

STRUCTURE DETERMINATION OF AMBUIC ACID WITH SYNCHROTRON POWDER DIFFRACTION AND SOLID-STATE NMR

Yuegang Zhang¹, James K. Harper², Peter L. Lee¹, Robert Von Dreele¹, David M. Grant²

¹ Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

² Department of Chemistry, University of Utah, Salt Lake City, Utah 84112

Ambuic acid is an antibiotic obtained from an endophytic fungus commonly found in tropical plants. The sample of this study was obtained from *P. microspora* cultured from the lesions of a pandanus leaf. The formula of this compound is C₁₉H₂₆O₆, totally 51 atoms with 25 non-hydrogen atoms. Solid-state NMR chemical shift tensor measurements for this compound provided the stereochemistry and the initial conformation. In order to confirm the structure and study the interaction of molecules inside the crystal lattice, powder diffraction studies of ambuic acid were carried out while the single crystal cannot be obtained.

The synchrotron x-ray powder diffraction data were measured at beamline 1-BM at the Advanced Photon Source using a Mar345 image plate detector system. The x-ray beam was focused onto the detector with wavelength of 0.619 Å. The sample-to-detector distance was calibrated with a NIST LaB₆ standard as 502mm. The data were average with five 20-second exposure images and the powder pattern was integrated with 30° azimuthal angle range using the Fit2d program. The powder pattern was indexed with the Dicvol program as monoclinic with a P2₁ space group having lattice parameters: a=15.514(1)Å, b=4.3937(2)Å, c=14.2012(5)Å, β=110.316(4)°, Z=2, vol=907.79(8)Å³. The structure was solved by the DASH and PSSP programs with the starting model from the NMR experiments. The Rietveld refinement was carried out by the GSAS program with all the bond distances and angles constrained at the values given by the solid-state NMR results. The model was fine tuned between the NMR model and x-ray model until the model fit both the NMR and x-ray data well. The final x-ray refinement gave wRp 6.46%, Rp 8.26%, R(F²) 5.21%.

This research demonstrates that the combination of the solid-state NMR and synchrotron powder diffraction can give accurate structures without ambiguity.

Acknowledgment: The work at the Advanced Photon Source was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract W-31-109-Eng-38. Support for the research of D. M. G. was provided by the National Institutes of Health under Grant No. GM 08521-40. Computer support for all tensor computations was provided by the Center for High Performance Computing at the University of Utah.