Poster Presentation

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Old structures revisited: Better insights from new data

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The need for improving the description of structural features with better quality data, at low temperature and with modern 2D detectors of certain materials, sometimes leads to surprisingly new insights into a previously reported structure. When attempting to grow single crystals of maleamic acid, good crystals of ammonium maleate, NH4(Mal), were obtained. Although the structure of this material has been reported at room temperature, in space group Pbcm with V=613.2(5) Å³ [1], synchrotron data were collected at low temperature to examine the behavior of the ammonium moiety. The data collected lead to a structure better described in Pbca, a=8.9687(12), b=8.1604(8), c=16.348(2) Å, V=1196.5(2) Å³, Z=8. The refinement converged to R=0.0438, wR2=0.1156, S=1.02. An examination of the new data indicates that reflections with h odd are systematically weak but, nevertheless, present. The Ca derivative of valproic acid (a common anticonvulsant) was reported as monoclinic, C2/c, with a=16.250(8), b=18.471(17), c=7.729(7) Å, β=109.71(5)°, V=2183.97 Å³, Z=4, and R=10.94% [2]. However, data collection at room temperature and under a stream of nitrogen on several newly prepared crystals always lead to a triclinic, P-1 cell, with approximately half the volume of the reported cell. Attempts to index the dataset using the known monoclinic cell resulted in high uncertainties for the unit cell parameters and high Rint values since reflection spots showed splitting and diffuse scattering. The new cell had dimensions a=7.6995(4) Å, b=11.7444(6) Å, c=11.7708(6) Å, α=91.089(3)°, β=101.643(3)°, γ=102.041(3)°, V=1017.47(99) Å³, Z=2. Although the initial refinement was discouraging (R=0.1172, wR2=0.360, S=1.12) the analysis with PLATON indicated the presence of twining and, after considering the twin law, the refinement improved significantly (R=0.059, wR2=0.1472, S=0.99). Several examples where a new data collection resulted in interesting results will be presented.

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