

High-Field NMR at Queen Mary

600 MHz

The 600 MHz spectrometer was re-consolidated in 2002 and is now a state-of-the-art instrument capable of performing almost any conceivable NMR experiment. It has four transmitter channels; a variable temperature controller; automatic sample changer; Z-gradient capability; and a Magic Angle Spinning (MAS) controller. It is equipped with the following probeheads:

5mm $^1\text{H}/^{13}\text{C}/^{15}\text{N}/^2\text{H}$ (z-grad) - useful for all ^1H observation experiments including most high resolution 1-, 2- and 3-dimensional experiments on organic and biological molecules. This probe was bought in 2001 and gives high sensitivity as well as access to gradient experiments such as self-diffusion measurements. On a typical sample (1-10 mM) average experiment times would be:

<u>Experiment</u>	<u>Time</u>
^1H 1D spectrum	1 minute
$^1\text{H}/^1\text{H}$ 2D COSY	5 minutes - 1 hour
$^1\text{H}/^1\text{H}$ 2D NOESY	1 hour - 4 hours
$^1\text{H}/^{13}\text{C}$ 2D correlation	20 minutes - 4 hours
$^1\text{H}/^{15}\text{N}$ 2D correlation	4 hours - overnight

This probe can also be used for ^{19}F observation including $^{19}\text{F}/^{13}\text{C}$ 2D correlation experiments. Because ^{19}F uses the same channel as ^1H it cannot do ^{19}F with ^1H decoupling.

5mm BroadBand/ $^1\text{H}/^2\text{H}$ – range ^{109}Ag to ^{31}P . Useful for direct observation of nuclei other than ^1H and ^{19}F . On a typical sample (as above) average experiment times are:

<u>Experiment</u>	<u>Time</u>
^{13}C with ^1H decoupling	20 minutes - 4 hours
^{31}P with ^1H decoupling	5 minutes

This probe can also be used for measuring ^{13}C spectra with both ^1H and ^2H decoupling.

5mm $^{19}\text{F}/^1\text{H}/^2\text{H}$ – used for measuring ^{19}F spectra with ^1H decoupling.

8mm $^{13}\text{C}/^1\text{H}/^2\text{H}$ – used for samples with low solubility where a greater volume may be needed.

10mm BroadBand/¹H/²H – broadband in the range ⁴¹K to ¹⁵N. Suitable for very low frequency nuclei (goes beyond the range of the 5mm probe).

4mm MAS – used for solid samples where cross-polarisation (CP) or high-power decoupling (HPDEC) is not possible. The maximum spin-rate is 12 KHz. Tuneable between ²H and ³¹P. This probe gives sharper linewidths than the CP/MAS probe and so should be used for any sample not containing ¹H.

4mm CP/MAS – as above but with a ¹H channel for CP/HPDEC. This probe can also perform variable temperature experiments.

7mm CP/MAS - Tuneable between ¹⁰³Rh and ¹⁷O. Used for CP/HPDEC and MAS for low frequency nuclei. The maximum spin rate is 6 KHz.