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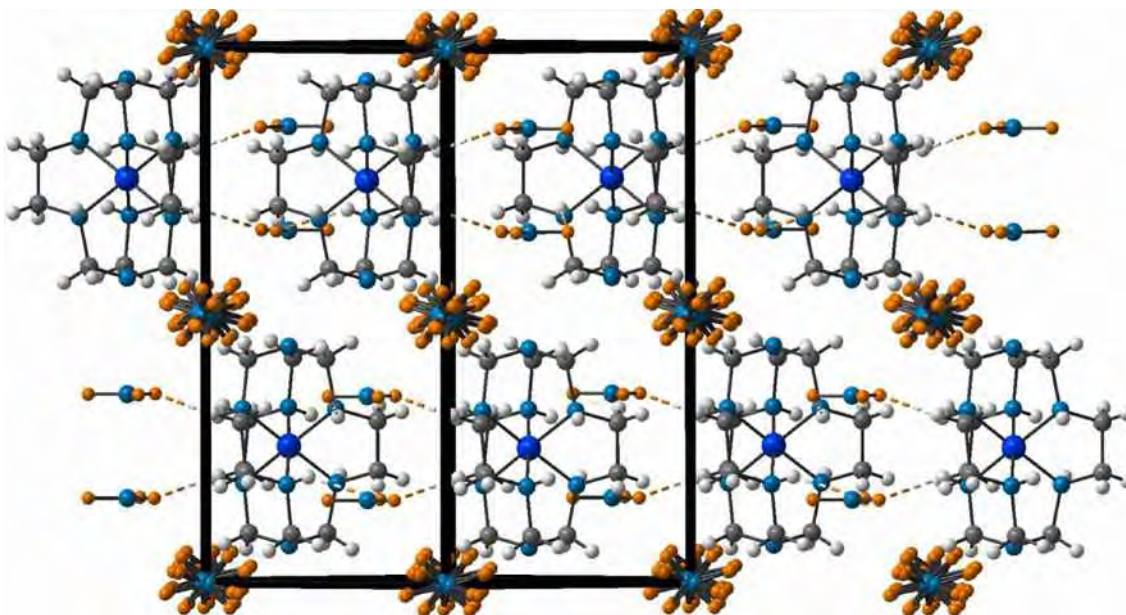
Disorder, modulation and twinning in Λ -Co(sepulchrate) trinitrate

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The organic compound Λ -Co(sepulchrate) trinitrate, $[\text{C}(12)\text{H}(18)\text{N}(8)\text{Co}](3+) \cdot 3[\text{NO}(3)](-)$, exhibits at room temperature a disordered structure in symmetry $P6(3)22$ [1,2]. The Co(sepulchrate) cation and two of the nitrate anions, all centred on three fold rotation axes, are linked via dense N–H \cdots O hydrogen bond networks, the third nitrate anion, centred on the intersection of the two fold rotation axes, shows orientational disorder (see Figure). Three phase transitions have been observed upon cooling by means of light microscopy and spectroscopic measurements [1] and by single crystal neutron diffraction [3] at $T(1) = 133$ K, $T(2) = 106$ K and $T(3) = 98$ K. These phase transitions are interpreted as ordering of the disordered nitrate anions [1] and as reduction of symmetry from hexagonal to orthorhombic associated with twinning. The appearance of satellite reflections in the diffraction pattern at $T(1) = 133$ K indicates a modulated structure; as the positions of those satellite reflections are temperature dependent [3], the modulation is incommensurate. By single crystal X-ray diffraction at beam lines D3 and F1 of Hasylab (DESY, Hamburg) at different low temperatures we found that all observed peaks are indexable in an hexagonal setting and two q -vectors $q(1)=(\sigma,\sigma,0)$ and $q(2)=(-2\sigma,\sigma,0)$ with $\sigma \approx 0.0882$. This setting is compatible with a three-fold orthorhombic twinning and one q -vector $q(\text{orth})=(2\sigma,0,0)$ for each of the three twin domains, which allows to reduce the symmetry. Structure refinement of all three low temperature phases allows to set them into relation to each other.

[1] L. Dubicki, J. Ferguson, B. Williamson, *J. Phys. Chem.*, 1984, 88, 4254-4258, [2] A. Schönleber, S. van Smaalen, F. K. Larsen, *Acta Cryst.*, 2010, C66, m107-m109, [3] F. K. Larsen, P. Jørgensen, R. G. Hazell, et al., in *Molecular structure: Chemical reactivity and biological activity*, Stezowski et al. (ed.) IUCr, Oxford University Press, 1988, 499-504



Keywords: superspace, incommensurate modulation, phase transition