DOWNLOAD PDF CRYSTALLOGRAPHIC INSTRUMENTATION

Chapter 1 : (IUCr) OpenlLabs

Innovations in crystallographic instrumentation and the rapid development of methods of diffraction measurement have led to a vast improvement in our ability to determine crystal and molecular structure.

Development from to [edit] Although diamonds top left and graphite top right are identical in chemical compositionâ€”being both pure carbon â€”X-ray crystallography revealed the arrangement of their atoms bottom accounts for their different properties. In diamond, the carbon atoms are arranged tetrahedrally and held together by single covalent bonds , making it strong in all directions. By contrast, graphite is composed of stacked sheets. Within the sheet, the bonding is covalent and has hexagonal symmetry, but there are no covalent bonds between the sheets, making graphite easy to cleave into flakes. The earliest structures were generally simple and marked by one-dimensional symmetry. However, as computational and experimental methods improved over the next decades, it became feasible to deduce reliable atomic positions for more complicated two- and three-dimensional arrangements of atoms in the unit-cell. The potential of X-ray crystallography for determining the structure of molecules and mineralsâ€”then only known vaguely from chemical and hydrodynamic experimentsâ€”was realized immediately. The earliest structures were simple inorganic crystals and minerals, but even these revealed fundamental laws of physics and chemistry. The first atomic-resolution structure to be "solved" i. Bragg was known to compare crystal formation to "curtains, wallpapers, mosaics, and roses". One of the leading scientists of the project was Dr. Megaw is credited as one of the central figures who took inspiration from crystal diagrams and saw their potential in design. The initial studies revealed the typical radii of atoms, and confirmed many theoretical models of chemical bonding, such as the tetrahedral bonding of carbon in the diamond structure, [28] the octahedral bonding of metals observed in ammonium hexachloroplatinate IV , [47] and the resonance observed in the planar carbonate group [31] and in aromatic molecules. These rules led to the structure of brookite and an understanding of the relative stability of the rutile , brookite and anatase forms of titanium dioxide. The distance between two bonded atoms is a sensitive measure of the bond strength and its bond order ; thus, X-ray crystallographic studies have led to the discovery of even more exotic types of bonding in inorganic chemistry , such as metal-metal double bonds, [52] [53] [54] metal-metal quadruple bonds, [55] [56] [57] and three-center, two-electron bonds. In material sciences, many complicated inorganic and organometallic systems have been analyzed using single-crystal methods, such as fullerenes , metalloporphyrins , and other complicated compounds. Single-crystal diffraction is also used in the pharmaceutical industry , due to recent problems with polymorphs. Mineralogy and metallurgy[edit] First X-ray diffraction view of Martian soil â€” CheMin analysis reveals feldspar , pyroxenes , olivine and more Curiosity rover at " Rocknest ", October 17, The application of X-ray crystallography to mineralogy began with the structure of garnet , which was determined in by Menzer. A systematic X-ray crystallographic study of the silicates was undertaken in the s. Machatschki extended these insights to minerals in which aluminium substitutes for the silicon atoms of the silicates. The first application of X-ray crystallography to metallurgy likewise occurred in the mids. The green, red, yellow and blue spheres represent atoms of carbon , oxygen , sulfur and nitrogen , respectively. The white spheres represent hydrogen , which were determined mathematically rather than by the X-ray analysis. The first structure of an organic compound, hexamethylenetetramine , was solved in A significant advance was the structure of phthalocyanine , [85] a large planar molecule that is closely related to porphyrin molecules important in biology, such as heme , corrin and chlorophyll. X-ray crystallography of biological molecules took off with Dorothy Crowfoot Hodgkin , who solved the structures of cholesterol , penicillin and vitamin B12 , for which she was awarded the Nobel Prize in Chemistry in In , she succeeded in solving the structure of insulin , on which she worked for over thirty years. Such proteins are long, linear molecules with thousands of atoms; yet the relative position of each atom has been determined with sub-atomic resolution by X-ray crystallography. Since it is difficult to visualize all the atoms at once, the ribbon shows the rough path of the protein polymer from its N-terminus blue to its C-terminus red. Crystal structures of proteins which are irregular and hundreds of times larger than cholesterol began to be solved in the late s, beginning with the structure of sperm whale

myoglobin by Sir John Cowdery Kendrew , [87] for which he shared the Nobel Prize in Chemistry with Max Perutz in X-ray crystallography is now used routinely by scientists to determine how a pharmaceutical drug interacts with its protein target and what changes might improve it. Such membrane proteins are a large component of the genome, and include many proteins of great physiological importance, such as ion channels and receptors. The structure assigned in to the antibiotic isolated from a marine organism, diazonamide A C40H34Cl2N6O6, molar mass Relationship to other scattering techniques[edit] Further information: X-ray scattering techniques Elastic vs. By contrast, inelastic scattering occurs when energy is transferred from the incoming X-ray to the crystal, e. Such inelastic scattering reduces the energy or increases the wavelength of the outgoing beam. Inelastic scattering is useful for probing such excitations of matter, but not in determining the distribution of scatterers within the matter, which is the goal of X-ray crystallography. X-rays range in wavelength from 10 to 0. Longer-wavelength photons such as ultraviolet radiation would not have sufficient resolution to determine the atomic positions. At the other extreme, shorter-wavelength photons such as gamma rays are difficult to produce in large numbers, difficult to focus, and interact too strongly with matter, producing particle-antiparticle pairs. Therefore, X-rays are the "sweetspot" for wavelength when determining atomic-resolution structures from the scattering of electromagnetic radiation. In general, single-crystal X-ray diffraction offers more structural information than these other techniques; however, it requires a sufficiently large and regular crystal, which is not always available. These scattering methods generally use monochromatic X-rays, which are restricted to a single wavelength with minor deviations. A broad spectrum of X-rays that is, a blend of X-rays with different wavelengths can also be used to carry out X-ray diffraction, a technique known as the Laue method. This is the method used in the original discovery of X-ray diffraction. Laue scattering provides much structural information with only a short exposure to the X-ray beam, and is therefore used in structural studies of very rapid events Time resolved crystallography. However, it is not as well-suited as monochromatic scattering for determining the full atomic structure of a crystal and therefore works better with crystals with relatively simple atomic arrangements. The Laue back reflection mode records X-rays scattered backwards from a broad spectrum source. This is useful if the sample is too thick for X-rays to transmit through it. The diffracting planes in the crystal are determined by knowing that the normal to the diffracting plane bisects the angle between the incident beam and the diffracted beam. A Greninger chart can be used [97] to interpret the back reflection Laue photograph. Electron and neutron diffraction[edit] Other particles, such as electrons and neutrons , may be used to produce a diffraction pattern. Although electron, neutron, and X-ray scattering are based on different physical processes, the resulting diffraction patterns are analyzed using the same coherent diffraction imaging techniques. As derived below, the electron density within the crystal and the diffraction patterns are related by a simple mathematical method, the Fourier transform , which allows the density to be calculated relatively easily from the patterns. However, this works only if the scattering is weak, i. Weakly scattered beams pass through the remainder of the crystal without undergoing a second scattering event. Such re-scattered waves are called "secondary scattering" and hinder the analysis. Any sufficiently thick crystal will produce secondary scattering, but since X-rays interact relatively weakly with the electrons, this is generally not a significant concern. Since this thickness corresponds to the diameter of many viruses , a promising direction is the electron diffraction of isolated macromolecular assemblies , such as viral capsids and molecular machines, which may be carried out with a cryo- electron microscope. Moreover, the strong interaction of electrons with matter about times stronger than for X-rays allows determination of the atomic structure of extremely small volumes. The field of applications for electron crystallography ranges from bio molecules like membrane proteins over organic thin films to the complex structures of nanocrystalline intermetallic compounds and zeolites. Neutron diffraction is an excellent method for structure determination, although it has been difficult to obtain intense, monochromatic beams of neutrons in sufficient quantities. Traditionally, nuclear reactors have been used, although sources producing neutrons by spallation are becoming increasingly available. Being uncharged, neutrons scatter much more readily from the atomic nuclei rather than from the electrons. Therefore, neutron scattering is very useful for observing the positions of light atoms with few electrons, especially hydrogen , which is essentially invisible in the X-ray diffraction. Neutron scattering also has the remarkable property that the solvent can be made invisible by

adjusting the ratio of normal water , H2O, and heavy water , D2O. Overview of single-crystal X-ray diffraction[edit] Workflow for solving the structure of a molecule by X-ray crystallography. The oldest and most precise method of X-ray crystallography is single-crystal X-ray diffraction, in which a beam of X-rays strikes a single crystal, producing scattered beams. When they land on a piece of film or other detector, these beams make a diffraction pattern of spots; the strengths and angles of these beams are recorded as the crystal is gradually rotated. For single crystals of sufficient purity and regularity, X-ray diffraction data can determine the mean chemical bond lengths and angles to within a few thousandths of an angstrom and to within a few tenths of a degree , respectively. The atoms in a crystal are not static, but oscillate about their mean positions, usually by less than a few tenths of an angstrom. X-ray crystallography allows measuring the size of these oscillations. Procedure[edit] The technique of single-crystal X-ray crystallography has three basic steps. The firstâ€”and often most difficultâ€”step is to obtain an adequate crystal of the material under study. The crystal should be sufficiently large typically larger than 0. In the second step, the crystal is placed in an intense beam of X-rays, usually of a single wavelength monochromatic X-rays , producing the regular pattern of reflections. The angles and intensities of diffracted X-rays are measured, with each compound having a unique diffraction pattern. Multiple data sets may have to be collected, with each set covering slightly more than half a full rotation of the crystal and typically containing tens of thousands of reflections. In the third step, these data are combined computationally with complementary chemical information to produce and refine a model of the arrangement of atoms within the crystal. The final, refined model of the atomic arrangementâ€”now called a crystal structure â€”is usually stored in a public database. Two limiting cases of X-ray crystallographyâ€”"small-molecule" which includes continuous inorganic solids and "macromolecular" crystallographyâ€”are often discerned. Small-molecule crystallography typically involves crystals with fewer than atoms in their asymmetric unit ; such crystal structures are usually so well resolved that the atoms can be discerned as isolated "blobs" of electron density. By contrast, macromolecular crystallography often involves tens of thousands of atoms in the unit cell. Such crystal structures are generally less well-resolved more "smeared out" ; the atoms and chemical bonds appear as tubes of electron density, rather than as isolated atoms. In general, small molecules are also easier to crystallize than macromolecules; however, X-ray crystallography has proven possible even for viruses and proteins with hundreds of thousands of atoms, through improved crystallographic imaging and technology. Crystals used in X-ray crystallography may be smaller than a millimeter across. Although crystallography can be used to characterize the disorder in an impure or irregular crystal, crystallography generally requires a pure crystal of high regularity to solve the structure of a complicated arrangement of atoms. Pure, regular crystals can sometimes be obtained from natural or synthetic materials, such as samples of metals , minerals or other macroscopic materials. The regularity of such crystals can sometimes be improved with macromolecular crystal annealing [] [] [] and other methods. However, in many cases, obtaining a diffraction-quality crystal is the chief barrier to solving its atomic-resolution structure. Small molecules generally have few degrees of conformational freedom, and may be crystallized by a wide range of methods, such as chemical vapor deposition and recrystallization. By contrast, macromolecules generally have many degrees of freedom and their crystallization must be carried out so as to maintain a stable structure. For example, proteins and larger RNA molecules cannot be crystallized if their tertiary structure has been unfolded ; therefore, the range of crystallization conditions is restricted to solution conditions in which such molecules remain folded. Three methods of preparing crystals, A: Microdialysis Protein crystals are almost always grown in solution. The most common approach is to lower the solubility of its component molecules very gradually; if this is done too quickly, the molecules will precipitate from solution, forming a useless dust or amorphous gel on the bottom of the container. Crystal growth in solution is characterized by two steps:

Chapter 2 : (IUCr) The crystallographic community

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In their introduction to this book, the authors cite the need for a book which describes the instrumentation used for single-crystal diffractometry and the principles underlying the technique. Despite the title of the book, much more space is devoted to the principles than to the instrumentation itself. The authors quote the rapid advances in the field and the number of inexperienced users entering the field as their motivation for writing this book. This is indeed a laudable reason to produce such a volume; however, it implies a completeness that is lacking in this work and which is acknowledged in the introduction: It is clear that the authors have based the book mostly on their own laboratory experiences. This has led to several in depth chapters which will serve as references for advanced users of the technique. There are, however, significant voids, especially in the area of modern instrumentation. The early chapters describe the generation of X-rays and the nature of the diffraction experiment using the Ewald construction. The description of four-circle geometry is very detailed and the discussion of the contributions to reflection broadening is excellent. Except for a brief description of the kappa geometry, no other common geometries are explicitly described. In particular, readers are left to interpret for themselves the implications of using any of the platform instruments commonly used with area detectors. The description of intensity data collection with a point detector is very complete including profile fitting ; however, the only discussion with respect to area detectors is in a short treatment of the Laue technique. Indeed, the treatment of the basic principles of detectors in general is rather sketchy. In the chapter on data reduction, the treatment of absorption corrections using empirical or integration methods with point detector data is suitably detailed. Only a discussion of the multipole methods currently employed for both point and area detectors is lacking. The treatments of primary beam inhomogeneity, the Renninger effect and thermal diffuse scattering are presented in great detail but, surprisingly, there is no mention of decay corrections. The chapter devoted to defects is really a discussion of the extinction problem. However, it does provide a strong argument against the grinding of crystals into spheres, contrary to popular dogma. The final chapter on hardware accessories provides a nice history of nitrogen cooling systems and a more sketchy description of helium cooling devices. The examples for high-pressure cells are well chosen but some of the details of the need for corrections for absorption or diamond extinctions would have been welcome. Although several chapters of this book will be valuable to experienced crystallographers, there are a number of gaps that will leave the experienced unsatisfied. On the other hand, the introductory chapters will be helpful to the proposed audience of inexperienced users. However, many of the chapters are written at a level that the inexperienced will find daunting.

Chapter 3 : X-ray crystallography - Wikipedia

In their introduction to this book, the authors cite the need for a book which describes the instrumentation used for single-crystal diffractometry and the principles underlying the technique.

Chapter 4 : Crystallographic Instrumentation : L. A Aslanov :

XÂ-Ray Crystallography: Procedure and Instrumentation A Look into the methodology of Xâ€•Ray Diffraction What is XÂ-Ray.

Chapter 5 : AsCA â€“ Asian Crystallographic Association Conference

The X-Ray Crystallographic Laboratory (XCL), C Kolthoff Hall, provides single crystal diffraction of small molecule

materials to the University's community of researchers, including chemists, medicinal chemists, pharmaceutics researchers, chemical engineers, physicists, and geologists.

Chapter 6 : Fetisov, G. V. [WorldCat Identities]

The Crystallography Facility offers a wide variety of instrumentation including facilities for x-ray data collection, light scattering, crystallization and data manipulation.

Chapter 7 : Crystallography Basics | Chemical Instrumentation Facility

The program will showcase outstanding science from Asia, Australia and New Zealand, and from around the world, and will be presented in three streams covering diverse topics in structural biology, chemical crystallography, crystal engineering, materials science, physics and fundamental science, and methods including instrumentation, techniques.

Chapter 8 : (IUCr) Information about Journal of Applied Crystallography

The crystallographic community. Crystallographers have always felt a strong sense of community. The creation of the International Union of Crystallography in was inspired by the desire to restore the bonds of scientific friendship amongst the nations of a world recovering from a major war.

Chapter 9 : X-Ray Crystallography - INSTRUMENTATION

The IUCr-UNESCO OpenLab is a network of operational crystallographic laboratories based in different countries worldwide, many in less endowed regions of Africa, South and Central America and South Asia.