

properties [1]. However, one of the mayor difficulties in the synthesis of these materials has been to prepare pro-ligands with structural conformations and chemical properties close enough to the ligands around the metal in the protein to avoid problems associated with molecular recognition in the catalytic process. In this work, we have attempted to study the molecular and crystalline structure by means of X-ray powder diffraction and theoretical calculations using density functional tight-binding methods (DFTB) [2, 3] of three new pro-ligands of the type [N<sub>2</sub>S<sub>2</sub>] tetradentate and [NS] bidentate: methyl *N,N'*-buthyl-bis(2-amino-1-cyclope ntendithiocarboxidithioate (compound I), (1-methyl)-proyl and methyl 2,4-bis(cyclohexane)dispiro-[1,2,3,4,4a,5,6,7]-octahydro-(1H,3H)quinazoline-8-carboxydi-thioate (compounds II and III). The molecular conformation for both compounds, which are subjected to internal hydrogen bonds and electron charge delocalization in the carboxyditioate group, and the crystal packing of the compounds will be discussed. Finally, the molecular structures obtained by X-ray powder diffraction are compared with the ones optimized by DFTB calculations.

[1] Solomon, E. I., Szilagy, R. K., DeBeer, G. S. & Basumallick, L. **2004**, *Chem. Rev.*, 104, 419. [2] Porezag, D., Frauenheim, Th., Köhler, Th., Seifert, G. and Kaschner, R. **1995**, *Phys. Rev.*, B51, 12947. [3] Elstner, M., Porezag, D., Jungnickel, G., Eisner, J., Haugk, M., Frauenheim, Th., Suhai, S. & Seifert, G. **1998**, *Phys. Rev.* B58, 7260.

**Keywords:** powder diffraction; structure solution; solid state DFTB calculations

#### FA5-MS01-P21

**The D20 Instrument at the ILL: A Versatile High-Intensity Two-axis Neutron Diffractometer. Recent Developments and Future Prospective.** Thomas Hansen<sup>a</sup>, Henry E. Fischer<sup>a</sup>, Paul F. Henry<sup>b</sup>, Pierre Convert<sup>a</sup>, Michela Brunelli<sup>a</sup>, Jacques Torregrossa<sup>a</sup>. <sup>a</sup>ILL Institut Laue-Langevin, BP 156, 38042 Grenoble cedex 9, France. <sup>b</sup>Helmholtz Zentrum Berlin, Glienicke Str., 100, D-14109, Berlin, Deutschland. E-mail: [brunelli@ill.eu](mailto:brunelli@ill.eu)

D20 is a high intensity neutron 2-axis diffractometer based at the high-flux reactor of the Institut Laue-Langevin (ILL), in Grenoble (France). In spite of ever increasing competition from powder diffractometers at new neutron sources, the D20 diffractometer at the ILL remains at the top of its class, mating high flux to high-resolution while offering the convenience and reliability of reactor-based operation. Extensive possibilities in instrument configuration and sample environment lead to a rich and varied scientific production, as attested by over 40 publications per year in refereed journals. It has undergone a variety of improvements and new developments during the years [1-4], and due to the extremely high neutron flux, it opens up a large range of possibilities for real-time experiments on very small samples. Typical experiments performed at D20 include accurate powder diffraction for structural refinement, magnetic structure determination,

thermodiffractometry, pressure dependence, crystallization kinetics, *in situ* chemical reactions, highly neutron-absorbing samples among others. Such success encourages the future improvements and developments envisaged for the D20 instrument.

[1] Hansen T, *Meas. Sci. Technol.* 19, **2008**, 034001 (6pp). [2] Hansen T C, **2004**, *Mater. Sci. Forum* 443-444 181-6. [3] Convert P, Hansen T, Oed A and Torregrossa J, **1997**, *Physica B* 241-243 195-7. [4] Convert P, Hansen T and Torregrossa J, **2000**, *Mater. Sci. Forum* 321-324 314-9.

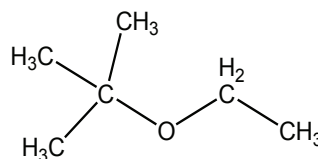
**Keywords:** neutron instrumentation; neutron diffraction; powder diffraction

#### FA5-MS01-P22

**Crystal-structure Prediction for Ethyl-*tert*-butylether.** Sonja M. Hammer<sup>a</sup>, Edith Alig<sup>a</sup>, Jürgen Glinnemann<sup>a</sup>, Martin U. Schmidt<sup>a</sup>. <sup>a</sup>Institute of Inorganic and Analytical Chemistry, University of Frankfurt, Max-von-Laue-Str. 7, D-60438 Frankfurt am Main, Germany.

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For the anti-knocking agent Ethyl-*tert*-butylether (ETBE, (CH<sub>3</sub>)<sub>3</sub>COC<sub>2</sub>H<sub>5</sub>, mp. 176 K) no crystal structure is reported up to now. The program CRYSCA<sup>[1]</sup> with a Dreiding/X6<sup>[2]</sup> force field was applied to predict possible crystal structures.



The prediction was performed in the most frequent space groups for organic crystals<sup>[3]</sup>: *P1* (*Z* = 1), *P-1* (*Z* = 2), *P2*<sub>1</sub> (*Z* = 2), *Cc* (*Z* = 4), *C2* (*Z* = 4), *C 2/c* (*Z* = 8), *P2*<sub>1/c</sub> (*Z* = 4), *P2*<sub>2</sub><sub>1</sub> (*Z* = 4), *Pna*<sub>2</sub> (*Z* = 4), *Pca*<sub>2</sub> (*Z* = 4) and *Pbca* (*Z* = 8), each with one flexible molecule per asymmetric unit. During the minimisation, supergroups of these spacegroups could occur.

Predicted crystal structures with low energies were subsequently optimised with the program package Cerius<sup>2</sup> (force field: Dreiding/X6). During these optimisations additional space groups were reached. The optimised structures within an appropriate energy range above the global minimum represent possible polymorphs.

Low-temperature X-ray diffraction experiments are carried out to confirm the results.

[1] M. U. Schmidt, H. Kalkhof, *CRYSCA, Program for crystal structure calculations of flexible molecules*, Frankfurt am Main **2002**. [2] S. L. Mayo, B. D. Olafson, W. A. Goddard III, *J. Phys. Chem.* **1990**, 94, 8897-8909. [3] Cambridge Structural Database Space group statistics, [http://www.ccdc.cam.ac.uk/products/csd/statistics/space\\_group\\_stats.php4](http://www.ccdc.cam.ac.uk/products/csd/statistics/space_group_stats.php4); update 1 January 2009.

**Keywords:** crystal structure prediction; lattice energy calculations; crystal structure solution