

dipolar zwitterionic form. The Cu(II) complexes seem to crystallize as isolated entities since only intramolecular H-bonds are found. However two disordered water positions can be found in a Fourier difference map, and two extra oxygen atoms were included in the refinement so that their occupancy adds to unity.

Keywords: X-ray crystallography of coordination compounds, copper coordination compounds, single crystals

P.07.01.28

Acta Cryst. (2005). A61, C301

Spectroscopic and Structural Investigation of $ZnI_2(\text{nicotinamide})_2$, $[Zn(\text{H}_2\text{O})_2(\text{picolinamide})_2]I_2$ and $ZnI_2(\text{isonicotinamide})_2$

Hümevra Paşaoğlu, Semra Güven, Orhan Büyükgüngör, *Ondokuz Mayıs University, Department of Physics, Samsun-Turkey*. E-mail: hpasa@omu.edu.tr

The zinc(II) complexes of nicotinamide, picolinamide and isonicotinamide with iodide were synthesized and characterized by FT-IR and XRD techniques. In both the nicotinamide (na) and isonicotinamide (iso) Zn(II) complexes the Zn(II) ion is coordinated by two iodide ligands and two N atoms either of na or of iso ligands in a distorted tetrahedral coordination environment. The zinc(II) complex with picolinamide (pa) has a different environment having a stable five-membered chelate coordination through the ring N and O atoms of pa ligand and has a slightly distorted octahedral geometry.

The $ZnI_2(\text{na})_2$, $[Zn(\text{pa})_2(\text{H}_2\text{O})_2]I_2$ and $Zn(\text{iso})_2I_2$ complexes are all crystallized in monoclinic system with space groups $C2/c$, $P2_1/n$, $C2/c$, respectively. All these complexes are stabilized through intermolecular hydrogen bondings together with $\pi \dots \pi$ interactions.

Keywords: IR and XRD, zinc complexes, amides

P.07.01.29

Acta Cryst. (2005). A61, C301

Green-yellow Thermochromism of (N-methyl-2,6-lutidinium) $_2$ CuCl_4

Marcus R. Bond, Allison Gerdes, Annette F. Kelley, *Department of Chemistry, Southeast Missouri State University, Cape Girardeau, MO*. E-mail: bond@mbond2.st.semo.edu

The previously reported, green, room temperature phase of (N-methyl-2,6-lutidinium) $_2$ CuCl_4 consists of layers of square-planar CuCl_4^{2-} anions interspersed with coplanar organic cations.

The temperature behavior of the compound was studied, and a thermochromic phase transition from green to yellow found at 67 °C. The crystal structure of the high temperature yellow phase of $(\text{C}_8\text{H}_{12}\text{N})_2\text{CuCl}_4$ was determined at 77(1) °C with unit cell parameters triclinic, $P\bar{1}$, $a = 7.9350(5)$ Å, $b = 9.1550(7)$ Å, $c = 16.144(2)$ Å, $\alpha = 75.467(4)^\circ$, $\beta = 86.975(4)^\circ$, $\gamma = 64.505(5)^\circ$, $V = 1022.64(15)$ Å³, $Z = 2$.

The structure of the high temperature phase consists of flattened CuCl_4^{2-} tetrahedra with the two unique organic cations now canted relative to one another rather than coplanar. The canting of the organic cations lengthens the short aromatic C-H...Cl contacts in the low temperature phase which appear to stabilize the square planar over the flattened tetrahedral anion geometry.

Previous examples of green-yellow thermochromism in the A_2CuCl_4 family have occurred only in the presence of strong N-H...Cl hydrogen bonding, which stabilizes the square planar anion geometry in the low-temperature phase. The title compound is the first known to exhibit this behavior in the absence of strong N-H...Cl hydrogen bonding.

Keywords: copper complexes, phase transitions, hydrogen bonding

P.07.01.30

Acta Cryst. (2005). A61, C301

Structure of Hydrido(carboxylato)Ir(III) Complexes and Catalytic Symmetric Hydrogenation of Imines

Tsuneki Yamagata, Kazunori Hoshida, Mitsuhiro Nagata, Kazushi Mashima, Kazuhide Tani, *Department of Chemistry, Graduate School of Engineering Science, Osaka University, Toyonaka, Osaka, Japan*.

E-mail: tyama@chem.es.osaka-u.ac.jp

Recently, we have succeeded in the isolation and characterization of $[\text{IrCl}(\text{binap})_2]_2$ **1** {binap = 2,2'-bis(di-phenylphosphino)-1,1'-binaphthyl}. [1] Complex **1** reacted easily with methanol and water to give the oxidative addition products. [2] Here we report on the highly diastereoselective oxidative addition of carboxylic acids to $[\text{IrCl}(\text{binap})_2]$, which gave mononuclear hydrido(η^2 -carboxylato)Ir(III) complexes.

Reaction of (*S*)-**1** with several carboxylic acids proceeded smoothly at r.t. to give $[\text{IrCl}(\text{H})(\text{O}_2\text{CR})\{(S)\text{-binap}\}]$ (*S*)-**2** (R = CH₃, *p*-tolyl, etc.) as air-stable almost colorless solids in good yields. The absolute configuration of $[\text{IrCl}(\text{H})(\text{O}_2\text{CC}_6\text{H}_4\text{CH}_3\text{-4})\{(S)\text{-binap}\}]$ was elucidated to be (*S*)-OC-6-23-A. Catalytic activity for these complexes for asymmetric hydrogenation of prochiral imines will also be discussed.

[1] Yamagata T., Iseki A., Tani K., *Chem. Lett.*, 1997, 1215-1216. [2] Yamagata T., Iseki A., Tani K., *Angew. Chem. Int. Ed.*, 1998, **37**, 3381-3383.

Keywords: absolute configuration, asymmetric catalysis, carboxylate complexes

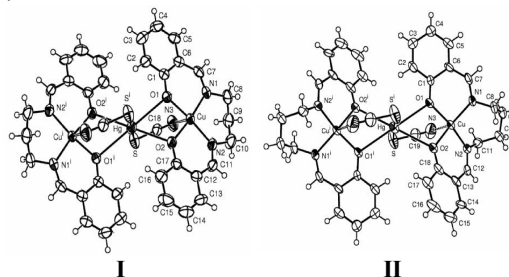
P.07.01.31

Acta Cryst. (2005). A61, C301

Synthesis, Characterization and Crystal Structure of new Transition Metal Compounds of Thiocyanate

Leyla Tatar Yıldırım¹, Raif Kurtaran², Orhan Atakol³, ¹*Hacettepe University, Department of Engineering Physics, Beytepe 06800, Ankara, Turkey*. ²*Department of Chemistry, Faculty of Arts and Science, University of Balikesir, Balikesir, Turkey*. ³*Department of Chemistry, University of Ankara, Ankara, Turkey* E-mail: tatar@hacettepe.edu.tr

Two similar hetero-trinuclear centrosymmetric Cu-Hg-Cu Schiff base complexes were prepared and characterized by elemental analysis, NMR, IR and UV-Vis spectroscopy. The crystal structures of the title compounds reveal that $\text{Hg}[\text{Cu}(\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_2)\text{SCN}]_2$ (I) crystallizes in the triclinic $P\bar{1}$ space group and $\text{Hg}[\text{Cu}(\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_2)\text{SCN}]_2$ (II) crystallizes in the monoclinic $P2_1/c$ space group. The central Hg atom of the compounds, which is located on an inversion centre, has a distorted octahedral coordination geometry. In both compounds, there are long range interactions between Cu and N atoms of the bridging SCN group at the axial position. The coordination geometry of the inversion-related terminal Cu atoms of the compounds is square-pyramidal. The Cu-Hg pairs are triple bridged by the O atoms of the Schiff base ligands and by the SCN groups. The Cu...Hg distance is 3.7623(9) Å (I) and 3.7778(17) Å (II).



Keywords: crystal structure, thiocyanate complexes, copper(II) and mercury(II) complexes

P.07.01.32

Acta Cryst. (2005). A61, C301-C302

Crystal Structure of Trimethyltin hydroxide, $(\text{CH}_3)_3\text{SnOH}$

Nobuko Kanehisa, Yasushi Kai, *Department of Materials Chemistry, Graduate School of Engineering, Osaka University, Japan*. E-mail: kanehisa@chem.eng.osaka-u.ac.jp

It has been reported that $(\text{CH}_3)_3\text{SnOH}$ has many incommensurate structures in crystalline state. So far, the precise structure analysis has not been reported. Only one X-ray crystal structure analysis without three dimensional coordinates available has been reported. [1]