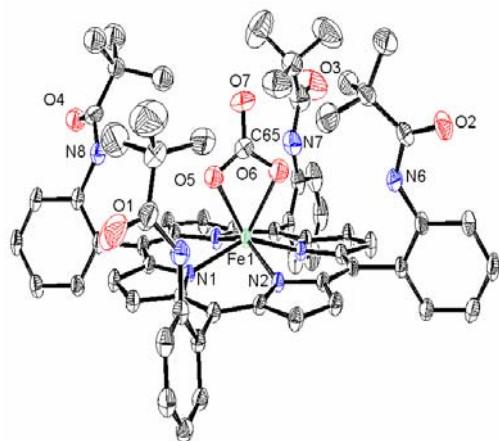


**MS15 P06**

**Preparation, Spectroscopic and Structural characterization of the hexa-coordinate, high-spin iron(III) carbonato « picket fence » porphyrin complex** Mondher Dhifet, Mohamed Salah Belkhiria et Habib Nasri, *Laboratoire de Physico-Chimie des Matériaux, Faculté des Sciences de Monastir, Avenue de l'environnement, 5019 Monastir, Tunisie.*  
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**Keywords: metalloporphyrins, carbonato-complexes, Heme model.**

The preparation and characterization of the (carbonato)( $\alpha,\alpha,\alpha,\alpha$ -tetrakis(*o*-pivalamidophenyl)porphyrinato)iron(III) complex is described. The synthesis procedure utilizes the cryptand-222 to solubilize potassium carbonate. The compound has been characterized by UV-vis, IR and  $^1\text{H}$  NMR spectroscopies. Proton NMR data for the isolated product is in accordance with high-spin ( $S = 5/2$ ) ferric porphyrin species. The X-ray molecular structure of this species has been also determined. The iron atom is hexa-coordinated by the four nitrogen atoms of the pyrrol rings and the two oxygen atoms of the  $\text{CO}_3^{2-}$  group. It lies at 0.695(1) Å out of the porphyrinato plane and 0.640(1) Å out of the four nitrogen plane. The Fe-O bond length is 2.067(10) Å and the average Fe-Np distance is 2.076(10) Å with is a stereochemical proof that the  $[\text{Fe}^{\text{III}}(\text{TpvPP})(\text{CO}_3)]^+$  is high-spin. Crystal data for  $[\text{K}(222)][\text{Fe}(\text{TpvPP})(\text{CO}_3)]\cdot\text{C}_6\text{H}_5\text{Cl}\cdot 2\text{H}_2\text{O}$  are:  $a = 18.085(5)$  Å;  $b = 21.505(10)$  Å;  $c = 23.160(5)$  Å;  $\beta = 100.52(3)^\circ$ , monoclinic, space group  $\text{P2}_1/\text{n}$ ;  $V = 8855$  Å $^3$ ;  $Z = 4$ ; 27435 unique reflections measured which were used in all refinements. The final agreement factors  $R(F)$  ( $I > 2\sigma(I)$ ) for 18421 reflections and  $wR(F^2)$  (all data) were 0.09 and 0.21, respectively.



**Fig.** *ORTEP* diagram of  $[\text{Fe}(\text{TpvPP})(\text{CO}_3)]^+$  ion complex showing thermal ellipsoids at 30 % probability level.

**MS15 P07**

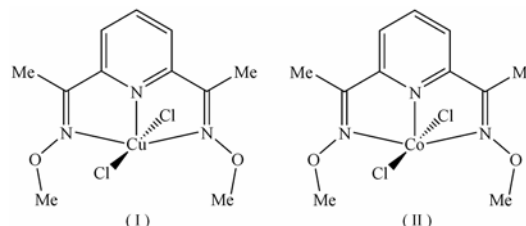
**Copper(II) and cobalt(II) complexes of 2,6-diacetylpyridine bis(*O*-methyloxime)** Muharrem Dinçer<sup>a</sup>, Namık Özdemir<sup>a</sup>, Osman Dayan<sup>b</sup>, Bekir Çetinkaya<sup>b</sup>, <sup>a</sup>*Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139, Samsun, Turkey.* <sup>b</sup>*Department of Chemistry, Faculty of Science,*

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**Keywords: crystal structure analysis, coordination complexes, catalysts**

The title complexes, dichloro[(1*E*,1'*E*)-1,1'-(pyridine-2,6-diyl)diethanone bis(*O*-methyloxime)- $\kappa^3\text{N}^1,\text{N}^2,\text{N}^6$ ]copper(II),  $[\text{CuCl}_2(\text{C}_{11}\text{H}_{15}\text{N}_3\text{O}_2)]$ , (I), and dichloro[(1*E*,1'*E*)-1,1'-(pyridine-2,6-diyl)diethanone bis(*O*-methyloxime)- $\kappa^3\text{N}^1,\text{N}^2,\text{N}^6$ ]cobalt(II),  $[\text{CoCl}_2(\text{C}_{11}\text{H}_{15}\text{N}_3\text{O}_2)]$ , (II), crystallize in space groups  $\text{P2}_1/\text{c}$  [1] and  $\text{Pnma}$  [2], respectively. In each compound, the metal atom is pentacoordinated by the two methyloxime N atoms, one pyridine N atom and two Cl atoms. Although the coordination polyhedra around the metal atom in (I) is a strongly distorted trigonal-bipyramid, the coordination polyhedra in (II) is a strongly distorted square-bipyramid. It is observed that the N(oxime)-M-N(pyridine) bond angle for five-membered chelate rings of 2,6-diacetylpyridine dioxime complexes is inversely related to the magnitude of the M-N(pyridine) bond. Both structures are stabilized by intra- and intermolecular C-H...Cl hydrogen bonds. Despite the similar chemical compositions of the  $\text{Cu}^{\text{II}}$  and  $\text{Co}^{\text{II}}$  complexes, the packing patterns of the molecules in the crystal structures are quite different. In contrast to the columnar packed structure observed in (I), the molecules of (II) pack in layers.



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**MS15 P08**

**Structural Study of Dendronized Palladium Scorpionate Complexes.** Pilar Gómez-Sal, Alberto Sánchez-Méndez, Ernesto de Jesús, Juan C. Flores, *Departamento de Química Inorgánica, Universidad de Alcalá. Alcalá de Henares (Madrid) Spain.*  
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**Keywords: crystal and molecular structure; dendrimers; palladium compounds.**

Most dendrimers are flexible molecules and therefore their structures have been largely debated. Several computational and experimental techniques have been used to gain structural information regarding dendrimers in solution and the solid state, but only X-ray diffraction techniques allow the acquisition of highly precise data regarding the internal molecular conformations. Unfortunately, the growth of single crystals of suitable quality has been restricted mainly to small dendrimers because of a variety of problems, in part due to the conformational flexibility of these molecules [1]. Poly

(aryl ether) dendrons based on 3, 5-dihydroxybenzyl alcohol, developed by Fréchet, are amongst the most popular dendritic structures and have been widely used in organometallic catalysis, usually after metal functionalization at the core or focal point. The conformation adopted by the dendritic arms of these catalysts in solution is important because determines the nanoenvironment and accessibility of the metal centers. [2]

In our communication novel palladium (II) complexes with bis (pyrazol-1-yl) methane ligands at the focal point, of generations G0 to G3 poly (aryl ether) Fréchet-type dendrons are reported. The molecular structures of the metallodendrimer series G0-, G1-, and G2-[(dend)CH(3,5-Me<sub>2</sub>pz)<sub>2</sub>(PdCl<sub>2</sub>)] have been determined by X-ray diffraction methods. The three structures show a similar three-dimensional organization of the metal complex, which is progressively engulfed by the branches with increasing dendrimer generation. Several interesting features are discussed and some new structural parameters to identify their configurations are defined. The higher generation (G2) is, to the best of our knowledge, the first transition-metal dendrimer G2 to be crystallographically characterized so far.

Details of the crystal structure based in weak hydrogen bonding interactions with the solvent and  $\pi$ - $\pi$  stacking are also examined.

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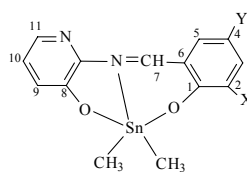
b) Andrés, R.; de Jesús, E.; Flores, J. C. *New J. Chem.* 2007

#### MS15 P09

**Structural features of some organotin(IV) complexes of ONO donor Schiff bases** Semra Ide<sup>a</sup>, Nilgün Ancin<sup>b</sup>, Hasan Malkaş<sup>a</sup>, Selma Gül Öztaş<sup>b</sup>, Hacettepe University, Department of Physics Engineering, 06800 Beytepe-Ankara, Turkey <sup>b</sup>Ankara University, Department of Chemistry, 06100 Besevler-Ankara, Turkey. E-mail: [side@hacettepe.edu.tr](mailto:side@hacettepe.edu.tr)

**Keywords:** Diorganotin(IV) complexes; Tridentate Schiff base; Crystal structure.

The Schiff bases of salicylaldehyde with aminopyridines are a well-known class of predominantly thermochromic compounds which have attracted a lot of interest from chemists for a long time [1]. Increasing attention has also been devoted to Schiff base complexes of organotin(IV) moieties in view of their potential applications in medicinal chemistry and biotechnology [2]. In this work, [N-(3-hydroxypyridine-2-yl)-3,5-dichlorosalicylideneiminato] dimethyltin (IV) was characterized by single crystal x-ray diffraction method and a coordination geometry that is nearly half-way between trigonal-bipyramidal and square pyramidal arrangement was found. Unit cell parameters are: a= 9.601(1), b= 15.861(3), c= 10.98(1) Å, B= 113.83(2)°, V=1529.5(2) Å<sup>3</sup>, Crystal system is monoclinic. The structure was solved by direct methods and refined by least squares on F<sup>2</sup><sub>obs</sub> by using SHELXL-97. The structural results were compared with its analogs in our previous studies [3,4]. It is found that the electronegativity of substituents influenced to the C=N, Sn-O and Sn-N bond lengths.



	X	Y
(1)	H	H
(2)	H	OH
(3)	H	Br
(4)	H	Cl
(5)	Cl	Cl

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#### MS15 P10

**Crystal structure of Hexa- $\mu_2$ -chloro- $\mu_4$ -oxo-tetrakis-(3,5-dimethylpyrazole)copper(II)** Željko K. Jačimović<sup>a</sup>, Zoran D. Tomić<sup>b</sup> and Vukadin M. Leovac<sup>c</sup> <sup>a</sup>Faculty of Metallurgy and Technology, University of Montenegro, 81000 Podgorica, Montenegro, <sup>b</sup>'Vinca' Institute of Nuclear Sciences, Laboratory of Theoretical Physics and Condensed Matter Physics, 1001 Belgrade, Serbia. <sup>c</sup>Institute of Chemistry, Faculty of Sciences, University of Novi Sad, Trg Dositeja Obradovica 3, 21000 Novi Sad, Serbia, E-mail: [zeljkoj@cg.ac.yu](mailto:zeljkoj@cg.ac.yu)

**Keywords** Synthesis, Cu crystal structure, pyrazole based ligand

We report the synthesis, and structural characterization, of a novel tetranuclear copper(II) complex Cu<sub>4</sub>OX<sub>6</sub>L<sub>4</sub> where L = 3,5-dimethylpyrazole.

**Synthesis** 3,5-dimethylpyrazole-1-carboxamide (0,14g, 1mmol) were dissolved in ethanol (5cm<sup>3</sup>) and added to the hot solution of CuCl<sub>2</sub> · 2H<sub>2</sub>O (0,17g, 1mmol) in ethanol (7 cm<sup>3</sup>). Green crystals were obtained after one week. Four copper atoms (separated by the 3.070 (2)-3.198 (3) Å) encapsulate central O atom in a distorted tetrahedral arrangement, with the Cu—O bond lengths of 1.903 (6)-1.914 (6) Å, and the Cu—O—Cu angles of 107.3 (3)-114.3 (3)°. Between each pair of copper atoms, there is a bridging chlorine atom with a Cu—Cl distances of 2,340(4) - 2,509(3) Å. Closer inspection of the Cu—Cl distances reveal grouping of values around every copper atom in a two 'short' and one 'long' distances. Average values are 2.375 and 2.459 Å for 8 short and 6 long Cu—Cl distances respectively. Copper coordination sphere is completed by the 3,5-dimethylpyrazole ligand with Cu—N distances of 1,952(7) - 1,957(9) Å. Coordination polyhedron can be described as a slightly distorted trigonal bipyramid where three chlorine atoms lie in the equatorial positions while the central oxygen and the 'pyridine' nitrogen from pyrazolyl ligand are placed at the axial sites.