



WP2 RESEARCH INFRASTRUCTURES

Task 2.3 Unlocking the collaborative potential of EDUC Research Infrastructures

Advancing healthcare using the Research Infrastructure
CeSAR of UniCa: Nutrition during the first two years of
life

Cagliari, 23th and 24th November 2023



This project has received funding
from the European Union's Horizon
2020 research and innovation
programme under grant agreement
No 101017526

NMR FACILITY

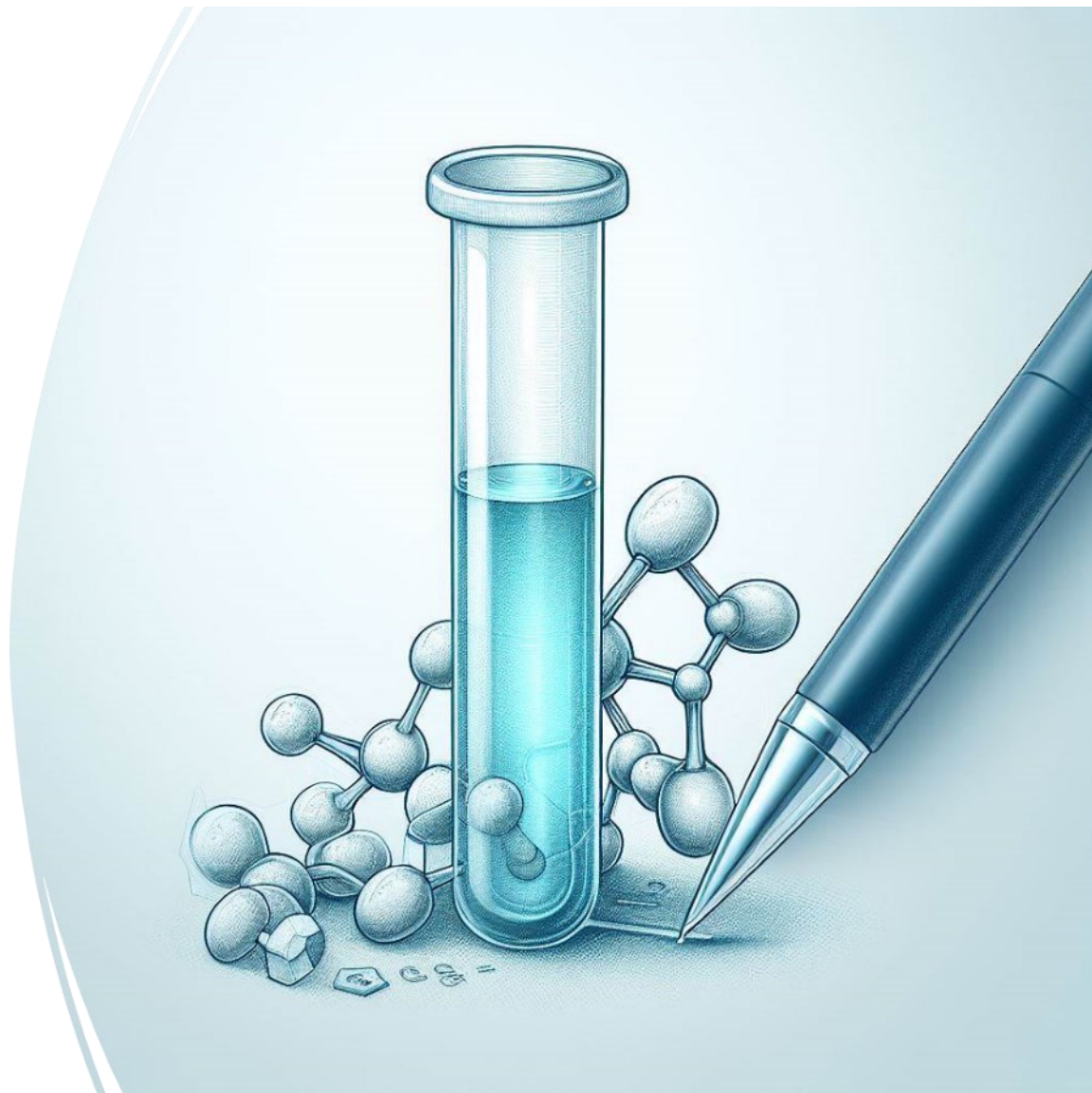
Nuclear Magnetic Resonance Spectroscopy



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OUTLINE

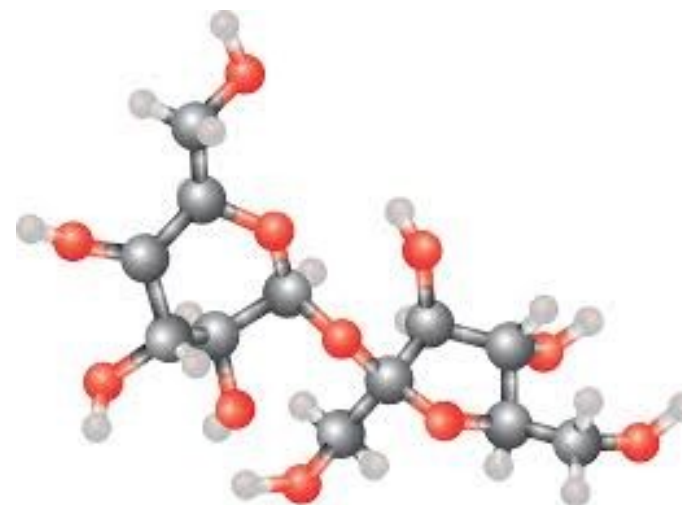
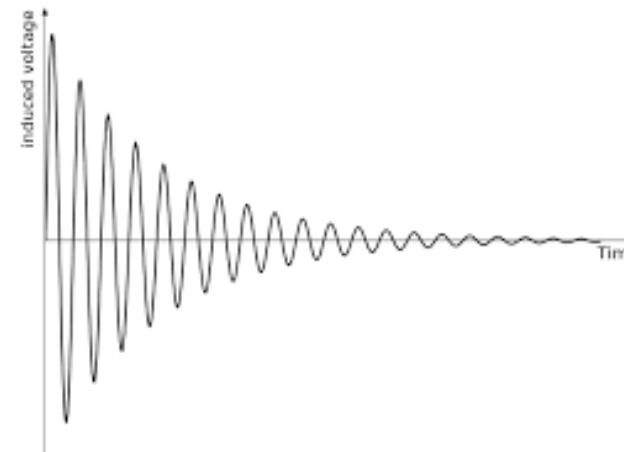
- NMR Laboratory Equipment
- Uses of NMR@UniCA
- Some advanced applications in biological samples and material science



WHAT CAN WE DO WITH NMR?

NMR is used to reveal the structure, identity, concentration, and behavior of molecules in liquid or solid samples:

- characterize molecular structures
- monitor the composition of mixtures
- study molecular interactions
- quantify known and unknown components



CESAR NMR FACILITY

The NMR facility is critical to many research programs and supports several research groups:

Chemistry

Life science

Biomedical science

Physics



EQUIPMENT

Bruker Avance III HD 600 MHz

We offer a state-of-art 600 MHz NMR spectrometer configured to meet the needs of our researchers with a high level of automation



Bruker Avance III HD 600 MHz

EQUIPMENT

Bruker Avance III HD 600 MHz

Bruker Avance III HD 600 MHz

- Superconducting magnet (14.1 Tesla), shielded
- 5 mm Smartprobe BBFO for liquid state (multinuclear)
- 4 mm HR-MAS probe for heterogeneous materials (^1H - ^{13}C)
- 2.5 mm CP-MAS probe for solid samples (multinuclear)



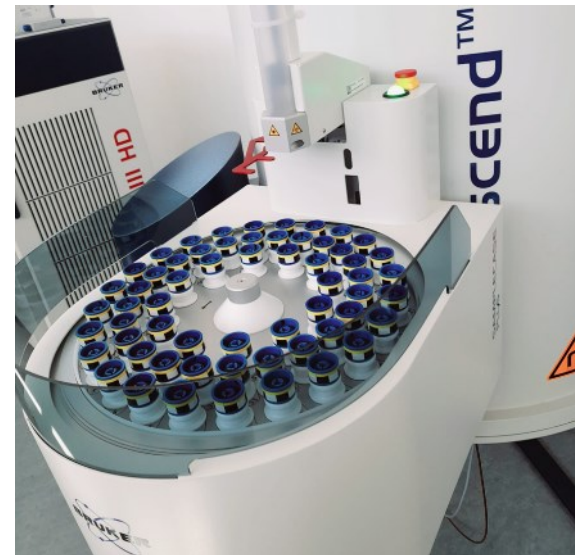
Bruker Avance III HD 600 MHz

EQUIPMENT

Bruker Avance III HD 600 MHz

The NMR spectrometer is equipped also with:

- 60-seat autosampler for the liquid state
- Variable temperature unit
- Magnetic field gradients
- Automated sample tuning, lock, and shimming

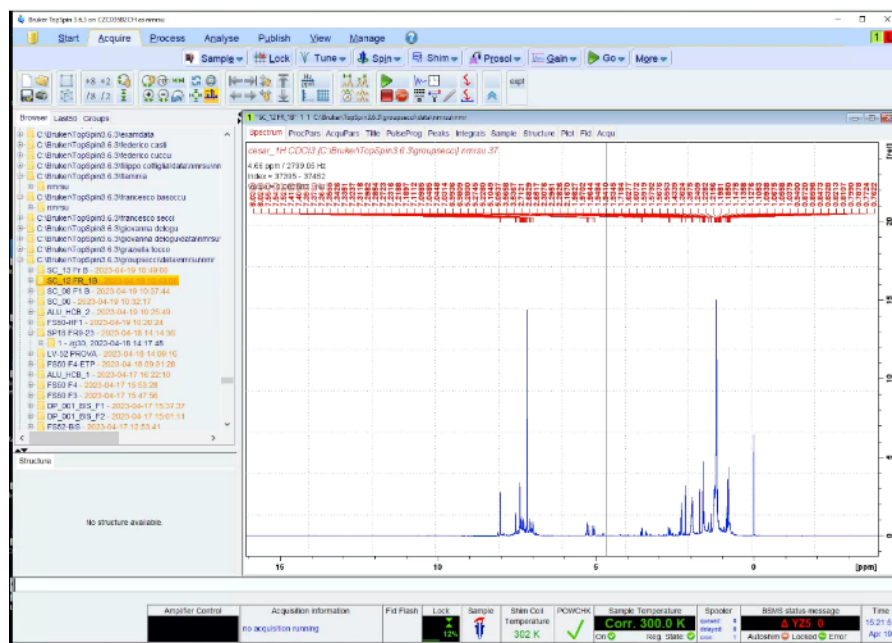


Bruker Avance III HD 600 MHz

EQUIPMENT

Bruker Avance III HD 600 MHz

Topspin 3.6 Bruker software for routine and advanced NMR acquisition and analysis



The screenshot displays the IconNMR automation software interface. The main window shows a list of experiments with columns for Name, No., Solvent, Equipment, Pa, Per, Title/Orig, Time, User, and Start Time. The experiments are listed as follows:

Exp. No.	Name	No.	Solvent	Equipment	Pa	Per	Title/Orig	Time	User	Start Time
1	ETH-3-methylster	31	ETH-3	N-PROTON	*		ETH-3-methylster		mnru	Set Start Time
1	ETH-3-methylster	31	ETH-3	CH2QCEGPPH	*		ETH-3-methylster		mnru	Set Start Time
2	Cytosine	31	ETH-3	N-PROTON	*		Cytosine		mnru	Set Start Time
3	Serotonin	21	ETH-3	N-PROTON	*		Serotonin		mnru	Set Start Time
3	Serotonin	21	ETH-3	CH2QCEGPPH	*		Serotonin		mnru	Set Start Time
4	Styehrine	41	ETH-3	N-PROTON	*		Styehrine		mnru	Set Start Time
4	Styehrine	41	ETH-3	CH2QCEGPPH	*		Styehrine		mnru	Set Start Time
4	Styehrine	42	ETH-3	CH2QCEGPPH	*		Styehrine		mnru	Set Start Time

IconNMR automation software

ACCESS TO NMR FACILITY

Full-service: internal users, externals (other Universities, Companies)

Self-service: internal trained users



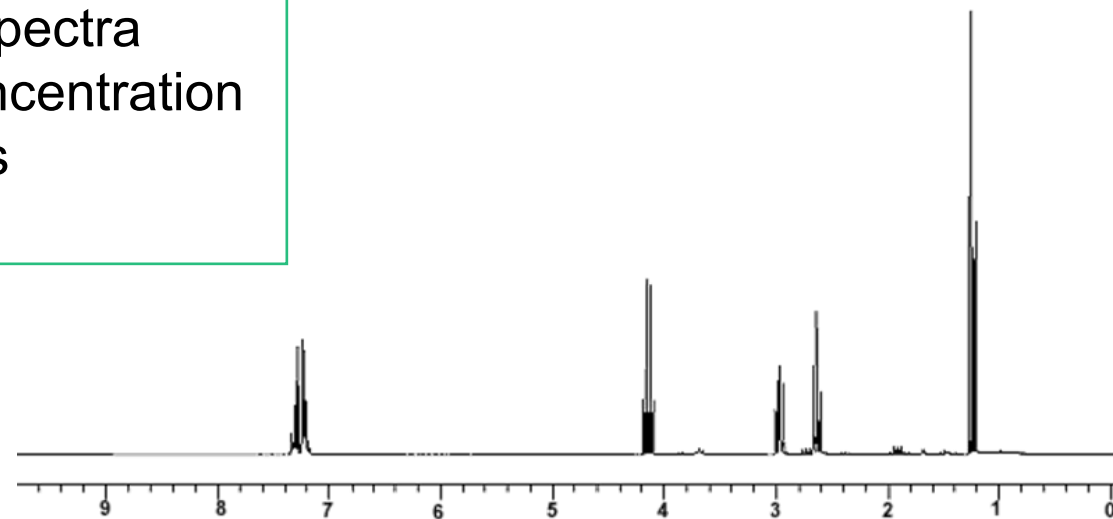
NMR ROUTINE WORK @CESAR

Typical investigations include:

molecular structure identification -> 1D/2D spectra
reaction kinetics -> variable temperature and concentration
inter-molecular interactions -> titrations

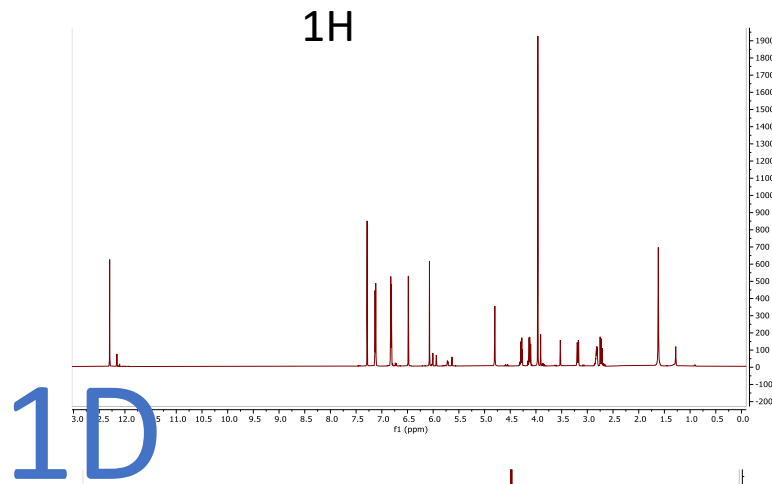
We routinely work with:

- small molecules (organic / inorganic)
- polymers
- materials
- biomolecules

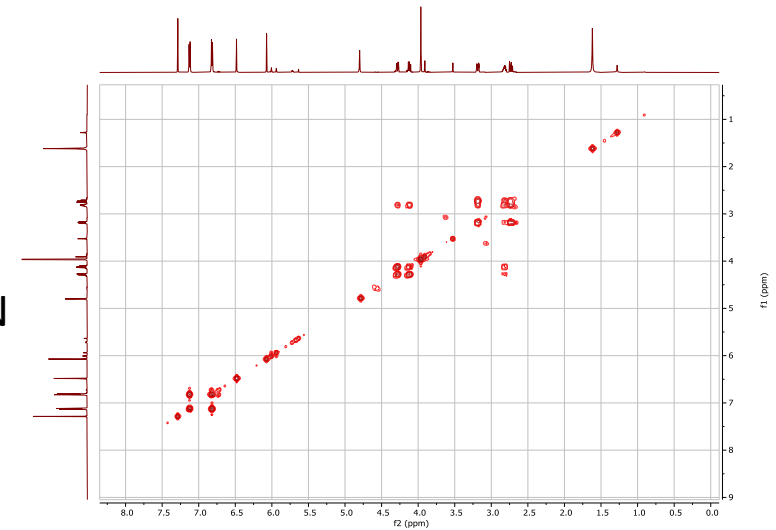


NMR ROUTINE WORK @CESAR

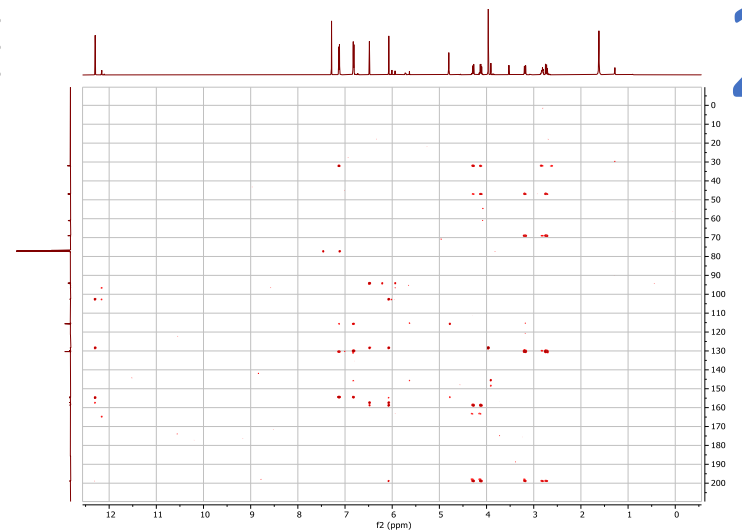
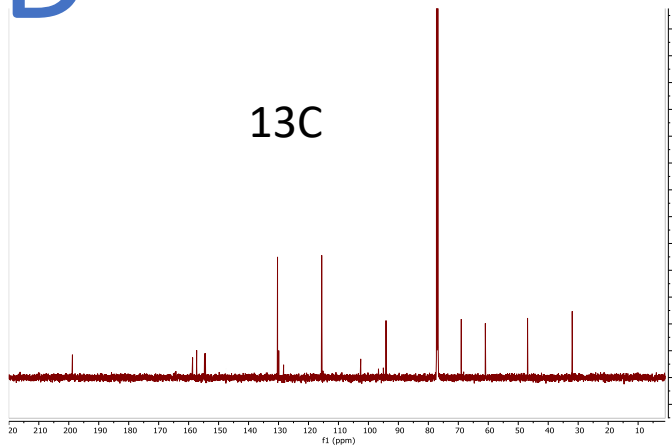
MOLECULAR STRUCTURE IDENTIFICATION



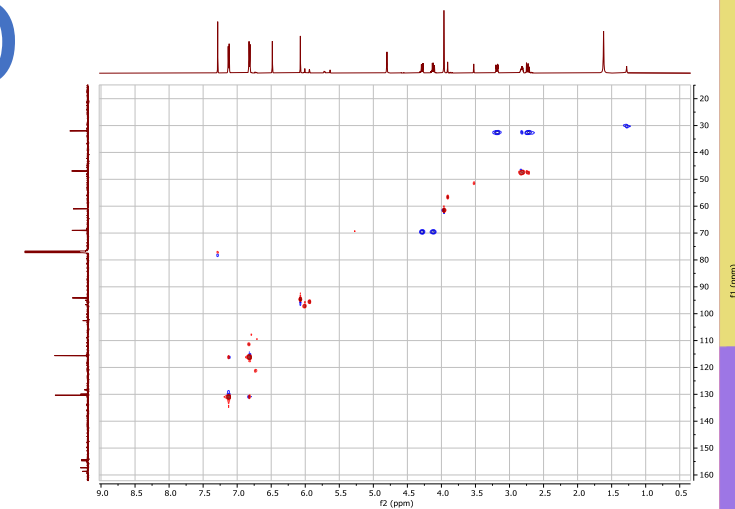
1H-1H DIRECT CORRELATION



2D



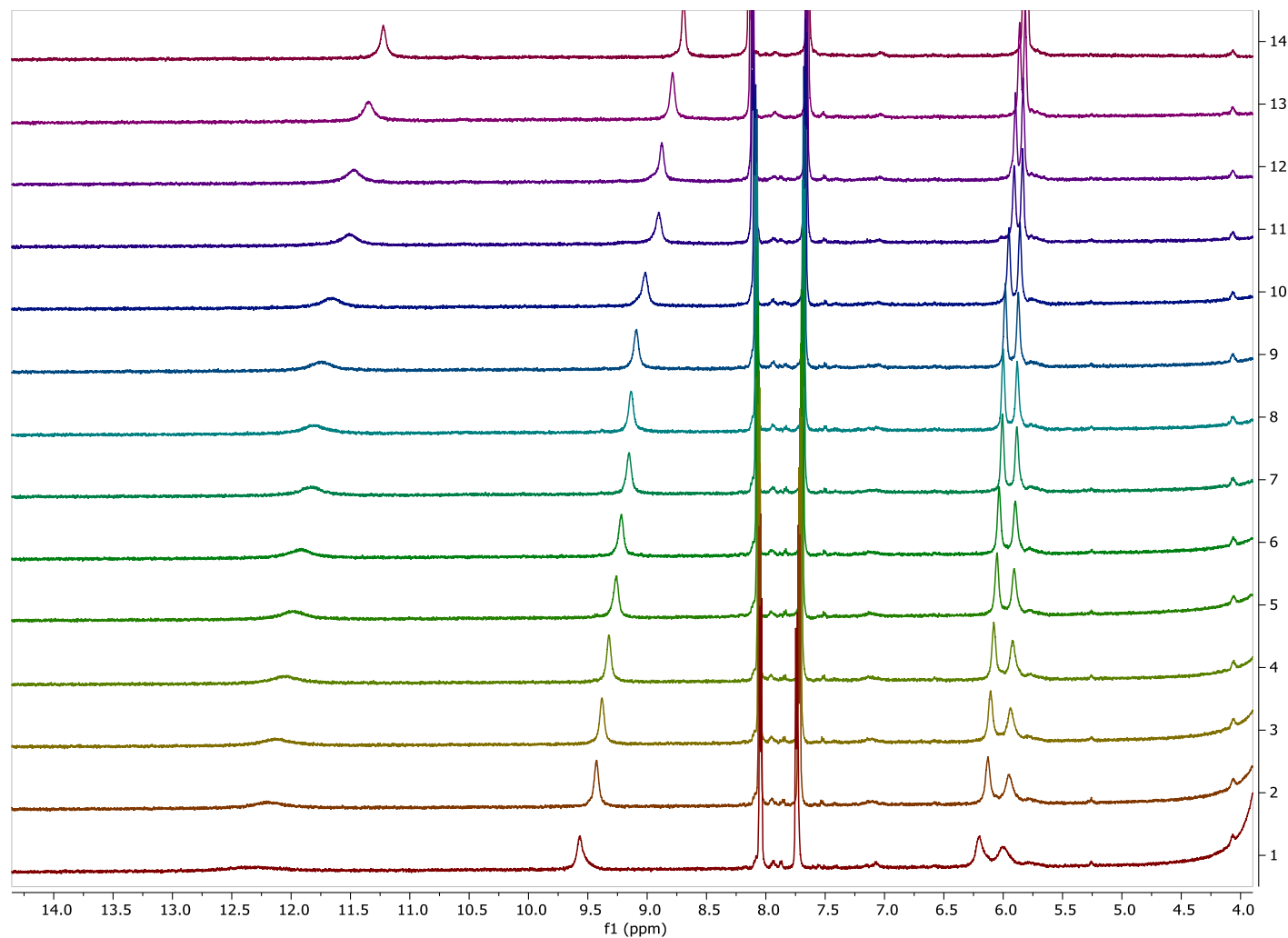
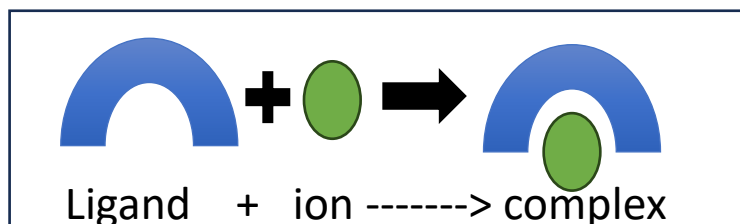
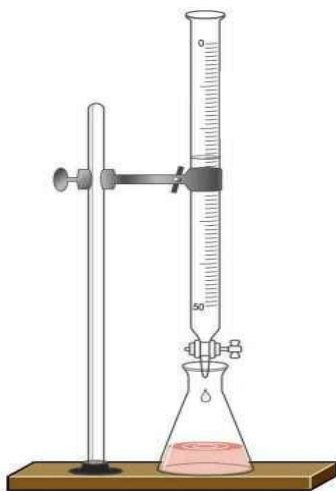
1H-13C LONG RANGE CORRELATION



1H-13C DIRECT CORRELATION

NMR ROUTINE WORK @CESAR

COMPLEX FORMATION - NMR TITRATIONS



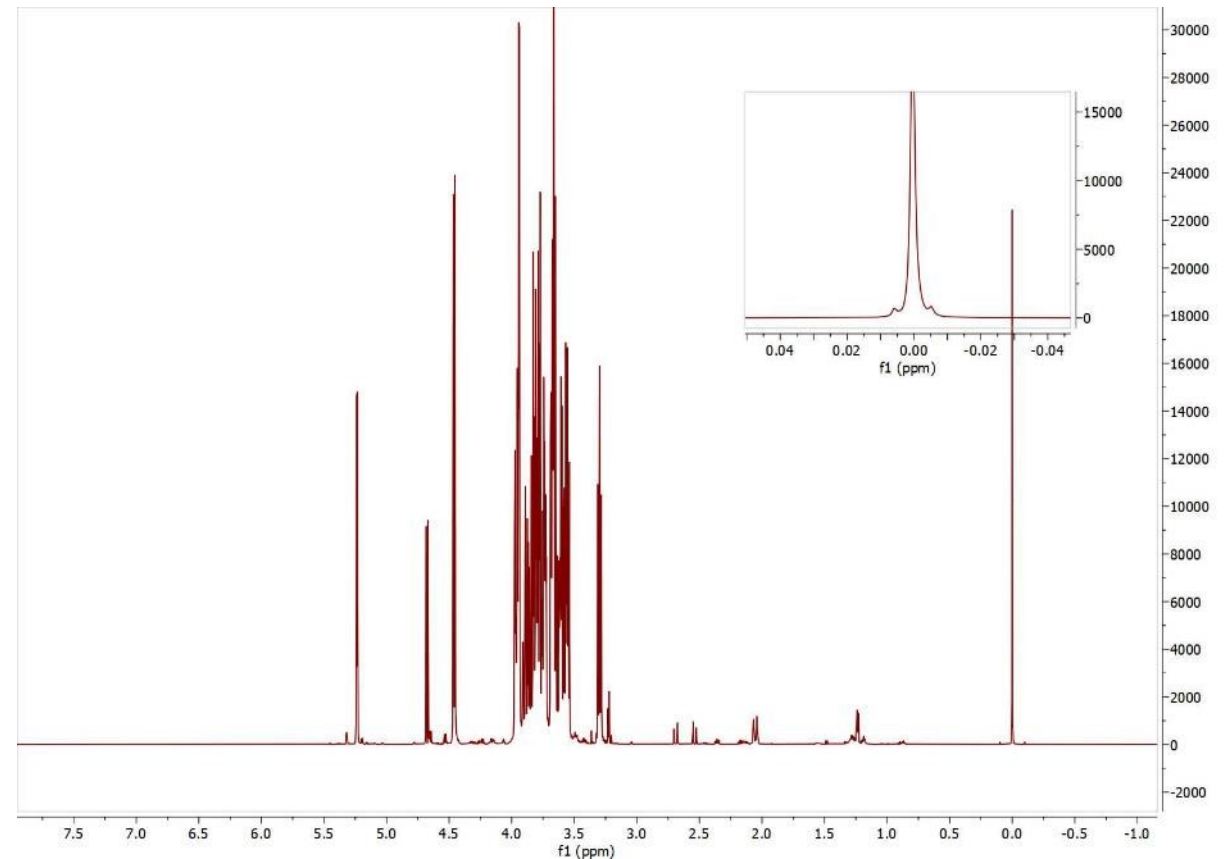
METABOLOMICS STUDIES

Quantitative and simultaneous analysis of large numbers of metabolites in biological systems

Typical investigation include:

High resolution proton spectra for identification and quantification of metabolites

- Urine
- Feces
- Milk



ADVANCED NMR APPLICATIONS

Focus on complex samples

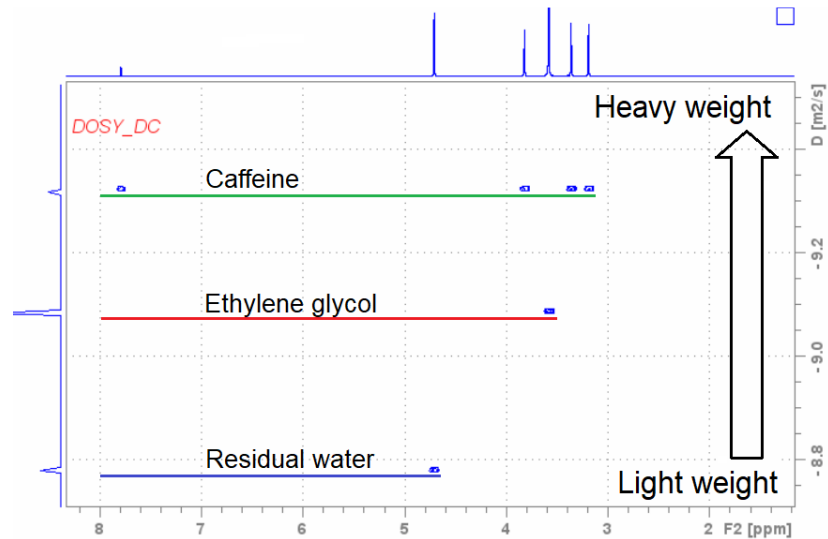
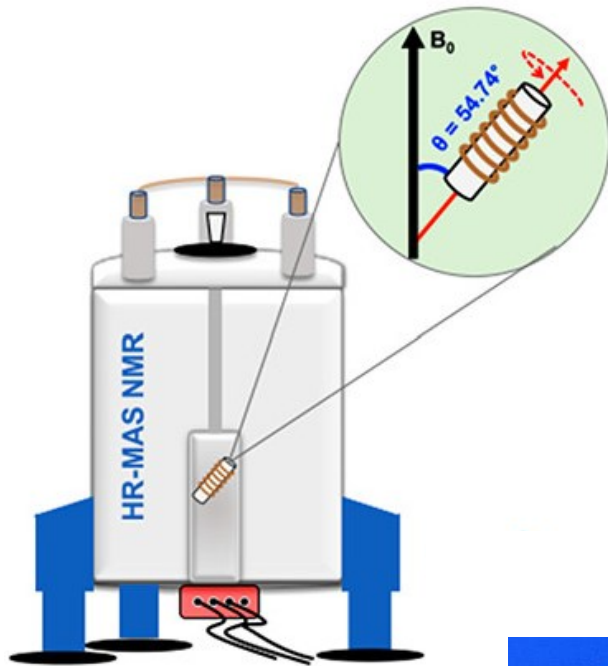


Figure: 1H NMR DOSY experiment

Diffusion (DOSY): measure of self-diffusion constants to obtain mass-resolved NMR spectra of mixtures

ADVANCED NMR APPLICATIONS



Magic Angle Spinning:

This mechanical sample spinning removes most of the anisotropic interactions which otherwise would cause strong line broadening and result in significant signal overlap.

CP-MAS: research on material science (fine powders)

HR-MAS: semisolid samples (e.g. intact tissues, gels, vegetables, foods)



MAS rotors

ADVANCED NMR APPLICATIONS

2017/18 September 20, 2018 18:00:00

Multinuclear NMR:

multinuclear probe obtains not just ^1H and ^{13}C spectra of organic compounds, but greatly extend the range of nuclei by observing ^7Li , ^{11}B , ^{15}N , ^{19}F , ^{29}Si , ^{31}P , ^{113}Cd , ^{119}Sn , ^{195}Pt , ^{199}Hg , and other nuclei.

Thanks for your attention