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QUASI-PERIODIC CRYSTALS - THE ROLE OF ELECTRON DIFFRACTION

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The discovery of the Icosahedral Phase in 1982 was one of the striking victories of transmission electron microscopy. Indeed, the first observations of dislocations and early stages of precipitation processes in the first days of electron microscopy were extremely important, and contributed a great deal to modern materials science, but the role of TEM has been mainly supportive, rather than ground breaking. Most of the crystalline defects, so elegantly studied by TEM, thanks to our understanding of contrast phenomena, were known before. The support of TEM, which produced dramatic images and diffraction patterns provided in most cases a final proof or rejection of existing theories and speculations. For the crystallographers' community, X-ray diffraction was the undisputed king, the reliable research tool in town. Enters the Icosahedral Phase, the first observed Quasi-Periodic crystal. Unexpected, not predicted, and flatly rejected by the paradigm: 'A crystal is ordered and periodic'. TEM was the only tool by which the I phase could have been discovered. The techniques needed for structural analysis of the I phase: contrast analysis, composition analysis and high-resolution images are all standard in modern transmission electron microscopy. X-ray diffraction could not provide any of the these, since the first produced I phase crystals were only a few microns in size, and the specimens contained several phases in addition to the I phase. The results provided in the first article on the I phase in 1984 were very convincing to the TEM community, but very controversial in the community of crystallographers. It was not until 1987 that large enough crystals were grown to enable single crystal x-ray diffraction, and the community of crystallographers was slowly convinced. Electron diffraction in the transmission electron microscope became a major tool for the discovery of new crystal structures. Most, if not all of the new quasi-periodic structures that were found since 1984 were discovered by electron diffraction in the TEM. In 1992 a committee of the International Union of Crystallographers changed the definition of a crystal to include Quasi-periodic crystals, but the new definition is more than that, for it defines 'Crystal', for the first time, through its reciprocal space.

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INHIBITOR DESIGN AND THE CHOLERA TOXIN FAMILY - FROM WATER PUZZLES TO 10 KD SPIDERS

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Bacterial Toxins employ ingenious mechanisms for assembly, entering target host cells, and wreaking havoc in those cells. Protein crystallographic studies in numerous groups have provided profound insight into the mode of action of these toxins at the atomic level. Cholera toxin (CT) and its relatives like heat labile enterotoxin from enterotoxigenic E. coli (LT), are heterohexameric AB5 multimers which are assembled in the periplasm and, in the case of cholera toxin, are secreted across the outer membrane in a folded stage. The B-subunits recognize GM1 receptors on the outer membrane of the host epithelial cells and the A subunit is launched on an adventurous journey and eventually reaches the cytoplasm where it ADP-ribosylates Gs-alpha. This leads to continuous stimulation of adenylyl cyclase, excess of cAMP, stimulation of ion channels and, in extreme cases, death within eight hours.

The crystal structure of the members of the cholera toxin family have been a rich source of inspiration for the design and synthesis of inhibitors. Examples will be given, in particular of the development of several generations of receptor binding antagonists. The displacement of certain water molecules appears only to be a prerogative of highly specific functional groups; combinatorial chemistry allowed exploration of a wide binding site; and multivalent ligands of over 10,000 Daltons and long flexible arms, have been obtained and appear to be 100,000 times more powerful than the monovalent starting point. These large receptor-antagonists could be co-crystallized with the toxin and show the way for further enhancements in affinity.

Keywords: DRUG DESIGN, CHOLERA TOXIN, MULTIVALENT INHIBITORS

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PHASING OF COHERENT X-RAY DIFFRACTION FROM NANOCRYSTALS

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Laue showed in 1936 that the shape of a small crystal appears as a fine structure to the peaks of its diffraction pattern. This explains why diffraction from surfaces occurs as a 'crystal truncation rod' intensity in between the Bragg peaks. A facetted nanocrystal will have such rods pointing in the directions of each of its surfaces, which can then mutually interfere. Using the high coherence of modern synchrotron radiation sources we observe this fine structure in the Bragg peaks from small gold crystals. Because such a diffraction pattern is a continuous function of reciprocal space, it can be over sampled with respect to the spatial Nyquist frequency. In this case the diffraction phase is over determined and can be obtained by iterative methods. We have employed Gershberg-Saxton-Fienup methods to invert such diffraction patterns to produce three-dimensional images of the interior structure of the gold nanocrystals.

Keywords: PHASING SURFACES IMAGING

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IN SITU CRYSTALLIZATION TECHNIQUES: TOOLS FOR CRYSTAL ENGINEERING AND CO-CRYSTALLIZATION OF GASEOUS COMPOUNDS

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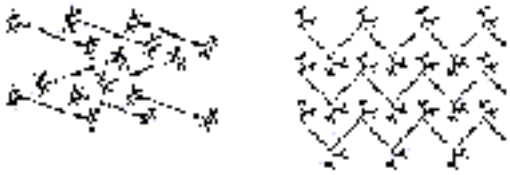
Single crystal growths in capillaries on the diffractometer, i.e. in situ crystallization, is desired or sometimes inevitable for low melting compounds, if intermediate phases exist or if the compound is temperature sensitive. Using an apparatus with an infrared laser beam for a miniature zone melting procedure allows having a maximum control on the crystallization process. Numerous examples exist for successful single crystal structure determinations applying such techniques, either for molecular structure evaluation or for crystal packing analyses. One of the challenges in crystal engineering is the co-crystallization of compounds utilizing specific intermolecular interactions to achieve well-defined motifs in the crystalline lattice. Based on the experience and knowledge gained from structures which involve C≡C-H···X interactions (X = O, N), we co-crystallized acetylene with acetone under elevated pressure, forming molecular complexes and networks in a 1:1 and a 1:2 complex, respectively (see below). In situ crystallization of gases can be extended to other systems ranging from molecular to inclusion complexes, e.g. complexes of acetylene and dioxane or methanol to clathrates and gas hydrates, which have a pronounced economical relevance.



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