

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

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

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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

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#### Green-yellow Thermochromism of (N-methyl-2,6-lutidinium) $_2$ $\text{CuCl}_4$

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The previously reported, green, room temperature phase of (N-methyl-2,6-lutidinium) $_2$   $\text{CuCl}_4$  consists of layers of square-planar  $\text{CuCl}_4^{2-}$  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,  $\alpha = 75.467(4)^\circ$ ,  $\beta = 86.975(4)^\circ$ ,  $\gamma = 64.505(5)^\circ$ ,  $V = 1022.64(15)$  Å<sup>3</sup>,  $Z = 2$ .

The structure of the high temperature phase consists of flattened  $\text{CuCl}_4^{2-}$  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  $\text{A}_2\text{CuCl}_4$  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

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

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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( $\eta^2$ -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 = CH<sub>3</sub>, *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

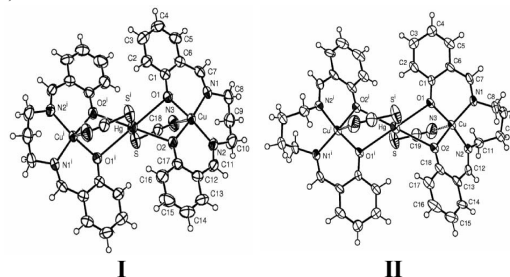
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#### Synthesis, Characterization and Crystal Structure of new Transition Metal Compounds of Thiocyanate

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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

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

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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]