Physical Chemistry

Brief Overview of X-ray Diffraction Experiment

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Requirements for Single-Crystal X-ray diffraction experiment

- Single crystal
- X rays from X-ray generator and X-ray tube, or synchrotron radiation
- Detector (X-ray film or electronic detector)
- Goniometer to move crystal and detector into position to satisfy Bragg's law
- Computer and software
- Crystallographer!

Information we measure from the diffraction pattern

Geometry of the diffraction pattern gives size and shape of unit cell

- measuring the spacing of the spots gives *a, b, c*
- measuring the angles between axes gives α, β, γ

Density of crystal + unit cell information gives the number of molecules in unit cell

By assigning *hkl* indices to each spot, we notice there are systematic absences of diffraction spots—these absences gives the symmetry of lattice

Intensity of each diffraction spot + phase of diffracted beam + many calculations gives the types of atoms and their *xyz* coordinates in the unit cell. This last step is the difficult one!

Structural information we get from X-ray diffraction experiment

The type and position of atoms in the unit cell = the *xyz* coordinates of atoms

From the atomic coordinates we can calculate:

Bond lengths and bond angles
Intermolecular contact distances give indication of hydrogen bonds, ionic interactions, van der Waals forces, ring stacking
Torsion angles
Overall conformation of molecule

From the thermal motion of the atoms we deduce:

Possible reactivity
Possible alternate conformations

Molecular Graphics and Molecular Modeling

The *xyz* coordinates of all the atoms in the structure are processed and displayed by computer programs such as Mercury, Spartan, Gaussian, and Chimera. Some of these programs have general information about typical bond distances, amino acid, and nucleic acid structures. Bonds are drawn between appropriate atoms in the unit cell. Individual atoms and residues can be selected, labeled, colored, etc.

Display, analysis and comparison of results

- Color and three-dimensional stereo viewing highlight important aspects of the structure of the molecule.
- Computer calculation of interatomic distances, partial charges, van der Waals surfaces disclose likely intramolecular and intermolecular interactions.
- From this structural information we can draw conclusions about reactivity.