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Re-investigating the structures of $[Cu(NO_3)_2(en)_2]$ and $[Cu(NO_3)_2(pn)_2]$; Tales of twinning and a reversible phase change.

The structure of *trans*-bis(1,2-diaminoethane)-dinitrato-copper(II), $[Cu(NO_3)_2(en)_2]$, has been reported twice previously,[1] including once as a private deposition to the CSD. On both occasions the data were collected at room temperature with a 4-circle serial diffractometer and the R factors at ca. 4% suggest there is little more to understand. Our recent re-determination at low temperature on a CCD area detector system revealed a twinned diffraction pattern. We will describe the handling of the twinning and hence a halving of the R factor.

The structure of *trans*-bis(1,2-diaminopropane)-dinitrato-copper(II), $[Cu(NO_3)_2(pn)_2]$, has also been reported twice previously.[2,3] As above, both determinations were at room temperature on serial diffractometers. The first determination did not include H atoms and had a R factor of 12.2%. The second however, did include H atoms and refined to R1 = 3.3%. A close inspection of the published ORTEP plot however, with hindsight, now provides clues to our new findings. A reduction in temperature leads to a loss of molecular symmetry and, primarily, a significant movement of the nitrate ligands. On re-warming the crystal the original structure is obtained, albeit with a little de-lamination of the crystal. We will describe our experiments and describe the structural changes observed.

References

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