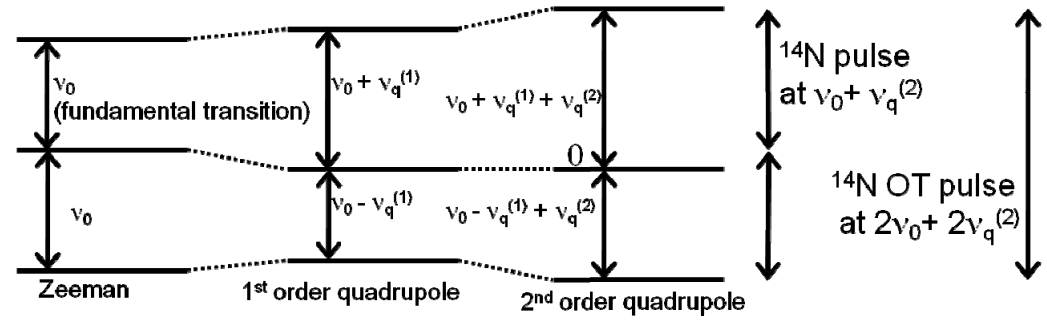
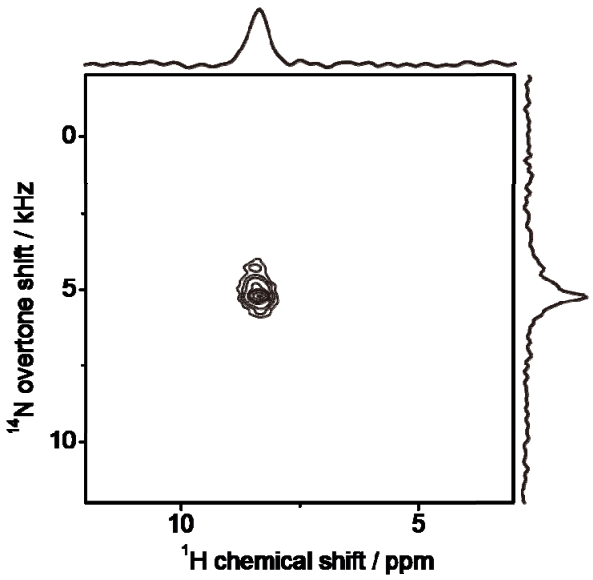
Overtone solid-state NMR spectroscopy on Nitrogen-14

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%), it's 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).



^{14}N overtone/ ^{1}H 2D correlation spectrum of glycine observed in 2.3 hours under 90 kHz MAS.

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