Solid-state NMR studies of micelle-templated mesoporous solids

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Electronic Supplementary information (ESI)

Glossary

CP

Cross Polarization is a method used to enhance the signal of low natural abundance nuclei (rare nuclei).[1] The RF pulse sequence consists in transferring the magnetisation from abundant nuclei such as ^{1}H (but can be another nucleus) to the rare nuclei (for example ^{13}C, ^{29}Si, ^{15}N...).[2] The transfer occurs during the CP contact time and depends on dipolar couplings.

DAS

Dynamic Angle Spinning involves rotation of the sample about an axis that is tilted sequentially at two different angles during the experiment in order to remove second-order quadrupolar broadening by vanishing second and fourth rank Legendre polynomials.[3] It allows improving the resolution of half-integer quadrupolar nuclei.

DNP

The Dynamic Nuclear Polarisation experiment is used to enhance the nuclear spin polarisation of paramagnetic samples.[4] It consists in a polarisation transfer from the unpaired electrons (e.g. of nitroxide free radicals) to nuclear spins through microwave irradiation near the electron paramagnetic resonance frequency. This experiment necessitates dedicated apparatus that are currently being commercialised.

DOR

DOuble Rotation involves rotation of the sample simultaneously about two axis tilted at 54.74° and 30.56° relative to B_0, in order to remove second-order quadrupolar broadening by vanishing respectively second and fourth rank Legendre polynomials.[5,6]It allows improving the resolution of half-integer quadrupolar nuclei.

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DQ

Double Quantum transitions are used in coupled spin systems. These transitions can be promoted in RF pulse sequences to identify or select pairs of nuclei that are bonded (indirect spin-spin *J* couplings) or spatially close (dipolar *D* couplings).

EXSY

EXchange SpectroscopY is a 2D homonuclear correlation experiment probing chemical exchange between nuclei.[7] The RF pulse sequence is the same as NOESY and can also be used for spin diffusion studies.

HETCOR

HETeronuclear CORrelation experiments are 2D NMR experiments correlating the chemical shifts of two types of nuclei (e.g. ^{1}H-^{13}C correlation) through a heteronuclear transfer of the magnetization. Usually, the transfer is made using an INEPT block in liquid-state NMR, and using a CP block in solid-state NMR. The resulting spectra inform about bond connectivity or spatial proximity, respectively.

INADEQUATE

Incredible Natural Abundance Double Quantum Transfer Experiment.[8] This experiment allows correlating signals through double quantum transitions between homonuclear *J* coupled states.

INEPT

Insensitive Nuclei Enhanced by Polarization Transfer experiment.[9] Heteronuclear correlation based on a polarisation transfer using heteronuclear *J* couplings.

MAS

Magic Angle Spinning. The sample is spinning at a sufficiently high frequency around an axis inclined at 54.74° (Magic Angle) with respect to the direction of the static magnetic field.[10,11] The MAS technique averages the anisotropic part of interactions described by a second-rank Legendre polynomial. It removes the line broadening leading to high resolution solid state NMR spectra. MAS may be combined with other techniques like CP, decoupling...

MQMAS

The Multiple-Quantum Magic Angle Spinning is a 2D NMR experiment used for obtaining well resolved spectra of half integer spin quadrupolar nuclei. By exploiting the effect of the quadrupolar interaction onto the multiple quantum transitions, it correlates an isotropic spectrum to the anisotropic central transition MAS spectrum.[12] This sequence has become very popular in solid-state NMR of quadrupolar nuclei, and there are now several improved related versions.

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NOESY

Nuclear Overhauser Effect SpectroscopY is a liquid-state 2D homonuclear correlation experiment probing spatial proximity thanks to cross-relaxation or spin diffusion processes.[13]

REAPDOR

The Rotational Echo Adiabatic Passage DOuble Resonance experiment is a multipulse sequence designed to estimate the distance between two different nuclei belonging to an isolated spin pair. It is based on the recoupling of the heteronuclear dipole interactions between $I = ½$ spin nuclei and $I > 1$ quadrupolar spin nuclei.[14]

REDOR

The Rotational Echo DOuble Resonance experiment is designed to estimate the distance between two different nuclei belonging to an isolated spin pair. It is based on the recoupling of the heteronuclear dipolar interaction in spin-echo based RF pulse sequences.[15]

QCPMG

Quadrupolar Carr-Purcell-Meiboom-Gill sequence. Name given to the CPMG sequence when applied to obtain spectra of quadrupolar spin nuclei, either $I = 1$[16] or $I = n/2$.[17] The standard CPMG spin echo trains are used to measure transverse relaxation times T_2.[18]

Spin-echo

If otherwise mentioned, the standard spin-echo experiment consists in a $\pi/2$ pulse followed by a π pulse after a delay τ. The NMR signal of ½ spin nuclei (or selective central transition for half integer quadrupolar spin nuclei) is refocused at 2τ.[19]

TRAPDOR

TRAnsfer of Population in DOuble Resonance is a 1D experiment that probes the spatial proximities between $I = ½$ spin nuclei and $I = n/2$ quadrupolar spin nuclei. It is based on a spin-echo performed on spin ½ combined with continuous irradiation applied alternatively on the quadrupolar spin $n/2$.[20]

T_1

Spin-lattice relaxation time or longitudinal relaxation time. Time required for the longitudinal component of the magnetization (along z axis) to return to equilibrium.

$T_{1\rho}$

Spin lattice relaxation time in the rotating frame.

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T_2

Spin-spin relaxation time or transverse relaxation time. Time required for the transverse component of the magnetization (in the x-y plane) to return to equilibrium.

WISE

The WIdeline Separation Experiment aims at correlating proton peaks with different linewidths to ^{13}C resolved peaks, thus probing the local mobility. It consists in a 2D CP-based HETCOR pulse sequence run at null or moderate MAS rates.[21]

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