XRAY CRYSTALLOGRAPHY 101: The Who's What's and Why's

So X-ray Crystallography happened to be taught on the lecture right before our midterm week, so it may have not been your main priority at the moment since it was not going to be on the midterm. But yes, it will be on the FINAL. No fear, I have compiled a fun crash course in X-ray Crystallography to teach ya what important and fun ways to memorize things….

I'm a hands-on learner who loves visuals, so I have come up with some little demos and created some visuals to make learning this stuff fun! Enjoy!

So What is X-Ray Crystallography?

-It is a type of analysis in which atomic positions are revealed through the scattering of x-rays that occurs because of electron clouds surrounding the atoms' tendency to diffract x-rays.

Memory Tip!
To remember that 100 is the maximum number of atoms for a molecule to fit under "small" category, just remember that the highest dollar bill amount is 100.

It is useful for small molecule structures (considered 'small' if up to only 100 atoms)

And also especially for structural biology that include: Nucleic acid structure, protein structure, enzyme/drug interactions

The Requirements for X-Ray Crystallography:

The molecule being observed must have a crystalline structure

To be eligible Crystals must:

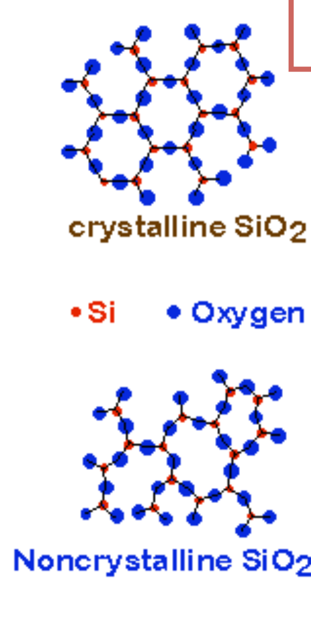
-Not have any cracks or flaws and be small enough to allow for photons from x-rays to interact with atoms (less than 1mm)

-The crystal must be mounted (aka just secured) to prevent vibration of atoms which will skew reflections!

-it's also useful to have a general idea of the structure of the crystal, X-ray crystallography is useful for the details of atom positioning in space

What is considered a 'crystal'?

-A regularly spaced array of atoms



Ex: Crystalline vs. Non-crystalline Structure of same molecule

Things to Memorize: Low Molecular Weight Molecules are easy to crystalize, but high molecular weight ones (Such as Biological molecules) take a lot longer to make.

HOW THE PROCESS WORKS: The basic mechanics

Each dot=reflection

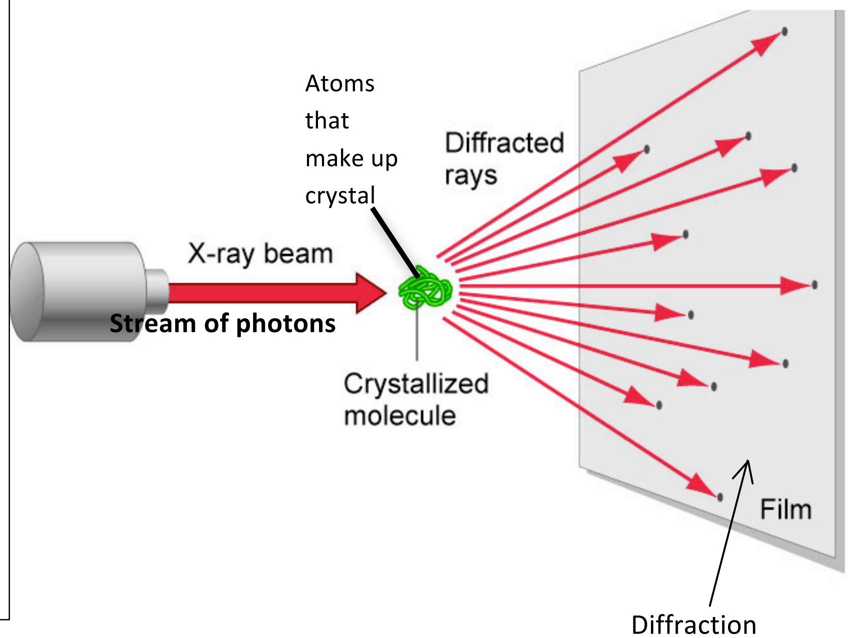
Compilation of Reflections
=Diffraction Pattern

The arrangement of the atoms within the crystal is what determines the specific diffraction pattern…

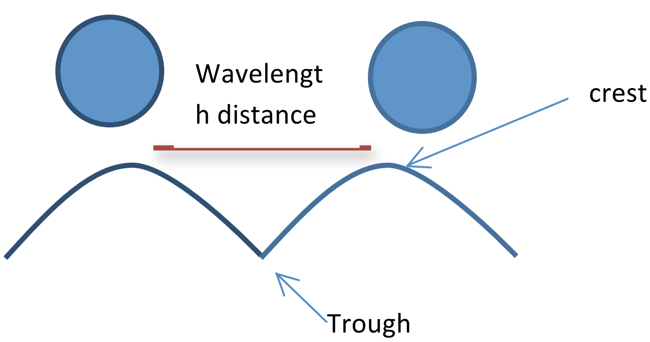
The dots projected can be light or dark depending on the amount of photon intensity (how many photons per cubic area)

Photon Intensity Depends on Wave Interactions (look below)

These varied photon intensities then help to find structure of molecule who's atoms' electron clouds are diffracting the photons



For diffraction to occur it is required that photons being shot out towards crystals have a wavelength that is approximately equal to the distance between atoms in the crystal.



Wave Interactions:

Constructive: Troughs and crests align=Amplitudes of waves add together

Destructive: Troughs and crests are not aligned and their amplitudes cancel each other out

Partial Interference: (most common) only slightly unaligned leads to complex pattern of high and low amplitudes

***Dr. H recommends thinking of the 2 interacting waves as actual ocean waves for easier understanding!*

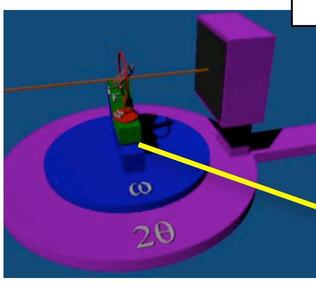
Wave Interactions That Affect Photon Intensity:

So once we have a diffraction pattern, what happens next?

Diffraction pattern→Electron Density Map→Solve Electron Density map→Structure

-Well once we collect one diffraction pattern, we must proceed to alter the angle at which x-rays hit the crystal to get yet another diffraction pattern. You continue to alter the angles until you have a 360 degree "view" of the crystal structure in terms of a ton of separate diffraction patterns.. Putting together all of these diffraction patterns, then allows you to create 3D diffraction pattern (which is translated into an Electron Density Map) to then discover structure.

The rotation of the crystal is achieved by using a tool called a "Goniometer" which can adjust the crystal's x,y,z



Goniometer

Click this link to watch image of rotating crystal!

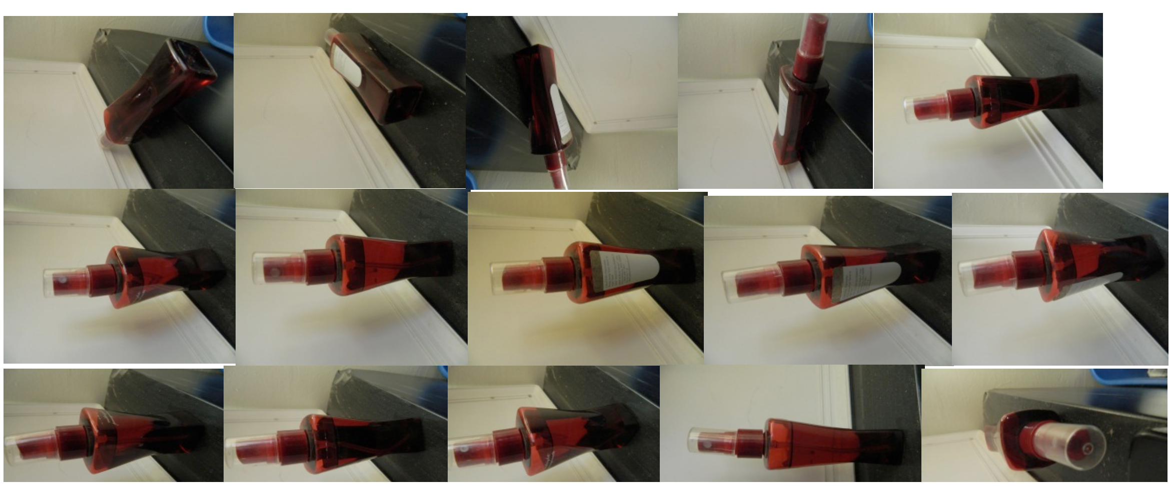
http://www.google.com/imgres?hl=en&sa=X&biw=1366&bih=643&tbm=isch&prmd=imvns&tbnid=24nMD6tYJ

If you find this method hard to visualize, just image this process…

Imagine you are given the task of figuring out the overall 3D shape of a random object. You are given one picture of just one angle of the object that is a pretty flat 2D image… This is hard to do right?

Well what if they then allowed you to look at pictures of the object in the same location just in all sorts of different angles? This would be a lot easier to get an idea of how this object looked right? It's the same idea behind X-ray Crystallography!

Demo: All of these images together from all sorts of angles are preferable to just one image, to figure out entire 3D structure.



Electron Density Map:

Still confused on how to visualize the map in 3-D terms?

-Similar to how a topographical map works, the Electron Density Map represents the electron cloud size through circular/elliptical shapes that are "layered" to represent how much electron density there is.

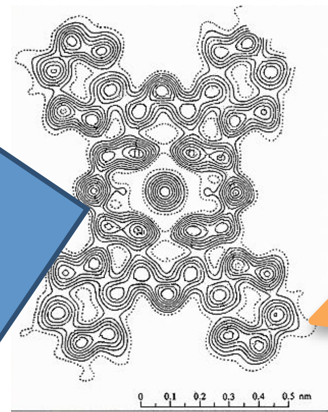
The more rings=Larger Electron Cloud--> More Electron Density → More diffraction (darker dots)

Real Life Explanation: To get a better idea of how the concentrical rings represent a 3-D shape, try doing the little demo below.

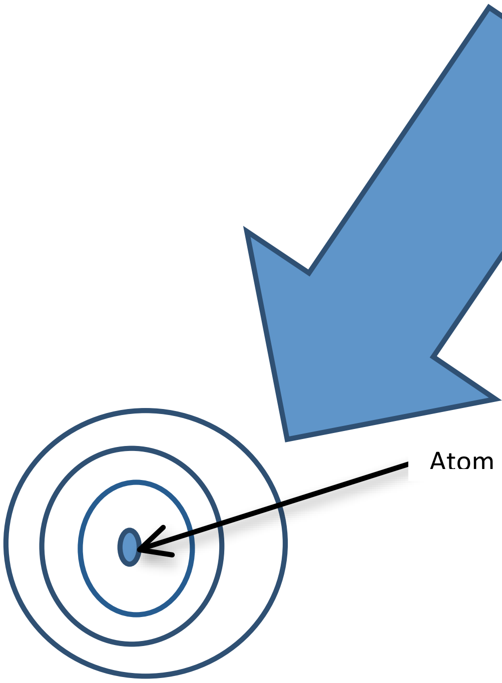
1) You just need one of each coin. Each coin represents a ring on the map.
2) Just stack each coin from largest to smallest as shown below.
3) Once stacked, you can now visualize how each ring represents a "slice" of a whole 3D structure.

This is what you get once you stack each coin in a concentric pattern. The side view shows you how it is a 3D shape… The taller the pile, the larger "the larger the electron cloud"

Here you can see how each individual coin representing a ring is depicted from looking from above, and almost in 2D like in the Electron Density Map. ... It looks flat, but really it represents a 3D shape (just like the electron densities are not 2D, but 3D surrounding around atoms)



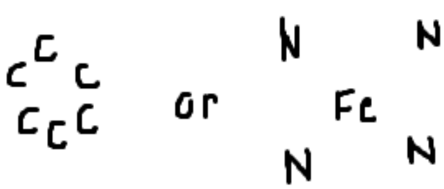
You can now look at an Electron Density Map like this one and just picture it in terms of coins! So simple.



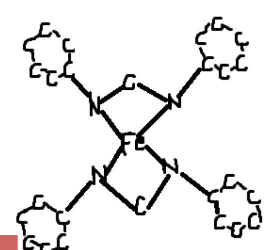
Remember that: The higher the atomic number the larger the electron cloud!

**This is why Hydrogen's (Very small atomic number and electron cloud) are hard to find in maps.

From this point you just look at the Electron Density Map and knowing a general idea of what general structure of the crystal, you can assign each atom a specific element name according to amount of electron density. .. You will get something like this for each cluster of ring groups….



Next, just play "connect the dots" in the sense that no bond rules are broken. You will end up with a crystal structure once you put everything together



STRUCTURE!

<u>Applications of XRay Crystallography Knowledge:</u>

-It will allow you to distinguish which is a proper reaction product structure (cis or trans)since crystals can have same molecular formula, functional groups, and splitting patterns making NMR, IR And MS useless

-For Biological molecules it allows for the creation of drugs that utilize enzyme/substrate complex functioning to alter behaviors and biological

CONGRATS YOU ARE NOW CULTURED IN THE ART OF X-ray Crystallography! **Good luck with studying !**

Sources:

-Xray Crystollography Process Image:

biochem.arizona.edu

-Examples of Crystals Images

http://www.quora.com/Nature/Is-any-surface-perfectly-flat-in-nature

-Rotation of Crystal on Goniometer Gif:

Wikipedia, http://en.wikipedia.org/wiki/File:Kappa_goniometer_faster_smaller.gif

-Coin Images : https://www.google.com/search?q=x+ray+crystallography+tutorial&hl=en&tbm=isch&prmd=imvns&source=lnms&ei=DmvBT6a1LIWQiQLe-LziBw&sa=X&oi=mode_link&ct=mode&cd=2&ved=0CDMQ_AUoAQ&biw=1366&bih=643#hl=en&tbm=isch&sa=1&q=quarter&oq=quarter&aq=f&aqi=g10&aql=&gs_l=img.3..0l10.55452.58726.0.58810.15.9.5.1.1.0.90.610.9.9.0...0.0.bfWu2mD89vA&pbx=1&bav=on.2,or.r_gc.r_pw.r_cp.r_qf.,cf.osb&fp=7c3ffc9b147c8b7f&biw=1366&bih=643

-***Stacked Coin Images***:

Original Photograph

-Sample Electron Density Map Image:

Organic Chemistry Illustrated Glossary, Steve Hardinger , http://www.chem.ucla.edu/harding/

-X-Ray Crystallography General Information:

Chemistry 14C Lecture Supplement, Steve Hardinger (UCLA)