2mm HXMAS probe : Multi-use probe capable of high speed MAS and high sensitivity measurements

Product used : Nuclear Magnetic Resonance (NMR)

JEOL 2mm HXMAS probe is a multi-use probe capable of high speed magic angle spinning (MAS) up to 40kHz and high sensitivity measurements. It is not only available for general use in standard ^{13}C measurements of organic materials, but also for highly sensitive ^{1}H indirect detection utilizing high resolution ^{1}H NMR. Since it is also suitable for ^{19}F measurements where spinning side bands are likely to appear (JEOL application note: NM18003) and MQMAS measurements of quadrupole nucleus, the 2mm probe is strongly recommended probe that can handle a variety of measurements as a single probe.

	1mm	2mm	3.2mm
Outer diameter	1mm	2mm	3.2mm
Inner diameter	0.5mm	1.55mm	2.2mm
Maximum spinning frequency	80,000Hz	40,000Hz	22,000Hz
Sample volume	0.8µl	17µl	49µl

^{13}C sensitivity

The 2mm probe can obtain ^{13}C CPMAS spectra with a sensitivity of about 20 to 30% less than the 3.2mm probe although the sample volume is about 1/3. This probe can be used for a daily use of ^{13}C measurement and is especially effective for measuring a small amount of sample.

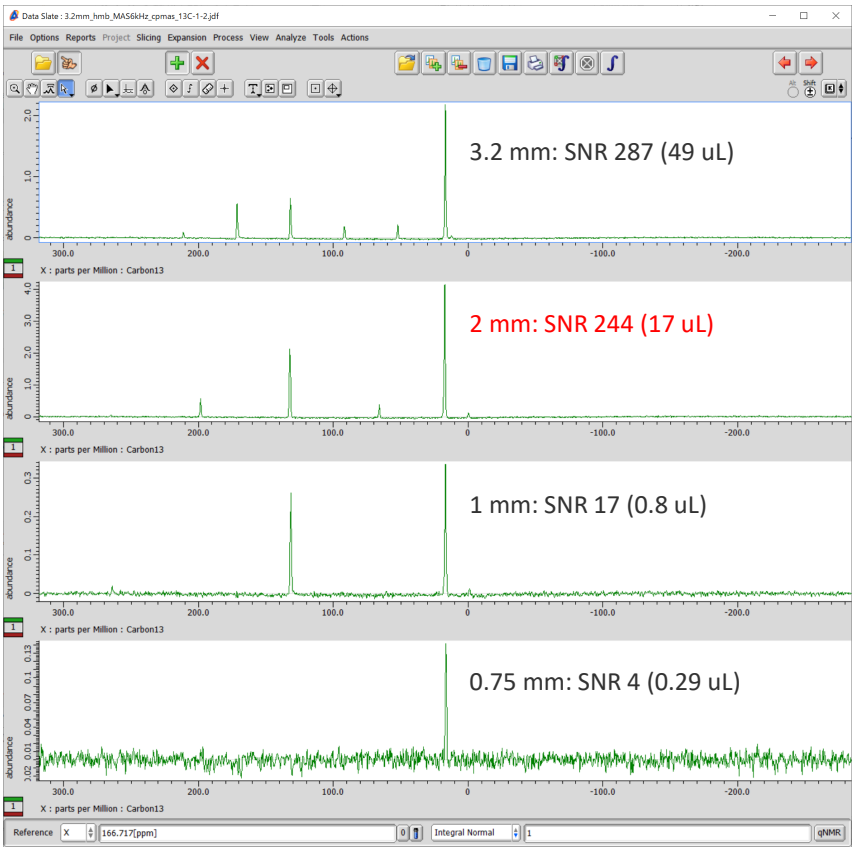


Fig.1 Comparison of ^{13}C sensitivity among various probes. ^{13}C CPMAS spectra for Hexamethylbenzen (HMB) were obtained using 3.2mm, 2mm, 1mm and 0.75mm HXMAS probes. SNRs were calculated using methyl signals of HMB.

Temperature increase caused by MAS

In solid state MAS measurement, the sample temperature rises due to the friction between the spinning control gas and the sample tube. Since the 2 mm probe can perform MAS more efficiently than the 3.2 mm probe, the rise in sample temperature can be suppressed. For example, 20kHz MAS raises nearly 40 °C in the 3.2mm probe, while it raises only 5 °C in the 2mm probe. The 2mm probe is recommended to obtain a ^{13}C spectrum with less spinning sidebands while suppressing the sample temperature rise.

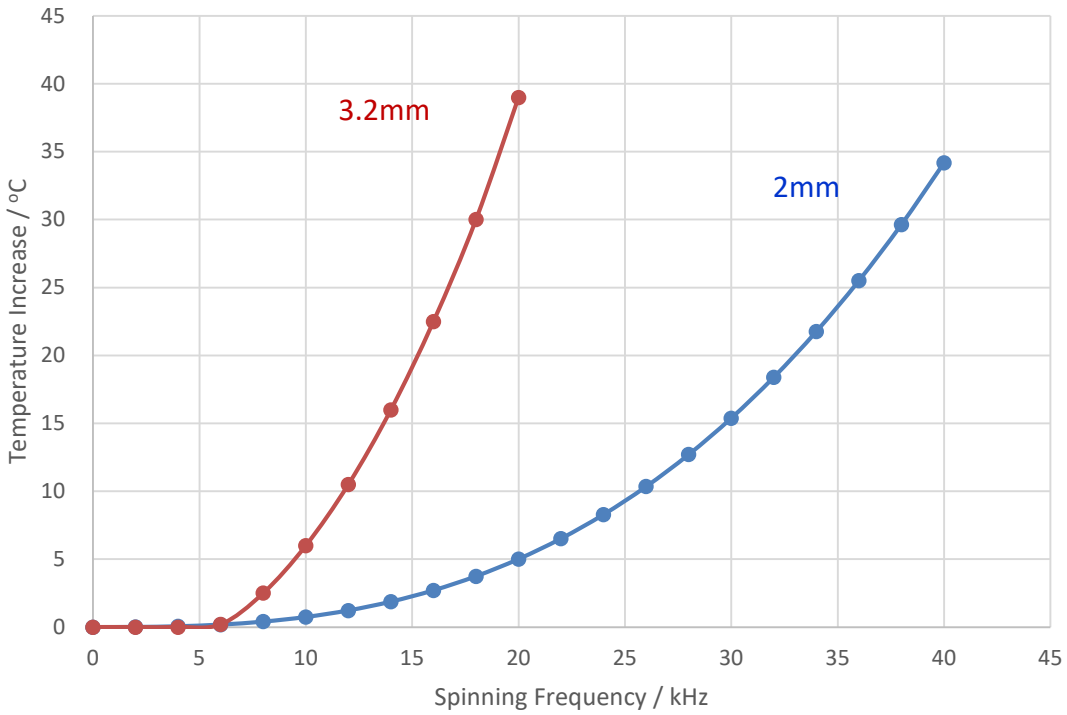


Fig.2 Relation between spinning frequency and temperature increase of samples for 3.2mm and 2mm probes.

^{1}H indirect detection

The sensitivities of nuclei with low gyromagnetic ratios can be dramatically improved by ^{1}H indirect methods owing to high sensitivity and resolution of ^{1}H at very fast MAS regime. 40kHz MAS achieved by the 2mm probe provides ^{1}H high resolution and allows to obtain ^{1}H indirect spectra. Although the sensitivity with the same sample volume is inferior to that of the 1mm probe, actual sensitivity of the ^{1}H indirect detection using the 2mm probe is much higher because of much larger sample volume. As an example, natural abundance ^{1}H/^{15}N CP-based HSQC spectra of cimetidine measured by the 1mm and 2mm probes are shown.

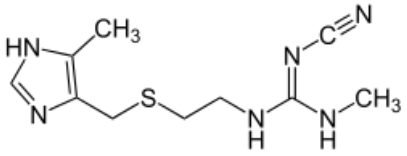
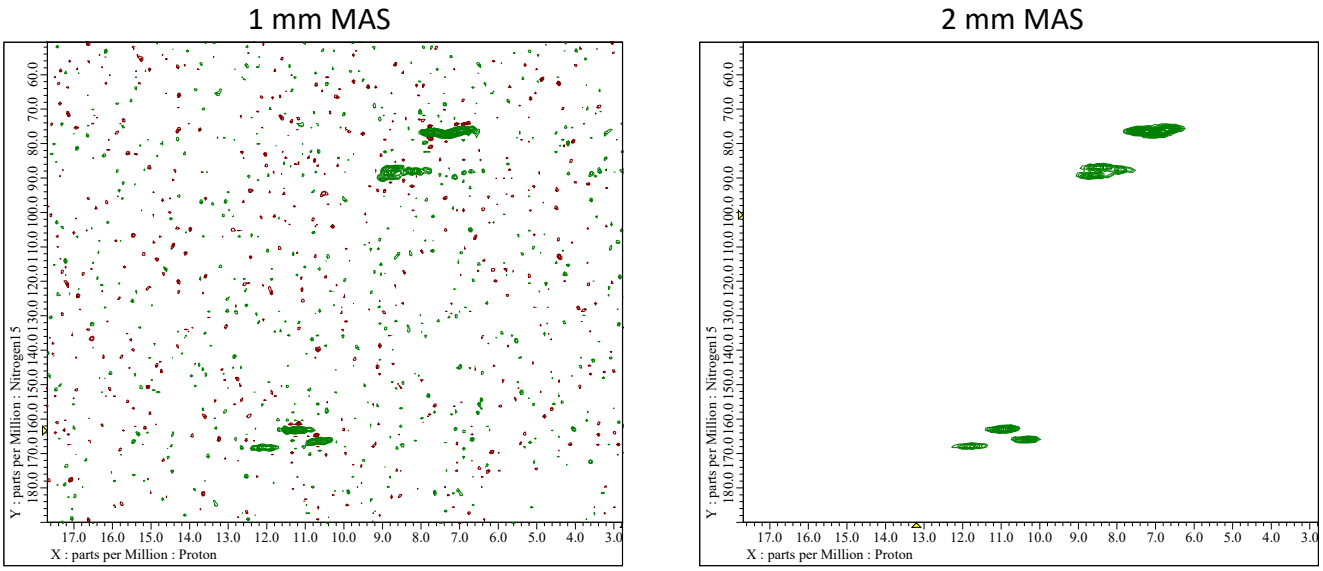


Fig.3. Chemical structure of cimetidine



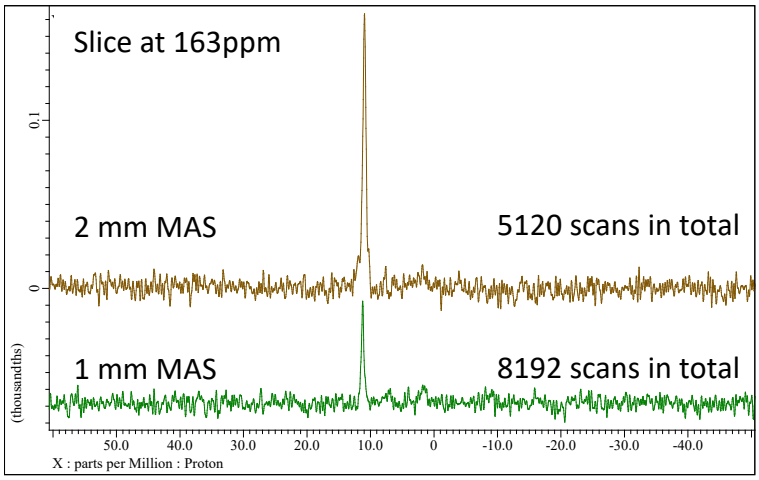


Fig.4 ^{1}H/^{15}N CP-based HSQC 2D spectra of cimetidine obtained by 1mm and 2mm HXMAS probes. ^{1}H sliced spectra at ^{15}N 163ppm are also shown.

MQMAS

The 2mm probe is also useful for MQMAS measurement of quadrupole nuclei. The MQMAS is a representative method for observing quadrupolar nuclei with high resolution, and is widely used because it is available with a standard solid state MAS probe. A strong rf magnetic field strength ,B_1, is essential for the MQMAS method because the excitation efficiency of multi-quantum excitation affects the sensitivity of MQMAS measurements. The 2mm probe can apply a stronger B_1 than the conventional 3.2mm probe, enabling highly sensitive MQMAS measurements. Here, we show the ^{27}Al 3QMAS spectra of kyanite (Al_2SiO_5) measured with the 3.2mm and the 2mm probes.

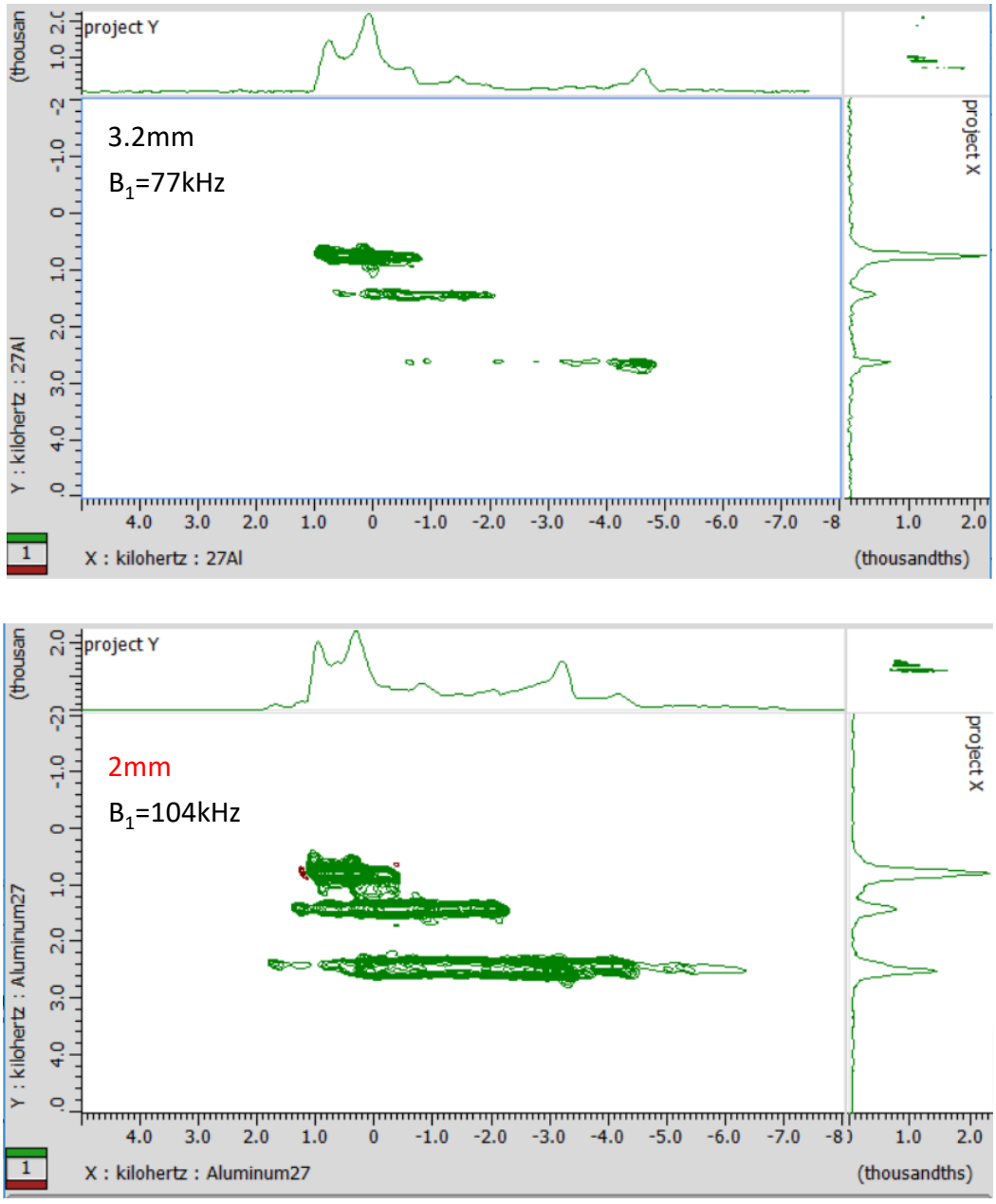


Fig.5 ^{27}Al 3QMAS spectra of kyanite (Al_2SiO_5) obtained by 3.2mm and 2mm HXMAS probes. The rf field strength B_1 of multi-quantum excitation are 77kHz for 3.2mm and 104kHz for 2mm, respectively.

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