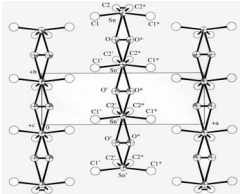


We took a X-ray structure analysis of title compound to get a information of incommensurateness using a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite Mo-K α radiation.

As this compound is very unstable in air, crystal was sealed in a capillary under nitrogen atmosphere in a glove box.

Crystal data of (CH₃)₃SnOH: Orthorhombic, *Pmmn*2₁, *a* = 11,207(1) Å, *b* = 4.171(1) Å, *c* = 6.652(1) Å, *V* = 310.9(1) Å³, *Z* = 2, *R* = 0.140.

The obtained crystal structure is shown in figure. This complex has an infinite chain structure through the bridging trimethyltin groups by hydroxide groups along the *b* axis. The co-ordination of each tin atom is approximate trigonal bipyramidal with two hydroxide groups and three methyl groups essentially. Due to the mirror symmetry perpendicular to the *a* axis passing through the tin atom, this complex takes the disordered structure consequently.



[1] Kasai N., et al., *J. Organometal. Chem.*, 1965, **3**, 172.

Keywords: tin, hydroxide, disorder

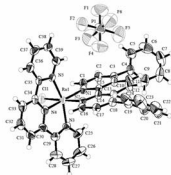
P.07.01.33

Acta Cryst. (2005). A61, C302

Crystal Structure of [RuCl(dpphen)(terpy)]PF₆ and [Ru(CH₃CN)(phen)(terpy)](PF₆)₂

Naokazu Yoshikawa^a, Hiroshi Takashima^a, Keiichi Tsukahara^a, Nobuko Kanehisa^b, Yasushi Kai^b, ^a*Department of Chemistry, Faculty of Science, Nara Women's University, Japan.* ^b*Department of Applied Chemistry, Graduate School of Engineering, Osaka University, Japan.* E-mail: naokazuu@dg.mbn.or.jp

The desired complex was prepared by a sequential procedure with ligand replacement. [RuCl₃]·H₂O and 2,2':6',2''-terpyridine (terpy) were mixed in ethyleneglycol (15 ml). The suspended mixture was refluxed for 5 min. in a microwave oven under a purging nitrogen atmosphere. 4,7-Diphenyl-1,10-phenanthroline (dpphen) was added to the refluxing red solution for 10 min. A saturated aqueous solution of KPF₆ (20 ml) was added, and a black-red product began to precipitate. We examined complexes **1** and **2** using X-ray analysis, CV, and UV.



Crystal data of [RuCl(dpphen)(terpy)]PF₆ (**1**) [1] Monoclinic, *C2/c*, *a* = 42.071(1) Å, *b* = 8.6042(1) Å, *c* = 19.9088(1) Å, β = 96.096(2), *V* = 7165.9(2) Å³, *Z* = 8, *R*(F₂) = 0.083, *wR*(F₂) = 0.201 for 28548 measured reflections.

Crystal data of [Ru(CH₃CN)(phen)(terpy)](PF₆)₂ (**2**) [2] Triclinic, *P* $\bar{1}$, *a* = 8.7861(3) Å, *b* = 10.3590(9) Å, *c* = 17.9636(7) Å, α = 99.192(7), β = 90.389(2), γ = 105.774(3), *V* = 1551.0 (2) Å³, *Z* = 2, *R*(F₂) = 0.089, *wR*(F₂) = 0.172 for 14369 measured reflections.

[1] Yoshikawa N., et al., *Acta Cryst.*, 2005, **E61**, m545-m547. [2] Yoshikawa N., et al., *Acta Cryst.*, 2005, **E61**, m55-m56.

Keywords: microwave, terpyridine, ruthenium compounds

P.07.01.34

Acta Cryst. (2005). A61, C302

Structural Studies on Copper(II) Carboxylate Complexes containing Pyrazole

Magda Monari^a, Luciano Pandolfo^b, Claudio Pettinari^c, ^a*Department of Energy Engineering, University of Bologna, Italy.* ^b*Department of Chemical Sciences, University of Padova, Italy.* ^c*Department of Chemical Sciences, University of Camerino, Italy.* E-mail: magda.monari@unibo.it

Polynuclear copper derivatives are the subject of an increasing number of studies [1, 2]. Trinuclear copper derivatives characterized by the presence of the triangular core [Cu₃(μ_3 -OH)(μ -pz)₃(RCOO)₂ (*R* = H, C₂H₅, C₃H₇)] have been prepared and characterized by X-ray studies. Copper(II) formate gives [Cu₃(μ_3 -OH)(μ -pz)₃(HCOO)₂-(Hpz)₂], **1**, whereas copper propionate and butyrate afford [Cu₃(μ_3 -

OH)(μ -pz)₃(C₂H₅COO)₂-(EtOH)], **2**, and [Cu₃(μ_3 -OH)(μ -pz)₃-(C₃H₇COO)₂(MeOH)(H₂O)], **3**, respectively, both containing solvent molecules coordinated to the copper atoms.

Here we report a comparison of their geometries and their supramolecular architectures which show the formation of interesting 1D coordination polymers.

[1] Solomon E.I., Sundaram U.M., Machonin T.E., *Chem. Rev.*, 1996, **96**, 2563. [2] La Monica G., Ardizzoia G.A., *Progr. Inorg. Chem.*, 1997, **46**, 151.

Keywords: copper complexes, X-ray crystal structure, carboxylates

P.07.01.35

Acta Cryst. (2005). A61, C302

Crystal Packing of Three-Aqua Sodium Maleate

Agneš Kapor^a, Suzana Cakić^b, Thomas Armbruster^c, Vladimir Divjaković^d, Bela Ribar^d, ^a*Department of Physics, Faculty of Sciences, University of Novi Sad, Serbia Montenegro.* ^b*Faculty of Technology, University of Nish, Leskovac, Serbia Montenegro.* ^c*Laboratory for Chemical and Mineralogical Crystallography, CH-3012 Bern.* ^d*Academy of Sciences and Arts of Vojvodina, Serbia Montenegro.* E-mail: akapor@uns.ns.ac.yu

Three-Aqua Sodium Maleate was crystallized as a side product in the process of the synthesis of the manganese complexes with mixed ligands [1]. The crystal of NaC₄H₅O₇ belongs to the space group *P*-1 with unit cell parameters *a* = 5.9609(14) Å, *b* = 6.3907(16) Å, *c* = 11.2308(27) Å, α = 104.178(4)°, β = 91.574(4)°, γ = 100.241(4)° and *D*_x = 1.567 Mg m⁻³ for *Z* = 2. The maleate monoanion is the ligand bonded through an oxygen atom to the Na⁺ ion. The intramolecular O-H...O hydrogen bond in the maleate monoanion was analysed and compared with literature data [2,3]. The central Na atom is surrounded by six oxygen atoms and forms an octahedral polyhedron with the shape of a deformed quadratic bipyramid. Molecules built in this manner form a polymerized infinite chain along the *a* crystallographic axis. Maleate monoanions alternate with Na ion chains and form a layer in the *ac* crystallographic plane. Viewed along the *a* axis, the packing shows that two chains are related by the inversion center, so that they form a channel along the crystallographic *a* axis.

[1] Stamenković J., Cakić S., Nikolić G., *Chemical Industry*, 2003, **57**, 559. [2] James M.N.G., Williams G.J.B., *Acta Crystallogr.*, 1974, **B30**, 1249. [3] James M.N.G., Williams G.J.B., *Acta Cryst.*, 1974, **B30**, 1257.

Keywords: coordination geometry, hydrogen bonds, crystal packing

P.07.01.36

Acta Cryst. (2005). A61, C302

Structural Parameters of Several Lanthanide Clusters

Dale Swenson, Chang-Tong Yang, Donald Nolting, Louis Messerle, *Department of Chemistry, University of Iowa, Iowa City, Iowa, 52242 USA.* E-mail: dale-swenson@uiowa.edu

This work presents key structural parameters for a series of amino acid-chelated μ_3 -hydroxide-containing polygadolinium (and lanthanide analog) complexes with potential as high relaxivity MRI contrast agents. The structures of perchlorate salts of *D*_{4h}-symmetry Ln₄(μ_4 -OH)₂(μ_3 -OH)₁₆(OH₂)₈(serine)₂₀³⁺, *D*_{3h}-symmetry Ln₁₅(μ_5 -Br)(μ_3 -OH)₂₀(μ_3 -histidinate)₁₅(μ -OH₂)₈(OH₂)₂₀⁹⁺, *T*_d-symmetry Ln₄(μ_3 -OH)₄(valine)₄(OH₂)₈⁸⁺ and Ln₂(alanine)₄(OH₂)₈⁶⁺ complexes will be presented.

Structural parameters (Gd-O and Gd...H distances, Gd-water tilt angles, and H-bonding) that may play a significant role in MRI relaxation mechanisms for these novel complexes will be presented. We will collect neutron diffraction data on the dysprosium analog of one of the complexes at the Intense Pulsed Neutron Source (IPNS) at Argonne National Laboratory in April and hope to include these results.

This research is supported in part by NIH/NIBIB, the Roy J. Carver Charitable Trust and DOE (grant of time on IPNS).

Keywords: clusters, lanthanides, materials magnetic resonance imaging