PS07.02.02 LOW TEMPERATURE NEUTRON AND X-RAY DIFFRACTION STUDIES OF Mn(eth)Cu(oxpn)(CF3SO3)2

The present investigation illustrates the complementarity of neutron and x-ray diffraction for studies of disordered and hydrogen-bonded heterodinuclear compounds. This has been performed to provide nuclear structure factors and to form the basis for a spin distribution study of Mn(eth)Cu(oxpn)(CF3SO3)2.

A neutron diffraction study at 40 K was first attempted; however, it was not possible to define all parameters. The number of atoms was 10 ions/unit cell including 48 hydrogen atoms per ion. The structure was solved by direct methods using SHELXTL (1) and two uncoordinated triflate anions were found to be disordered. We therefore believe that a combination of x-ray and neutron diffraction data was the best way to solve this problem.

The unconventional space group P21/a was used to be consistent with the room temperature study (3). The lattice parameters at low temperature are a = 17.525(3), b = 17.555(4), c = 12.804(2) Å, and β = 104.97(2)° with Z = 4. The structure consists of an oxamido-bridged MnCu²⁺ part and two uncoordinated triflate anions. The Mn(II) ion is in an elongated octahedral surrounding, and the Cu(II) ion is in a square-planar surrounding. The intermolecular Mn—Cu separation is 5.440(1) Å whereas the shortest intermolecular metal-metal (Mn—Cu) is equal to 7.825(1) Å.

The number of atoms was not possible to determine due to disorder. Mn—O: 1.908 and 2.448 Å for (1); 1.910 and 3.355 Å for (2). Coordination geometry around Mn atoms in these two complexes is a distorted trigonal bipyramidal fashion by two methyl groups (a), two oxo substituents (a) and the N atom of the amine (equatorial). In 1-3 the axial Mn-O bond lengths are significant shorter than the equatorial one. In 1, the center of the Al2O2 ring coincides with a crystallographic inversion center (space group P21/c). The absolute structure of 2 (space group P21/c) was found to be (S) (Flack parameter = 0.04). Compound 3 crystallizes with two non-interacting molecules of toluene per formula unit.

Figure 1: Molecular structure of (S)-(+-)-amino-2-propoxide 2

PS07.02.03 STRUCTURES OF THREE NOVEL DINUCLEAR COMPLEXES OF Eu(III), Pr(III) and Nd(III) WITH BRIDGING ALANINEHYDROXYAMIC ACID

The structures of bis(α-L-α-alaninehydroxyamic acid-1,2kO,1kO')bis(hexaaquaeuropium)(6+)tetracarbonylcobalt(1)-Eu(DL-αAlaH₂O)(ClO₄)·6H₂O (1), and the isostuctural complexes of neodymium (2), and praseodymium (3) have been determined. The compounds were synthesized by J. Legzdins and P. Gavryszewski at the Inorganic Chemistry of the Wrocław University and this study is a part of a broader investigation of novel complexes of lanthanides as model substances in biological systems.

The investigated complexes are binuclear, the lanthanide ions coordinate 9 oxygen atoms: 6 water molecules, one oxygen atom of the carboxyl group and two bridging oxygen atoms of hydroxyamic groups. Unexpectedly the bridging oxygen atom in the complexes is not the carboxyl-oxygen atom, but the hydroxamic oxygen atom. X-ray data were collected with a KM-4 diffractometer with Mo Kα radiation at 293 K. The structures were solved by direct methods and refined by full-matrix least squares using SHELXL-XL PC programs, on F values, with anisotropic temperature factors for non-hydrogen atoms. Positions of hydrogen atoms were taken from σF syntheses and refined with isotropic temperature factors. The space group of the isostuctural complexes is P2₁/a (Z=4) and the crystal data and R factors are given below:

<table>
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<tr>
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<tbody>
<tr>
<td>1</td>
<td>10.04(2)</td>
<td>11.31(2)</td>
<td>16.98(3)</td>
<td>95.90(3)</td>
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<tr>
<td>2</td>
<td>10.35(2)</td>
<td>11.32(2)</td>
<td>16.87(4)</td>
<td>95.90(3)</td>
</tr>
<tr>
<td>3</td>
<td>10.46(2)</td>
<td>11.32(1)</td>
<td>16.96(3)</td>
<td>95.90(3)</td>
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On the basis of the author's results and of the Cambridge Structural Database, the crystal chemistry of the investigated compounds in comparison with known lanthanides complexes will be discussed in detail.

PS07.02.04 CRYSTALSTRUCTURES OF ORGANO METALLIC ALUMINUM AMINO-ALKOXIDES T. Gelbrich, J. Sieler, E. Hecht, U. Dümichen

Aluminum compounds can be stabilized by donor ligands. This stabilization is especially pronounced when chelating ligands are present [1].

Compounds of general formula Me₂AlOR* [OR* = (+)-(+)-dimethylamino-2-propoxo] 1, (S)(+)-amino-2-propoxo 2, (±)-2-piperidylmethoxo 3 have been characterized by single crystal X-ray structure analysis at 200 K. In the solid state, compounds 1-3 are dimeric.

Two binuclear manganese(III) complexes containing bridging ketonic oxygen atom, Shi-Xiong Lu* and Yun-Long Feng, Department of Chemistry, Fuzhou University, Fuzhou, Fujian 350002, China.

Two binuclear manganese(III) complexes containing bridging ketonic oxygen atom have been characterized by X-ray crystallography. They are [Mn₂(bazacen)₂(MeOH)₂(CIO₄)₂] (1) and [Mn₂(bazacen)₂(MeOH)₂(CIO₄)₂]*2H₂O (2), where bazacen² is NN'-ethylenebis(1-phenyl-1-imino-1-butanone). These Schiff base compounds crystallize in Space group P 2₁/n for (1) and P bca for (2). Coordination geometry around Mn atoms in these two complexes is a distorted octahedron with an equatorial plane N₂O₂ from the bazacen², while the axial sites are occupied by two oxygen atoms of methanol molecules. The very strong axial elongation has usually found in a few complexes, for example, Mn-O(axial) bond being 2.493 Å in (1) and 2.448 Å in (2).

The [Mn₂(bazacen)₂(MeOH)₂]²⁺ cation in these complexes is a centrosymmetric dimer where two Mn(III) ions are joined by two ketonic oxygen atoms (Mn-O: 1.908 and 2.448 Å for (1); 1.910 and 2.493 Å for (2)). Therefore, MnO₂ cores in the dimers being a parallellogram with Mn—Mn separation (3.387 Å for (1) and 3.355 Å for (2)) and Mn-O-Mn angles (99.7° for (1) and 100.0° for (2)). It was reported in Mn Schiff base complexes that the bridging oxygen atoms were from the μ₂-O₂⁻ anion, μ₂-phenoxo, and μ₂-alkoxo. To our knowledge, the two structures are the first structure containing bridging ketonic oxygen atom in Mn Schiff base complexes.