

s2.m1.p1 Crystal structure of [Li(L-proH)(H₂O)]Cl – a high resolution study. M. Lutz, A.L. Spek, *Utrecht University, Bijvoet Center for Biomolecular Research, Dep. Crystal & Structural Chemistry, Padualaan 8, 3584 CH UTRECHT, The Netherlands.*

Keywords: charge density, lithium bond, datacollection.

Zwitterionic amino acids and peptides can react with inorganic metal salts to form neutral salts [1]. The metal cation binds to the negatively charged carboxylate group while the anion binds to the positive ammonium group. We describe here the crystal structure of the monohydrated adduct of lithium chloride and L-proline. This study extends earlier results obtained in normal resolution [2] to high resolution.

The compound crystallizes in the monoclinic space group P2₁. The lithium centers link the carboxylate groups to form a one dimensional chain along the crystallographic [0 1 0] direction. Thereby, carboxylate oxygen O1 coordinates to one lithium atom, while O2 binds to two lithiums. This results in significantly different C-O bond lengths. The fourth coordination position of the tetrahedral lithium is occupied by a water molecule.

Hydrogen bonds link these chains to form a two dimensional network parallel to the crystallographic b,c-plane, also involving the chloride anions. Temperature dependent crystal structure determinations show, that the expansion/contraction of the a-axis is higher than of the b and c-axis.

We describe here the results of the SHELXL (spherical) and the XD (multipole) refinements. Effects of different data collection (KappaCCD area detector, CAD4T point detector), evaluation (DENZO, EVAL14, HELENA, REFPK), merging and scaling (SHELXL, SORTAV) procedures are discussed. Thermal motion analysis and the Bader-type topological analysis are used to judge the results of the refinements, also with respect of phasing problems in the non-centrosymmetric space group. A comparison of the "lithium bonds" with "hydrogen bonds" in the structure is presented.

s2.m1.p2 Changes in structure and electron distribution between the high and low temperature phase of V₆O₁₃. J. Höwing, T. Gustafsson and J. O. Thomas. *The Ångström Laboratory, Inorganic Chemistry, Lagerhyddsvägen 1, Box 538, SE-751 21 Uppsala, Sweden.*

Keywords: charge spin densities.

The structure of the transition metal oxide V₆O₁₃¹, used as cathode material in Li-ion batteries², has been studied at low temperature. The structure can be described as distorted VO₆ octahedra sharing edges and corners, forming channels through which lithium ions can migrate. Up to twelve lithium can be inserted in each unitcell forming Li₆V₆O₁₃. To understand how the bonding electrons are distributed and to calculate the charge of the atoms, deformation electron density refinements can be done³.

Diffraction data has been collected at 160 K with a single-crystal diffractometer and the structure refined. Deformation electron density refinements were performed to provide information about covalent bonds, lone pairs and atomic oxidation states. Oxygens are found to have a negative charge between -0.2 e and -0.7 e; vanadium atoms carry a positive charge in the range 0.8 e to 1.0 e. Covalent bonding is found to dominate in the structure.

At about 150 K the compound undergoes a phase transition where all the atoms move out from a mirror plane, thus lowering the symmetry. The structure has been reported with the space group P 2₁/a⁴. For this low temperature phase data has been collected at 95 K. An attempt to make deformation electron density refinements will be done in order to see the changes in electron configuration between the to phases.

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