

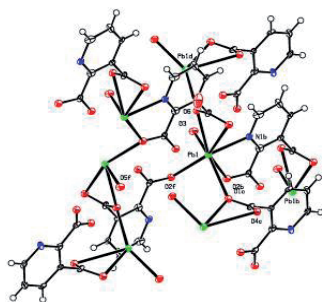
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Acta Cryst. (2011) A67, C617

Preparation and crystal structure lead(II) coordination polymers with pyridine-2,3-dicarboxylic acid

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An especially active research area in recent years has involved the preparation of metal-organic frameworks (MOFs) due to their tunable properties and functions. These compounds have been applied in many fields, such as molecular recognition, adsorption and separation processes, catalysis, ion exchange and molecular magnetism [1]. Polycarboxylate ligands have attracted an interest as the potential bridging ligands with a variety of connection modes with transition metal centers and abundant structural motifs [2]. pyridine-2,3-dicarboxylic acid often acts as a bidentate chelating ligand through nitrogen and one oxygen atom of one carboxylic group, while another carboxylate oxygen atom can act as a bridging atom between metal ions to form coordination polymer. Recently, We reported synthesis and crystal structure of Cd(II) and Co(II) polynuclear compounds with pyridine-2,3-dicarboxylic acid and have been reported [3]. In continuation of our research on the synthesis of metal complexes with pyridinedicarboxylic acids and heterocyclic amines such as 2-aminopyrimidine [4] or 2,2'-bipyridine [5], the reactions of lead nitrate with pyridine-2,3-dicarboxylic acid were done in the presence of 2-amino-6-methylpyridine. In this communication, we report synthesis and characterization of a coordination polymer with a main group element $\{[(\mu-2,3\text{-pydc})\text{Pb}]_2 \cdot \text{H}_2\text{O}\}_n$ (**1**). Compound **1** crystallizes in the monoclinic $C2/c$ space group. Polymeric chains of **1** is composed of Pb(II) ions bridged by pyridine-2,3-dicarboxylate ions (2,3-pydc) in N,O,O' fashion. Each Lead(II) ion is coordinated by two nitrogen and five oxygen atoms from four different 2,3-pydc ions and The distorted pentagonal bipyramidal coordination geometry is observed around the each metal ions. The two Pb^{II} atoms are bridged by 2,3-pydc ligands through their carboxylate groups to form a dimeric unit. As a result, the dimers are further connected by pdc ligands to furnish an extended neutral two-dimensional layer.



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Keywords: coordination polymer, lead complex, pyridine-2,3-dicarboxylic acid

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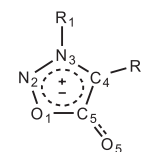
Substituent effects in the structural parameters of the sydnone ring

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Sydnone is a mesoionic 5-membered, heterocyclic compound, that demonstrates aromatic character and thereby is observed to undergo electrophilic substitution at the N(3) and C(4) positions. Of interest is the stability of the ring system and the influence of the substituents at the N(3) and C(4) positions on that stability.

The structure of three new sydnone compounds have been determined and are reported herein.

The new structures as well as structures previously determined in this laboratory, and those found in the Cambridge Structural Database [1] have been examined so as to attempt to understand the stability of the sydnone ring.



[1] F. H. Allen, *Acta Cryst.*, **2002**, *B58*, 380-388

Keywords: sydnone, structure, stability

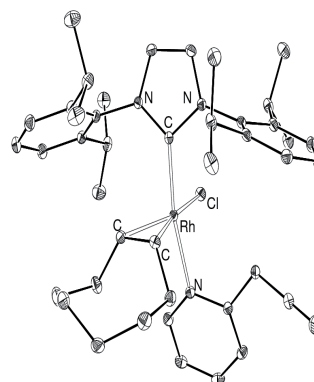
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Structural aspects of new n-heterocyclic carbene-rhodium catalysts

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Transition metal carbene complexes are currently tools of utmost relevance in organic chemistry and organometallics, as they exhibit appropriate properties as catalysts or intermediates in several highly valuable processes. 'Fischer-type' carbenes contain one or two heteroatoms directly connected to the metal-bonded carbon atom conferring greater stability to this kind of compounds. In these particular carbenes, the multiple bonding nature of the M-C link involves a synergetic donation from the carbon sp^2 hybrid orbital to the metal together with a back-donation from the metal. The whole interpretation of the details of the metal-carbene and the carbene-heteroatom interactions is usually evaluated from structural bonding parameters [1].



Currently we are exploring the use of N-heterocyclic carbenes in the chemistry of rhodium complexes. Our idea is to increase the stability of potential rhodium catalysts by incorporating this sort of electron-rich ligands, whereas we try to direct selectivity by introducing bulky substituents exerting steric control over the interaction of substrates with catalysts [2].

We have chosen the 1,3-bis-(2,6-diisopropylphenyl)imidazol-2-carbene (IPr) ligand in the synthesis of a family of rhodium complexes of the types $[\text{RhCl}(\text{IPr})\text{PyL}]$ or $[\text{RhCl}(\text{IPr})\text{L}]_2$ (Py = pyridine or substituted pyridines; L = ethylene, cyclo-octene, oxygen) (see figure). The additional presence in all these complexes of an unsaturated labile ligand L confers them a good suitability to be used as catalyst precursors in different processes [3]. In the present communication, we will discuss the structural features of these complexes trying to understand the relationships between these features with their chemical and catalytic behavior.

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Keywords: organometallic, carbene, rhodium complexes

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Molecular iodine trapped as alternating layers with amphiphilic salts

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Halogen bonding is a term used to describe a non-covalent interaction between a halogen atom acting as a Lewis acid and a Lewis base.¹ As the bonding parameters of this interaction are very predictable halogen bonding can be exploited to create some intriguing solid state structures using both halogen containing hydrocarbons (aliphatic or aromatic) and dihalogen species (eg. I₂).

We have investigated the insertion of molecular iodine (I₂) to amphiphilic salts where mono-alkylated DABCO (1,4-diazabicyclo[2.2.2]octane) moiety acts as the cationic species and the halogen bond acceptor. The solid state structures of the I₂⋯DABCO complexes will be presented in addition to the complexation reactions which include crystallization of the complexes from solutions as well as solid state complexation.

[1] P. Metrangolo, G. Resnati, *Chem. Eur. J.* **2001**, *7*, 2511-2519.

Keywords: halogen bonding, x-ray diffraction, amphiphilic salts

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Crystal Structure of PrAgAlGe_{3-x}

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A systematic investigation of the Pr-Ag-Al-Ge system led to the

discovery of a new quaternary compound.

An alloy of the nominal composition Pr_{0.125}Ag_{0.250}Al_{0.250}Ge_{0.375} was synthesized by arc-melting of the constituent elements under argon atmosphere. For homogenization the sample was annealed in an evacuated silica tube at 873 K for 720 h and subsequently quenched into cold water. X-ray powder diffraction data were collected on a diffractometer PANalytical (Cu K α radiation). The profile and structural parameters were refined using the WinCSD [1] program package.

X-ray phase analysis of the alloy indicated a three-phase sample. It contained: PrAgAlGe_{3-x} (87.32%), Ag₃Ge (9.26%) and Ge (3.42%). Automatic indexing of the peaks of the phase PrAgAlGe_{3-x} resulted in an *I*-centered tetragonal unit-cell with the parameters $a \approx 0.423(1)$ and $c \approx 2.489(1)$ nm. The crystal structure of the quaternary compound was solved in the space group *I4/mmm*. It was established that its crystal structure belongs to a new type. For the refined composition PrAg_{0.94}(¹)Al_{1.06(2)}Ge_{2.17(2)} (Pearson symbol *tI32*-8.70, space group *I4/mmm*) the unit-cell parameters are: $a = 0.42332(2)$, $c = 2.4902(1)$ nm. The final values of the reliability factors ($R_1 = 0.1068$, $R_p = 0.1690$, $R_{wp} = 0.1449$, $R_{dbw} = 0.0763$) were obtained for the following distribution of atoms: Pr $4e$ 0 0 0.14281(9) ($B_{iso} = 0.43(3) \cdot 10^{-2}$ nm²), 0.440(6)Ag + 0.560(6)Al $4d$ 1/2 0 1/4 ($B_{iso} = 0.68(4) \cdot 10^{-2}$ nm²), Ge1 $4e$ 0 0 0.4141(5), occ. 0.354(8), ($B_{iso} = 0.84(4) \cdot 10^{-2}$ nm²), 0.500(9)Ag + 0.500(9)Al $4e$ 0 0 0.3150(2) ($B_{iso} = 0.70(4) \cdot 10^{-2}$ nm²), Ge2 $16n$ 1/2 0.063(2) 0.0474(2), occ. 0.455(2), ($B_{iso} = 1.04(4) \cdot 10^{-2}$ nm²).

The crystal structure of PrAgAlGe_{3-x} is a quaternary substitution derivative of the closely-related types ScNi₂Si₃ [2] and UNi₂Si₃ [3] with splitting of one small-size atom site. The structure types ScNi₂Si₃ and UNi₂Si₃ differ by the distribution of the small-size atoms. In ScNi₂Si₃ the small-size atoms are distributed in an orderly manner, whereas in UNi(Ni,Si)₂Si₂ both $4e$ sites are occupied by a statistical mixture of Ni and Si atoms in the ratio 1:1. In comparison with these prototypes, in the structure of PrAgAlGe_{3-x} splitting of the $8g$ site takes place; the new $16n$ site is occupied by Ge atoms. Moreover, another kind of small-size atom distribution is observed. The crystal structure of PrAgAlGe_{3-x} (like the ScNi₂Si₃ and UNi₂Si₃ structure types) is an intergrowth of structural segments of the simple type BaAl₄ (or its ordered ternary variant CeAl₂Ga₂), which are separated along the crystallographic [001] axis by segments of the α -Po type.

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Keywords: alloy, X-ray powder diffraction, crystal structure

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Synthesis and crystal structure of mer-[Co(C₈H₄O₄)(C₃H₄N₂)₃(H₂O)₂] H₂O

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The design and synthesis of metal-organic frameworks (MOFs) have received attention in recent years due to their potential applications in diverse areas such as electrical conductivity, magnetism, host-guest chemistry, molecular separation, gas storage, sensors and catalysis [1][2].

Recently, great success has been achieved by the combination