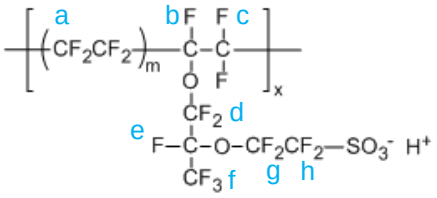
^{19}F solid state NMR by using 2mmMAS probe

Product used : Nuclear Magnetic Resonance (NMR)

JEOL 2mm MAS probe enables MA Spinning at nearly twice MAS speed of the conventional 3.2mm and 4mm MAS probe with 20 times the sample volume of the 1mm MAS probe. An attractive application of the 2mm MAS probe is ^{19}F NMR. Strong ^{19}F homonuclear dipolar coupling and wide chemical shift range cause a series of spinning side band (SSB) which make it difficult to analyze ^{19}F spectra obtained by using the conventional 3.2mm and 4mm probes. The 2mm probe can achieve 40kHz MAS speeds, the resulting ^{19}F spectra will had small well managed SSB's.

Here, we introduce ^{19}F solid state NMR spectra of Nafion known as a solid polymer electrolyte for fuel cells. Fig.1 shows ^{19}F MAS spectra of Nafion at various MAS speeds. 40kHz MAS gives a clear ^{19}F spectrum without overlapping of SSBs whereas overlap occur between center bands and their SSBs at MAS speeds less than 40kHz. Moreover, the much greater sensitivity of the 2mm probe than the 1mm probe enables direct observation of low sensitive nuclei such as ^{13}C .Thus, ^{13}C{^{19}F} CPMAS(Fig.2) and ^{13}C-^{19}F 2D-HETCOR (Fig.3) can easily be obtained.



Chemical structure of Nafion

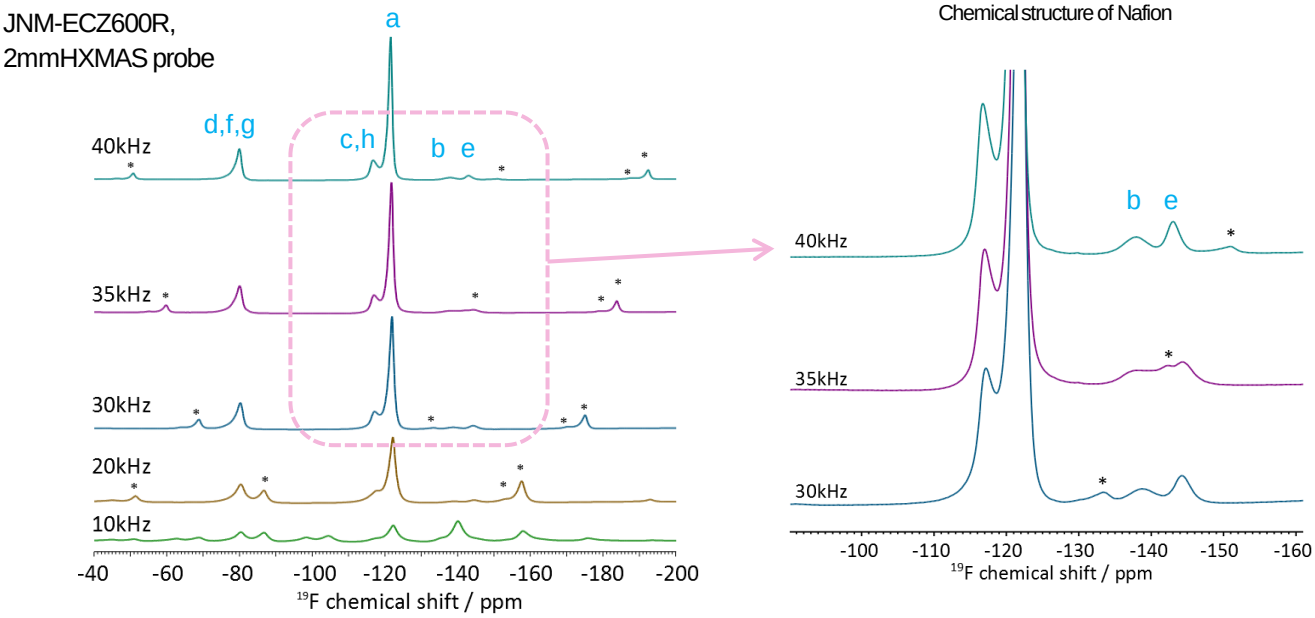


Fig.1 ^{19}F single pulse at increasing MAS frequencies. Peaks marked by * represent SSBs.

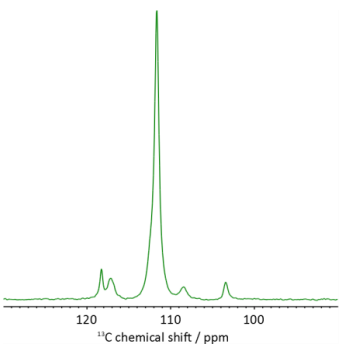


Fig.2 ^{13}C {^{19}F} CPMAS

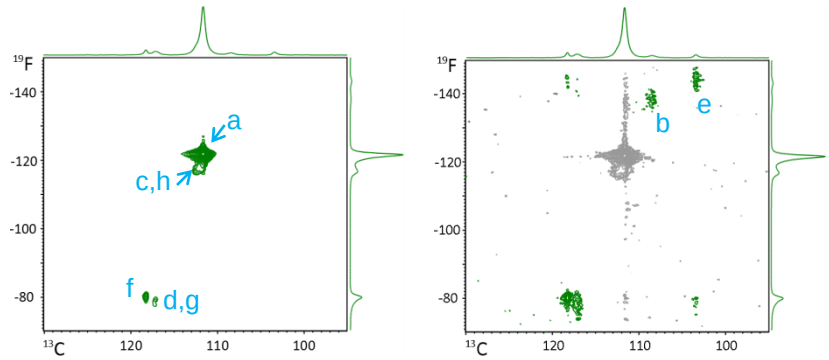


Fig.3 ^{13}C-^{19}F HETCOR with high (left) and low (right) level thresholds.

• Reference : Q. Chen and K. Schmidt-Rohr, *Macromolecules* 2004, **37**, 5995-6003

• Nafion is a trademark of The Chemorus Company

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