This is confirmed by the DFT Bader charge of ~+3.7 e in the present compound; in the phosphonates the charge was ~+3.4 e. This difference may be attributed to the presence of an additional oxygen atom in the phosphate group. In the experimental approach the carboxylic O and O(H) oxygen atoms show almost equal charges of 0.92 and 0.97 e respectively, which is not supported by the DFT results - higher on the average by ~0.3 e for these atoms. The phosphate O(H) atom displays a lower or slightly lower charge than the other phosphate oxygen atoms. This is consistent with previous findings [3].
The respective values are -1.43 e for O(H) and -1.46 – 1.67 e for the remaining oxygen atoms in the experimental approach.

The reported topological properties are comparable to those of hydrogen methylphosphonates of calcium and lithium with P-O ρ_c values being near 1.55 e·Å^{-3} and P-O(H) near 1.31 e·Å^{-3}. Interestingly the value of ρ_c of P-O(C) bonds is also near to 1.30 e·Å^{-3} as in the P-O(H) bonds.

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Keywords: charge density, phosphoglycolate, phosphorus

L.A.07

Acta Cryst. (2011) A**67,** C813

A novel high-throughput approach for purification and reconstitution of large multi-protein complexes
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Proteins are both building blocks and molecular motors for virtually all cellular functions. To perform and regulate complex cellular processes, proteins form interacting heterogeneous assemblies (henceforth referred to as multi-protein complexes or MPCs) whose components exchange continuously during the process. As a result of the transient nature of their interactions, monomeric and multimeric components of MPCs typically have low binding affinities, making their recovery almost impossible during purification. Therefore assembly of MPCs often requires expression and purification of individual (monomeric and multimeric) components in a form (mono-dispersed and properly folded) conducive to reconstitution; furthermore, individual constituents of such large MPCs are frequently insoluble in the absence of the associated components. Thus, identifying conditions leading to MPC reconstitution represents a highly desirable goal in structural biology. To this end we have developed novel solubilization and purification protocols that have allowed us to: 1) solubilize individual monomeric and multimeric components of MPCs; 2) reconstitute (using previously solubilized components), fully active (and stoichiometric) MPCs. Such techniques work equally well on cytosolic and membrane proteins. Despite the intrinsic difficulties that MPCs represent to the field of structural biology, it is crucial to tackle these structural giants since we will only begin to comprehend their biology when the structures of the whole machines have been determined.

Keywords: Biochemistry, Protein, Complex

L.A.08

Acta Cryst. (2011) A**67,** C813

Crystallization of a serpin with a insulin-sensitizing function
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Serpins (serine protease inhibitors) are proteins with a molecular mass of about 50 kDa that inhibit chymotrypsin-like serine proteases via the formation of a covalent complex with the target protease. We purified and crystallized SERPINA12, that acts as an adipokine with insulin-sensitizing effects. It could be found in visceral adipose tissue of a rat model of type 2 diabetes. Sequence identity shared with α-1-antitrypsin (41%) suggests a putative serpin function but its molecular target remains to be identified.

To confirm and further analyze the predicted serpin function we intend to determine the three-dimensional structure of the serpin. Thus, His-tagged protein was produced in *Escherichia coli* and purified by Ni-affinity chromatography and size exclusion chromatography. High-troughput screening crystallization trials were prepared in a nanoliter scale with a MicroSys pipetting system using the sitting-drop technique. First diffracting crystals were observed with polyethylene glycol of a molecular weight of 3350 as the precipitant. Crystallization conditions were improved in hanging-drop setups to yield two different crystal forms (space groups $P2_1$ or C2) with similar habitus. C2 crystals show the highest diffraction limit (2.08 Å) and gave rise to a complete dataset collected at BESSY beamline 14.1 at Helmholtz-Zentrum Berlin (Germany).

Acknowledgements: This publication is supported by LIFE – Leipzig Research Center for Civilization Diseases, Universität Leipzig. LIFE is funded by means of the European Union, by the European Regional Development Fund (ERDF) and by means of the Free State of Saxony within the framework of the excellence initiative.

Keywords: protein, crystallization

L.A.09

Acta Cryst. (2011) A**67,** C813-814

Crystal structure of bacteriophage T4 fibre protein gp37
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Some viruses and bacteriophages attach to their host cell via specialised fibre proteins, like adenovirus, reovirus and tailed bacteriophages. The bacteriophage T4 short and long fibre proteins, the T5 L-shaped fibres and T7 fibre all have the same basic architecture: they are trimeric and contain an N-terminal phage attachment domain, a long, thin, but stable shaft domain and a more globular C-terminal cell attachment domain. Our goal is to determine the structures of these proteins and thus to make an inventory of stable trimeric folds present in nature.

Phage fibre proteins may also find applications in nanotecnology or in recognition of pathogenous bacteria and their elimination (phage therapy).

Bacteriophage T4 contains six short and six long fibres. The long fibres are responsible for initial host cell recognition. They contain a trimer of gp34, a single copy of gp35, a trimer of gp36, and a trimer of gp37. T4 fibre proteins need the chaperone gp57 for correct folding. Gp37 also needs a second chaperone, gp38. We have expressed gp37 by co-expression with gp57 and gp38 and solved the structure of the gp37 receptor-binding needle domain.

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Keywords: virus, fibre, structure

L.A.10

Acta Cryst. (2011) A**67,** C814

New copper based MOF´s built up from 1,2,3,4-cyclobutanetetracarboxylate ligand.

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One of the areas of solid-state chemistry that has shown remarkable growth over the last two decades concerns the synthesis and characterization of metal-organic magnetic materials (MOFs). As part of our ongoing study of molecular materials, we reported several studies of correlation between crystal structure and magnetic properties in a tetracarboxylate ligand with different metal ions [1]. In this communication we report the current studies with the 1,2,3,4-cyclobutanetetracarboxylate ligand $(cbt)^{-4}$. Three new copper(II) metal-organic compounds have been synthesized using the gel technique varying the stoichiometry of the reaction in an effort to prepare low density frameworks [2].

The $[Cu_2(cbt)(H_2O)_4]\cdot 2H_2O$ **(1)** complex is built up by two different inorganic tapes: one formed by dinuclear copper(II) assemblies, and a second one formed by triple-bridge copper(II) regular chains. These inorganic tapes are kept together through the cbt that acts as a connector in a hexaquis-monodentate way, giving rise to a 2D structure. The $[Cu_2(cbt)(H_2O)_4]\cdot 2H_2O$ **(2)** compound presents a 3D structure where the cbt acts as pentakis-monodentate, giving rise to a porous structure with two different types of square channels running along the *c*-axis. Crystallization and coordination water molecules are allocated in the bigger channels, while in the small ones there are only coordination water molecules. The $[Cu_5(cbt)_2(OH)_2\ (H_2O)_8]\ \cdot 8H_2O$ **(3)** compound presents a crystallographically independent cbt acting simultaneously as hexakis-monodentate and bidentate. This intricate coordination mode produces a 3D porous crystal structure, with cavities along the *b*-axis where the crystallization water molecules are allocated.

Here we present the X-ray single-crystal diffraction studies, the thermogravimetric analysis, and the magnetic studies of this new family of MOFs that opens new possibilities in the field of multifunctional materials.

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L.A.11

Acta Cryst. (2011) A**67,** C814

Preparation and molecular structure of the complex $[Sn^{IV}(C_{44}H_{28}N_4)(OCN)(OH)]$

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For a review of porphyrin complexes [1] and For the synthesis of tin(IV) porphyrin species [2] .

The title complex, $[Sn^{IV}(C_{44}H_{28}N_4)(OCN)(OH)]$, exhibits substitutional disorder of the OH^- and OCN^- axial ligands. The Bis[(cyanato-*O*)/(hydroxo)(0.5/0.5][(5,10,15,20-tetraphenylporphyrinato-κ^4N)tin(IV)] has been synthesized and characterized by UV-vis, IR and proton NMR and the tin ion presents the oxidation state IV, Thus, the cyanato-*O* ligand and the hydroxyl group bonded to the central Sn^{IV} atom share statistically the axial position. The X-ray molecular structure shows that the Sn^{IV} ion is hexacoordinated by the four N atoms of the pyrrole rings of the tetraphenylporphyrin (TPP) and the O atoms of the two disordered OCN^- and OH^- axial ligands. The equatorial tin–pyrrole N atom distance (Sn—Np) is 2.100 (2) A ° and the axial Sn—O(OCN) or Sn—O(OH) bond length is 2.074 (2) A°. The complex $[Sn^{IV}(C_{44}H_{28}N_4)(OCN)(OH)]$ crystallizes in the monoclinic space group $P2_1/c$ with unit cell dimensions a=11.2943(6) Å, b=12.6972(7) Å, c=13.0711(8) Å, β=114.251(2)°. The structure was refined to R=3.9%, WR2=9.7% and S=1.13.

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Keywords: Sn-porphyrin, cyanato-*O* ligand

L.A.12

Acta Cryst. (2011) A**67,** C814-815

Development of Cubic Anvil Type High Pressure Apparatus for Neutron Scattering at Low Temperature

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In recent years, as an important physical measurement device, various types of high pressure apparatus have been developed for each experimental purposes [1]. Among them, cubic (multi) anvil type apparatus is more suitable for good-hydrostatic pressure [2]. Another merit of this apparatus is that which has relatively large sample space. On the other hands, in the field of neutron experiments, high pressure technique is less common compared to other experiments due to the inevitable difficulty, such is a significant decrease in intensity by absorption and scattering when the neutrons pass through a pressure device which surrounds the sample: it is difficult to conduct experiments with reliability and accuracy.