MS27 P07

Complexes of M(carbaldehyde-oxime)(M=Pd and Hg): Synthesis and Structural Characterization

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Syntheses and X-ray crystal structure analyses of Hg(II) and Pd(II) complexes with pyridine-2-carbaldehyde-oxime ligand are reported. The complexes are obtained from the reaction of simple Hg(II) and Pd(II) salts ($PdCl_2$ and $HgCl_2$) with pyridine-2-aldoxime (HL). The molecular structures of complexes have been determined by single-crystal X-ray analyses. Structural characterization reveals in Hg(II) case the presence of HgL_2 complex but in Pd(II) case the formation of PdL(HL)Cl complex is observed.

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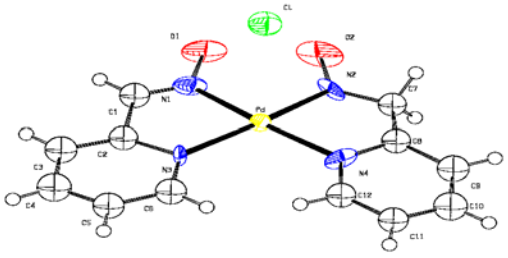

Fig. 1. ORTEP diagram of a symmetric unit of $[PdH(C_6N_2OH_5)_2Cl]$(**1**).

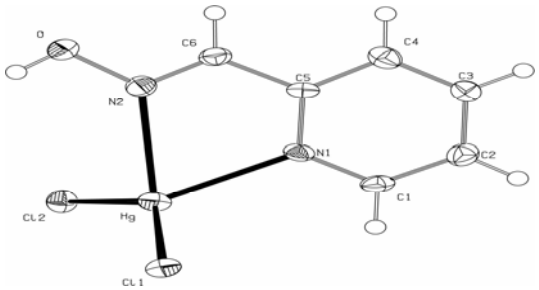

Fig. 2. ORTEP diagram of a unit of $[HgC_6N_2OH_6Cl_2]$(**2**)

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Synthesis and Crystal Structure of 4-Aminoquinolinium Bis(citrato)borate Monohydrate

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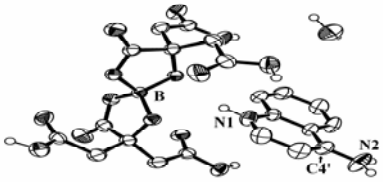
Keywords: single-crystal X-ray, boron compounds, hydrogen bonds

In the course of structural studies of amine salts of boron coordination compounds a new complex – 4-aminoquinolinium bis(citrato)borate monohydrate (I) has been synthesized and its X-ray structural investigation has been carried out. Complex I was prepared by mixing boric and citric acids with 4-aminoquinoline in proportions (molar ratio) 1:2:1 in water solution. Crystals of compound I were obtained by slow evaporation of water from solution.



The structure of the title compound I is formed by univalent cations $[(4\text{-}NH_2)C_9H_6NH]^+$, spiran-type complex anions $[(C_6H_6O_7)_2B]^-$ and crystallization water molecules (fig.). Boron atoms are tetracoordinated as confirms the sp^3 hybridization sphere around them. The bonds B–O(carb.) (average length 1.495(3) Å) are systematically longer than B–O(hydr) (average length 1.448(3) Å). The values of the dihedral angles corresponding to the crossing of two main-square planes of boron-containing heterocycles –C–O–B–O–C– in the two independent complex anions amount to 87.88(6)º and 87.87(6)º. The molecule of 4–aminoquinoline molecule is protonated at the ring N1 atom but not at the amino group nitrogen atom N2 in acidic solutions. The C4'–N2 bond length (1.326(3) Å in average) is approximately equal to the C=N double bond lenght indicating that atom N2 of the amino group must also be sp^2- hybridized. The C–C bonds at C4' atom are somewhat longer (1.424(3) Å in average). The N2 atoms lie almost in the quinoline ring plane (maximum deviation of 0.029(3) Å). Bond angles $C–N^+–C$ are 118.6(5)º. The hydrogen atoms of amino groups, protonated nitrogen atoms, as well as those of hydroxyl groups from two independent complex anions and three of four hydrogen atoms from two independent water molecules are involved in formation of 17 independent O–H...O and N–H...O type hydrogen bonds. Nitrogen

atoms N1 from two independent heterocyclic cations participate in the bifurcatic hydrogen bonds N–H...O, O'. Three dimensional spatial package of crystals of the compound I is more dense (ρ_c=1.547 g.cm^{-3}) than lamellar package of 8-aminoquinolinium bis(citrato)borate terahydrate crystals (ρ_c=1.453 g.cm^{-3}) [1]. Crystals I are triclinic, space group $P\bar{1}$: a=10.6678(3) Å, b=14.2920(3) Å, c=16.4891(4) Å, α=78.106(1)º, β=75.368(1)º, γ=85.919(1)º, V=2379.8(1) Å^3, Z=4, R=0.054, wR2=0.133 for 10562 independent reflections with R(int)=0.0266.

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Isothermal section at 800°C of the Gd-Fe-Ge ternary system R. Ben Hassen[a], M. Jemmali[a,b], S. Walha[a], O. Tougait[b], H. Noël[b], [a]*Unité de chimie des matériaux, Université Tunis El Manar, Tunis, Tunisia.* [b]*Sciences chimiques de Rennes UMR 6226, CNRS-université de Rennes 1, Avenue du Général Leclerc, F-35042 Rennes, France.*
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Keywords: intermetallic phase equilibrium, intermetallic compounds crystal structure, intermetallic compounds synthesis

The investigation of the Gd-Fe-Ge system is part of an ongoing research project with the aim to clarify the phase equilibrium in ternary system of Gd and iron with a p-block element. This ternary system has been partially investigated; the phase diagrams established at 800°C show two compounds. **A**: $Gd_1Fe_2Ge_2$ (a = 3.995, c = 10.46 Å, I4/mmm, $ThCr_2Si_2$ structure type) [1], **B**: $Gd_1Fe_6Ge_6$ (a = 5.128, c = 4.076 Å, P6/m 2/m2/m, YCo_6Ge_6 structure type) [2]. We present here our experimental results on the Gd-Fe-Ge ternary system, studied in the whole concentration range at an isotherm of 800° C. All the samples were prepared by arc-melting the elemental components, followed by heat-treatment of one week. The phases in alloys were determined by electron-probe microanalysis and examined by X-ray powder diffraction analysis (X-ray diffractometer with CoKα radiation with iron filters) in order to determine the phase compositions and the equilibrium lines within the ternary system. In addition to the known phases, a new ternary phase, $Gd_3Fe_1Ge_6$, has been found and its crystal structure was refined from powder. $Gd_3Fe_1Ge_6$, this new ternary phase crystallizes in the orthorhombic space group Cmcm (n° 63) with the lattice parameters a = 4.151, b= 16.062 and c= 4.0239 Å, structure type $CeNiSi_2$. This phase shows a significant homogeneity range which extends between the compositions $GdFe_{1-x}Ge_2$ ($0.55 \leq x \leq 0.75$).

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