PS07.02.02 LOW TEMPERATURE NEUTRON AND X-RAY DIFFRACTION STUDIES OF Mn(eth)Cu(Ox)(CF3SO3)2 (eth = (2)-5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazacyclocotetradecane)(OX)n=5,6,N,N'-bis(triminopyroxyl)oxamide, V. Baron, H. Rundlöf, R. Tellgren, B. Gillon, O. Kahn, NPL Studsvik, S-611 82 Nyköping, Sweden, Msc. of Chem. Uppsala University, Box 531, S-751 21 Uppsala, Sweden., ELL, CEA-CNRS, CEN Saclay, 91191 Gif-sur-yvette cedex, France, 4ICMCB, Château de Brivaizac, 33600 Pessac, France.

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(Ox)(CF3SO3)2.

A neutron diffraction study at 40 K was first attempted; however, it was not possible to refine all parameters. The number of atoms was 100 atoms/unit cell including 48 hydrogen atoms and the 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 = 17.752(5), b = 17.555(4), c = 12.804(2) Å, and β = 104.97(2)° with Z = 4. The structure consists of an oxamide-bridged MnCu5 part and two uncoordinated triflate anions. The Mn(II) is in an elongated octahedral surrounding, and the Cu(II) in a square-planar surrounding. The intramolecular Mn-Cu separation is 5.440(1) Å whereas the shortest intermolecular metal-metal (Mn-Cu) is equal to 7.825(1) Å.

(2) V.Baron these de l'université de Paris XI -Orsay (10 December 1994).

PS07.02.03 STRUCTURES OF THREE NOVEL DINUCLER COMPLEXES OF Eu(III), Pr(III) and Nd(III) WITH BRIDGING ALANINEHYDROXYAMIC ACID. By Zdzisław Galdecki, Institute of General and Ecological Chemistry, Technical University of Łódź, ul. Zwirki 36, 90-924 Łódź, Poland.

The structures of bis(m-D,L-α-hydroxyamid acid-1,2kO1kO')bis(hexaammineeuropium)(6+)tertacarboxylate(1-), [Eu(D,L-αAla)(H2O)6][ClO4]2 (1), and the isosstructural complexes of neodymium (2), and praseodymium (3) have been determined. The compounds were synthesized by J. Legzdinskiwicz and P. Gawrzewszkwa at the Institute of 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 atoms coordinate 9 oxygen atoms: 6 water molecules, one oxygen atom of the carboxyl group and two bridging oxygen atoms of hydroxamic 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 on 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 SHELXTL-PC programs, on F values, with anisotropic temperature factors for non-hydrogen atoms. Positions of hydrogen atoms were taken from Δψ syntheses and refined with isotropic temperature factors. The space group of the isosstructural complexes is P21/c (Z=4) and the crystal data and factors are given below:

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<tr>
<td>1</td>
<td>10.245(2)</td>
<td>11.312(2)</td>
<td>16.987(3)</td>
<td>95.90(3)</td>
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<tr>
<td>2</td>
<td>10.317(5)</td>
<td>11.332(4)</td>
<td>16.876(4)</td>
<td>95.90(3)</td>
<td>0.046</td>
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<tr>
<td>3</td>
<td>10.464(2)</td>
<td>11.320(1)</td>
<td>16.963(3)</td>
<td>95.90(3)</td>
<td>0.035</td>
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</table>

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.

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

PS07.02.04 CRYSTAL STRUCTURES OF ORGANO METALLIC ALUMINUM AMINO-ALKOXIDES T. Gelbrich, J. Sieler, E. Hecht, a) Institut für Anorganische Chemie der Universität, D-64103 Leipzig, Limnestr. 3. b) Institut für Anorganische Chemie der Martin-Luther-Universität Halle-Wittenberg, D-06217 Merseburg, Germany.

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-propoxyl 1, (S)(+)-amino-2-propoxyl 2, (±)-2-piperidylmethoxyl 3] have been characterized by single crystal X-ray structure analysis at 200 K. In the solid state, compounds 1-3 are dimeric. The central four-membered Al2O2 rings are planar and each aluminum is coordinated in a distorted trigonal bipyramidal fashion by two methyl groups (axial), two alkoxy substituents (axial and equatorial) and the N atom of the alminocarboxylate (equatorial). In 1-3 the Al-O bond length is 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.


PS07.02.05 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(bacen)2(MeOH)2](ClO4)2 (1) and [Mn2(bacen)2(MeOH2)](ClO4)2*2H2O (2), where bacen2- is N,N'-ethylenebis(1-phenyl-3-imino-1-butanonato). 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 complexes is a distorted octahedron with an equatorial plane coincides with a crystallographic inversion center (space group P21/n). The absolute structure of 2 (space group P21/c) was found to be (S) (Flack parameter = 0.04). Therefore, Mn=O cores in the dimers being a parallelogram 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-phenoxy, and μ2-alkoxo. To our knowledge, the two structures are the first structure containing bridging ketonic oxygen atom in Mn Schiff base complexes.