

Poster Sessions

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Some Examples of Polyoxometalates Crystal Structure Defects

Gregory Leitus,^a Linda J.W. Shimon,^a Yael Diskin-Posner,^a Alexander M. Khenkin,^b Ronny Neumann,^b ^a*Chemical Research Support and* ^b*Organic Chemistry Department, Weizmann Institute of Science (Israel)*. E-mail: gregory.leitus@weizmann.ac.il

The study of Molybdenum and Tungsten polyoxometalate complexes is of fundamental importance in the context of both oxidation catalysis and biology. The possibility to build complexes with different structures and to attach to them varied organometallic molecules is related to oxygen and electron transport during enzymatic reactions [1].

X-ray crystal structure solution and refinement of poly-oxometalate single crystals often involves some difficulties usually related to different disorders in their crystal packing. The structural framework of these compounds is formed by joining complexes W, Mo – O, which can be presented in variety of different shapes, like spheres, ellipsoids, wheels and others. The complexes can be connected to each other by a network of solvent water molecules and cationic counter ions or by different organic or organometallic groups. Large accessible voids between the complexes usually allow inserting additional atoms or groups. The sufficiently weak bonds between different structural groups and the inherent high symmetry of the polyoxometalate can cause several types of disordering. Metal atoms of the complexes may also be partially substituted by other metal atoms, which in turn can be disordered distributed inside of the complexes.

Different types of disordering and partial or mixed occupancies at certain positions can cause small changes in the symmetry of the crystals, which can give rise to supersymmetry or even to an aperiodic structure. Lack of small reflections with high resolution can prevent from finding correct symmetry group and thereafter from obtaining structure solution.

[1] A.M. Khenkin, G. Leitus, R. Neumann *J. Am. Chem. Soc.* **2010**, *132*, 11446–11448.

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Supercell approximation for an incommensurately modulated crystal of profilin:actin

Jason C. Porta*, Jeffrey J. Lovelace, Gloria E.O. Borgstahl, *Eppley Institute for Research in Cancer and Allied Diseases, University of Nebraska Medical Center, Omaha, NE*. E-mail: jporta@unmc.edu

A crystal structure from an incommensurately modulated protein crystal has never been reported, mainly due to a lack of methods for solving these unique structures. Here we report the first modulated protein structure that was determined using q-vectors to accurately integrate the diffraction data followed by a commensurate supercell approximation. Crystal modulation is characterized by a loss of short-range translational symmetry, where a single unit cell is no

longer sufficient to accurately describe the structure. Such a loss of periodicity is often caused by dynamic processes within the crystal arising from, for example positional modulations. Experimentally, the incommensurately modulated state is characterized by the appearance of distinct satellite reflections surrounding the main Bragg reflections on the diffraction pattern that cannot be indexed with a supercell. In order to fully describe the modulated structure, and hence the dynamic processes within the crystal, one must explore higher-order space over multiple unit cells. By careful examination of atomic positions over higher dimensional space, a modulation function can be calculated that traces the atomic disorder. Such a function is periodic, but incommensurate with the crystal lattice. Another form of modulation occurs when the atomic disorder is commensurate with the main lattice. In this case, the intensities can be indexed as a large, or 'supercell'. In the case of incommensurately modulated profilin:actin crystals, the data was not able to be indexed as a supercell by use of existing software. The diffraction data had to be indexed and integrated using the q-vector approach. Data were observed to 2.4 Å and were processed with a single q-vector with *EVAL15* software. The main and satellite reflections were indexed and described by an orthorhombic main unit cell of a=37, b=71, c=185 Å and the q-vector $q=0.2829\dots b^*$. From this q vector we know that the modulation repeats at approximately, but not exactly, three and a half unit cells in the b direction. To approximate the supercell, the data was then reindexed using a supercell approximation (assuming the q vector is close to 2/7; then $h=h$, $k=7k+2m$, $l=l$). The supercell dimensions were a=37, b=498, c=185 Å with space group $P2_12_12_1$. A previously refined periodic PA structure was used as a search model and molecular replacement using *MOLREP* produced a ribbon structure with 6 PA copies. Analysis of electron density maps made clear the position of the 7th copy, which was fit manually. Then refinement was performed with *REFMAC* using 7 rigid groups for each actin and one for profilin. In the resultant map there was interpretable density for ATP. In this preliminary structure the motion appears to involve a rotation in the ac plane and is in general agreement with the analysis of the average structure, which by analysis of Fourier electron density maps indicate major motion in actin subdomains 2 and 4. The position of profilin seems to change the least. The structural information gleaned from this approximation will help bootstrap the solution of the incommensurate structural refinement in (3+1)D superspace.

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Service crystallography at the university of santiago de compostela

Antonio L. Llamas-Saiz, Bruno Dacuña-Mariño, Oscar Lantes, Guillermo Zaragoza, Inés Fernández, Juan Rama. *Unidad de Rayos X, RIAIDT, Edificio CACTUS, Campus Vida, Universidad de Santiago de Compostela, E-15782 Santiago de Compostela (Spain)*. E-mail: antonio.llamas@usc.es

The latest technological developments in X-Ray production, optics and detection, the automation of crystallization, sample handling and conditioning, and the improvement in computing facilities combined with new methodologies have converted crystallographic analysis to routine in many cases, mainly for small and medium-size molecules. The standardization of these techniques allowed their use by non-specialized users inside Departments and to be included in the general services offered by Universities and other research institutions. However, there are still many situations, even in simple systems, where the assistance of an expert crystallographer is required.

We describe here our experience with the set up and development of the X-Ray Unit at the University of Santiago de Compostela (USC)