**MS61.P32**  

**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 its carboxylate groups to form a dimeric unit. As a result, each metal ions are bridged by 2,3-pydc ligands to furnish an extended neutral two-dimensional layer. The two PbII ions bridged by pyridine-2,3-dicarboxylate ions (2,3-pydc) in monoclinic C2/c space group. Polymeric chains of I 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 coordination around each metal ions. The two PbII 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.

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

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**MS61.P33**  

**Substituent effects in the structural parameters of the sydnone ring**  
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Sydones are mesoionic 5-membered, heterocyclic compounds, that demonstrate aromatic character and thereby are 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.


**Keywords:** sydnone, structure, stability

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**MS61.P34**  

**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. 'Fisher-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 sp² 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].


**Keywords:** coordination polymer, lead complex, pyridine-2,3-dicarboxylic acid
Currently we are exploring the use of N-heterocyclic carbene 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 [RhCl(IPr)PyL] or [RhCl(IPr)L], (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.

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

MS61.P35

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. 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 complication reactions which include crystallization of the complexes from solutions as well as solid state complexation.


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

MS61.P36

Crystal Structure of PrAgAlGeₓ₁₋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 Pr₁₋ₓAgₓBiₓ₁₋ₓAl₁₋ₓGeₓ 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 Kr 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: PrAgAlGeₓ, (87.32%), Ag,Ge (9.26%) and Ge (3.42%). Automatic indexing of the peaks of the phase PrAgAlGeₓ, resulted in an I-centered tetragonal unit-cell with the parameters a = 0.423(1) Å and c = 2.489(1) Å. 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 PrAgₓₓ₁₋ₓAl₁₋ₓBiₓ₁₋ₓGeₓ (Pearson symbol m32-8.70, space group I₄/mmm) the unit-cell parameters are: a = 0.4232(2) Å, c = 2.4902(1) Å. The final values of the reliability factors (R.I. = 0.1068, R.I. = 0.1690, R.w = 0.1449, R.1 = 0.0763) were obtained for the following distribution of atoms: Pt 4e 0 0 0 1.4281(9) (B(I) = 0.43(3)Å²; 10⁻² Å²), 0.440(6)Ag + 0.560(6)Al 1/2 0 1/2 1/4 (B(Å) = 0.68(4)Å²; 10⁻² Å²), Ge 1e 0 0 0 0.0414(5), occ. 0.354(8), (B(Å) = 0.84(4)Å²; 10⁻² Å²), Ag 0.500(9)Ag + 0.500(9)Al 0 0 0 0.3150(2) (B(Å) = 0.70(4)Å²; 10⁻² Å²), Ge 2e 1/2 1/2 0.063(2) 0.0474(2), occ. 0.455(2), (B(Å) = 1.04(4)Å²; 10⁻² Å²).

The crystal structure of PrAgAlGeₓ, is a quaternary substitution derivative of the closely-related types ScNiₓSi₁₋ₓ [2] and UNiₓSi₁₋ₓ [3] with splitting of one small-size atom site. The structure types ScNiₓSi₁₋ₓ and UNiₓSi₁₋ₓ differ by the distribution of the small-size atoms. In ScNiₓSi₁₋ₓ, the small-size atoms are distributed in an orderly manner, whereas in UNiₓ(Ni,Si)Si, 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 PrAgAlGeₓ, 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 PrAgAlGeₓ, (like the ScNiₓSi₁₋ₓ and UNiₓSi₁₋ₓ structures) is an intergrowth of structural segments of the simple type BaAl₃ (or its ordered ternary variant CeAl₃,Ga₃), which are separated along the crystallographic [001] axis by segments of the α-Po type.


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

MS61.P37

Synthesis and crystal structure of mer-[Co(C₃H₇O)₃(H₃N₃)][H₂O] A. Benkanoun,a Balegroune Fadéla,a Guerbah-Laidou Achour,a Dahautou Slimane,a Leconte Claude,a Laboratoire de Cristallographie-Thermodynamique, Faculté de Chimie, USTHB, BP32, El-Alia, Alger, (Algérie). b CRM, CNRS-UPRESA 7036, Université Henry Poincaré, Faculté des Sciences et Techniques, BP 70239, 54506 Vandoeuvre Les Nancy Cedex, (France). E-mail: abenkanoun@gmail.com

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