spectra we will discus the electric structure, distortion of polyhedra related to ferroelectricity and thermal vibration.

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Keywords: ferroelectric, XANES, phase transition

MS56.P09

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Temperature dependence of pre-edge feature in Ti K-edge XANES spectra for ATiO$_3$, A$_2$TiO$_4$ (A=Mg, Ca, Fe, Sr and Ba) and TiO$_2$ compounds

Tatsuya Hiratoko,[a] Tomotaka Nakatani[a] ,Hiroki Okudera,[b] Akihiko Nakatsuka[c] and Akira Yoshias,[a] [a]*Graduate School of Sciences and Technology, Kumamoto University, Kumamoto 860-8555 (Japan).* [b]*Graduate School of Natural Science, Kanazawa University, Kanazawa, 920-1192 (Japan) .*[c]*Department of Advanced Materials Science and Engineering, Yamaguchi University, Ube 755-8611 (Japan).* E-mail: 118d8052@st.kumamoto-u.ac.jp

X-ray absorption near edge structure (XANES) provides important information on the electronic structure and local symmetry around X-ray absorption atom. There are three distinct pre-edge peaks correspond to electronic transitions in Ti K-edge XANES spectra. The composition, structure and temperature dependence of XANES spectra on three peaks was investigated. We measured Ti K-edge XANES spectra of various titanates, MgTiO$_3$, CaTiO$_3$, SrTiO$_3$, BaTiO$_3$, Mg$_2$TiO$_4$, Fe$_2$TiO$_4$, TiO$_2$ rutile and anatase, in the temperature range from 20K to 800K. Ti atoms are placed in TiO$_6$ octahedral and TiO$_4$ tetrahedral sites in crystal structures. The measurements of Ti k-edge XANES spectra were carried out in transmission mode at beam line BL-7C and BL-9A of the Photon Factory in KEK, Tsukuba. High temperature X-ray absorption measurements were made under a helium atmosphere. XANES spectra of all sample on each peaks is increasing as the temperature increases except for tetragonal BaTiO$_3$ and tetragonal SrTiO$_3$ phases. TiO$_2$ rutile and anatase have largely different rate of rising pre-egde absorption to the temperature. The XANES spectra in the high temperature region were strongly affected by the harmonic thermal vibration of the atoms. There is an interesting relation between electronic transition and local distortion of coordination environment coming from the harmonic thermal vibration. We will discuss harmonic and anharmonic thermal vibration models by XAFS method.

Keywords: XANES, pre-edge peak, titanate

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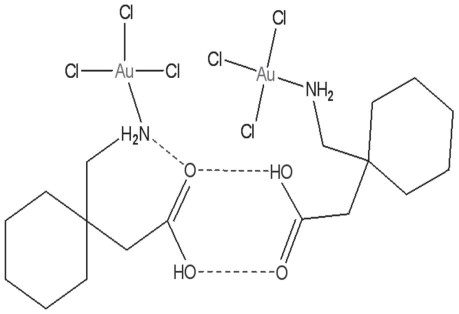
A gold(III) complex of the neuroepileptic drug gabapentin

Demetrius. C. Levendis, Ahmed Shaikjee, Helder M. Marques and Richard Mampa, *Molecular Sciences Institute, School of Chemistry, University of the Witwatersrand, PO WITS, 2050, Johannesburg, (South Africa).* Email: demetrius.levendis@wits.ac.za

Gabapentin, a neuro-epileptic drug, has been the subject of interest lately as new polymorphs, [1] salts and hydrates have been reported, as well as the high pressure crystallisation of a novel heptahydrate [2]. The first complexes with transition metal complexes, Cu(II) and Zn(II) were reported recently by Braga and co-workers [3]. Since gold is known to have pharmaceutical applications [4] we were interested to see if we could prepare a Au(III)-Gp complex.



Auric acid was reacted with gabapentin in an aqueous solution. Two distinct types of crystals precipitated from this solution: pale yellow needle shaped crystals (**I**) and dark yellow blocks (**II**, not shown here). Their structures were determined by single crystal structure analysis. The MeOD solution ^{1}H and ^{15}N-NMR were then recorded to confirm that the gold remains coordinated to the nitrogen of the amino group in solution (**I**).

The crystal structure of (**I**) clearly shows the Au-N coordination (Au-N bond 2.043(2) Å) in the hydrogen bonded dimer. The cyclohexane is disordered over two positions, both having a chair conformation. The ^{1}H -NMR shows peaks at δ = 2.45, 2.88 ppm for the free gabapentin –CH$_2$- groups (–CH$_2$-COOH and -CH$_2$-NH3$^+$ respectively). On dissolving (**I**) in MeOD four peaks are apparent in the ^{1}H-NMR; at 2.45, 2.51, 3.05 and 3.12 ppm. Our current interpretation of this is that (**I**) exists as two different isomers in solution and that on crystallisation these persist in the solid state.

In conclusion, auric acid reacts with gabapentin in aqueous solution to form a Au(III)-gabapentin complex in which the Au(III) is coordinated to the amino nitrogen [5]. It appears that in this reaction an intermediate complex has been trapped given that Au(III) is known to oxidise amino acids.

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Crystal structure of Zn complex with chelidamic acid and acridine

Zohreh Derikvand,[a] Andya Nemati,[b] Masomeh Mansouriki,[a] [a]*Department of Chemistry, Faculty of Science, Islamic Azad University, Khorramabad Branch, Khorramabad, (Iran).* [b]*Iran Compiling Encyclopedia Foundation, Tajrish, Tehran, (Iran).* E-mail: zderik@yahoo.com

4-Hydroxypyridine-2,6-dicarboxylic acid as carboxylate derivative has drawn extensive attention in coordination chemistry. This ligand could potentially provide various coordination motifs to form both discrete and consecutive metal complexes under appropriate synthesis condition [1,2]. We prepared new mononuclear complex of Zn(II) containing 4-hydroxypyridine-2,6-dicarboxylic acid ($hypydcH_2$) and acridine (acr). The compound, (acrH)[Zn(hypydc)$_2$].10H_2O, was characterized by elemental analysis, ^1H NMR, ^{13}C NMR, IR spectroscopy, single crystal X-ray diffraction method. Zn(II) compound crystallized in monoclinic system with 8 molecules per unit cell. The crystallographic analysis revealed that the complex consist of $[Zn(hypydc)_2]^{2-}$ anion, two $acrH^+$ cations and ten uncoordinated water molecules. Zn(II) atoms in the complex, is coordinated in a distorted octahedral geometry by two nitrogen and four oxygen atoms of 4-hydroxypyridinedicarboxylate ligand. Acridine is known as proton acceptor ligand. In the crystal structures of the complex extensive O–H···O, N–H···O and C–H···O hydrogen bonds as well as electrostatic forces, C–O···π and π–π stacking play important roles in stabilizing structures.

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Keywords: Zn complex, 4-hydroxypyridine-2,6-dicarboxylic acid

MS56.P12

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Effects of substituents on the conformational diversity of the new *N*-phosphinylureas

Nilufar Dorosti,[a] Narges Dorosti,[b] Foroogh Molaei,[c] Khodayar Gholivand[c] [a]*Razi Herbal Medicines Reaserch Center, Lorestan University of Medical Sciences, Khoramabad, (Iran).* [b]*Lorestan University of Medical Sciences, Khoramabad (Iran).* [c]*Department of Chemistry, Faculty of science, Tarbiat Modares University, Tehran, (Iran).* E-mail: nilparabi@yahoo.com

N-phosphoniylureas are some important instances of phosphoramidates that little attention has been given to their biological properties [1] and structural studies [2]. These compounds can cause attractive biological activities due to having urea and peptide moieties. Recently, we evaluated anticancer [3] and antibacterial [4] properties of some of these compounds. Further, in our previous studies, we were synthesized and characterized two novel *N*-phosphinylureas with general formula $RC_6H_4NHC(O)NHP(O)NHCH_2C(CH_3)_2CH_2NH$ [4]. The crystal structures of compounds containing (R = H (**1**), NO_2 (**2**)) are composed of two and one conformers, respectively. These molecules showed *anti* and *syn* conformations with respect to the C=O and P=O bonds for (**1**) and (**2**). In order to further investigate, quantum chemical calculations were applied. The results predict a structure with *anti* conformation as the most stable form for **2** due to the packing effect.

In connection with our current work in the field of *N*-phosphinyl ureas chemistry and investigate of effect of substituent, here we synthesized and characterized two novel derivatives with R = CH_3 (**3**) and F (**4**). The 3D structures of these molecules have been determined by X-ray crystallography. The title molecules contain four and two conformers in crystalline lattice, respectively. In molecule **3**, four types of hydrogen bond are established among these conformers, intramolecular P=O…H-NPh, intermolecular C=O…NHP, C=O…NH and P=O…NH hydrogen bonds. These conformers create two types of chains with different arrangements in the crystal lattice of **3**. Linking of these chains by hydrogen bonding leads to form a two-dimensional polymeric chain in the crystal lattice. In molecule **4**, intramolecular P=O…H-NPh, intermolecular C=O…NHP, P=O…NH hydrogen bonds cause to form a two-dimensional polymeric chain. It is noteworthy; the new *N*-phosphinylureas crystallize in the triclinic and monoclinic crystal systems with space groups $P\bar{1}$ and $P2_1/c$, respectively. The symmetry of the unit cell decreases with increase in the multiplicity of conformers that demonstrate our previous study [5]. The mean P=O distances fall in the range 1.4803-1.4845Å that are slightly longer than the normal P=O bond length (1.45A°). The phosphoryl oxygen atoms (O(2), O(22), O(23), O(24), O(1) and O(1A)) occupy a pseudoaxial position, and the 4-methyl and 4-flurophenylureido groups adopt a pseudoequatorial orientation.

In all of the compounds, the $P-N_{amide}$ bond length is longer than the $P-N_{amine}$ bond lengths. All of these bonds are in the range 1.608 (2) - 1.698(18) A° and thus are significantly shorter than a typical P-N single bond (1.77 A°). The phosphorus atoms have slightly distorted tetrahedral configuration.

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Keywords: synthesis, 3D, substituent

MS56.P13

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Structural characterization of $Mo_3Fe(NO)S_4$ Cubane-type clusters with different electron populations

Iván Sorribes,[a] Rosa Llusar,[a] Cristian Vicent,[b] Francesc Lloret,[c] and Victor Polo,[d] [a]*Dpt. Química Física i Analítica, Universitat Jaume I, 12071 Castellón, (Spain).* [b]*Serveis Centrals d'Instrumentació Científica, Universitat Jaume I, 12071 Castellón, (Spain).* [c]*Dpt. Química Inorgánica, Instituto de Ciencia Molecular, Universitat de Valencia, E-46980 Paterna, (Spain).* [d]*Dpt. Química Física, Universidad de Zaragoza, 50009 Zaragoza, (Spain).* E-mail: isorribe@qfa.uji.es

Molecule Nitric Oxide plays an important role in biological systems. [1] Many of the important biological functions of NO are iron-mediated, being the synthesis and characterization of new iron nitrosyl complexes a priority target for the understanding of its biological functions. [2] Cubane-type clusters $[Mo_3(FeNO)S_4(dmpe)_3Cl_3]^{0/+}$ (dmpe = 1,2- bis(dimethylphosphine-ethane)) have been isolated (see figure 1). Here we analyze the influence of the different electron population on the molecular structure. A detailed spectroscopic study (NMR, magnetic susceptibility, EPR and IR) is presented to evaluate the effect derived from the non-innocent behaviour of the nitrosyl ligand attached to iron atom on the molecular structure of these clusters.

Figure 1: **Molecular structure of the molybdenum iron nitrosyl cubane-type clusters $[Mo_3(FeNO)S_4(dmpe)_3Cl_3]$ (left) and $[Mo_3(FeNO)S_4(dmpe)_3Cl_3]^+$ (right)**

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