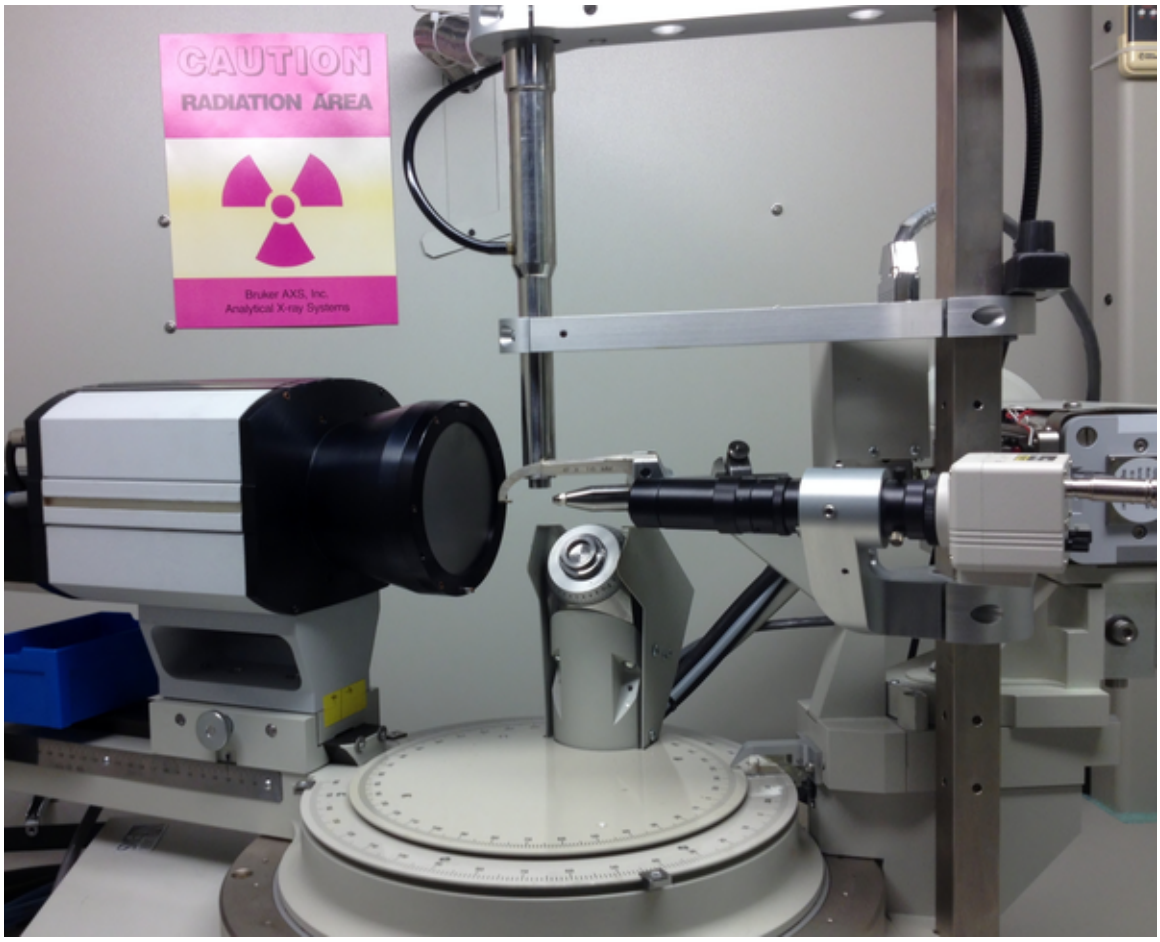
Introduction

History

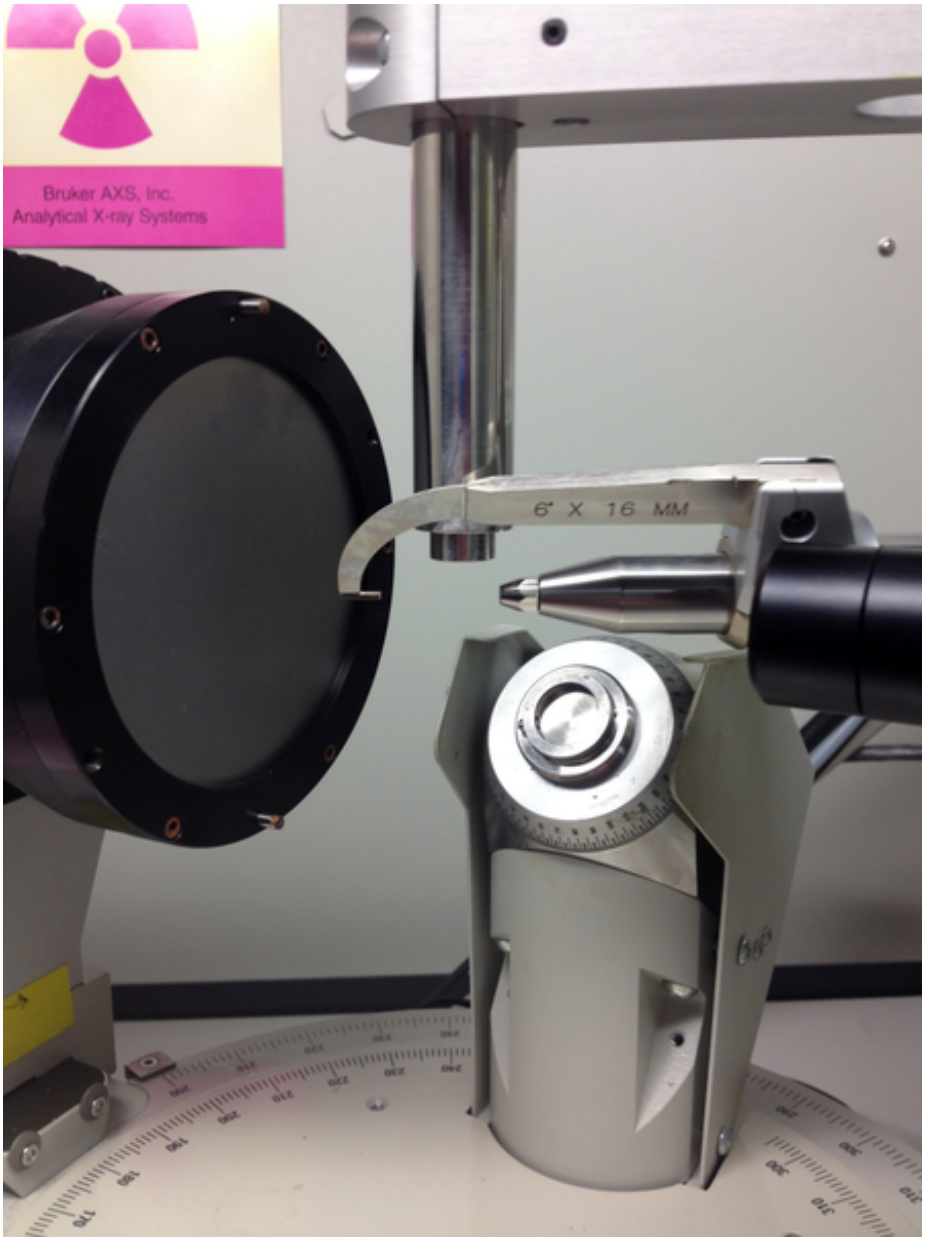
Very brief history of technique

Discovery; when it came into use

Use today



Modern single-crystal x-ray diffraction machine; the x-ray source can be seen at the right edge as the gray box that extends into the background. Note that the goniometer that holds the crystal in place is not shown.

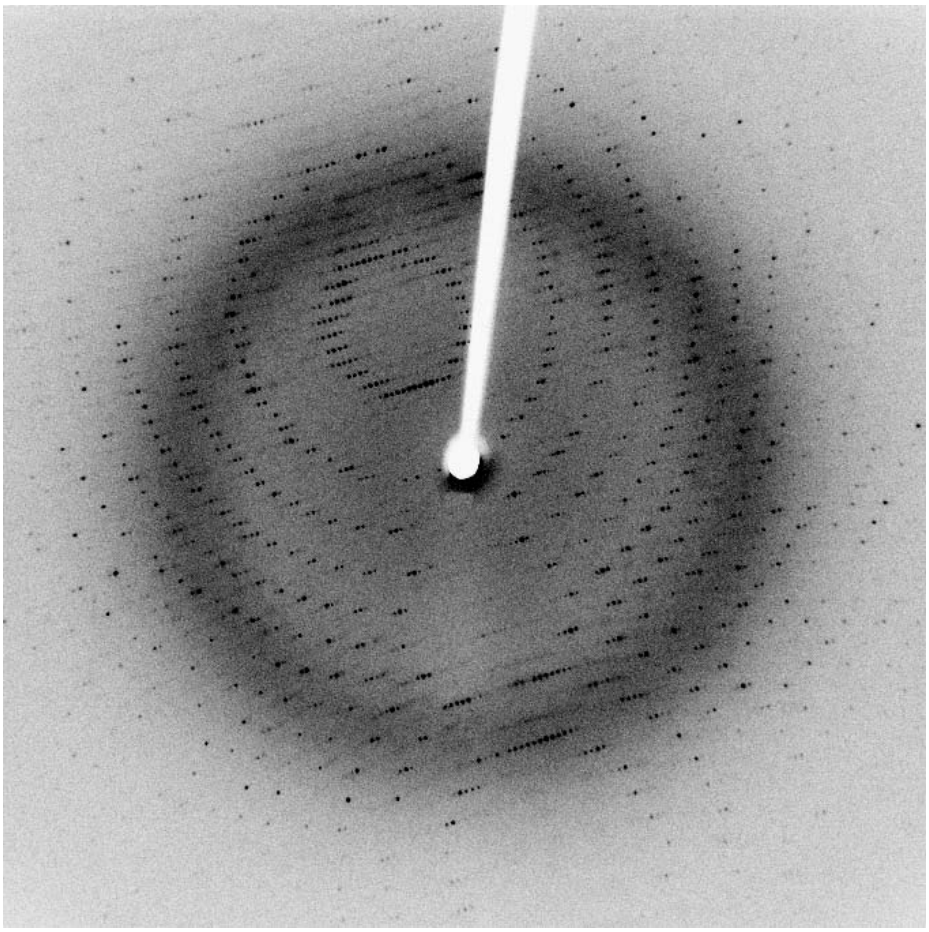


Close-up view of a single-crystal x-ray diffraction instrument. The large black circle at the left is the detector, and the x-ray beam comes out of the pointed horizontal nozzle. The beam stop can be seen across from this nozzle, as well as the gas cooling tube hanging vertically. The mounted crystal rests below the cooling gas supply, directly in the path of the beam. It extends from a glass fiber on a base (not shown) that attaches to the third, smaller goniometer.

Additional features (multiple radiation sources- Cu/Mb dual source, gas-cooling devices,

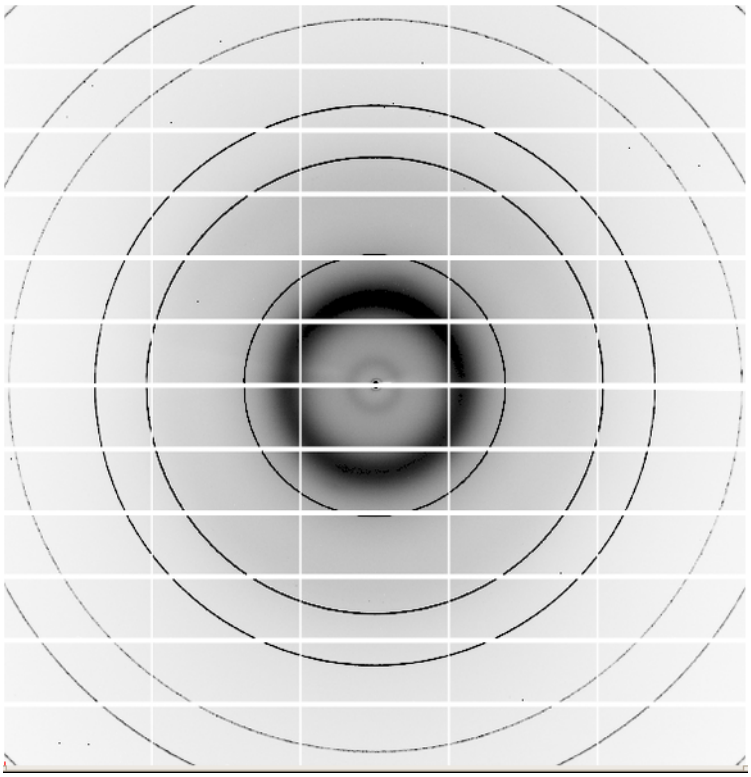
Single-Crystal vs. Powder

Image of single-crystal and powder XRD spectra side-by-side:



Single-crystal diffraction pattern of an enzyme.

http://en.wikipedia.org/wiki/File:X-ray_diffraction_pattern_3clpro.jpg



Powder X-ray diffraction spectrum of silicon. Taken by XanaG; used under PD license.

http://commons.wikimedia.org/wiki/File:Si_powder_diffraction_pattern.png

Brief comparison between single-crystal and powder XRD

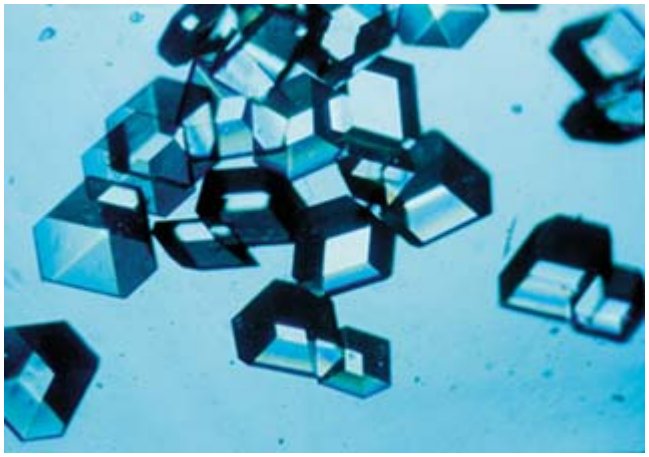
Advantages/disadvantages of single-crystal XRD over powder XRD

Technique

Summary of device and technique [Will link to module 38289 (Intro to XRD)]

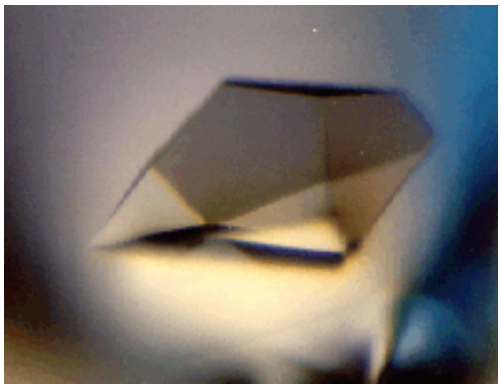
What makes a good crystal; how to obtain them

Images of ideal crystals:



http://en.wikipedia.org/wiki/File:Insulincrystals.jpg

Single crystals of insulin, grown in space; taken by NASA. Released under PD license.



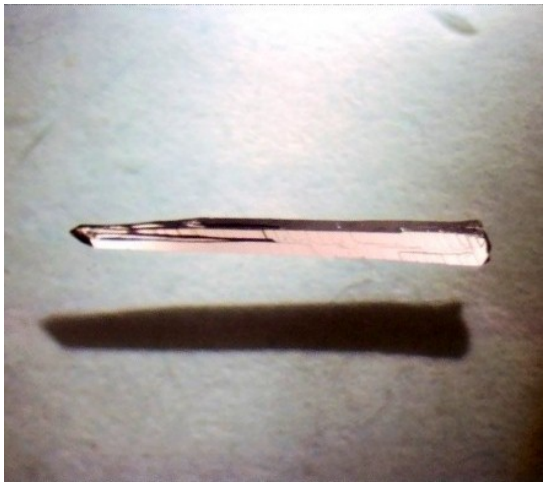
http://en.wikipedia.org/wiki/File:Protein_crystal.jpg

Single crystal of a protein; taken by NASA. Released under PD license.

An octahedral-shaped single crystal of synthetic chrome alum.

"Chromium Alum - top view.jpg" © 2008 Ra'ike, used under a Creative Commons Attribution-Share Alike 3.0 Unported license.

http://en.wikipedia.org/wiki/File:Chromium_Alum_-_top_view.jpg



Single FeSi crystal showing long bar morphology. Cropped from image by Paul Canfield, released under PD license.

http://core.materials.ac.uk/search/detail.php?id=3740

Explanation

Explanation of data and parameters for (single-crystal) XRD

Figure of a typical report of XRD data

~~[Imitate a publication to generate a mock table for NaCl?]~~

Line-by-line discussion of each element of the reported data:

General information about the crystal analyzed

how it was obtained

size

chemical formula and weight

crystal system

[link to module 16927 (Crystal Structure)]

space group

unit-cell parameters (dimensions, angles, volume)

Image for unit cell parameters:

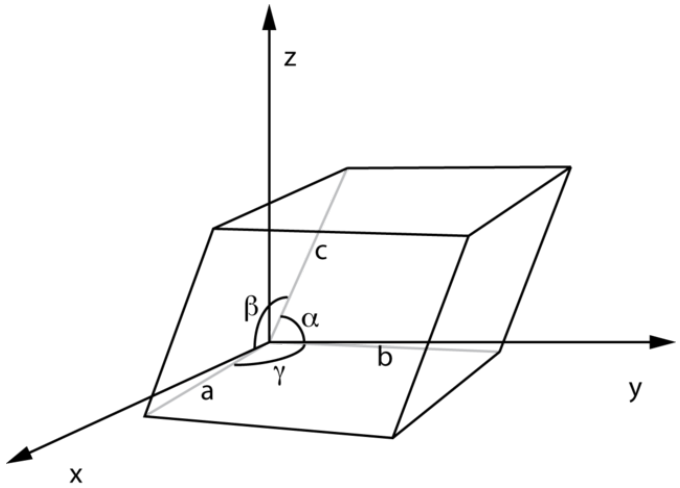


Diagram of a unit cell with side lengths a, b, and c and angles between those sides indicated by alpha, beta, and gamma. Image created by Mcpazzo; released under PD license. http://en.wikipedia.org/wiki/File:UnitCell.png

number of assymetric units

calculated and measured density

linear absorption coefficient

Information on how the data was collected

instrument used

temperature

radiation source

monochromator

number and 2(theta) ranges of reflections used to determine unit-cell

range of h, k, l studied

range of theta

number of independent data points (discussion of redundancy)

number of reflections observed, criteria for being "observed"

internal agreement between measurements of equivalent reflections

checks for intensity decay

absorption correction (why or why not applied, how determined)

Lorentz and polarization factors

How the information was processed

method

program/software

Conclusion

Summary of what has been learned

Bibliography