Overtone solid-state NMR spectroscopy on Nitrogen-14

Product used: Nuclear Magnetic Resonance Spectrometer (NMR)

$^{15}$N NMR is widely used because of the importance of nitrogen in chemistry, materials, biology, environment etc. However, very low abundance of $^{15}$N ($<0.4\%$) results in poor sensitivity and thus makes observation time-consuming. On the other hand, the rest of nitrogen atoms are also NMR sensitive nucleus of $^{14}$N. Despite the high abundance of $^{14}$N ($>99\%$), its application is rather limited due to the huge quadrupolar interactions and its spin quantum number $I = 1$. The introduction of very fast MAS (>70 kHz) enabled the $^1$H detected $^{14}$N/$^1$H correlation spectroscopy (Nishiyama et al., JMR 208 (2011) 44-48). However, it demands precise magic-angle adjustment, moreover, the sensitivity is largely affected by the molecular motion. It was shown that the use of $^{14}$N overtone transition avoids these difficulties under MAS (O’Dell et al. Chem. Phys. Lett. 514 (2011) 168-173) with a cost of sensitivities. Since the overtone transition between -1 and +1 energy level is forbidden, the low sensitivity fundamentally comes from small transition moment between these levels. Here, we have developed $^1$H detected $^{14}$N overtone/$^1$H correlation spectroscopy under ultrafast MAS conditions > 70 kHz to achieve highly sensitive $^{14}$N NMR spectroscopy of rigid solid.

![Energy levels of $^{14}$N (I = 1).](image)

![$^{14}$N overtone/$^1$H 2D correlation spectrum of glycine observed in 2.3 hours under 90 kHz MAS.](image)