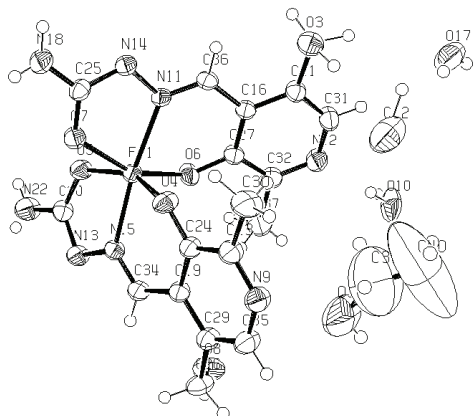


configuration with two non-equivalent tridentate ligands with O₃N₂O donors:

one of the ligands having deprotonated semicarbazide fragment, whereas for the other additional deprotonation involves the pyridine nitrogen.

The compound crystallizes in the P-1 space group with $a=8.7273(3)\text{Å}$, $b=12.3030(4)\text{Å}$, $c=14.3589(6)\text{Å}$, $\alpha=109.118(3)$, $\beta=100.490(3)$, $\gamma=101.860(3)$, $V=1372.79\text{Å}^3$.



[1]Leovac V.M., Jevtoviv V.S., Jovanovic Lj.S., Bogdanovic G.A, *J.Serb.Chem.Soc.* **2005**, 70 (3) , 393.

Keywords: iron(IV) complex; pyridoxal semicarbazone; octahedral configuration

FA4-MS06-P20

Study of Solid State Reaction in Pb (Zr, Ti) O₃ Ceramics Doped with (Fe²⁺, Ni²⁺, Sb³⁺) Ions.

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Lead zirconate titanate Pb(Zr_x, Ti_{1-x})O₃ or (PZT) ceramics are of great technological interest due to their excellent piezoelectric and ferroelectric properties. Such piezoelectric materials often used in ultrasonic generators, hydrophones, electronic buzzers and ringers, pressure and stress sensors, etc [1]. In general, the PZT are processed by the traditional solid-state reaction. However, there are undesirable features such as nonstoichiometry, compositional fluctuation and poor microstructure because of the high-temperature processes [2]. Therefore, it is necessary to process PZT ceramic at a temperature as low possible. Low firing temperature processing ceramic fabrication demands fine precursor powders of high homogeneity of components. The formation of Pb(Zr, Ti)O₃ has been studied with several

kinds of starting materials by several authors who suggested a large number of reaction mechanisms[3], [4]. The phase assemblages and appearance of intermediate reaction products will depend on the precursor powders that have been employed (specifically, their purity and particle size) and the time and temperature at which the solid state reaction has been allowed to proceed. To investigate the reaction sequence through which PZT solid solutions are formed by solid-state reactions in a mixture of PbO+TiO₂+ZrO₂, researchers have performed several series of calcinations tests, but came up with different conclusions. There have been contradictory observation, particularly with respect to the presence or absence of intermediate products like PbZrO₃, PbTiO₃ solid solution (PT)_{ss} and a PbO solid solution (P)_{ss}.

The purpose of this study was to investigate lead zirconate titanate PZT and addition a small amount of Fe₂O₃, Ni, Sb₂O₃ were synthesized by single phase perovskite PZT powders were obtained after treatment at low temperature, the effects of calcination temperature on lattice parameters and tetragonality of PZT powders have been discussed, differential thermal analysis (DTA) and thermal gravimetric analysis (TGA).

The formation of PZT is accomplished through several steps: decomposition of PbO₂ to PbO; formation of PbTiO₃ above 400°C and of its solid solution at 600°C.

[1] Uchino, K., *Piezoelectric Actuators and Ultrasonic Motors*. Kluwer Academic Publishers, Massachusetts, 1997. [2] B. Jaffe, W.R. Cook, H. Jaffe, *Piezoelectric Ceramics*, Academic Press Inc., New York, 1971. [3] T. Ohno, M. Takahashi, N. Tsubouchi, *J. Jap. Soc. Powder Metall.* 20, 1973, 154. [4] T. Yamaguchi, S. H. Sho, M. Maomori, H. Kuno, *Ceramurgia Int.* 2, 1976, 76.

Keywords: PZT; RX; thermal properties

FA4-MS06-P21

Crystal Structure of Bipyridine Type Ligand LL=N,N'-bis-pyridin-2-ylmethylene-naphthalene-1,5-diamine. Niloofer Akbarzadeh^T, Ali R. Rezvani^a. ^aDepartment of Chemistry, University of Sistan and Baluchestan, P. O. Box 98135-674, Zahedan, Iran
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The current interest in the crystal engineering of polymeric coordination networks stems from their potential applications as zeolite like materials for molecular selection, ion exchange and catalysis as well as in the variety of architectures and topologies [1-2]. One of the basic strategies for crystal engineering utilizes metal-ligand bonding between transition metals and organic ligands to create coordination polymers.

In designing these polymers the properties of linking ligands such as various coordination modes, variable lengths and relative orientation of donor atoms obey a fundamental role in determining the structural outcomes of target polymers.

A novel bipyridine-type linking ligand L ((2-py)-CH=N-C₁₀H₆-N=CH-(2-py)) (Fig. 1) was prepared by the Schiff-base condensation and fully characterized by spectroscopic methods and single-crystal X-ray diffraction. The unit cell parameters are: space group P21/c with $a = 4.8666(8)\text{Å}$, b

= 16.780(3) Å, c = 10.0559(16) Å, $\alpha = 90^\circ$, $\beta = 90.504(3)^\circ$, $\gamma = 90^\circ$, and $Z = 2$. These parameters suggested a monoclinic unit cell. This Ligand has a monoclinic crystal system and it can be seen there are π - π stacking interaction between both naphthalene and pyridin groups. The N...N separation between the pyridyl groups in L is 15.3 Å.

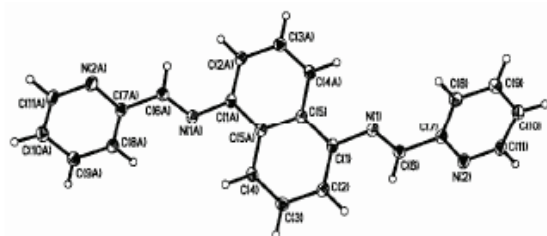


Fig 1. ORTEP of (2-py)-CH=N-C10H6-N=CH-(2-py)

[1] G.F.Swieger, T.J. Malefetse, *Chem. Rev.* 100, 2000, 3483. [2] M. J. Zaworotko, *Chem. Soc. Rev.* 1994, 283.

Keywords: schiff-base; monoclinic; naphthalene

FA4-MS06-P22

Crystal Structures of μ -bridged Trinuclear and Polynuclear Cadmium Metal Complexes. Orhan Atakol^a, Dinçer Ülkü^b. ^aAnkara University Chemistry Department, Ankara-Turkey, ^bHacettepe University Physics Department, Ankara-Turkey.

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Linear and angular homo- or heteronuclear metal complexes based on Schiff base ligands are of interest because of their magnetic super-exchange interactions between bridged metal ions [1]. In these complexes, various combinations of metal ions in the central and terminal locations, as well as the μ -bridges, such as acetate or nitrite anions, are possible. The two new tri- and polynuclear structures described here are an extension of our previous structural studies [2] of bridged metal complexes.

(I) $[C_{44}H_{54}CdCu_2N_{12}O_4]$, Pccn, $a = 13.8382(12)$, $b = 15.3944(11)$, $c = 22.5753(13)$ Å, $Z = 4$, $D_x = 1.46$ g.cm⁻³, $\lambda = 0.71073$ Å, $I > 2\sigma$ 4229, $N_{\text{par}} = 295$, $R, R_w \rightarrow 0.028, 0.061$, $\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}} \rightarrow 0.27, -0.27$ e Å⁻³. The central metal atom Cd has an octahedral environment consisting of four bridging O atoms from the ligand and two N atoms from the azide groups. The terminal Cu atoms have square-pyramidal coordination, involving two N and two O atoms from a ligand and one O atom from a dimethyl formamide group. The bridging angle Cd-O-Cu between the central and terminal metal atoms is 74.87(2)°. The Cd...Cu and Cu...Cu distances are 3.3239(5) and 4.0412(6) Å, respectively.

(II) $[C_{38}H_{40}Cd_2I_4N_4O_4]$, P1, $a = 10.3890(12)$, $b = 13.1950(11)$, $c = 16.2350(13)$ Å, $Z = 2$, $D_x = 2.07$ g.cm⁻³, $\lambda = 0.71073$ Å, $I > 2\sigma$ 8239, $N_{\text{par}} = 465$, $R, R_w \rightarrow 0.08, 0.175$, $\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}} \rightarrow 1.30, -1.88$ e Å⁻³. Cd atom has a distorted tetrahedral coordination consisting of two O atoms from two neighbouring ligands and two I atoms. The O atoms in the Cd coordination sphere belonging to two different ligands lead to polymerisation. It is actually a molecular μ bridge connecting the Cd atoms

via phenolic oxygens. The Cd-I bond distances range from 2.6980(14) to 2.7547(14) Å.

[1] Fukuhara C., Tsuneyoshi K., Matsumoto N., Kida S., Mikuriya M., Mori M. *J. Chem. Soc. Dalton Trans.* 1990, 3473. [2] Arıcı C., Ercan F., Atakol O., Akay A., Ülkü D., *Acta Cryst.* 1999. C55, 928-930

Keywords: metalloorganic structures; coordination compounds; polynuclear complexes

FA4-MS06-P23

Synthesis and Spectroscopic Properties of a Five Coordinate Cyanato Iron(II) "Picket Fence" Porphyrin Complex. Mondher Dhifet^a, Mohamed Salah Belkhiria^a, Jean-Claude Daran^b, Habib Nasri^a. ^aLaboratoire de Physico-Chimie des Matériaux, Faculté des Sciences de Monastir, Avenue de l'Environnement, 5019 Monastir, Tunisia. ^bLaboratoire de Chimie de Coordination, UPR CNRS 8042, 205 route de Narbonne, 31077 Toulouse cedex, France.

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The reaction of four-coordinate picket fence iron(II) porphyrin complex [Fe(TpivPP)] with cryptand-222 solubilized NaOCN in organic solvents, yields the five-coordinate porphyrin species [Fe(TpivPP)(NCO)]⁻. The five-coordinate, high-spin ($S = 2$) (cyanato-*N*)($\alpha, \alpha, \alpha, \alpha$ -tertakis(*o*-pivalamidophenyl) porphinato)iron(II) "picket fence" porphyrin derivative [Na(222)][Fe(TpivPP)(NCO)] has been synthesized and characterized by UV-vis, IR and magnetic susceptibility spectroscopies as well as single-crystal structure determinations. The X-ray molecular structure of the chlorobenzene solvate of the metalloporphyrin derivative has been determined. The $S = 2$ spin state has also been confirmed from the measurement of the temperature-dependant magnetic susceptibility. [Na(222)][Fe(TpivPP)(NCO)] crystallize in the monoclinic system with $a = 24.0027(8)$ Å; $b = 14.7581(6)$ Å; $c = 23.6448(8)$ Å; $\beta = 106.078(2)^\circ$; $V = 8048.18(49)$ Å³, $T = 180$ K, space group C2, $Z = 4$, μ (Mo-K α) = 0.258 mm⁻¹. 18133 unique reflections measured ($R_{\text{sigma}} = 0.0496$) which were used in all refinements. The final agreement factors $R(F)$ ($I > 2\sigma(I)$) for 14508 reflections and $wR(F^2)$ (all data) were 0.0602 and 0.1631, respectively. The average Fe-Np bond distance is 2.121(1) Å. The iron atom is pentacoordinated by the four nitrogen atoms of the pyrrole rings and the nitrogen atom of the NCO⁻ group. It lies at 0.7096(9) Å out of the porphinato plane and 0.5940(15) Å out of the four nitrogen plane of the porphyrin ring. The Fe-N(NCO) bond length is 2.007(2) Å and the Fe-N-C(NCO) bond angle is 176.62(7)°.

The Figure 1 is an ORTEP diagram of the [Fe(TpivPP)(NCO)]⁻ complex.