**A Fast Magic-Angle Spinning Three-Dimensional NMR Experiment for Simultaneously Probing H-H and N-H Proximities in Powdered Solids**

G N Manjunatha Reddy,*a* Michal Malon,*b,c* Andrew Marsh,*d* Yusuke Nishiyama*b,c* and Steven P. Brown*a*\*

*a Department of Physics and dDepartment of Chemistry, University of Warwick, Coventry, CV4 7AL, United Kingdom*

*b JEOL RESONANCE Inc., Musashino, Akishima, Tokyo 196-8558, Japan*

*c RIKEN CLST-JEOL Collaboration Centre, Yokohama, Kanagawa 230-0045, Japan*

**Experimental data as reported in Analytical Chemistry 2016**

All data recorded by G N Manjunatha Reddy (JEOL NMR data together with Michal Malon)

**L-Histidine.HCl**

**Figure 2** 1D Single pulse NMR data (recorded on 05/10/2015)

2D 1H DQ-SQ Correlation NMR data (recorded on 05/10/2015)

2D 1H(DQ)- 14N(SQ)-1H(SQ) Correlation NMR data (recorded on 05/10/2015)

**Figure 3** 2D 1H(DQ)- 14N(SQ) HMQC Correlation NMR data (recorded on 07/10/2015)

3D 1H(DQ)- 14N(SQ)-1H(SQ) Correlation NMR data (recorded on 13/10/2015)

**Figure S3** DFT refinedcrystallography information file (cif) (performed on 02/10/2015)

 NMR shielding calculations in .Magres format (performed on 02/10/2015)

 Experimental powder X-ray diffraction data (recorded on 01/01/2016)

**Guanosine.2H2O**

**Figure 5** TGA-DSC data in .xls format (recorded on 29/07/2014 and 09/02/2016)

**Figure 6** 1D Single pulse NMR data (recorded on 05/10/2015)

2D 1H DQ-SQ Correlation NMR data (recorded on 05/10/2015)

2D 1H(DQ)- 14N(SQ)-1H(SQ) Correlation NMR data (recorded on 05/10/2015)

**Figure 7** 2D 1H(DQ)- 14N(SQ) HMQC Correlation NMR data (recorded on 05/10/2015)

3D 1H(DQ)- 14N(SQ)-1H(SQ) Correlation NMR data (recorded on 05/10/2015)

**Figure S4** 1D 13C CPMAS NMR data (recorded on 13/02/2015, 16/04/2015 and 06/04/2016)

Experimental powder X-ray diffraction data (recorded on 22/07/2016)