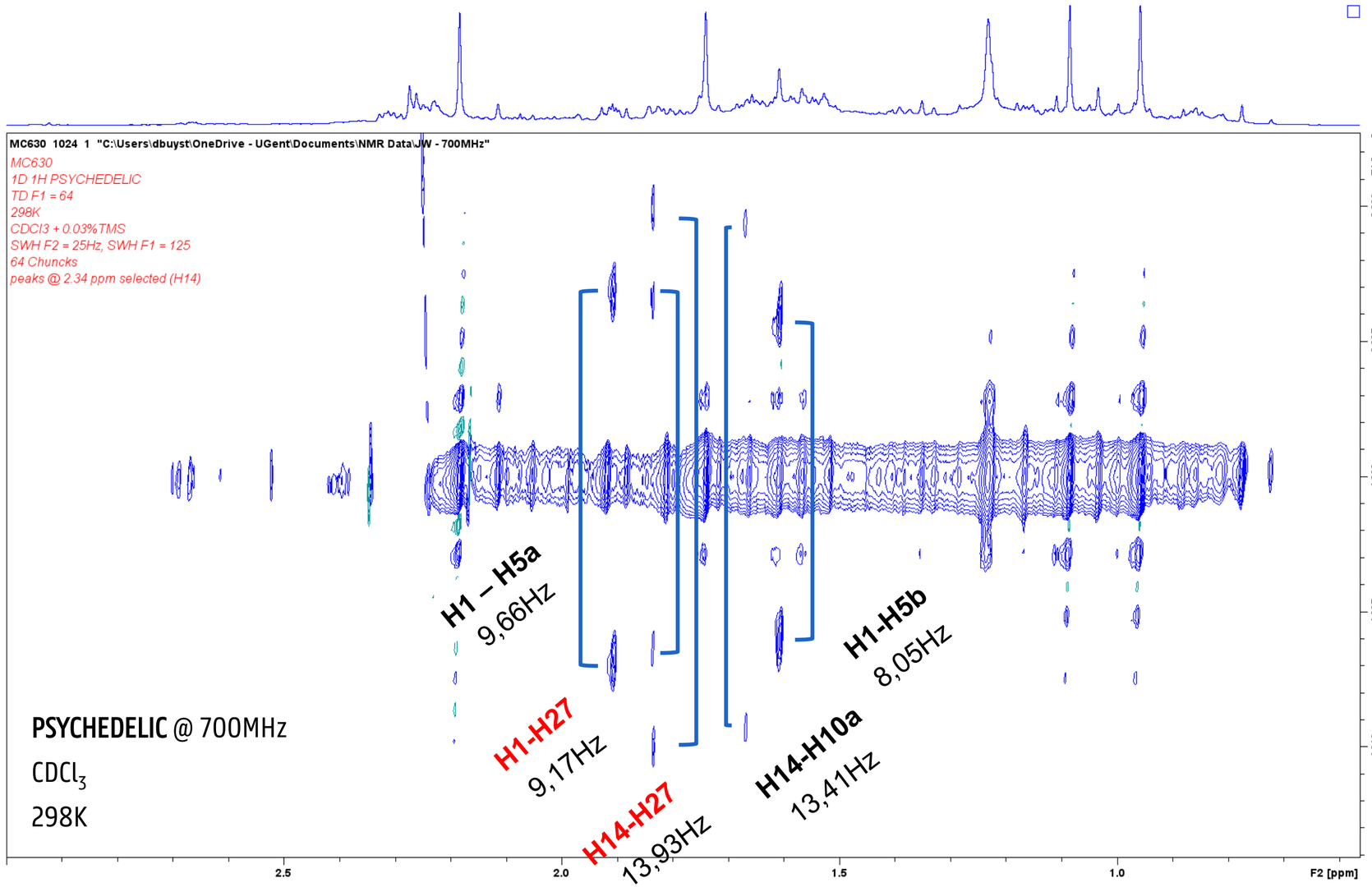
J. Winne

From 2D to 3D NMR structure elucidation in terpenes

Step 2 – Determine scalar couplings and resulting stereochemistry

Chemical Formula: $C_{23}H_{32}O_2$





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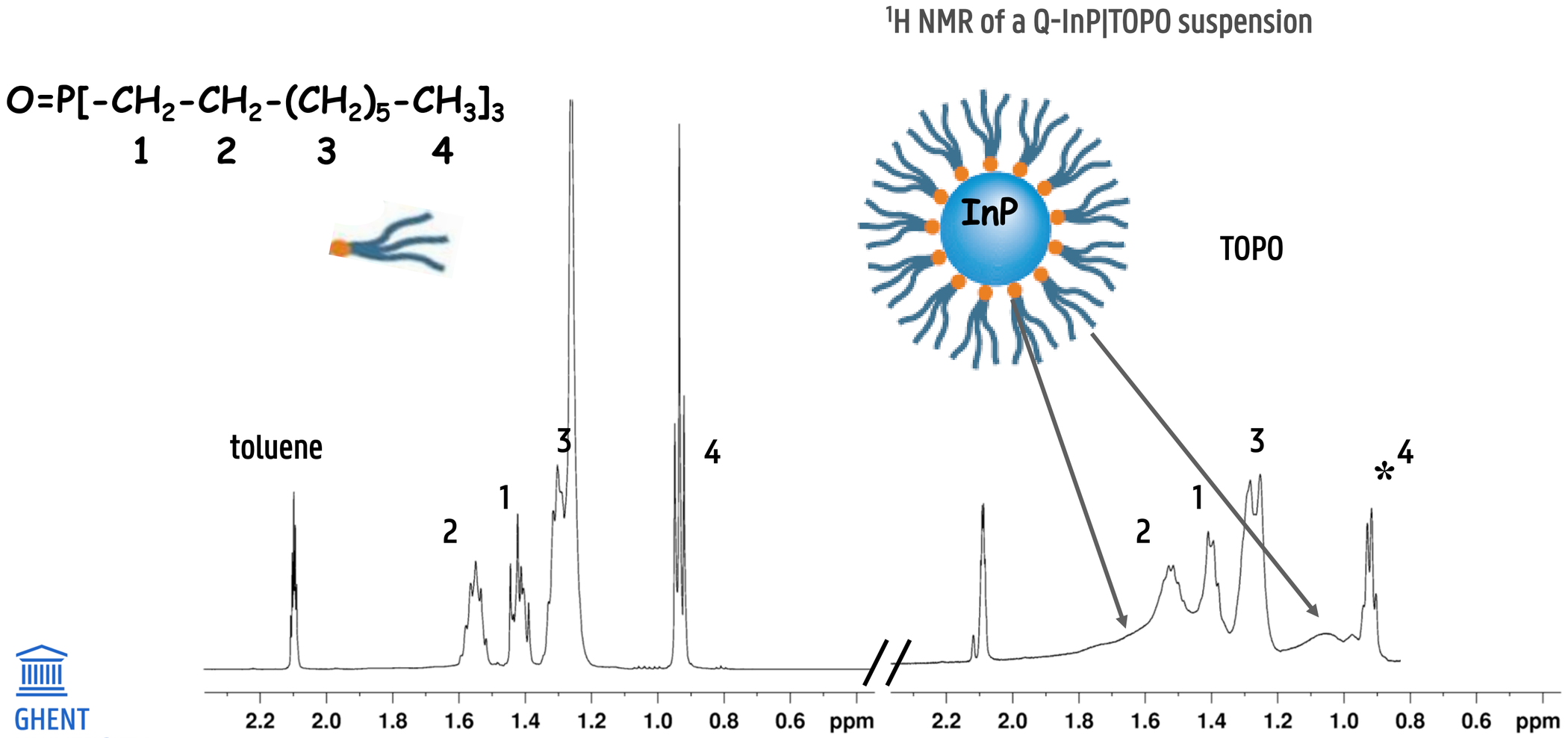
EXPERTISE

Diffusion NMR for “free” vs “bound” ligand discrimination

Z. Hens & I. Moreels

Solution stabilized quantomdots for solar cell applications



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EXPERTISE

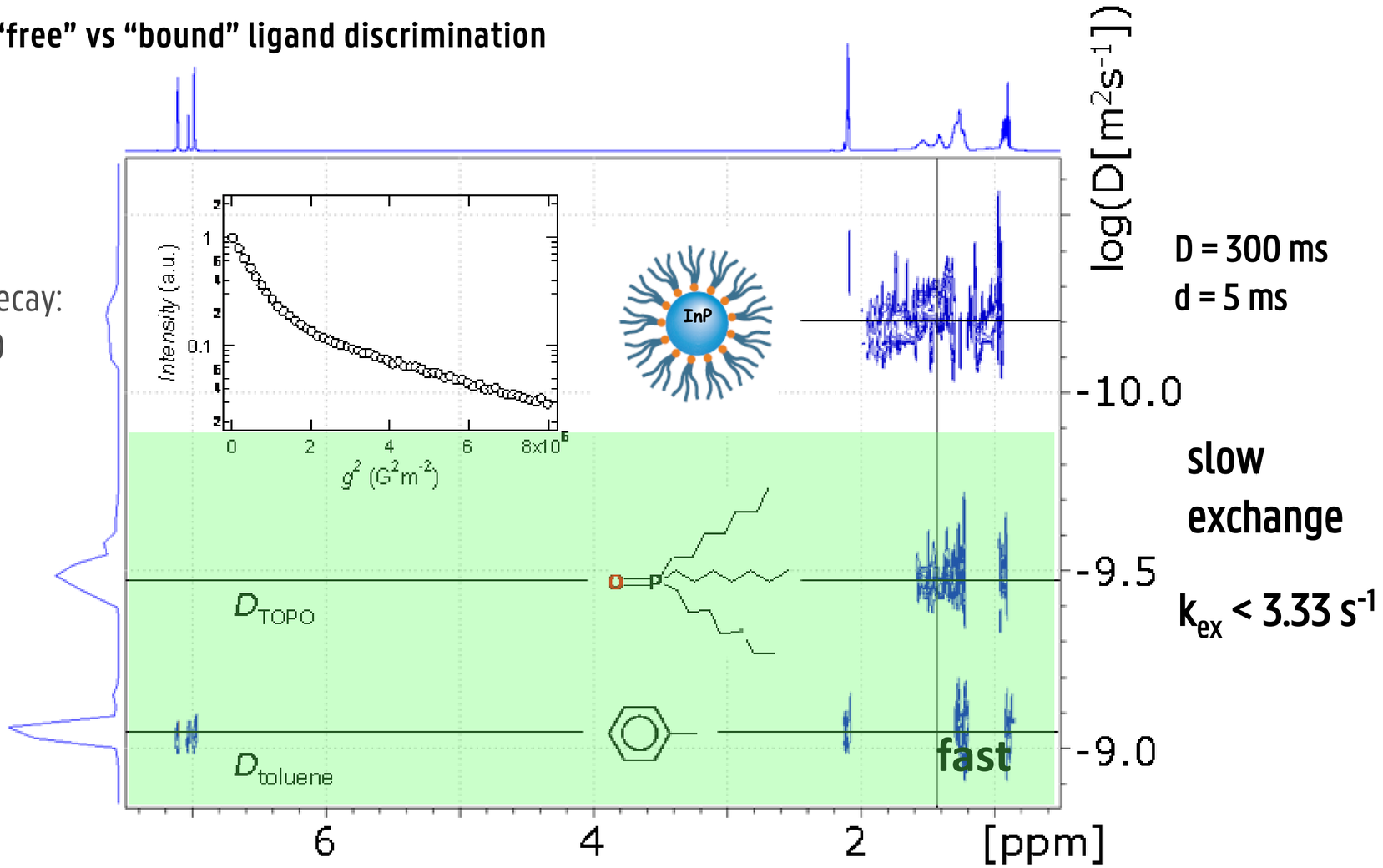
2D Diffusion Ordered SpectroscopY or **DOSY**

Diffusion NMR for “free” vs “bound” ligand discrimination

slow

Two componont decay:
free + bound TOPO

fast



- Sharp resonances are shown to be attributed to free TOPO ligand, while the broad TOPO resonances are attributed to TOPO bonded to the nanoparticle surface
- Bonded and non-bonded TOPO are separately visible in DOSY spectrum (biexponential decay). The kinetics of exchange between both forms is thus much slower than the diffusion NMR experiment !

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NMR TOOLBOX

Bruker 700MHz Avance NEO-X

"Hera" – INMRF infrastructure

Liquid state spectrometer

Triple channel probes

- 5mm TCI Prodigy N_2 cryoprobe
- 1mm TXI (^{1}H; ^{13}C; ^{15}N)

Topspin 5.0

Sample Case sample changer (60 samples)

Bruker 500MHz Avance III

"Hercules"

Liquid/Solid state & soft matter spectrometer

Dual channel probes

4mm dual channel HR-MAS iProbe - (^{1}H; ^{13}C)

4mm dual channel CPMAS iProbe - (^{1}H; BB)

5mm BB (^{1}H; BB)

MAS III pneumatic unit

Topspin 3.7

4mm Rotor Sample changer (10 samples)

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NMR TOOLBOX

Bruker 500MHz Avance III HD

"Osiris"

Liquid state spectrometer

Dual & Triple channel probes

- 5mm BBFO (1H; BB-^{19}F)
- 5mm TXI (^{1}H; ^{13}C; ^{31}P)
- 5mm TXO (^{1}H; ^{13}C; ^{19}F)

Topspin 3.8

Sample Xpress sample changer

(60 samples)

Bruker 400MHz Avance NEO

"Atlas"

Liquid state spectrometer

Dual channel probe

5mm BBO (^{1}H; BB) Progidy N2 cryoprobe

Topspin 4.4 in ICONNMR

Sample Xpress sample changer

(60 samples)

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NMR TOOLBOX

Bruker 300MHZ Avance II

"Deimos"

Liquid state spectrometer

Dual channel probe

5mm BBO (^{1}H; BB)

Topspin 3.2 in ICONNMR

Sample Xpress sample changer

(60 samples)

GHENT UNIVERSITY

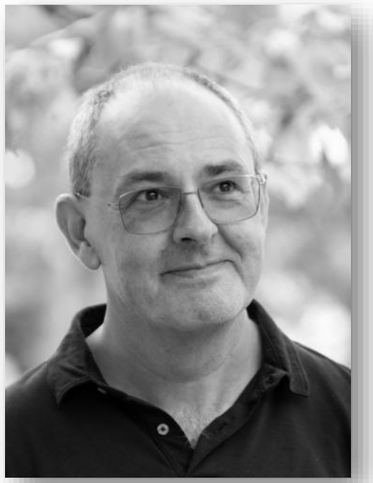
TEAM

A triad of experts at your disposal

Dieter Buyst, PhD

Core Facility Manager (CFM)

- Point of Contact
- Custom Analysis & Measurements
- Management & Organization
- ICT infrastructure
- Training



Prof. José Martins

NMRSTR Group leader

NMR EC ZAP responsible

- Scientific support

Emile Ottoy, PhD

Core Facility Assistant (CFA)

- Point of Contact
- Open Access & Routine Measurements
- Custom Analysis & measurements
- Training for Open Access

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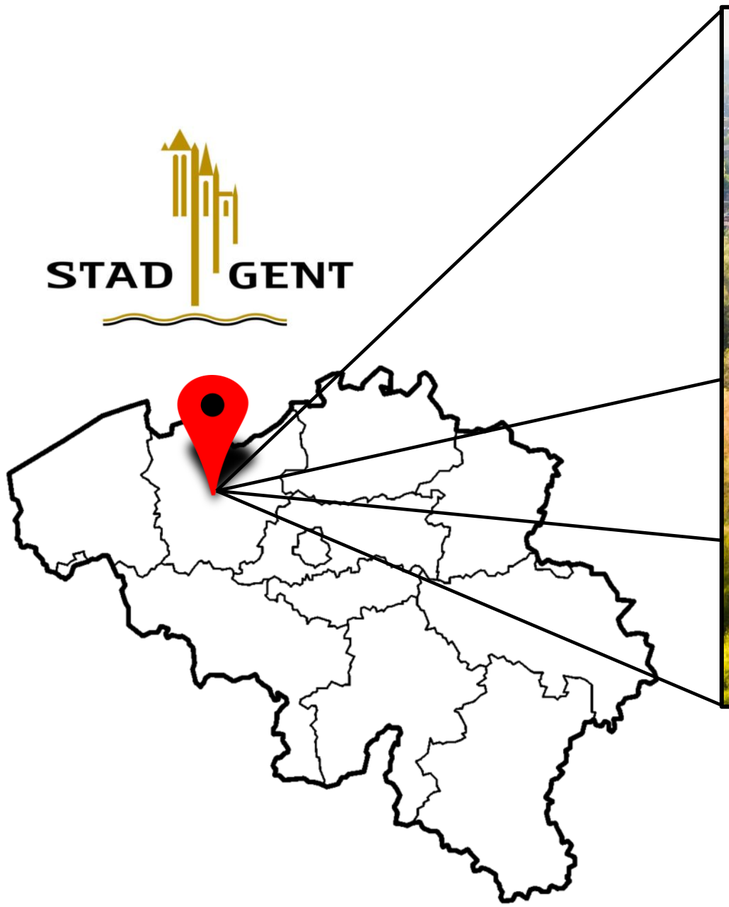
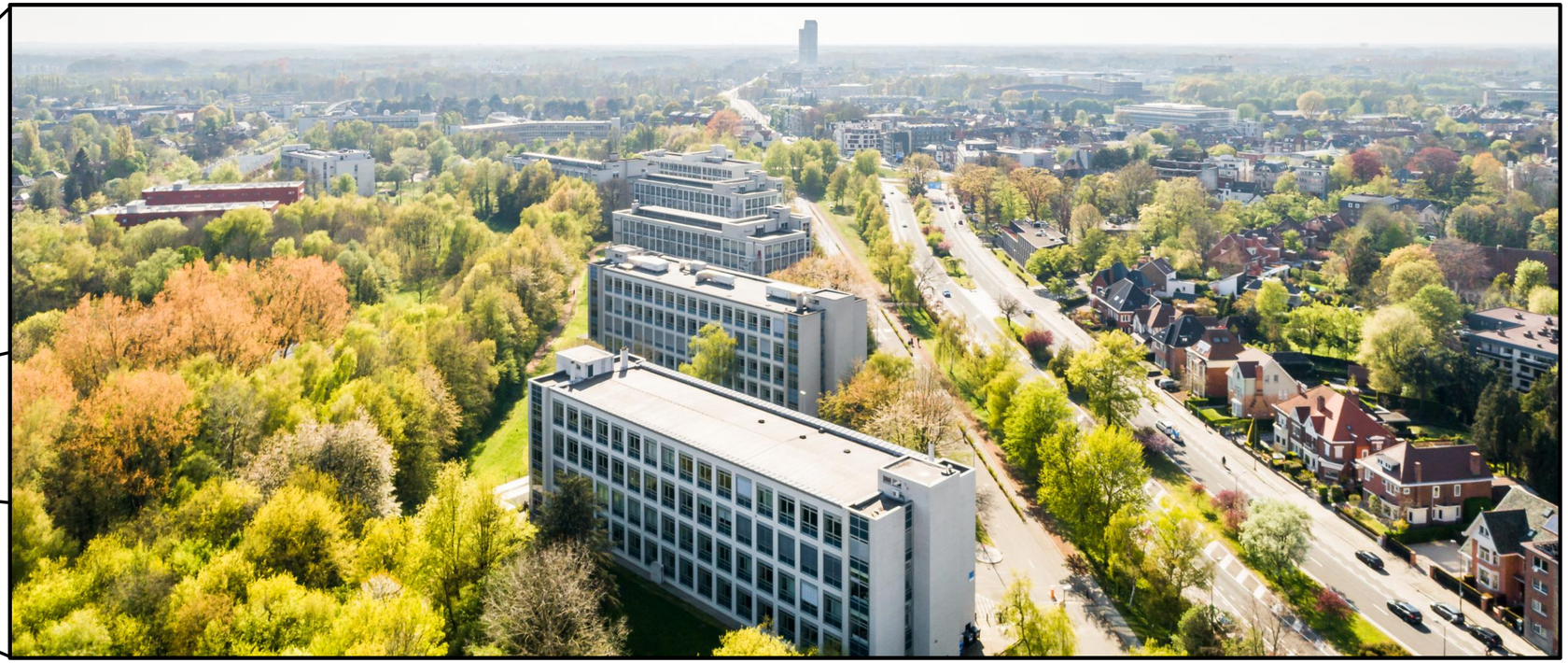
LOCATION





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