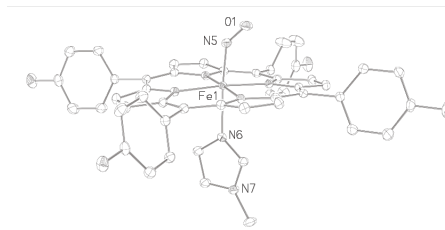


W0392

[Fe(TPP)(1-MeIm)(NO)], A Tale of 1 Crystal, 3 Cells and Too Many Data Collections. Multiple Temperature Studies to Resolve Disorder and Twinning. Bruce C. Noll, Nathan J. Silvernail, W. Robert Scheidt, Univ. of Notre Dame, Notre Dame, IN.

In our ongoing study of the environment of axial ligands in six-coordinate Fe porphyrins we commonly pursue a series of experiments at various temperatures to map out the conformational landscape of these groups. During the course of such an experiment, it was observed that the crystal under study, an iron-nitrosyl porphyrin, indexed at 100 K to a triclinic cell of twice the volume of the cell of the 293 K study. A cell of 3× the room-temperature volume was found at 224 K. The crystal could be indexed as a non-merohedral twin with two components at all temperatures. Cell dimensions of the twin cells were equivalent to the small 293 K cell. The system was refined as a twin at all three temperatures. In addition, both the 2× and 3× cells were solved and refined. Multiple orientations of the NO oxygen were present in all but the 100 K data sets. Tripling the cell at 224 K did nothing to isolate these orientations. No differences could be observed in the conformations of the two porphyrins of the asymmetric unit of the doubled 100 K cell.



A second crystal showed no indications of either twinning or a phase change. Complete experiments at 100 K, 224 K, 293 K and 350 K, where the crystal decomposed, exhibited alternate orientations in all but the 100 K data set.