ANALYTICAL CHEMISTRY

X-ray Crystallography

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X-ray Crystallography

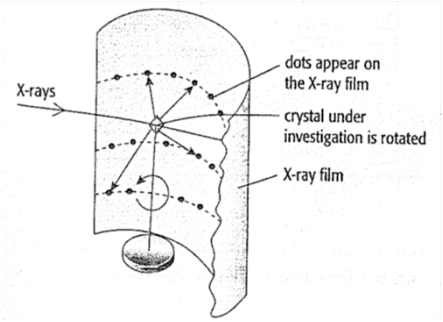
- X-rays are very short (about 0.1 nm) wavelength electromagnetic rays.
- An X-ray spectrometer consists of three main parts:
  - an X-ray source
  - a stable support on which the crystal is mounted, and which can be rotated by a measured angle around all three axes
  - a system for detecting the reflected X-rays. This can be either a photographic film or a scintillation counter.

X-ray Crystallography

- Principle : X-rays are diffracted by the electron clouds in the atoms of the crystal.
- All atoms except hydrogen contain enough electrons to diffract the X-rays.
- The heavier the atom, has more electron → the more intense is the spot produced in its diffraction pattern.
- Diffraction of each atom is small but effect is magnified by regular repeating patterns of atoms and ions in crystal.

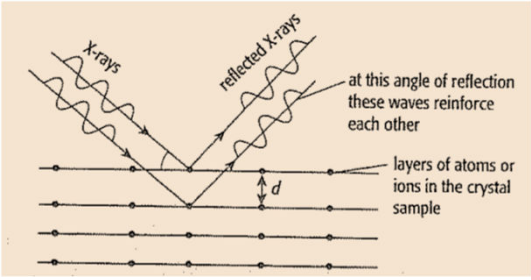
X-ray Crystallography

Process : crystal irradiated → image of diffraction pattern recorded → crystal rotated slightly (1°) and image recorded again. Process repeated until every angle of crystal is irradiated.



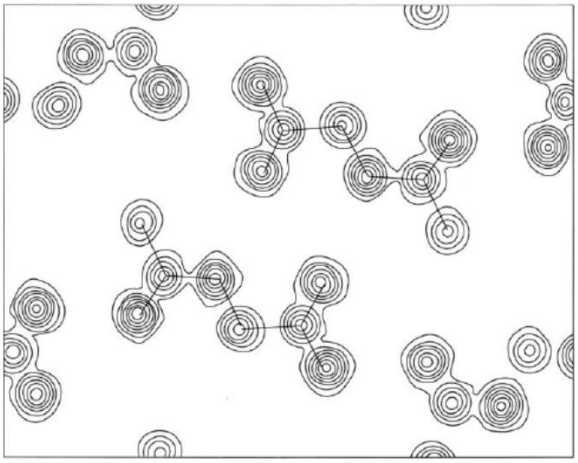
- By measuring the angles between the incident beam and the diffracted beams, and the relative intensities of those beams, we can piece together a picture of the electron density at all points in the unit cell.

- Electron density map constructed by computer.



- ethanoylaminoethanoic acid

The result is shown in Figure 2.19



Information obtained

- Shows the **position** of most of the atoms in the structure.
- Allows measurement of **bond length**.
- **Bond angle**.
- **Shape** of molecule.

- Application : Analysis of large biological molecules (e.g DNA).

- **Disadvantage** : a very pure crystal of sample needed.

X-ray Crystallography

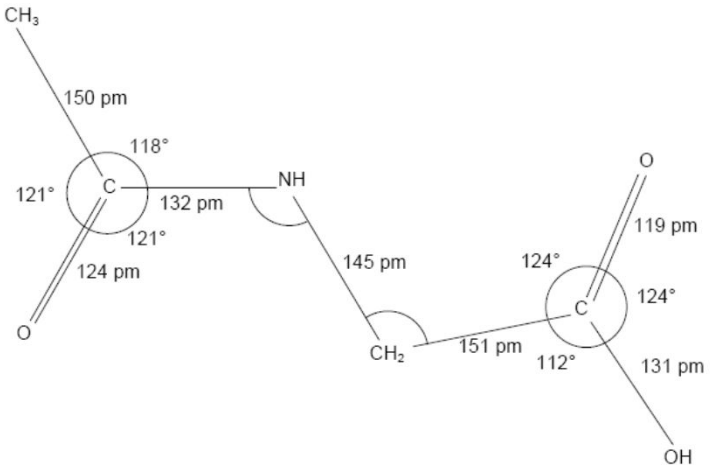


Figure 2.19 – electron density map of N-ethanoylaminoethanoic acid

X-ray Crystallography

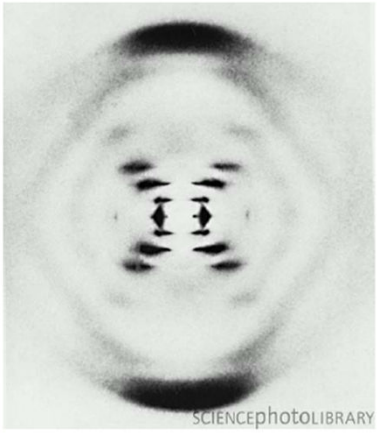


Figure 2.20 – X-ray diffraction photograph of a hydrated DNA fibre