JEOL

Scientific / Metrology Instruments
Solid State NMR

Solutions for Innovation

Solid State **NMR**

JEOL Ltd.

New ideas from JEOL

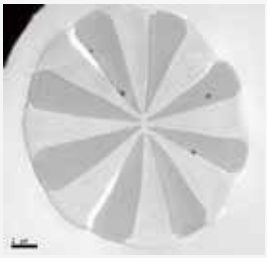
JEOL offers a full range of Magic-Angle-Spinning (MAS) probes and tools matched to a wide variety of solid-state NMR applications. JEOL MAS probes feature sample tube diameters to match the user sample and sensitivity needs. JEOL narrow bore MAS probes offer improved stability for high-speed spinning or for very large volumes.

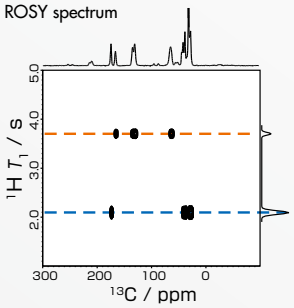
The JNM-ECZR and JNM-ECZL Series NMR Spectrometer automatically updates the relevant spectrometer settings for all NMR probes for fast and easy switching between solids and liquids NMR operation.

	Features and Applications	Max Speed	Guaranteed Speed	Sample Volume
8 mm	The large volume allows high-sensitivity NMR measurements. This size is best suited for measurements at moderate spinning speeds–for example ^{29}Si of synthetic minerals.	8 kHz	7.5 kHz	616 µl
4 mm	This 4 mm rotor provides a balanced combination of sample volume and spinning speed ideal for routine ^{13}C-CPMAS, and other measurements at 400 MHz.	18 kHz	17 kHz	69 µl
3.2 mm	The combination of sample volume and increased spinning speed makes the 3.2 mm rotor ideal for NMR measurements at 400 MHz and above.	22 kHz	21 kHz	49 µl
2 mm	This rotor is optimum for CPMAS at 700 MHz and above, small-volume samples, ^{19}F and ^{1}H high-resolution experiments.	40 kHz	38 kHz	17 µl
1 mm	Very-high sample spinning speeds up to 80 kHz attained by the 1 mm rotor are required for high resolution ^{1}H or multinuclear measurements of very small sample volumes (800 nl).	80 kHz	75 kHz	0.8 µl
0.75 mm	The 0.75 mm rotor provides the ultra-high sample spinning speeds to 100 kHz required for high resolution ^{1}H or multinuclear measurements of extremely small sample volumes (300 nl).	100 kHz	95 kHz	0.3 µl

Solid State NMR Applications:

Synthetic polymers





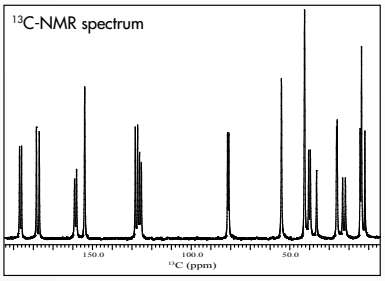
Middle size HXMAS probe p.3

Small size HXMAS probe p.4

ROSY p.10

New extended VT HXMAS probe p.6

Pharmaceuticals

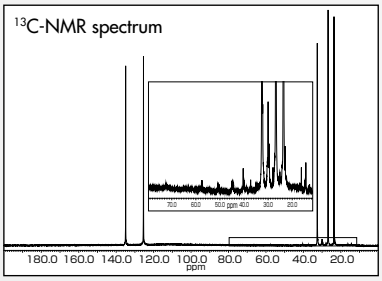


Middle size HXMAS probe p.3

Small size HXMAS probe p.4

ROSY p.10

Elastomers

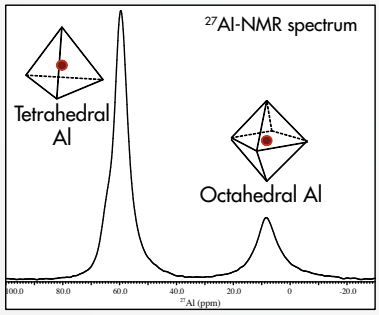


Large size HXMAS probe p.5

MAGIC SHIMMING p.9

FGMAS probe p.8

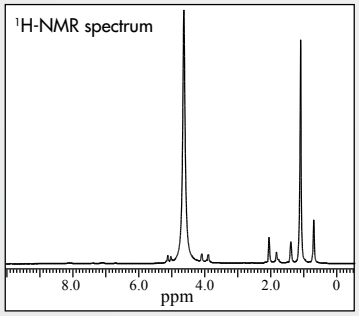
Inorganic materials



Large size HXMAS probe p.5

Cryocoil MAS probe p.7

Foods

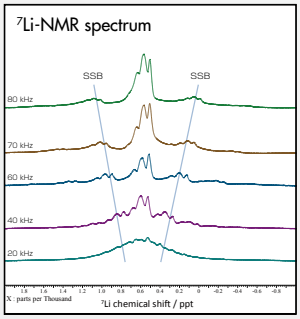


Large size HXMAS probe p.5

FGMAS probe p.8

MAGIC SHIMMING p.9

Batteries



Small size HXMAS probe p.4

Large size HXMAS probe p.5

Cryocoil MAS probe p.7

Middle Size HXMAS Probe (2 mm, 3.2 mm & 4 mm)

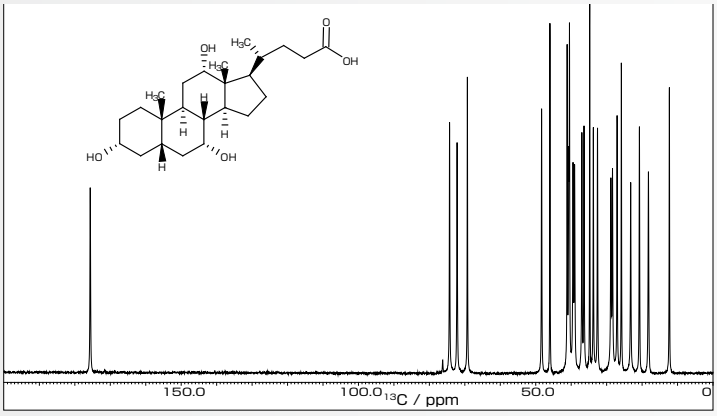
HXMAS Probes to Match Your Application

CPMAS is the most widely used solid-state NMR experiment. The 4 mm, 3.2 mm and 2 mm HXMAS probes are ideally suited for these measurements. Probes with sample tubes up to 4 mm in diameter balance optimal CPMAS data collection with respectable high-resolution ^{1}H NMR and MQMAS NMR performance.

^{13}C-CPMAS

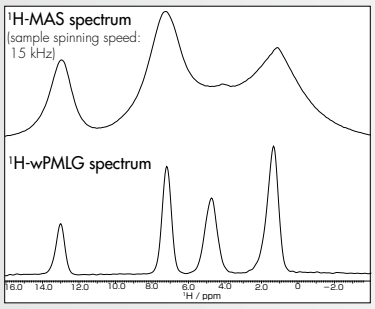
SSB free ^{13}C CPMAS spectra can be easily collected at 19 kHz spinning.

Sample : Cholic acid
Spectrometer : JNM-ECA600
Probe : 3.2 mm HXMAS probe
Experiment : ^{13}C-CPMAS
Sample spinning speed : 19 kHz
Number of accumulations : 640



High-Resolution ^{1}H Solids

The 4 mm, 3.2 mm, and 2 mm HXMAS probes support high-resolution ^{1}H measurements with wPMLG CRAMPS experiments.



Sample:
Monoethyl fumarate

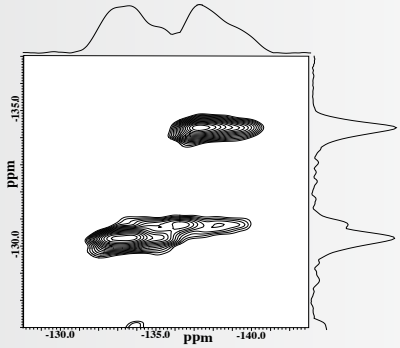
Spectrometer:
JNM-ECA500

Probe:
4 mm HXMAS probe

Moderate MAS of 15 kHz is not sufficient to narrow the line broadening caused by the strong ^{1}H-^{1}H dipole-dipole interactions; however, wPMLG CRAMPS NMR measurements easily yield high-resolution solid state ^{1}H spectra.

^{87}Rb-MQMAS

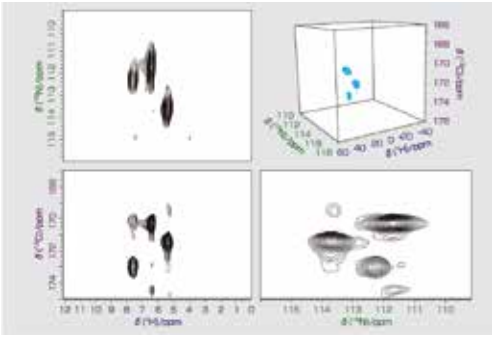
The 4 mm, 3.2 mm, and 2 mm HXMAS probes support MQMAS measurements.



Sample : Rubidium nitrate
Spectrometer : JNM-ECA600
Probe : 4 mm HXMAS probe

HCN MAS Probes

JEOL offers a variety of triple-resonance HCN probes for study of biological samples in solid state. HCN MAS probes come in various diameters, 0.75 mm, 1 mm, 2 mm, 3.2 mm, and 4 mm, in order to deliver optimum performance in either ^{1}H-, ^{13}C- or ^{15}N-detected experiments. Accessory to keep biological samples at safe, low temperature under MAS during the long experiments is available.

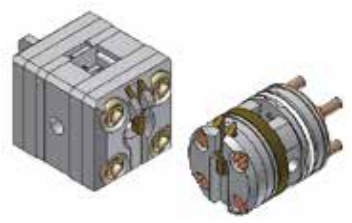


3D CO (CA) NH spectrum
Sample : ^{15}N, ^{13}C-labeled f-MLF tripeptide
Instrument : JNM-ECZ600R
Probe : 0.75 mm HCN MAS probe

Small Size HXMAS Probe (0.75 mm & 1 mm)

Ultra-Fast MAS Spinner Technology Achieves the Highest Level Sample Spinning Speed of 100 kHz and Opens Doors to New Solid-State NMR Experiments.

JEOL's advanced high-speed spinner technology, announced in April 2015, sets a new benchmark for solid-state NMR spinning speed: 120 kHz for a 0.75 mm, 300 nl sample rotor. The corresponding speed for a 1 mm sample tube holding 800 nl is 80 kHz. These two HXMAS probes for extremely small sample volumes and ultra-fast sample spinning permit high-sensitivity microcoil measurements of limited samples and open doors to new solid-state NMR experiments that make effective use of ultra-fast sample spinning and very high RF fields.

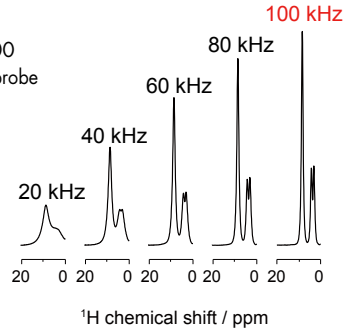


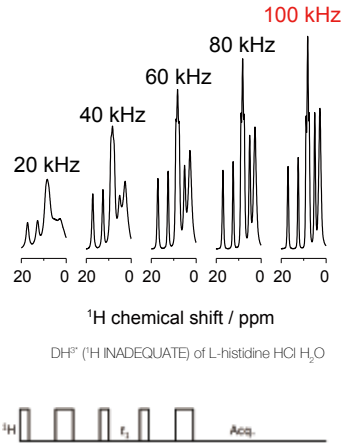
High-resolution ^{1}H measurements at Ultra-Fast Magic Angle Spinning

Ultra-fast sample spinning at 100 kHz and above enables high resolution ^{1}H measurements without the need for CRAMPS decoupling. High-resolution quantitative measurements and ^{1}H individual peak relaxation time determinations are only possible at very high spinning speeds. These speeds also make high-resolution ^{1}H two-dimensional measurements practical without degrading sensitivity.

^{1}H-MAS spectra

Sample: Glycine
Spectrometer: JNM-ECA600
Probe: 0.75 mm HXMAS probe



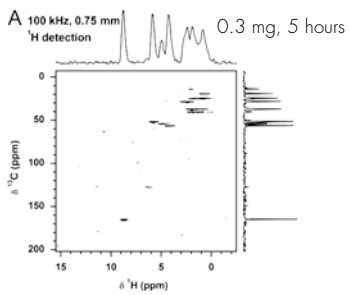


* DH*: M. Deschamps, et al., *Phys. Chem. Chem. Phys.*, **13,** 8024-8030 (2011).

^{1}H Indirect Detection of Heteronuclei at Ultra-Fast Magic Angle Spinning

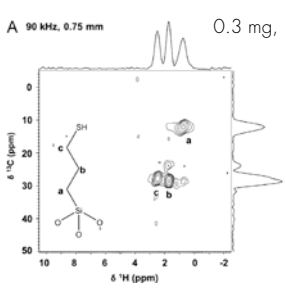
Ultra-fast sample spinning makes it possible to measure ^{1}H-^{13}C HSQC and ^{1}H-^{14}N HMQC of solid samples in natural isotopic abundance. This allows high resolution two-dimensional solid NMR measurements similar to solution NMR measurements, without a reduction in sensitivity or resolution.

f-Met-Leu-Phe-OH tripeptide in natural abundance

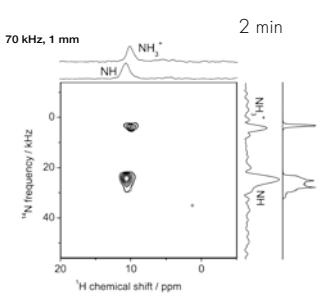


Y. Nishiyama et al., *Solid State Nucl. Magn. Reson.*, **66-67,** 56-61 (2015).

Organically functionalized mesoporous silica nanoparticles in natural abundance



Glycyl-L-alanine in natural abundance

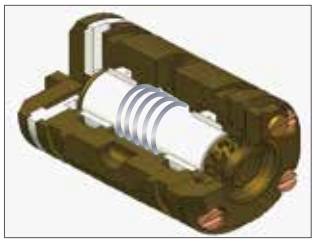


Y. Nishiyama et al., *J. Magn. Reson.*, **208,** 44-48 (2011).

Large Size HXMAS Probe (8 mm)

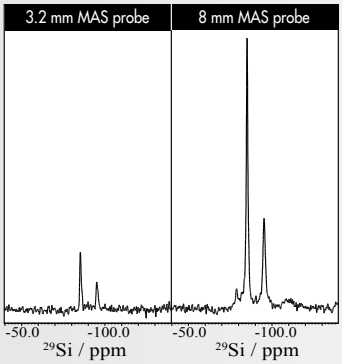
High-Sensitivity Measurements with Largest Available Sample Rotor in a Narrow Bore Magnet

Increasing the sample volume is a simple and cost effective way to boost the sensitivity of samples with weak NMR properties. JEOL therefore utilized the latest fluid dynamics simulation technologies to develop stable 8 mm MAS Solid State NMR probes with the largest sample volume available for narrow bore (54 mm) magnets. These probes are especially effective for ^{29}Si, low-gamma nuclei, and other nuclei with low sensitivity. The JEOL 8 mm MAS State NMR probes are available in single tune X only or double tune HX configurations for optimal NMR sensitivity.



High-Sensitivity Measurements with Large Sample Volume Rotors

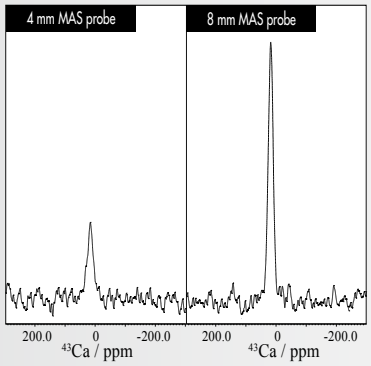
8 mm ^{29}Si-MAS spectrum with ^{1}H Decoupling



Sample: Synthesized tobermorite
Spectrometer: JNM-ECA500
Repetition time: 10 s
Number of accumulations: 128

Samples courtesy of
Asahi Kasei Corporation

8 mm ^{43}Ca-MAS spectrum



Sample: Synthesized tobermorite
Spectrometer: JNM-ECA600
Repetition time: 0.5 s
Measurement time: 2 days (approximately)

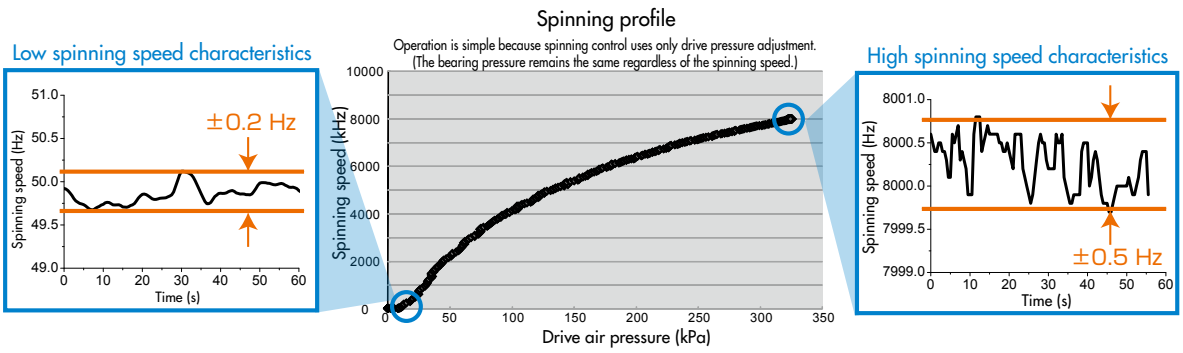
^{43}Ca nucleus characteristics

Low gyromagnetic ratio
-1.8×10^{7} rad/s
(approximately 1/15 that of ^{1}H)
Low natural abundance: 0.145%
spin quantum number: I = 7/2

Samples courtesy of
Asahi Kasei Corporation

High Spinning Speed & Highly Stable Spinning

Increasing the sample volume can reduce the solid-state NMR Magic Angle Spinning performance at high spinning speeds. The JEOL 8 mm MAS probes can achieve spinning speeds up to 8 kHz without compromising spinning stability in spite of the large 616 µl sample volume. The 8 mm MAS spinner module is stable over a broad range of spinning speeds from 50 Hz up to 8 kHz.



Low Frequency Unit for Solid NMR Probes

Measurement of Low Frequency Nuclei Below ^{15}N Available

The Low Frequency Unit NM-02380LFU extends the LF tuning range of the extended VT HXMAS probes and 8 mm HXMAS probes to low gamma nuclei (below ^{15}N) .
A wide range of nuclei can be covered with one MAS probe and the Low Frequency Unit.

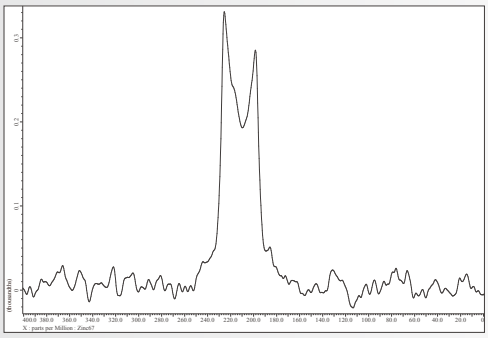
Observation nuclei include: ^{15}N, ^{35}Cl, ^{33}S, ^{14}N, ^{43}Ca, ^{25}Mg, ^{47}Ti, ^{49}Ti

Solid-state NMR probe

Low Frequency Unit

Only specific MAS NMR probes can take advantage of the optional Low Frequency Unit. Please check with your JEOL sales representative for information on compatible probes.

^{67}Zn NMR spectrum of ZnO



Probe: 3.2 mm VT Solid-state NMR probe and Low Frequency Unit
^{67}Zn resonance frequency at 14.1 T : 37.5 MHz
Scans: 30,000

New Extended VT HXMAS Probe

Wide Variable Temperature Range from -100 °C to +200 °C for Narrow Bore Magnets

Wide temperature range HXMAS probe has realized sample temperature of -100 °C to 200 °C at fast spinning, due to newly designed spinning module. In addition, after examination of heat-resistance evacuation system during temperature change, an experiment in such a wide range from low temperature to high temperature became possible by using a narrow bore magnet. Furthermore, this probe does not require nitrogen gas even at 200 °C high temperature measurement.
By combination with a highly accurate temperature control unit of JNM-ECZ series, a detailed control of rising and falling temperature is possible. Additional use of a low frequency unit makes it possible to measure low frequency nuclei lower than ^{15}N.

	400 MHz	500 MHz	600 MHz
Sample tube diameter	4 mm	3.2 mm	3.2 mm
Maximum MAS speed*1	18,000 Hz	22,000 Hz	
Variable temperature range*2 *3	– 100 °C ~ + 200 °C		
Observation nuclei	LF nuclei : ^{31}P, ^{7}Li, ^{11}B, ^{23}Na, ^{27}Al, ^{13}C, ^{79}Br, ^{207}Pb, ^{29}Si, ^{6}Li, ^{15}N		
	HF nuclei : ^{1}H, ^{19}F		
When low frequency unit is used	Major observation nuclei : ^{15}N, ^{35}Cl, ^{33}S, ^{14}N, ^{43}Ca, ^{25}Mg, ^{47}Ti, ^{49}Ti		
Irradiation nuclei	^{1}H, ^{19}F		

*1 When measured in room temperature
*2 Pre-set temperature
*3 Heat-resistant sample tube is needed at temperatures higher than 150 °C

Single-Tuned 4 mm Cryocoil MAS Probe

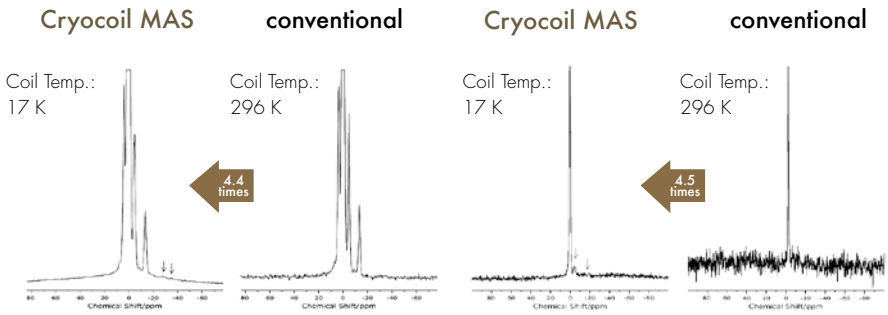
Sensitivity 4.5 Times Greater Than That of Conventional Room Temperature MAS Probe Achieved

A tunable single-tuned 4 mm Cryocoil MAS probe has been developed. The sensitivity enhancement factor attained by the Cryocoil MAS probe is 4.5 times compared to a conventional RT probe.

Typical Specification

- Detection Coil Temperature: 14 K
- Sample Temperature: 273 - 353 K
- Rotor O.D.: 4 mm (volume: 50 ul)
- Spinning Speed: 18 kHz max.
- Tuning Range: 73 - 192 MHz
- Input Power: 250 W max.

Comparison of detection sensitivity



^{6}Li-NMR spectrum **^{29}Si-NMR spectrum**

AUTOMAS Probe (3.2 mm & 8 mm)

The newly developed AUTOMAS probe and ROTORCARRIER™ bring the usability of solution NMR probes to solid-state NMR spectroscopy (1).
The ROTORCARRIER™, which is of similar shape as a sample holder for solution NMR, stores the solid-state NMR sample tube (rotor), and carries it from the top of SCM or ASC to the probe.
The AUTOMAS probe receives the sample tube from the rotor carrier and automatically adjusts Magic Angle.
As the AUTOMAS probe also has the automatic tuning function, all operations including sample loading, sample spinning, temperature control, probe tuning and experiment can be done in a fully automated way.
The innovative point of AUTOMAS probe and ROTORCARRIER™ is that solution and solid state can share the same auto sample changer (2). There are two types of sample tube diameters : 3.2 mm, which is well-balanced, and 8 mm which is suitable for measuring low-sensitivity nuclei.

✓ **Auto Tune**
Can use the same Auto Tune unit as the liquid-state probes
✓ **Sample load and eject**
Samples can be smoothly loaded/ejected from above the magnet via the ROTORCARRIER™
✓ **Auto Sample Changer**
The functions of the existing ASC24, JackBean 30, 64, and 100 can be used as-is

ROTORCARRIER™

(1) Compatible with the JNM-ECZ series only. MASCONT improvements may be required in some cases.
(2) Compatible Auto Sample Changers are ASC24 and JackBean 30, 64, 100 only.

FGMAS Probe

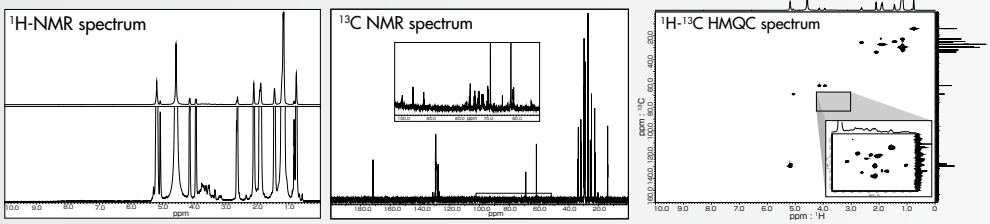
High-Resolution Field Gradient MAS NMR Probes for Semi-Solids, Tissue Biopsies, and Elastomers.

FGMAS Food Emulsion

Soy milk mayonnaise

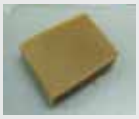
Primary ingredients: Canola oil, soy milk, apple cider vinegar, table salt, sugars

Spectrometer: JNM-ECA600
Probe: 4 mm FGMAS probe
Sample spinning speed: 5 kHz

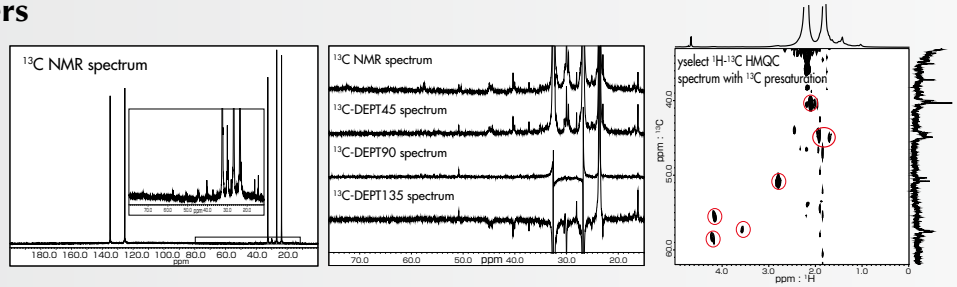


FGMAS Elastomers

Rubber



Crosslinking density: 1.36×10^{-4} mol/cm^{3}
Spectrometer: JNM-ECA600
Probe: 4 mm FGMAS probe
Sample spinning speed : 17 kHz



Samples courtesy of Prof. Kawahara Seiichi, Nagaoka University of Technology

Accessories

Bench spinner

Solid-state NMR frequently uses high sample spinning speeds. Spinning an unbalanced samples can damage the MAS NMR probe requiring expensive repairs. A bench spinner for test spinning outside the probe avoids this danger. It is a necessary accessory for films, pellets, and other samples that are especially difficult to balance.

Solid-state sample packing tools

JEOL's unique solid-state NMR sample packing tools simplify sample packing and unpacking for all rotors from 8 mm to 0.75 mm.

Power amplifiers

Many solid-state NMR applications require large RF fields. JEOL provides RF amplifiers for solid state NMR at 3 power levels: Standard, Middle, and High. These are designed to fit all experimental RF needs from routine CPMAS to high performance MQMAS experiments.

	H F	L F
Standard	100 W	300 W
Middle	200 W	500 W
High	500 W	1000 W

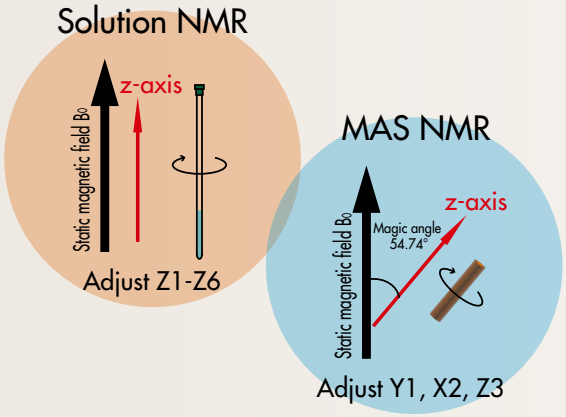
Easy, quick, & reliable shimming for Magic-Angle-Spinning NMR

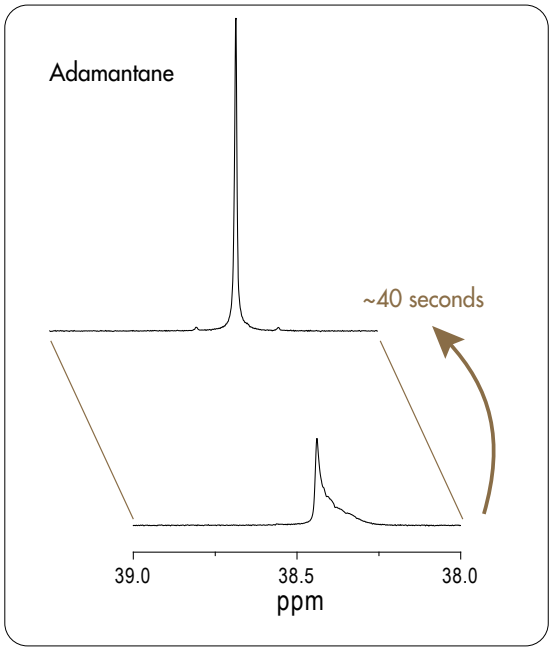
Magic Shimming

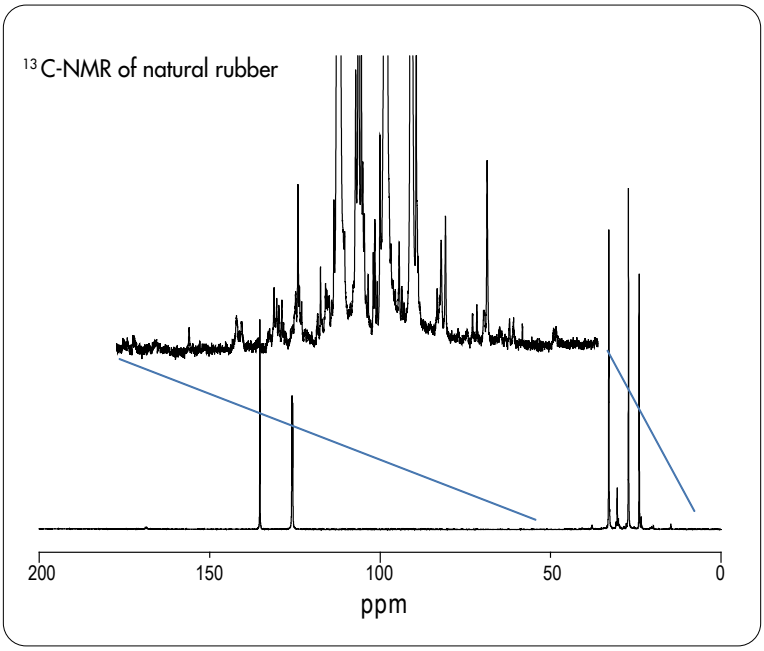
Unlike solution NMR, solid-state NMR has lacked access to automatic shim adjustment (auto shim) or gradient shimming resulting in large amounts of time spent shimming by hand with less than optimal results. Magic shimming is an innovative approach that achieves high resolution with field gradient mapping as in solution NMR. This allows the operator to easily and quickly adjust the shims to levels that are difficult to obtain with manual shimming. This advancement in sample shimming technology from JEOL will improve the quality of your measurements.

What is Magic Shimming?

Magic shimming is gradient shimming for magic angle spinning NMR spectroscopy. Like solution NMR gradient shimming Magic Shimming uses a field gradient or homo-spoil pulse to map the magnetic field inside the sample rotor. The axial Z1-Z6 shims used for solution NMR are very weak along the magic angle spinning rotor axis, therefore the tesseral (radial) Y1, X2, axial Z3, and other shims which are stronger along the magic angle spinning axis are used to optimize the magnetic field (Patent 2013-29366A).







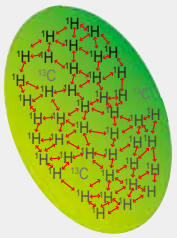
Latest answer for solid mixture analysis from JEOL

ROSY = Relaxation Ordered SpectroscopY

Analyzing mixtures by NMR is difficult; however, DOSY is well established for analyzing mixtures in solution. Recently JEOL developed ROSY to analyze mixtures in the solid state. ROSY, Relaxation Ordered SpectroscopY, relies on differences in ^{1}H T_1 relaxation in the solid state to separate the components of a mixture. This eliminates the complications and time spent to physically separate the mixture into pure substances. This new technology, from JEOL, promises to find application in analyzing crystal polymorphism, crystalline-amorphous mixing, and other solid state mixtures (Patent 2010-71839A).

Why is the ^{1}H longitudinal magnetization relaxation time T_1 uniform in solid samples?

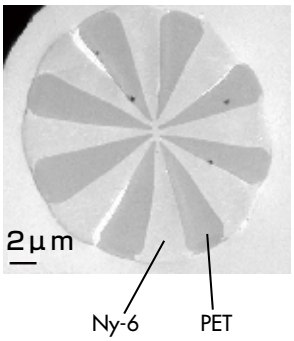
Within a single domain of a sample the homonuclear dipole-dipole interactions between the ^{1}H nuclei form strongly coupled spin networks. At moderate spinning speeds this causes the ^{1}H magnetization rapidly to diffuse over the entire network of spins in the domain. The domains can be a single crystal form of a pure chemical or an isolate amorphous region in a sample mixture. The ^{1}H spin diffusion causes the domain to have a single uniform ^{13}C detected ^{1}H T_1 value for all sites that are part of the domain.

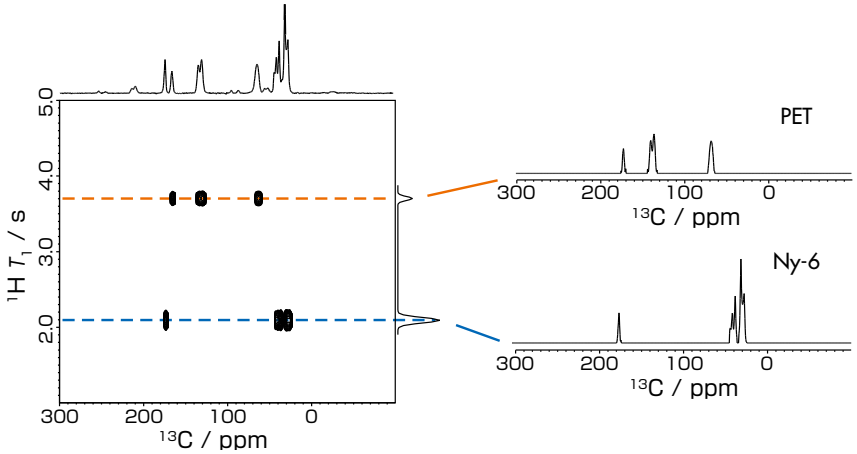


Spin diffusion simulation

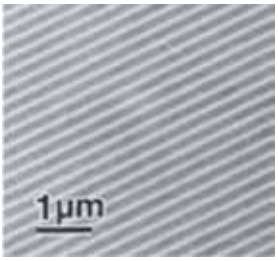
High Molecular Weight Composite Polymers Applications

PET/Ny-6 Compound Fiber

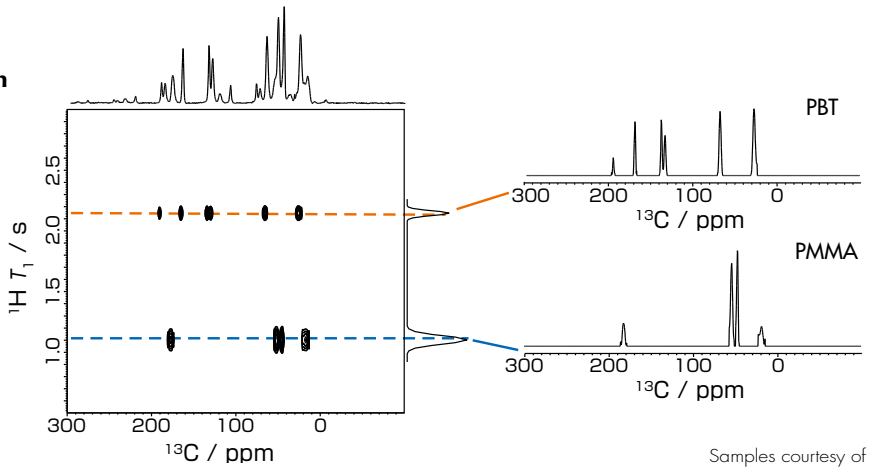




PBT/PMMA Nanolayer Film



Dark: PBT
Light: PMMA



Samples courtesy of Teijin Limited

*Specifications are subject to change without notice

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