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Keywords: ferroelectric oxides; synchrotron x-ray powder diffraction; size-strain

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Structural Phase Transitions in Nanoscaled Systems with Jahn-Teller Ions. Sophia Petrova^c, Eugenia Vykhodets^d, Robert Zakharov^c, Mikhail Ivanov^b, Nikolai Tkachev^a, Anatolii Fishman^c. ^a*Institute of High-Temperature Electrochemistry UD RAS.* ^b*Institute of Metallophysics NAS of Ukraine.* ^c*Institute of Metallurgy UD RAS.* ^d*Urals' State Technical University (USTU-UPI).*

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Specific features of the structural phase transitions of the first order were investigated in nanosized crystals with Jahn-Teller (JT) ions. As an example the phase transitions of martensite type, with changes in symmetry from a cubic (S) to a tetragonal (H) one, have been considered. It was shown the temperature and the latent heat of the transition decrease significantly for the nanoscaled grains. A possibility of multi-phase state with a coexistence of low-symmetry (H) and high-symmetry (S) JT-phases was predicted for the nanocrystalline materials [1] and proved for the nanoscaled Mn-O system (figure). With a certain ratio among parameters of cooperative interactions and random crystal fields at JT ions the structural phase transition can be reduced or suppressed totally. To explain the results the Kanamori model taking into account the size of nanocrystallites, the distribution of cations over non-equivalent crystallographic sublattices as well as the presence of random crystal fields in such systems was used. Figure. Temperature dependence of crystal lattice parameters and a subsequence of phase transitions during heat treatment of the course-grained (closed marks) and nanoscaled (open marks) oxide Mn₂O₃.

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Keywords: structural phase transitions; X-ray high-temperature powder diffraction; nanocrystalline materials

FA5-MS01-P19

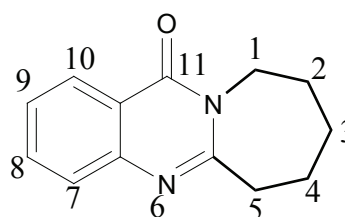
Structure Determination on Asthmatic Drugs by Synchrotron Powder Diffraction. Jey-Jau Lee^a, S. Thamotharan^b, J. Jeyakanthan^a, R. Bansal^c, R. Yadav^c. ^a*National Synchrotron Radiation Research Center, Taiwan.R.O.C.* ^b*Institute of Bioinformatics and Structural Biology, Department of Life science,*

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Asthma is a popular disease nowadays. There are many factors which are responsible for inducing symptoms of asthma. Many classes of drugs namely bronchodilators and anti-inflammatory agents have resulted in substantial improvement in survival and quality of life of asthmatic patients. Here we aim to search for a new chemical entity possessing better bronchodilatory effects and find out the structure solution by mean of synchrotron powder diffraction.

2,3,4,5-tetrahydroazepino[2,1-*b*]quinazolin-11(1*H*)-one(1) has been studied as a template. In addition, a number of nitrogenous functions were introduced at position 8, 9. The synthesized compounds were studied for their bronchodilatory activity using isolated guinea pig tracheal chain. The 3-D structure is also a key feature to understand their function. In this report, three series asthmatic drug targets structures are successfully solved by synchrotron powder diffraction data and combined simulated annealing method.



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Keywords: structure-aided drug design; structure from powder diffraction; quinazoline

FA5-MS01-P20

Study of the Molecular and Crystalline Structure of Three Nitrogen-Sulphur Pro-Ligands by X-Ray Powder Diffraction and Solid State Dftb Calculations. Asiloé J. Mora^a, Edward E. Ávila^a, Gerzon E. Delgado^a, Andrew N. Fitch^b, Michela Brunelli^c, Ricardo R. Contrera^a, Luis Rincón^a. ^a*Departamento de Química, Facultad de Ciencias, Universidad de Los Andes, Mérida, Venezuela.* ^b*European Synchrotron Radiation Facility, BP220, F-38043 Grenoble CEDEX, France.* ^c*ILL Institut Laue-Langevin, BP 156, 38042 Grenoble cedex 9, France.*

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In the last decades, inorganic chemist has faced the need to mimic the properties of metal sites in metalloproteins to synthesize new catalytic materials with advantageous

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 [N₂S₂] 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 carboxydithioate 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.

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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^a, Henry E. Fischer^a, Paul F. Henry^b, Pierre Convert^a, Michela Brunelli^a, Jacques Torregrossa^a. ^aILL Institut Laue-Langevin, BP 156, 38042 Grenoble cedex 9, France. ^bHelmholtz Zentrum Berlin, Glienicke Str., 100, D-14109, Berlin, Deutschland. E-mail: 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.

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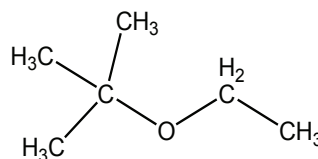
Keywords: neutron instrumentation; neutron diffraction; powder diffraction

FA5-MS01-P22

Crystal-structure Prediction for Ethyl-*tert*-butylether. Sonja M. Hammer^a, Edith Alig^a, Jürgen Glinnemann^a, Martin U. Schmidt^a. ^aInstitute 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, (CH₃)₃COC₂H₅, mp. 176 K) no crystal structure is reported up to now. The program CRYSCA^[1] with a Dreiding/X6^[2] force field was applied to predict possible crystal structures.



The prediction was performed in the most frequent space groups for organic crystals^[3]: *P1* (*Z* = 1), *P-1* (*Z* = 2), *P2₁* (*Z* = 2), *Cc* (*Z* = 4), *C2* (*Z* = 4), *C 2/c* (*Z* = 8), *P2₁/c* (*Z* = 4), *P2₂2₁* (*Z* = 4), *Pna2₁* (*Z* = 4), *Pca2₁* (*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 Cerius² (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.

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