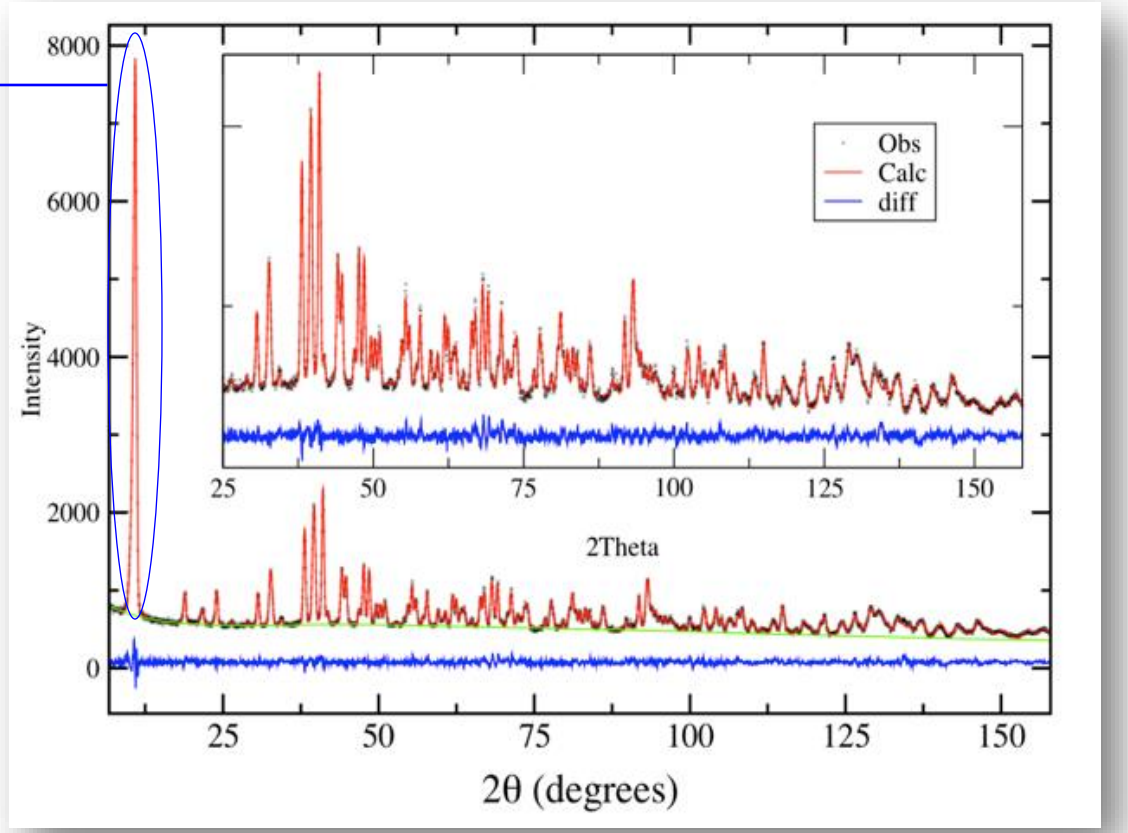
Module 2
Synthesis and Characterization

Solid State Chemistry and Energy Applications

X-ray diffraction

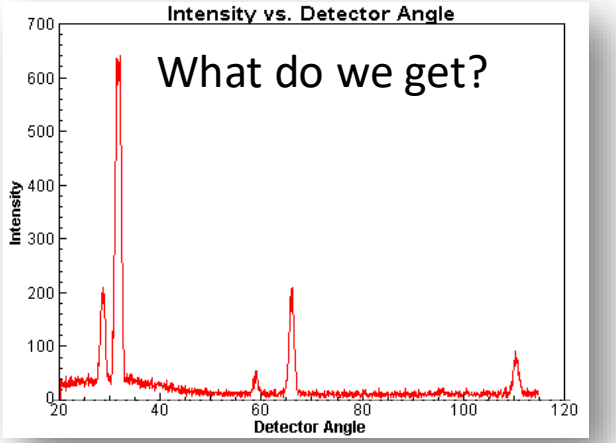
Representing a parallel set of planes

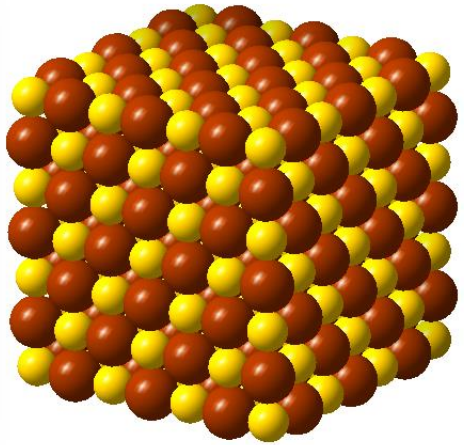
hkl



Things that effect Intensities and Peak positions

- Crystal System (triclinic, monoclinic, etc.…)
- Lattice Type (P, I, F, or C)
- Symmetry
- Unit Cell Parameters
- Atom distributions and types
- Preferred orientation



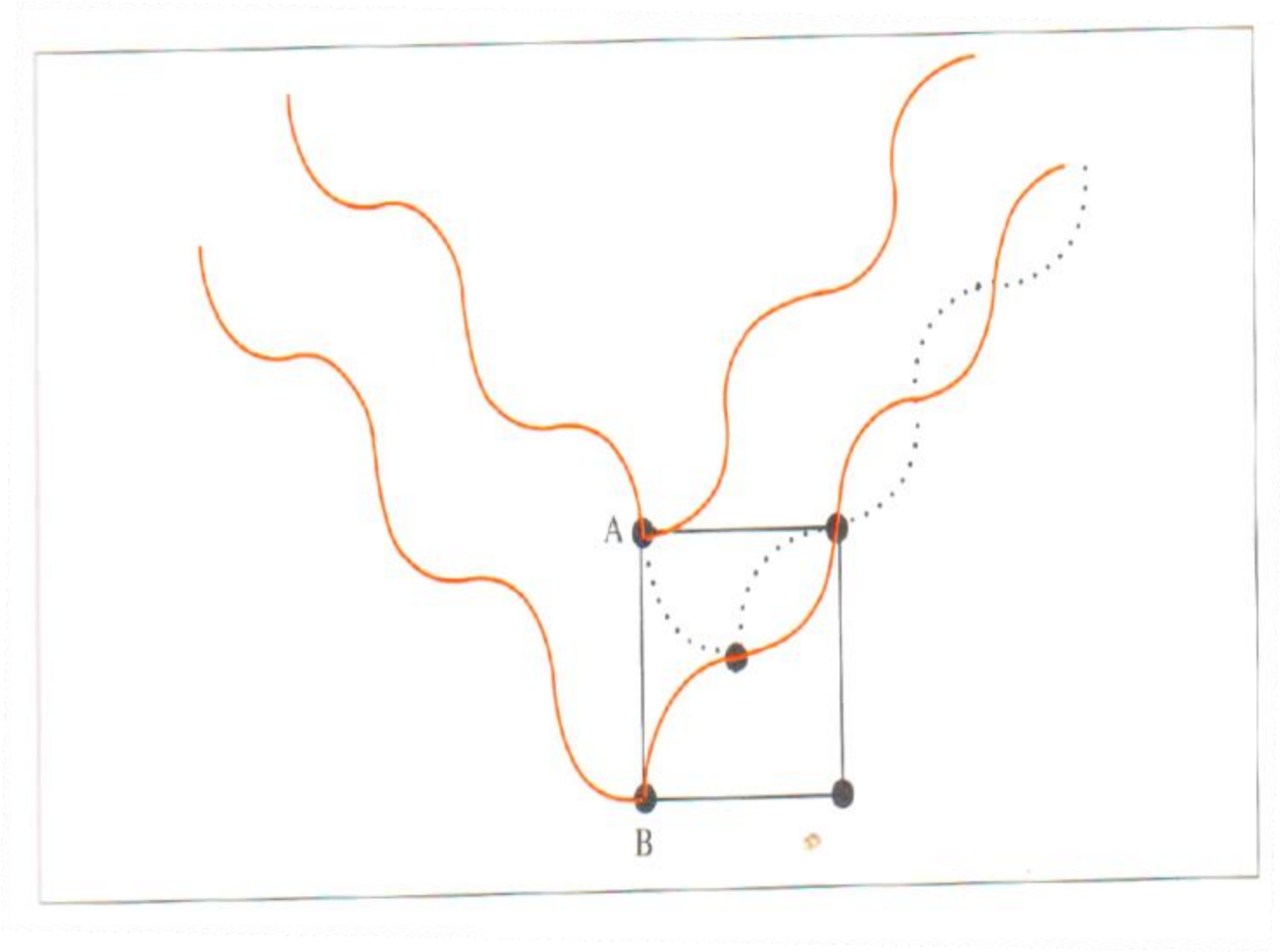


Bragg's Law

$n\lambda = 2d\sin\theta$

Exercise: If a crystal has large d-spacing, what do you expect to see? What types of materials might you see large d-spacing?

Indexing for a body centered cell



Exercise: For the following primitive lattice, determine the h, k, and l values for the 48.266 reflection.

Utilize the following equation.

$$\sin^2\theta/\sin^2\phi = (h_1^2 + k_1^2 + l_1^2)/ 1^2$$

2θ	$\sin^2\theta$	Ratio	Miller indices
19.213	0.0279	1	100
27.302	0.0557	2	110
33.602	0.0836	3	111
38.995	0.1114	4	200
43.830	0.1393	5	210
48.266	0.1671		
56.331	0.2228	8	220
60.093	0.2507	9	300
63.705	0.2785	10	310
67.213	0.3064	11	311
70.634	0.3342	12	222

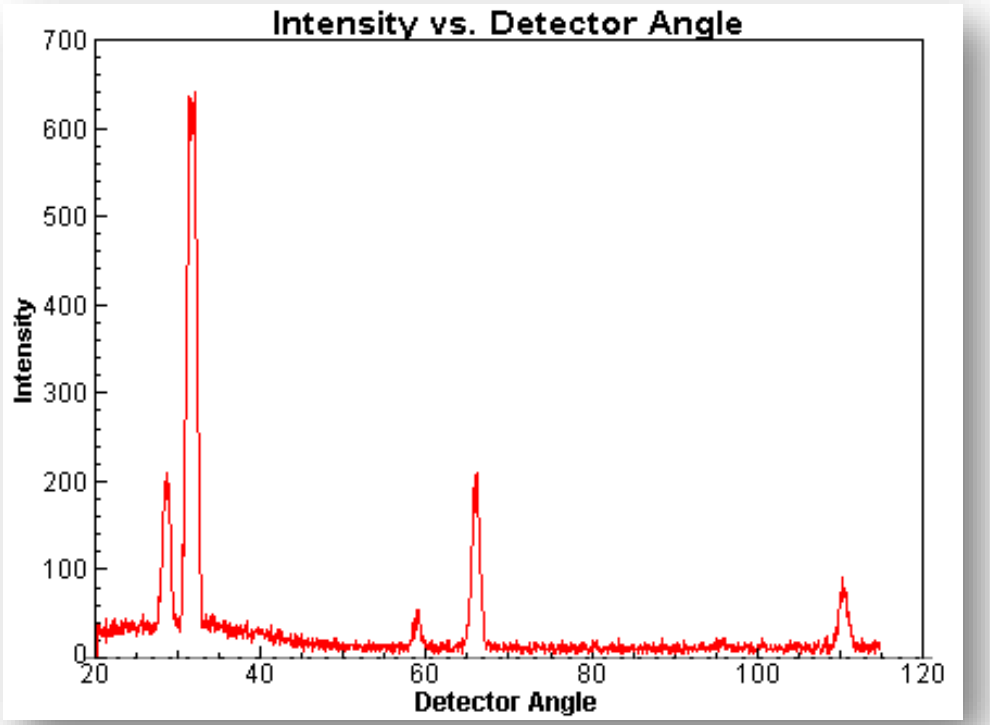
Exercise: given the source is copper, use the 222 reflection to calculate the unit cell dimensions.

$\sin^2\theta = \lambda^2 (h^2 + k^2 + l^2) / 4a^2$

Answer:

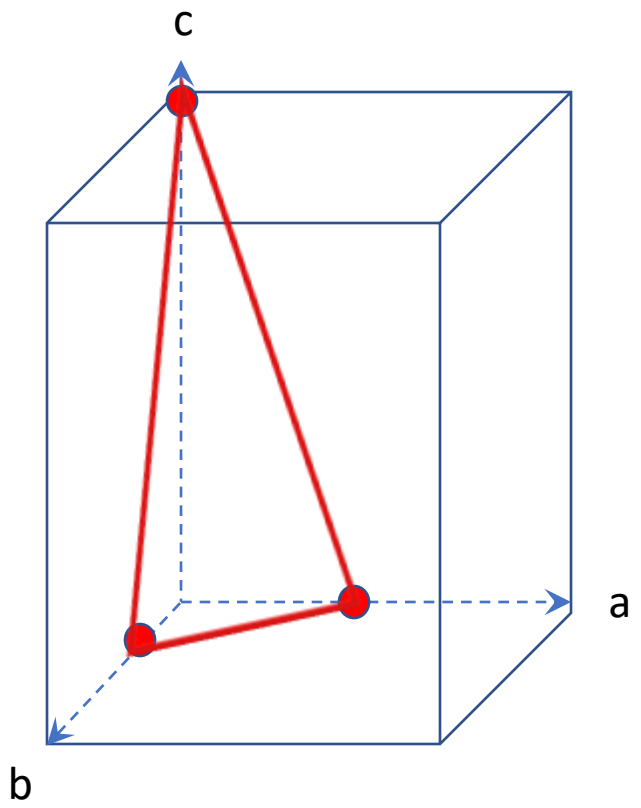
So $a^2 = (1.54)^2(12)/0.3342(4)$

So a = 4.614 Å



Exercise: Draw a tetragonal cell and then draw the the plane with hkl of (231).

If the unit cell dimensions are a, b =5.3 Å and c =7.2 Å, please tell me where the plane intersects the unit cell.



You can calculate the place it intersects the a, b, and c using the following equation

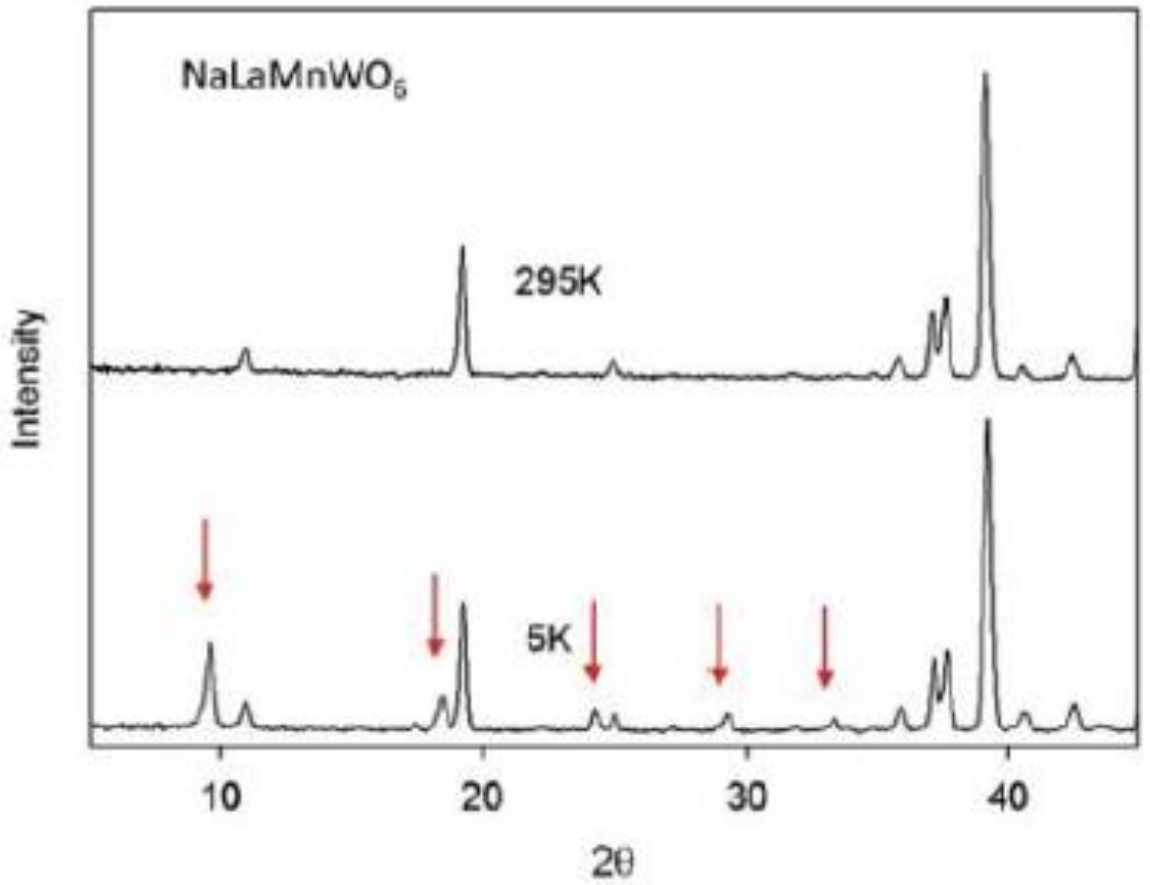
a/h, b/k, c/l

So 5.3/2, 5.3/3, and 7.2/1

Answer: 2.65 Å, 1.77 Å, and 7.1 Å

From this I want you to understand how the hkl relates to the unit cell dimensions.

Neutron diffraction pattern of magnetic material



Indexing for a body centered cell

Exercise: Determine the lattice parameter using table 3.2.

$\sin^2\theta/\sin^2\phi = (h_1^2 + k_1^2 + l_1^2)/ 1^2 + 1^2 + 0^2$

As such $\sin^2\theta/\sin^2\phi = (h_1^2 + k_1^2 + l_1^2)/2$ so you must multiply the ratio by 2.

Answer: $\sin^2\theta = \lambda^2 (h^2 + k^2 + l^2) / 4a^2$
So $a^2 = (1.54)^2(16)/0.0730(4)$
So a = 11.4 Å

Table 3.2 Ratio of $\sin^2\theta$ for K_6C_{60}

2θ	$\sin^2\theta$	Ratio	x 2
10.97	0.0091	1	2
15.54	0.0182	2	4
19.06	0.0274	3	6
22.05	0.0366	4	8
24.69	0.0457	5	10
27.09	0.0549	6	12
29.30	0.0640	7	14
31.38	0.0730	8	16

(321)