There is increasing evidence that active sites in zeolites, which comprise one of the most important classes of industrial catalysts, are more complex than previously recognized. Indeed, active sites that are in close proximity are known in some catalysts to produce advantageous catalytic effects. Differentiating these sites, which are often structurally similar, can be extremely challenging, presenting obstacles for the investigation of their synergistic effects.

In this work, ultra-high-magnetic field solid-state nuclear magnetic resonance (ssNMR) platforms and state-of-the-art probes, available only at the MagLab, were employed to investigate oxygen and aluminum sites in zeolites, with a focus on $^{17}$O and $^{27}$Al, which are quadrupolar nuclei (nuclear spin > $\frac{1}{2}$). High magnetic fields greatly reduce the breadths of $^{17}$O and $^{27}$Al ssNMR patterns, enhancing signal and resolution, and yielding rich structural information. This structural information is able, for the first time, to correlate structural and spatial aspects of active sites with their synergistic catalytic activities.

These findings will inspire new approaches for characterizing the nature of complex catalytic sites. They offer insights into acid site activities toward new rational design and engineering strategies for developing more effective catalysts.

**Facilities and instrumentation used:** NMR Facility: 14.1T/600 MHz and 19.6T/830 MHz; DC Facility: 36T SCH

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