ADVANCED NMR TECHNIQUES IN SOLUTION AND SOLID-STATE

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PRE-REQUISITES : Basic knowledge of NMR Spectroscopy and Analysis of Spectra

INTENDED AUDIENCE : Postgraduates and Research Students

INDUSTRIES APPLICABLE TO : All pharma industries which have NMR facilities

COURSE OUTLINE :

The course provides conceptual understanding of NMR spin physics, J couplings, multiplicity patterns, analysis of NMR spectra of 1H, 13C and couple of other nuclei, Fourier Transformation, theorems of Fourier Transformation. Pople Notation, wave functions, Hamiltonian, quantum mechanical analysis of 2 coupled spin systems, pulse phase, receiver phase, phase cycling, evolution of chemical shifts and couplings under the influence of different pulses. Coupled and decoupled INEPT and DEPT sequences, coherence pathway selection, pulse field gradients, coherence path way selection using PFGs for homo and heteronuclear cases, selective pulse, purge pulse. Relaxation processes, measurement of T1 and T2, homo and heteronuclear decoupling, conceptual understanding of NOE. Product operators and their use in understanding of free precession, evolution of chemical shifts and couplings. Two dimensional NMR, Analysis of 2D COSY, TOCSY, HSQC, HMBC spectra. Multiple quantum excitation and detection, spin system filtering, measurement of relative signs of couplings. Fundamentals of solid state NMR, magic angle spinning, cross polarization, high speed spinning, 2D experiments in solids.

ABOUT INSTRUCTOR :

Prof. N. Suryaprakash is currently a CSIR Emeritus Scientist at the Solid State and Structural Chemistry Unit, Indian Institute of Science. Previously he held various positions at IISc including Tatachem Chair Professor, before superannuation as a Professor and Chairman of NMR Research Centre. His academic career has proven to be nothing but exceptional in all aspects and his research achievements have received worldwide recognition, appearing in more than 155 publications in peer-reviewed journals of National and International repute, which include several invited book chapters. His research work is focused on diverse wings of NMR spectroscopy, with a major focus on the manipulation of spin dynamics to design novel experimental techniques to combat inherent challenges encountered in NMR spectroscopy, viz., weak sensitivity, poor resolution, and spectral crowding. He has also designed methods for the rapid acquisition of data, discerning degenerate transitions, and facile extraction of spectral parameters. His designed single and multiple quantum methodologies significantly transformed the chiral analysis of molecules in weakly aligned liquid crystalline media using 1H NMR, a field that remained unchallenged for decades. His research work led to the discovery of three novel water-compatible weak chiral aligning media that surpassed many inherent limitations of enantiomeric differentiation. His group has introduced a number of novel chiral auxiliaries, for enantiodiscrimination of molecules with diverse functionalities, accurate determination of enantiomeric contents, and also for the assignment of their absolute configurations, an area of very high significance in pharmaceutical industries. His innovative NMR approaches for the investigation of hydrogen bonds involving organic fluorine pave the way for the artificial architectural design of macromolecules. His seminal contributions have catapulted the research activities in NMR spectroscopy, structural chemistry, chiral chemistry, and in the area of weak molecular interactions.

COURSE PLAN :

Week 1: NMR spin physics, concepts, chemical shifts
Week 2: J couplings, splitting patterns, analysis of 1H, 13C and the spectra of other heteronuclei
Week 3: Fourier series, transformation, FT of different functions, Theorems of FT, Use of FT theorems in NMR, Pople nomenclature, Quantum mechanical analysis of spectra of two coupled spins
Week 4: Wave functions, product functions of weakly coupled spin systems, othronormalization, Hamiltonian for uncoupled spins, coupled spins, eigen values and eigen functions, strongly coupled spin systems. RF pulse, pulse phase, precession of magnetization under 90, 180 and 360 pulses and their phases, absorption and dispersive signals, FT of cosine and sine functions, phase cycling
Week 5: Polarization transfer, SPT and SPI for homo and heteronuclear spin systems, INEPT sequence, coupled and decoupled INEPT, DEPT sequences, coherence transfer pathways, selection of particular pathway in different sequences, phase cycling
Week 6: Phases of pulses and receivers, multiple pulse sequences, PFGs, and selection of coherence transfer pathways using gradients, in homo and heteronuclear cases, use of gradients for removal of pulse artifacts, purge pulse, etc. selective inversion, relaxation concepts

Week 7: T1 relaxation phenomenon and its deeper concepts, correlation times, relaxation processes, measurement of T1, T¬2 relaxation, spin echo experiment

Week 8: Decoupling and NOE, concepts and the factors governing NOE. Product operators, concept of product operators, product operators in understanding rotations. Hamiltonians and evolution of couplings, chemical shifts Hamiltonians and free precession

Week 9: Product Operator analysis of one pulse experiment, spin echoes in coupled and uncoupled spins, operators for two coupled spins, free precession Hamiltonian for two coupled spins, evolution of inphase and antiphase terms, heteronuclear spin echoes, coherence transfer, INEPT using product operators

Week 10: Concepts of different 2D experiments, general interpretation of 2D experiments, discussion on the COSY, limitations of COSY experiment, different COSY sequences, DQF COSY, TOCSY, coupled and decoupled HSQC, HMBC, INADEQUATE

Week 11: Multiple Quantum NMR, DQ and SQ transitions, and their detection. Higher quantum spectra in coupled spin systems, spin system filtering, spin state selection, measurement of relative signs of the couplings, Introduction to NMR spectra of solids

Week 12: Solid state NMR, MAS, spin space averaging, decoupling in solids, cross polarization, CRAMPS, side bands, VSMAS, CP at high speeds