

NMR signals. Eyring parameters for underlying dynamic processes were determined and compared to values modeled from a combined X-ray diffraction and computational study on selected *N*-(acyloxy)-4-methylthiazole-2(3*H*)-thiones [1,2,3]. The data suggest that the dynamic process occurs via N,O-rotation toward the less hindered face of the thiohydroxamate subunit. Topomerization toward the sterically more encumbered site, i.e. the thione sulfur, seems to require a stepwise two fold  $\sim 90^\circ$  N,O-rotation with an inserted back and forth flip about the *N*-acyloxy C,O-bond. The proposed mechanism has implications for interpreting decomposition chemistry, which is of tremendous importance for this product class [1].

[1] Hartung, J., Altermann, S., Bergstraesser, U., Gottwald, T., Kempter, I., Schur, C., Heubes, M., manuscript in preparation for Tetrahedron. [2] Hartung, J., Altermann, S., Svoboda, I., Fuess, H., *Acta Cryst.* **2005**, E61, o1738. [3] Hartung, J., Schwarz, M., Svoboda, I., Fuess, H., Duarte, M.-T., *Eur. J. Org. Chem.* **1999**, pp. 1275–1290.

**Keywords:** modelling; thiazolethione; variable temperature NMR

#### FA4-MS05-P27

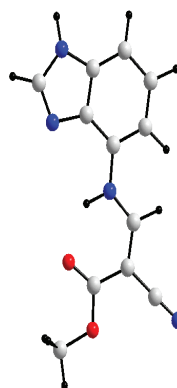
**Methyl 3-((benzimidazol-4(7)-yl) amino)-2-cyano-prop-2-enoate-powder Diffraction and DFT Study.** Lubomír Smrčok<sup>a</sup>, Rosanna Rizzi<sup>b</sup>, Pavel Mach<sup>a</sup>, Michela Brunelli<sup>c</sup>, Viktor Milata<sup>d</sup>. <sup>a</sup>*Institute of Inorganic Chemistry, Slovak Academy of Sciences, Dúbravská cesta 9, SK-845 36 Bratislava, Slovak Republic.* <sup>b</sup>*Istituto di Cristallografia - Sede di Bari Via Amendola, 122/o 70126 Bari, Italy.* <sup>c</sup>*European Synchrotron Radiation Facility, B.P. 220, F-38043 Grenoble CEDEX, France.* <sup>d</sup>*Faculty of Chemical and Food Technology, Slovak University of Technology, Radlinského 9, SK-812 37 Bratislava, Slovak Republic.* \*present address: ILL, Institut Laue-Langevin, B.P. 156, 38042 Grenoble cedex 9, France.  
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Monoclinic ( $P2_1/c$ ) crystal structure of the title compound was solved using RT synchrotron powder diffraction data (ID31/ESRF). The *ab initio* crystal structure determination was carried out by EXPO2009, the updated version of the EXPO2004 program [1]. A total of 879 extracted integrated intensities (450 independent observations because of the reflection overlapping) were used in the direct methods procedure to solve the crystal structure. The *E*-map corresponding to the best figure of merit (CFOM=0.999) was selected and optimized by applying cycles of least-squares to the molecular models derived from the electron density maps, followed by  $2F_o - F_c$  electron density calculations. In each electron density map peaks were automatically located and their connectivity established by the standard EXPO2004 routines. The structure was refined by geometry optimization done by energy minimization in solid state using DFT/plane waves approach [2]. Molecules in the structure are assembled in ribbons running approximately parallel to (010). Each molecule (see figure below) in a

ribbon is connected to the adjacent with three hydrogen bonds: two moderate N-H...N1 and a weaker C-H...O. The ribbons are linked by three weak C-H...N bonds with the –CN groups playing the role of acceptor.

[1] Altomare, A., Caliandro, R., Camalli, M., Cuocci, C., Giacovazzo, C., Moliterni, A.G.G. and Rizzi, R., **2004**. *J. Appl. Cryst.* **2004**, 37, 1025. [2] Smrčok, L., Jorik, V., Scholtzová, E. and Milata, V., **2007**. *Acta Cryst* **2007**, B63, 477; Smrčok, L., Brunelli, M., Boča, M., and Kucharik, M. *J. Appl. Cryst.* **2008**, 41, 634.

**Keywords:** powder diffraction; DFT



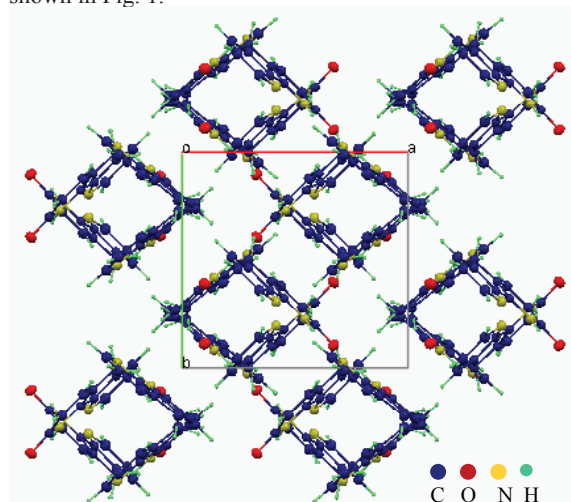
#### FA4-MS05-P28

**Synthesis, Characterization and Crystal Packing-Geometry of 1,5-dihydro-2H-cyclopenta [1,2-b:5,4-b']dipyridin-2-One.** Leyla Tatar Yıldırım<sup>a</sup>, Akın Baysal<sup>b</sup>, Feyyaz Durap<sup>b</sup>, Saim Ozkar<sup>c</sup>. <sup>a</sup>*Hacettepe University, Department of Engineering Physics, Beytepe, TR-06800, Ankara, Turkey.* <sup>b</sup>*Dicle University, Department of Chemistry, TR-21280 Diyarbakir, Turkey.* <sup>c</sup>*Middle East Technical University, Department of Chemistry, TR-06531 Ankara, Turkey.* E-mail: [tatar@hacettepe.edu.tr](mailto:tatar@hacettepe.edu.tr)

Chemistry of diazafluorene has been rarely reported [1, 2]. Diazafluorene is a weaker ligand than bipy (in spectrochemical series), which translates into a significant energy change in the ligand-field (LF) states [3]. Consequently, diazafluorene complexes are expected to exhibit spectroscopic properties different from those with 2,2'-bipyridine (bipy) and 1,10-phenanthroline (phen) ligands.

Using hydrazine as reducing agent, 1H-cyclopenta[1,2-b:5,4-b']dipyridine-2,5-dione [2,4] was selectively reduced to 1,5-dihydro-2H-cyclopenta[1,2-b:5,4-b']dipyridin-2-one. 1,5-dihydro-2H-cyclopenta[1,2-b:5,4-b']dipyridin-2-one was isolated as an analytically pure, colorless, solid and characterized by IR, <sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectroscopy, as well as elemental analysis. The crystal-packing have been structurally characterized by the single crystal X-ray crystallography. The crystal structure of the compound is involved intermolecular hydrogen bonds and, the short van der Waals interactions including helical and linear strand formation. The molecular packing in the solid state is square pyramidal porous structure along the c axis, as

shown in Fig. 1.



**Fig.1.** Packing diagram of the title compound.

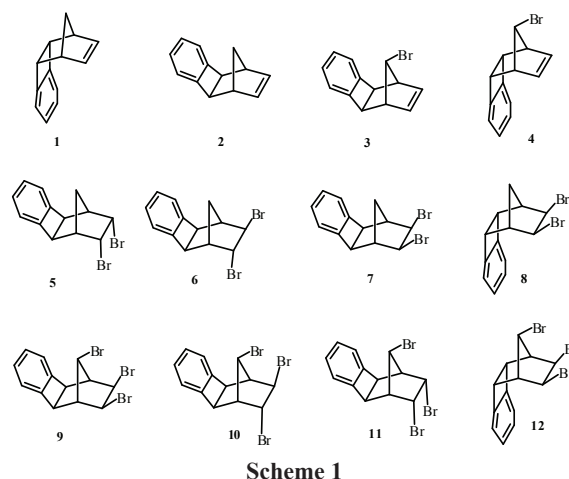
[1] M. Riklin, A. von Zelewsky, A. Bashall, M. McPartin, A. Baysal, J.A. Connor, J.D. Wallis, *Helv. Chim. Acta*, **1999**, 82, 1666. [2] A. Baysal, F. Durap, B. Gümgüm, L. T. Yıldırım, D. Ülkü, D. A. Boğa, S. Özkar, *Helv. Chim. Acta*, **2007**, 90, 1211. [3] L. J. Henderson, Jr., F. R. Fronczek, W. R. Cherry, *J. Am. Chem. Soc.*, **1984**, 106, 5876. [4] D. E. Marx, A. J. Lees, *Inorg. Chem.*, **1987**, 26, 620.

**Keywords:** diazafluorene; bipy; phen; crystal packing

#### FA4-MS05-P29

**Structural Analysis of *endo*- and *exo*-Benzocyclobutenonorbornene-Dibromides by Using  $^1\text{H}$ ,  $^{13}\text{C}$  NMR and X-Ray Diffraction Techniques.** Ertan Şahin<sup>a</sup>, Baris Anıl<sup>a</sup>, Arif Daştan<sup>a</sup>, Cavit Kazaz<sup>a</sup>. <sup>a</sup>Atatürk University, Faculty of Sciences, Department of Chemistry, 25240-Erzurum Turkey. E-mail: [ertan@atauni.edu.tr](mailto:ertan@atauni.edu.tr)

Several monobromides, dibromides and tribromides derived from *endo*- and *exo*-benzocyclobutenonorbornene were synthesised and published by Daştan co-worker for several purposes [1]. This kind of products are also important to investigate “the  $\gamma$ -gauche effect” in NMR spectroscopy [2]. In this study, we study on spectroscopic data of isomeric compounds by correlating exact conformations obtained by X-ray diffraction analysis.  $^1\text{H}$ -NMR,  $^{13}\text{C}$ -NMR, DEPT, gCOSY, gHMBC and GHMBC and double resonance techniques were used to determine the exact signal for each nucleus.



**Scheme 1**

[1] a) E. Uzundumlu, Bromination of *Endo*- and *Exo*-Benzocyclobutenonorbornene at different conditions. Master Thesis, Atatürk University Graduate School of Natural and Applied Sciences Department of Chemistry, Erzurum 2003. b) A. Dastan, E. Uzundumlu, M. Balci, F. Fabris, O. De Lucchi *Eur. J. Org. Chem.* **2004**, 183-192. [2] a) M. D. Gheorghiu, E. Olteanu, *J. Org. Chem.* **1987**, 52, 5158-5162. b) C. Kazaz, A. Dastan, M. Balci. *Magn. Reson. Chem.* **2005**, 43, 75-81.

**Keywords:** isomeric compounds; dibromides; single crystal

#### FA4-MS05-P30

**Crystal Structure of 1,1,3-Trioxo-2,3-dihydro-1,2-benzisothiazol-2-ylmethyl 4-phenyl piperazine-1-carbodithioate**,  $\text{C}_{19}\text{H}_{19}\text{N}_3\text{O}_3\text{S}_3$  Mehmet Akkurt<sup>a</sup>, Serife Pinar Yalçın<sup>a</sup>, Özlen Güzel<sup>b</sup>, Aydın Salman<sup>b</sup>, Orhan Büyükgüngör<sup>c</sup>. <sup>a</sup>Erciyes University, Graduate School of Natural and Applied Sciences, Kayseri, Turkey. <sup>b</sup>Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Istanbul University, 34116 Istanbul, Turkey. <sup>c</sup>Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey. E-mail: [serifepinar@gmail.com](mailto:serifepinar@gmail.com)

Title compound (Fig 1) is imported that Dithiocarbamates which found in its structure are appreciated as fungicidal [1-4], antibacterial and anticancer agents. In this compound, the mean planes of the benzisothiazole system and the phenyl ring make a dihedral angle of 8.87 (8)<sup>0</sup>. The piperazine ring has a chair conformation. The crystal structure is stabilized by weak intermolecular C-H...O interactions and weak intramolecular C-H...S interactions.

Using Stoe IPDS II diffractometer system, it was found that Crystal system of  $\text{C}_{19}\text{H}_{19}\text{N}_3\text{O}_3\text{S}_3$  was Triclinic, space group

$P\bar{1}$ ,  $a = 8.0390(5)\text{Å}$ ,  $b = 11.7619(7)\text{Å}$ ,  $c = 11.8796(8)\text{Å}$ ,  $\alpha = 109.029(5)^\circ$ ,  $\beta = 103.791(5)^\circ$ ,  $\gamma = 102.326(5)^\circ$ ,  $Z = 2$ ,  $D = 1.472 \text{ Mgm}^{-3}$ ,  $\mu = 0.41 \text{ mm}^{-1}$ ,  $R = 0.0291$ ,  $wR_2 = 0.0764$ ,  $S = 1.04$ .