# Solid State $^{13}C$ NMR Characterization of an Incommensurately Modulated Structure

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## Introduction

Incommensurately modulated structures (IMS) have aperiodic lattices that have some structural or magnetic feature that changes from unit cell to unit cell. The modulated feature never comes back on itself, thus the origin of the term. These structures are found to occur most often in inorganic compounds, though a few organic IMS are known. The telltale signature of an IMS is a set of weak satellite peaks in the diffraction pattern. We have synthesized a 2:1 co-crystal of urea:cyclo(gly-gly) that exhibits a highly unusual IMS. Furthermore, we have made this compound using solvent drop grinding in a ball mill as well as traditional solvent evaporation. The single-crystal diffraction data set points to an IMS. The compound is currently under investigation by single-crystal X-ray diffraction at the Advanced Light Source in Berkeley and powder X-ray diffraction at the Advanced Photon Source in Argonne. The high-resolution diffraction data set will help our collaborators solve the crystal structure. We seek complementarity between the diffraction and NMR data sets by characterizing the co-crystal IMS using solid-state magic angle spinning chemical shift spectroscopy.

## Experimental

With the help of NHMFL staff, we have successfully collected a $^{13}C$ magic angle spinning solid-state NMR spectrum of the co-crystal on the 750 MHz instrument at AMRIS in Gainesville and the 900 MHz instrument in Tallahassee. Data collected at the Magnet Lab complement and extend the data set collected on our 600 MHz instrument at Oberlin College. As expected, the increased dispersion of the higher-field instruments helped resolve the overlapping resonances of the carbonyl carbons. However, complications in the line shape arising from the structural modulation still remain. In order to fully interpret the chemical shift data, we will need to undertake high-level quantum chemical calculations, as well as single-crystal NMR experiments. This work is still in progress. Preliminary results, including the high-field NMR data, were presented in an invited talk at a meeting in Aveiro, Portugal in May 2011.[1]

## Acknowledgements

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## References

[1] Mehta, M.A., *et al*., SMARTER 2, Aveiro, Portugal, (2011).