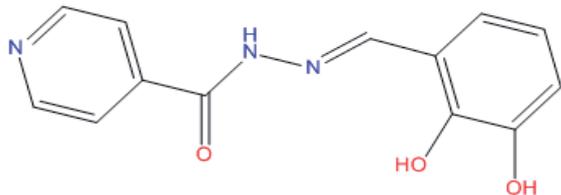


bases play an important role in inorganic chemistry as they easily form stable complexes with most transition metal ions. The development of the field of bioinorganic chemistry has increased the interest in Schiff base complexes, since it has been recognized that many of these complexes may serve as models for biologically important species [1], [2].



[1] S. Khlood, Abou-Melha, *Spectrochimica Acta A* **2008**, *70*, 162–170. [2] Z.H. Chohan, S.K. Sheazi, A. Synth. React. *Inorg. Met-Org. Chem.* **1999**, *29*, 105.

Keywords: single-crystal X-ray study, isonicotinic acid

MS61.P21

Acta Cryst. (2011) **A67**, C612

Preparation and Evaluation of Anti-inflammatory Potential of Some Sesquiterpene lactones: X-ray Structure of Psilostachyin

Rajnikant,^b Renu Chib,^a Bhabwal Ali Shah,^a Anjali Pandey,^a Kamini Kapoor,^b Sarang Bani,^a Vivek K. Gupta,^b Vijay K. Sethi,^a and Subhash Chandra Taneja,^a ^aNatural Products Microbes Division, Indian Institute of Integrative Medicines, Canal Road, Jammu Tawi-180 001, (India). ^bX-ray Crystallography Laboratory, Department of Physics and Electronics, University of Jammu, Jammu Tawi- 180 006, (India). E-mail: rkant.ju@gmail.com

The natural sesquiterpene lactones (SLs) have displayed a diverse range of bioactivities from antiinflammatory to antibacterial, ameobicidal to anticancer, therefore, they constitute interesting targets for further investigations in the area of drug development. The spiro lactone psilostachyin (**1**) and acetylated pseudoguaianolides (**2**, **3**) are the SLs isolated from *Ambrosia psilostachya* and *Parthenium hysterophorus* respectively (Fig.1).

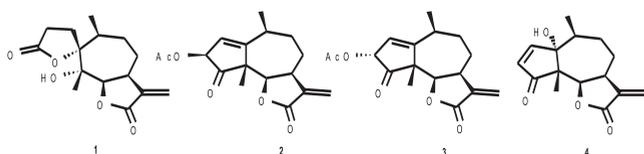


Fig.1 Naturally occurring sesquiterpene lactones

In continuity to our research programme of isolation and structural modifications of natural products to develop bioactive lead molecules particularly in the area of inflammation and cancer, we report the semi-synthesis of naturally occurring acetylated pseudoguaianolides (**2** and **3**) and spiro lactone psilostachyin (**1**) including their analogues from the major pseudoguaianolide parthenin from *Parthenium hysterophorus* for the evaluation of their anti-inflammatory potential estimated through *in vitro* expression of TNF- α , IL-1 β and IL-6 in murine neutrophils. The structure of semi-synthetic psilostachyin was also confirmed by X-ray crystallography.

Keywords: sesquiterpenes lactones, Parthenin, crystal structure

MS61.P22

Acta Cryst. (2011) **A67**, C612-C613

Crystal structures of novel CB[6] complexes with *p*-Xylylenediammine derivatives

Mark Botoshansky, Ofer Reany and Ehud Keinan, *Schulich Faculty of Chemistry, Technion-Israel Institute of Technology, Technion city, Haifa. (Israel)*. E-mail: botoshan@tx.technion.ac.il

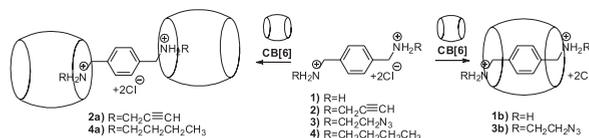
The ability of cucurbit[6]uril, **CB[6]**, to form host-guest complex with diammonium salts, in particular with linear aliphatic diammonium salts, has been widely used for the construction of pseudorotaxanes and rotaxanes that exhibited interesting dynamic, structural and functional properties. By contrast, the ability of **CB[6]** to host aromatic compounds has been less obvious. It has been shown that **CB[6]** and the dihydrochloride salt of bis-1,4-(allylaminomethyl)benzene, form only [3] pseudorotaxane. This mode of binding with two molecules of **CB[6]** hosting the allylic side chains of the guest was led to the interpretation that the aromatic moiety is too big to be encapsulated in the cavity of **CB[6]**. [1] However, it has already been reported that **CB[6]** and *p*-xylylenediammonium salt, **1**, form a stable inclusion complex, **1b**. [2] Furthermore, 4-aminobipyridine, and 4-(2-pyridyl)aniline, form strong, 1:1 inclusion complexes with **CB[6]**, whereas the bifunctional analog forms strong 1:2 complex with **CB[6]**, exhibiting remarkably large enhancements of fluorescence intensity and quantum yields. [3] These led us to speculate that the encapsulation of aromatic guest molecules, within the inner cavity of **CB[6]** can be thermodynamically favored but their formation may involve unexpectedly high kinetic barriers.

Novel pseudorotaxane compounds based on **CB[6]** and *p*-xylylenediammonium salts derivatives, **1**, **3-4**, were obtained by crystallization from aqueous solutions that contain a mixture of each diammonium guest and **CB[6]** at room temperature. The X-ray diffraction analysis of their complexes, **1b**, **3b** and **4a** was also supported by ¹H NMR in solution. The 1:2 complex of guest **2**, namely **2a**, was evident by NMR, but suitable for X-ray analysis single crystal were not be able to obtain. Nevertheless, upon heating and crystallization, the 1:1 complex, **2b**, was obtained.

Further support for these two different binding modes was obtained from ITC measurements at room temperature.

Each 1:1 complex exhibits two types of hydrogen bonding between the portal oxygen atoms and the guest. The more obvious bonds are formed with the ammonium hydrogen atoms (NH...O=C, 1.980(3)–2.420(3)Å), The less obvious, but quite visible from the solid state structure, are the hydrogen bonding between the carbonyl oxygens of **CB[6]** and the benzylic methylene hydrogens of the guest: CH...O=C, 2.317(3)–2.508(2) Å. Complex **4a** exhibited two distinguished hydrogen bond interactions: one corresponds to donor water molecule that is bridged by carbonyl oxygen of each cucurbituril unit (C=O(10)..O_w..(6A)O=C, 2.84-- 2.97Å); second, where two water molecules formed a string bridge between the two cucurbituril units. These strong interactions (C=O(8)..O_w..(O4A)O=C, 2.73-2.88Å) could explain the asymmetry observed in the structure of **4a** where the **CB** units are obviously attracted to one another, creating an angle of 42.56° between them.

Our results show that 2:1 host-guest complexes, **2a** and **4a**, are kinetic products, which may be converted to the thermodynamic products upon prolonged heating.



[1] S.Y. Kim, J.W. Lee, S.C. Han, K. Kim, *Bull. Korean Chem. Soc.* **2005**, *26*,

1265. [2] W. Freeman, *Acta Cryst.* **1984**, *B40*, 382. [3] M.K. Sinha, O. Reany, G. Parvari, A. Karmakar, E. Keinan, *Eur. J Chem.* **2010**, *16*, 9056

Keywords: Host, guest, inclusion

MS61.P23

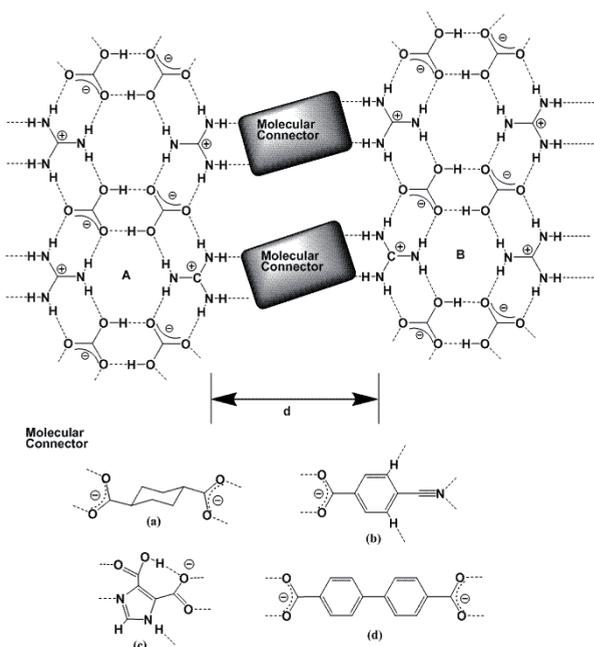
Acta Cryst. (2011) **A67**, C613

Layer-type Supramolecular Networks from Rosette Ribbons and Bridging Connectors

Jie Han,^{a,b} Thomas C. W. Mak,^a ^a*Department of Chemistry and Center of Novel Functional Molecules, The Chinese University of Hong Kong, Shatin, New Territories, Hong Kong SAR, (P. R. China).* ^b*School of Science & Technology, The Open University of Hong Kong, Kowloon, Hong Kong SAR, (P. R. China).* E-mail: chan@ouhk.edu.hk.

Over the years, chemical systems bearing a rosette motif (often described as hexagonal honeycomb unit or grid) have attracted considerable interest in connection with supramolecular self-assembly.[1] A rosette ribbon consists of a linear arrangement of hexagonal structural units that share fused opposite sides, and a sheet-like network can be generated by bridging an array of parallel ribbons with molecular connectors.[2]

We report the synthesis and X-ray analysis of a family of robust layer-type supramolecular networks featuring an uncharged, linear GM⁺HCO₃⁻ (GM⁺ = guanidinium cation) fused-rosette ribbon hemmed with multiple N-H hydrogen-bond donor sites. Bridging of such parallel rosette ribbons by hydrogen-bond acceptors (1,4-cyclohexanedicarboxylate, 1,4-benzenedicarboxylate, 4-cyanobenzate and 1H-imidazole-4,5-dicarboxylate) as anionic molecular connectors is tolerant to a wide inter-ribbon separation ranging from 10.00 to 19.42 Å. Various tetraalkylammonium cations serve as guest templates to construct this series of four inclusion compounds.



Acknowledgment. The first author acknowledges financial support from The Open University of Hong Kong (Grant No.10/1.1)

[1] C.-K. Lam, F. Xue, J. P. Zhang, X. M. Chen, T. C. W. Mak, *J. Am. Chem. Soc.* **2005**, *127*, 11536; J. Han, C.-W. Yau, C.-K. Lam, T.C.W. Mak, *J. Am.*

Chem. Soc. **2008**, *130*, 10315; M.D. Ward, *Struct. Bond* **2009**, *132*, 1. [2] T.C. W. Mak, F. Xue, *J. Am. Chem. Soc.* **2000**, *122*, 9860.

Keywords: hydrogen bonding, inclusion compound, supramolecular assembly

MS61.P24

Acta Cryst. (2011) **A67**, C613-C614

Crystal structures of hydrazinecarbothioamide derivatives

Irena Wawrzycka-Gorczyca,^a Agata Siwek,^b ^a*Faculty of Chemistry, Marie Curie-Skłodowska University, Lublin, (Poland).* ^b*Faculty of Pharmacy, Medical University, Lublin, (Poland).* E-mail: irena.wawrzycka-gorczyca@poczta.umcs.lublin.pl

Thiosemicarbazide compounds exhibit various biological activities such as anti-bacterial, anti-fungal and especially antituberculosis. Besides, thiosemicarbazide derivatives offer special affinity to inhibit corrosion of metals in acidic solutions [1, 2]. In order to search for new thiosemicarbazides, the compounds **I-III** have been synthesized and their crystal structures are reported here:

I: *N*-(4-chlorophenyl)-2-[(4-methyl-1,3-thiazol-5-yl)carbonyl]hydrazinecarbothioamide,

II: 2-(2-methyl-3-furoyl)-*N*-(4-methylphenyl)hydrazinecarbothioamide,

III: *N*-{[2-(1*H*-pyrrol-2-ylcarbonyl)hydrazino]carbonothioyl}benzamide hemihydrate.

	I	II	III
crystal system	monoclinic	triclinic	monoclinic
space group	$P2_1/n$	$P-1$	$P2/c$
<i>a</i> [Å]	9.899(2)	9.441(3)	14.494(5)
<i>b</i> [Å]	8.059(1)	11.883(4)	4.835(2)
<i>c</i> [Å]	17.702(2)	14.463(6)	19.915(6)
α [°]		74.46(4)	
β [°]	97.51(1)	69.56(3)	96.51(3)
γ [°]		73.93(3)	

In the crystal lattice of **I**, inversion related molecules are joined by chelated N-H...O hydrogen bonds forming the cyclic dimer. The interactions are thioamide...carbonyl and hydrazine...carbonyl, and within this dimer they could be described by the cyclic first-level $R_2^2(10)$ and $R_2^2(14)$ motifs. Moreover, in this crystal, second type of cyclic dimer is observed; molecules are linked by C-H...S (chlorophenyl...thioamide) interactions – $R_2^2(12)$ graph set. Additionally, the molecular structure is stabilized by intramolecular N-H...O (hydrazine...carbonyl) hydrogen bond.

Crystals of **II** are triclinic ($P-1$) with $Z = 4$. Two symmetrically independent V-shaped molecules are linked by N-H...O (thioamide...carbonyl), C-H...O (methyl-furoyl...carbonyl and methylphenyl...carbonyl) and N-H...S (hydrazine...thioamide) hydrogen bonds to form a ribbon.

As in **I**, dimeric arrangement is also observed in the crystal structure of hemihydrate **III**. Nearly planar molecules are associated by inversion-related N-H...O (pyrrol...carbonyl) hydrogen bonds, the dimer is denoted by the cyclic $R_2^2(10)$ graph set. Further, the dimers connected by N-H...O (hydrazine...carbonyl) and $\pi\cdots\pi$ interactions built the three-dimensional crystal net. There is second kind of N-H...O hydrogen bond – an intramolecular (hydrazine...carbonyl) which stabilized the molecular structure. In addition to these interactions water...**III** hydrogen bonds are observed, they are N-H...O (thioamide...water) and O-H...S (water...thioamide).