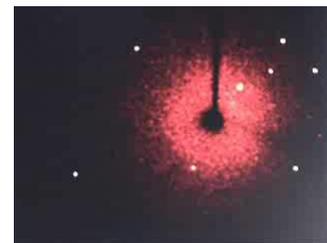


George L. Clark
X-ray
Laboratory

3M Materials
Chemistry
Facility



Danielle L. Gray
dgray@illinois.edu
244-1708



Toby Woods
tobyw@illinois.edu
300-1081



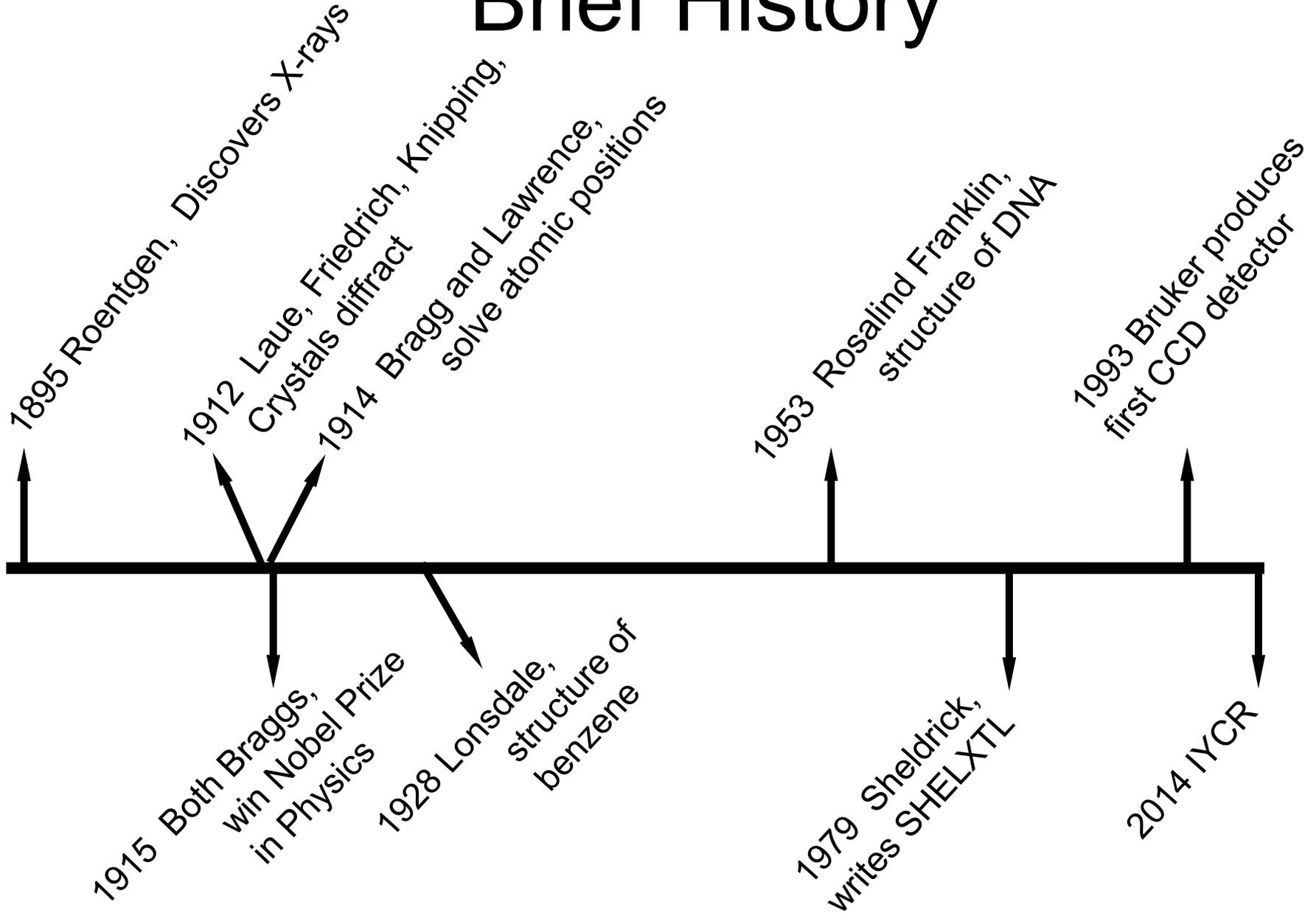
Biocrystallization
Yi Gui Gao
y-gao@illinois.edu
244-1131

Business Hours 8:30a - 12:00p and 1:00p - 4:30p Monday - Friday

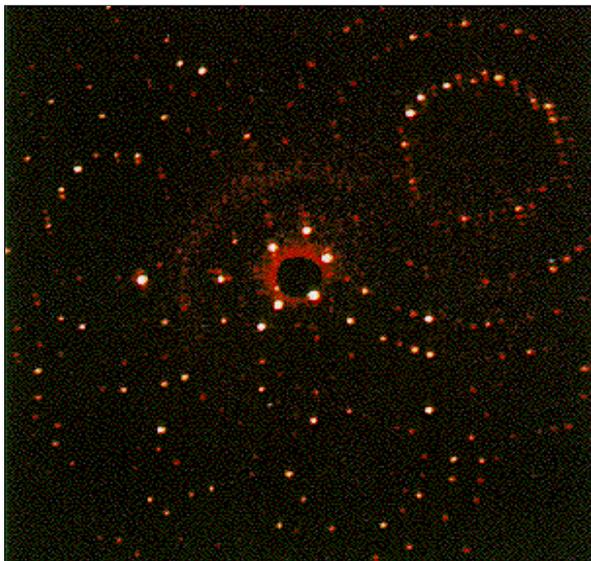
Consulting Hours 10:00a - 11:00a and 2:00p - 3:00p Monday - Friday

Laboratory Entry Northeast Ground Floor Noyes, Room 60

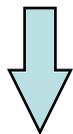
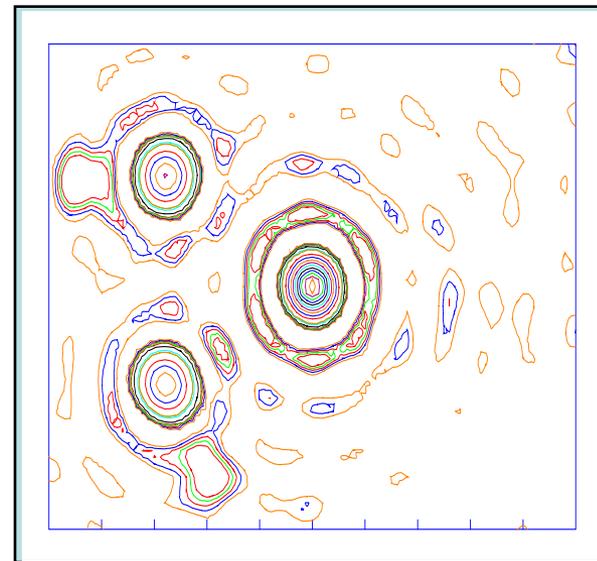
Brief History



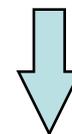
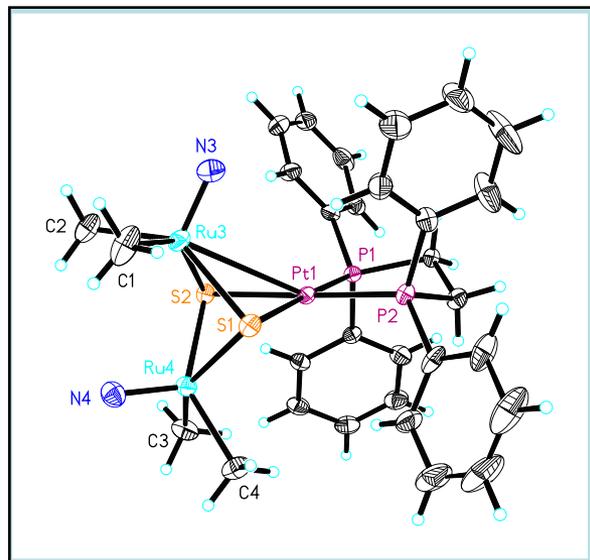
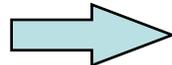
Overview



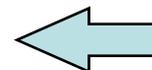
SHELXTL
XPREP
XS
XP



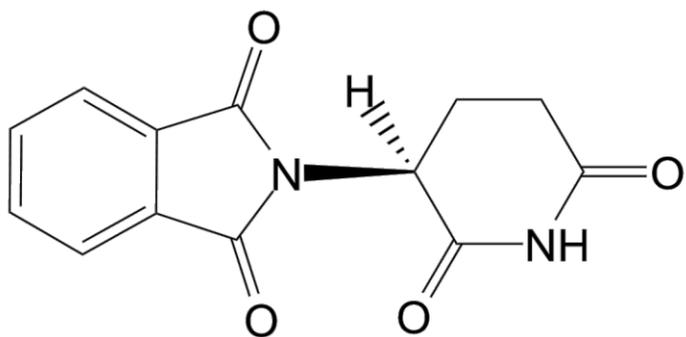
P 21/n
a = 23.3481(4)
b = 12.3868(2)
c = 23.8191(1)
beta = 92.756(1)



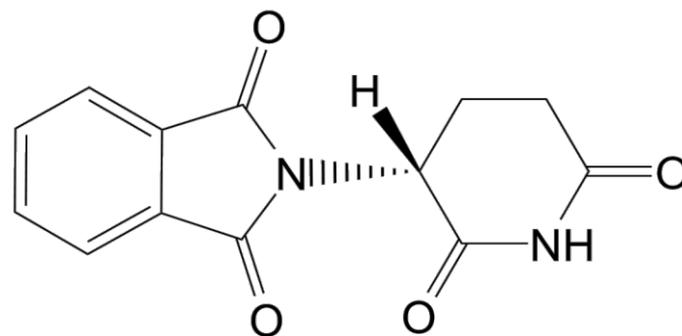
**Chemistry
XL**



Organic Chemistry and Crystallography it matters



(S)-thalidomide, identified
as the teratogenic agent



(R)-thalidomide, the therapeutic agent
for the treatment of morning sickness

Absolute structure without a heavy atom

1. Crystallize the compound with a component of known stereochemistry
2. Bijvoet Pairs ($h\ k\ l$ and $-h\ -k\ -l$) need to be examined for anomalous scattering effects

Centrosymmetric vs. Non-centrosymmetric

$$F_{hkl} = F_{-h-k-l}$$

$$F_{hkl} \neq F_{-h-k-l}$$

Flack and/or Hooft

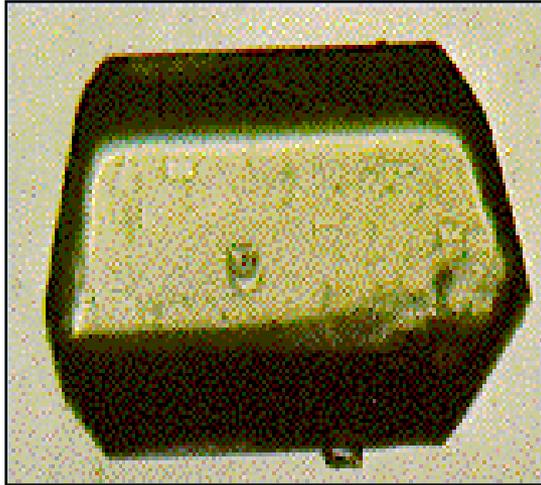
Analysis

In order to measure anomalous scattering effects you must be using a radiation near the absorption edge of the atom types. You **MUST** have Cu radiation for light atom structures!

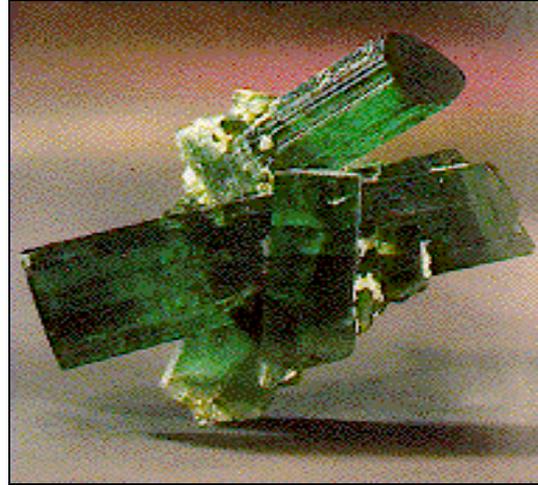
The main pitfall of absolute structures

- Is the crystal examined a representative of the bulk crystals?
 - Racemic mixtures can crystallize
 1. As a centrosymmetric crystal
 2. As a non-centrosymmetric crystal that is twinned
 3. Each individually as non-centrosymmetric crystals
 - Circular dichroism or enantioselective chromatography to test if crystal is the same as bulk

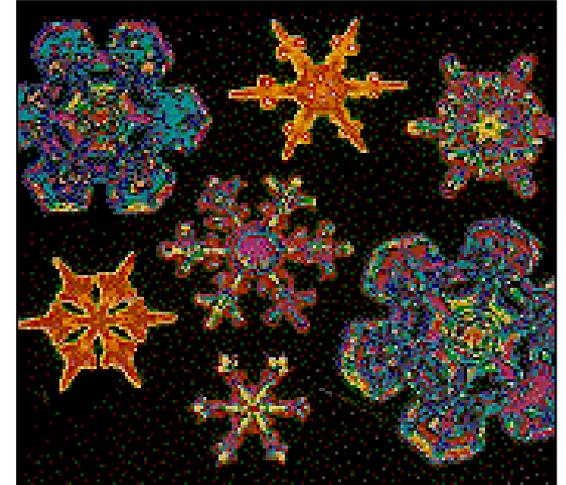
Crystal Quality is Essential



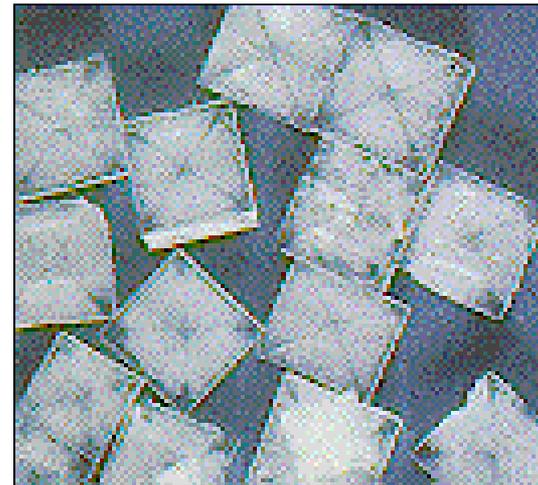
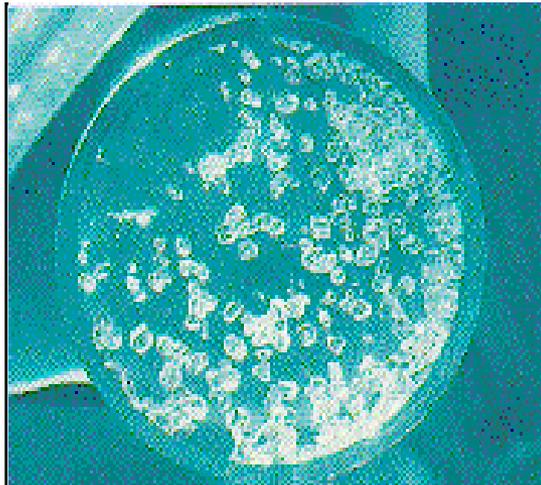
YES



MAYBE

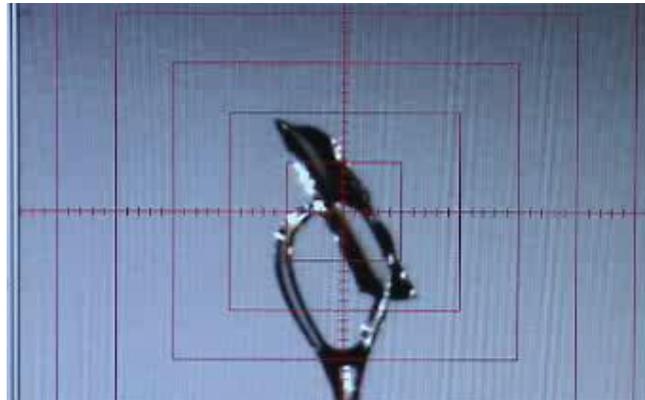


NO



The Ideal Sample

The ideal sample, like this 40 x 70 x 360 μm crystal mounted on a 300 μm cryo-loop, is attached using an inert oil, frozen in place using a cold gas stream, and recovered after the diffraction experiment is finished.



Like many spectroscopic techniques, the overall amplitude of the diffraction pattern is proportional to sample volume. There are no limits on crystal morphology, but the practical range of linear dimensions is 5 to 500 μm . Air, temperature, and light sensitive samples are routinely used.

Growing “good” crystals

- Solvent Cooling
- Solvent Evaporation
- Layering Liquid/Liquid diffusion
- Vapor Diffusion
- Sublimation

**No matter how you grow the crystals,
bring them to the X-ray facility the way
you grew them!**

You too can learn to collect and solve your own structures!



- How to pick a good crystal
- How to collect a data set
- How to integrate a data set
- How to solve a structure using OLEX2/SHELX

	<u>User run</u>	<u>Staff run</u>
Screening the crystals (Unit Cell)	\$33.71	\$57.26
Data Set	\$103.36	\$175.58
Structure Solution	\$212.30	\$360.64