When the unusual is the normal: making non-standard structural studies routine.

A Wheaton[1], I Guzei[2]
[1]University of Wisconsin at Madison Department of Chemistry, Madison, WI, [2]Chemistry, UW Madison
amwheaton@wisc.edu

We present several problematic small-molecule structures that required non-routine handling of data collection, processing and/or structural refinement. The structure of an octahedral Ni(II) complex could not be properly refined until the crystal was identified as a pseudo-merohedral twin. A highly absorbing complex [C[sub]6[/sub]H[sub]5[/sub]CH[sub]2[/sub]CH[sub]2[/sub]NH[sub]3[/sub]][sub]2[/sub][CH[sub]3[/sub]NH[sub]3[/sub]][Pb[sub]2[/sub]I[sub]7[/sub]] (μMo = 25.54 mm[sup]-1[/sup]) required a careful choice of crystal, radiation source, and subsequent positional disorder modelling. A sample of an organic salt contained non-merohedrally twinned crystals only; upon exploring multiple absorption correction choices with TWINABS, the structural model necessitated use of the Platon SQUEEZE protocol for modeling of disordered solvent electron density. A Co(II) complex required lowering the crystallographic symmetry from space group [i]C[/i]2/[i]c[/i] to [i]C[/i][i]c[/i], identifying solvent of crystallization, and then finally establishing the chemical identity of the metal center. Finally, a chiral [i]P[/i]1 organic salt whose crystals decomposed due to radiation damage during the X-ray measurement required two data collections to achieve a data set that had a sufficient data completeness for the subsequent structural refinement process.