

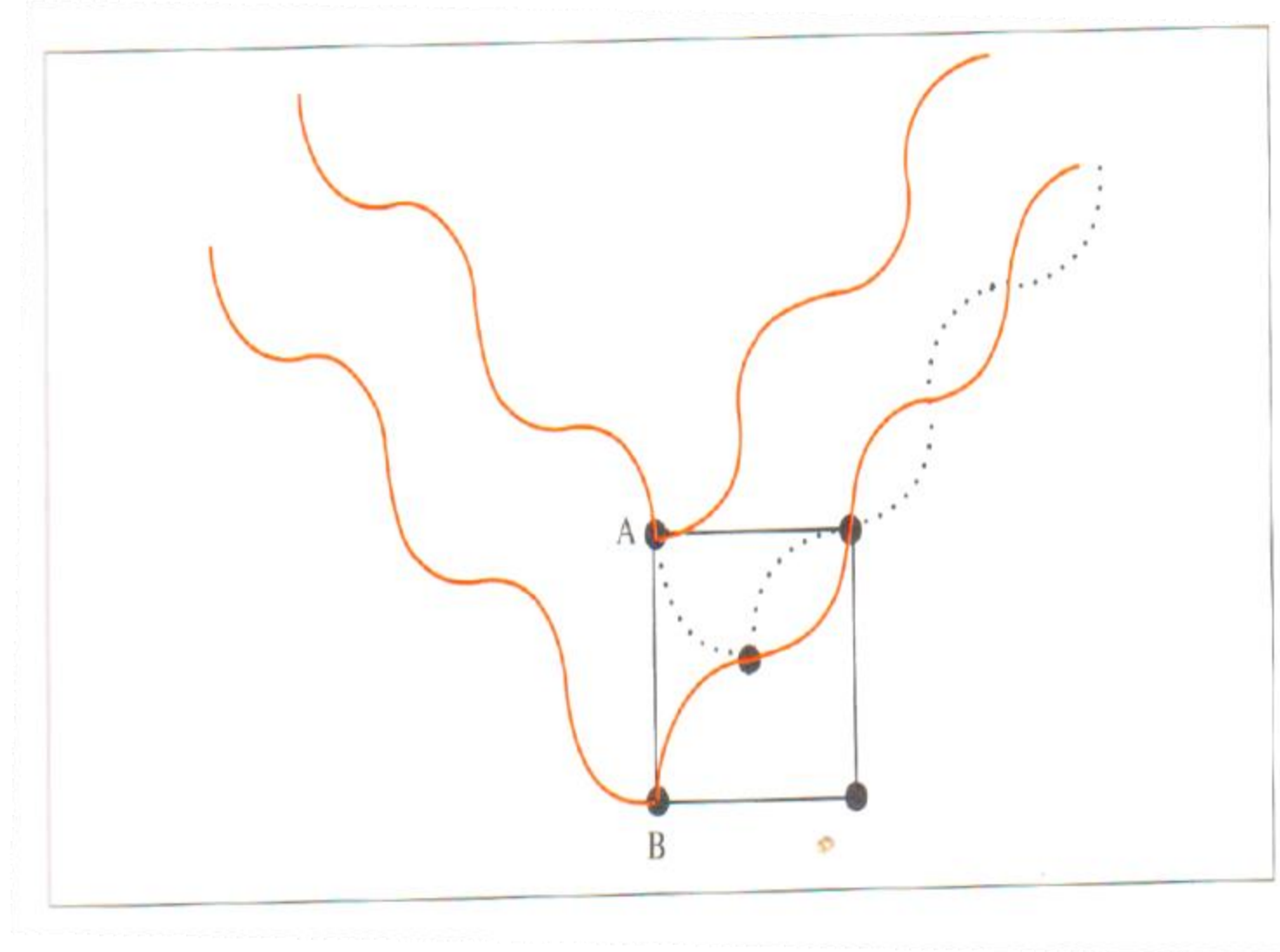
Module 2

Synthesis and Characterization



Solid State Chemistry and Energy Applications

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

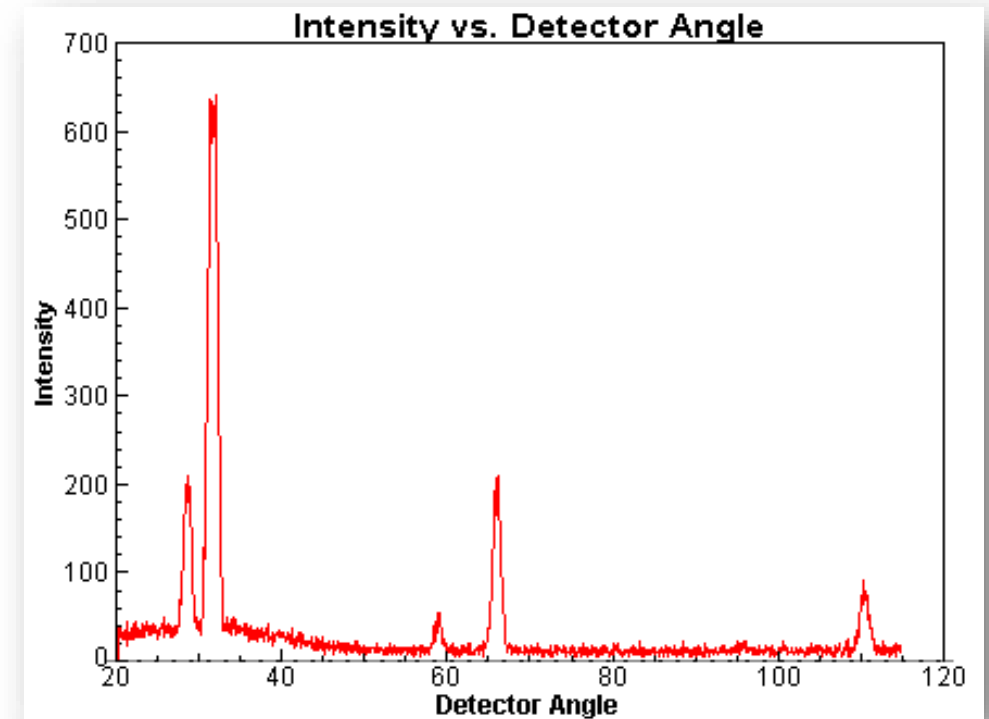
Exercise: given the source is copper, use the 222 reflection to calculate the unit cell dimensions.

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

Answer:

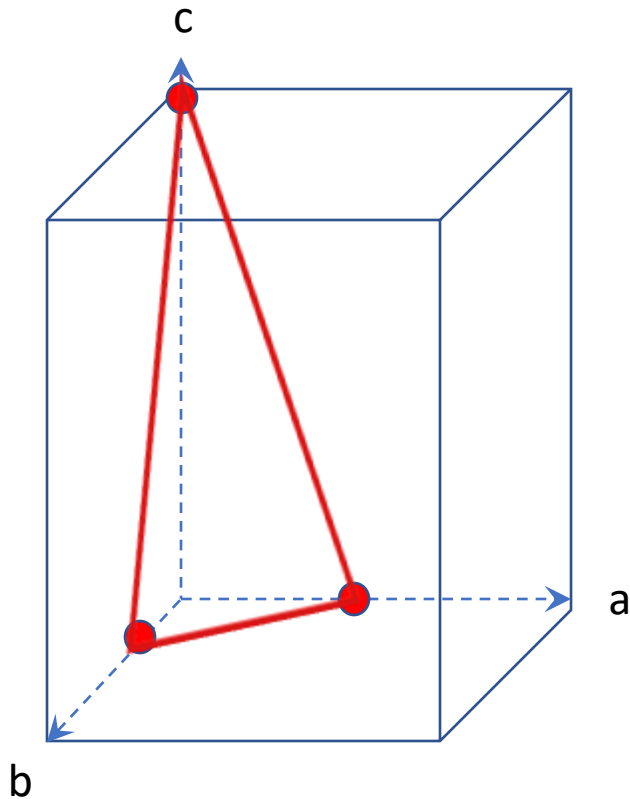
$$\text{So } a^2 = (1.54)^2(12)/0.3342(4)$$

$$\text{So } a = 4.614 \text{ \AA}$$



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

If the unit cell dimensions are a , $b = 5.3 \text{ \AA}$ and $c = 7.2 \text{ \AA}$, 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 $7.2/1$

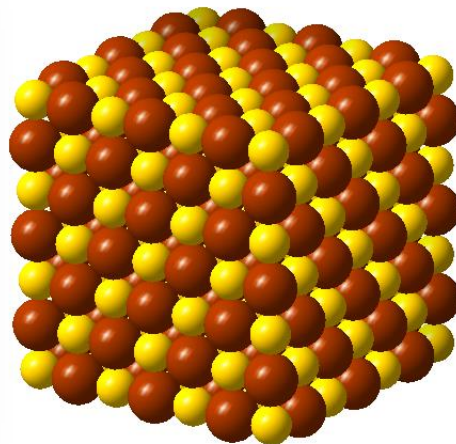
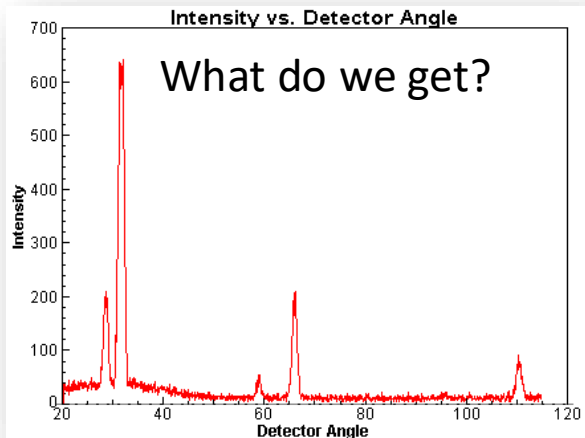
Answer: $2.65 \text{ \AA}$, $1.77 \text{ \AA}$, and $7.1 \text{ \AA}$

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

X-ray diffraction

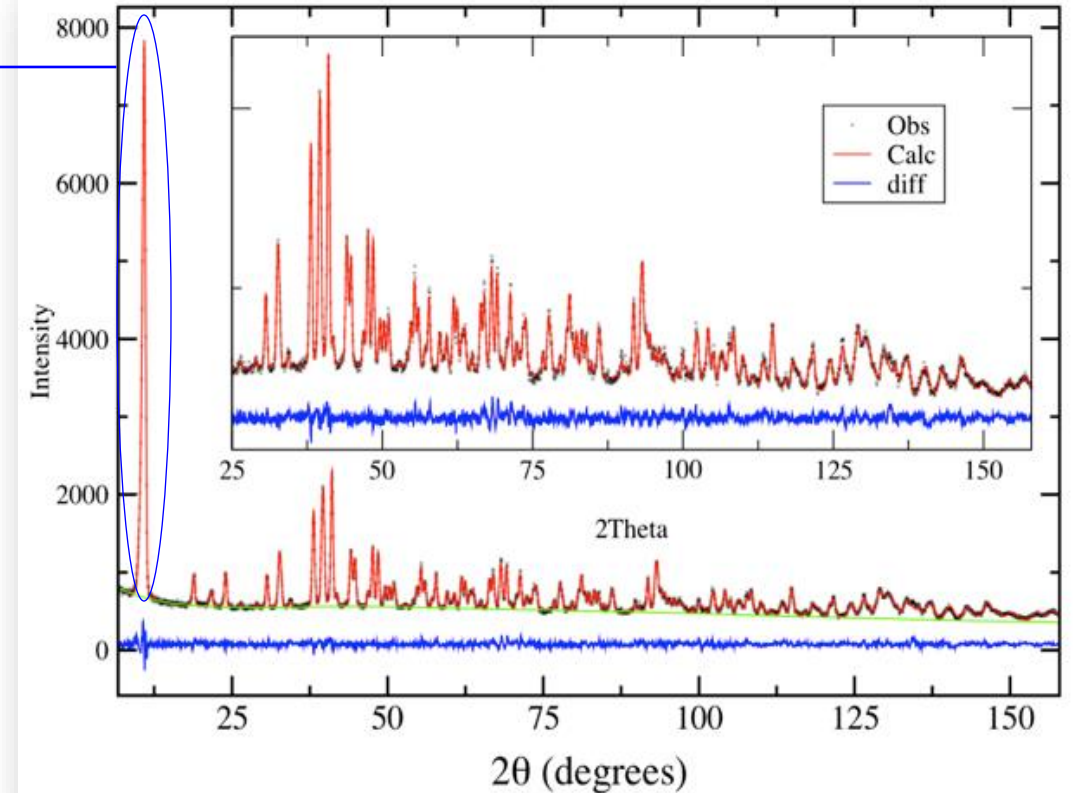
Things that effect Intensities and Peak positions

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



Representing a parallel set of planes

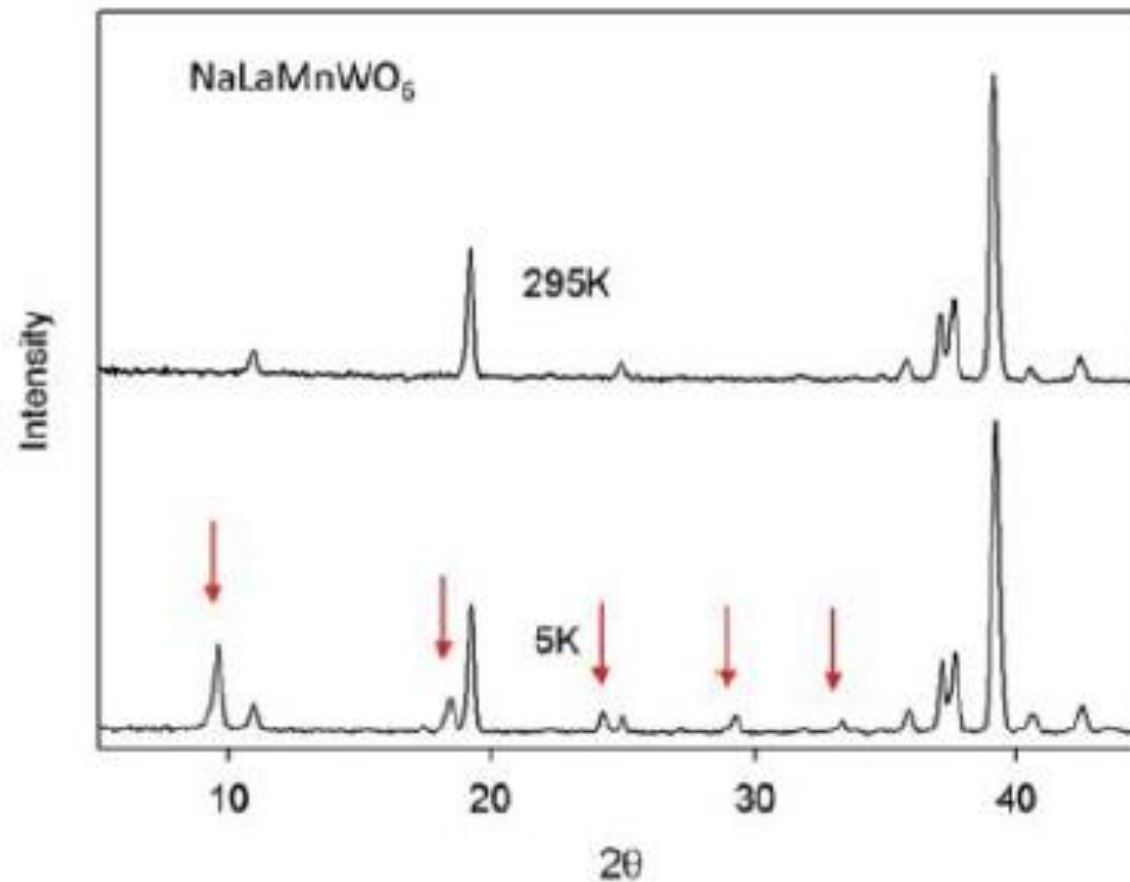
hkl



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?

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

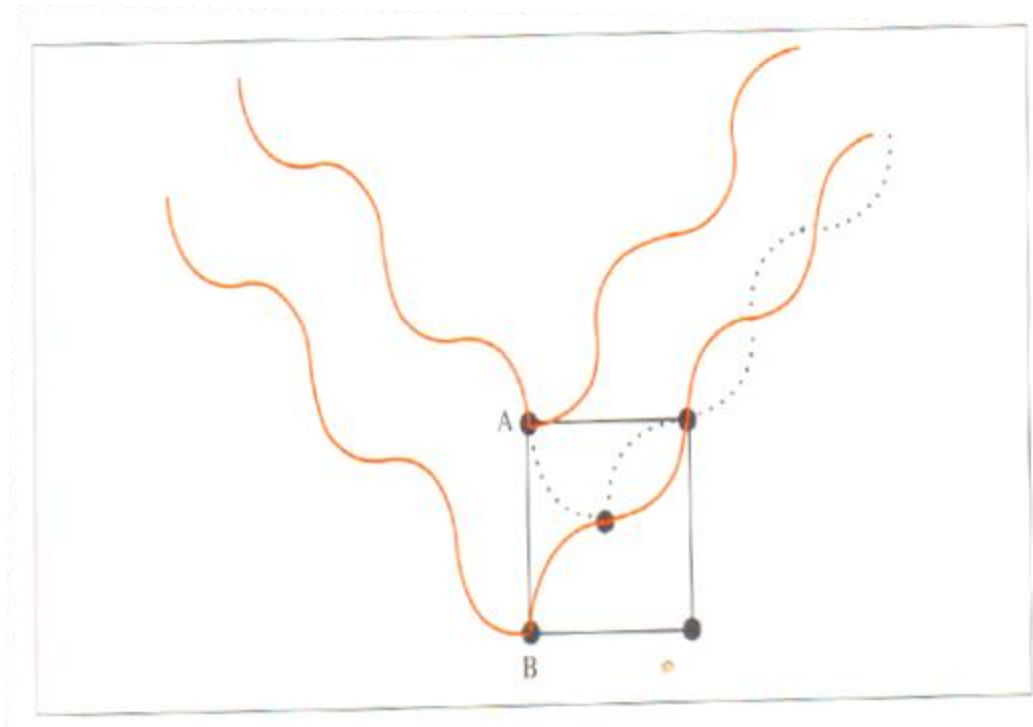
$$\text{So } a^2 = (1.54)^2(16)/0.0730(4)$$

$$\text{So } a = 11.4 \text{ \AA}$$

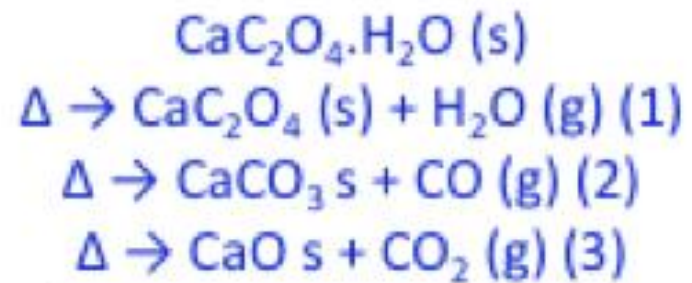
Table 3.2 Ratio of $\sin^2\theta$ for K_6C_{60}

2θ	$\sin^2\theta$	Ratio	$\times 2$
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

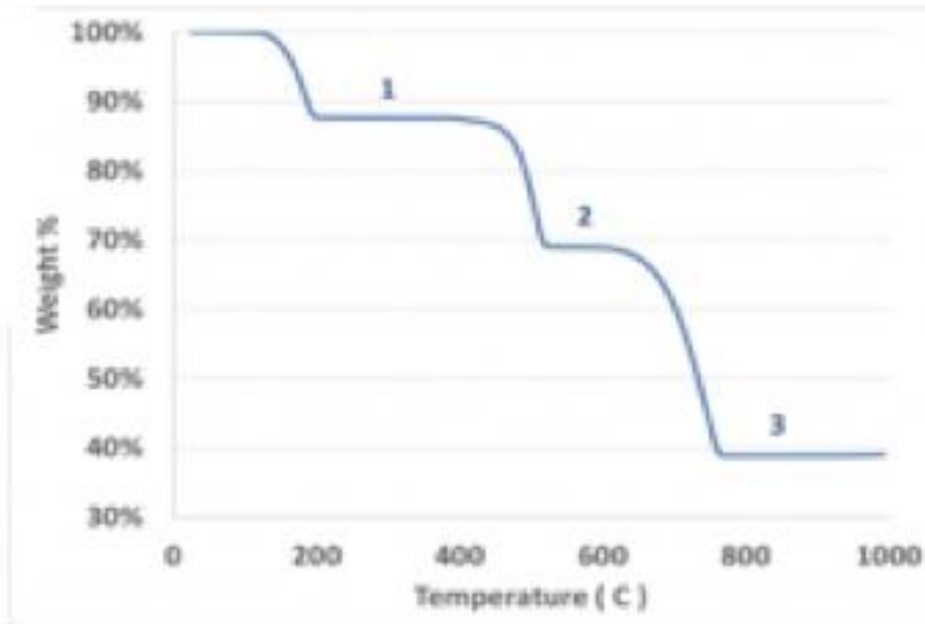
(321)



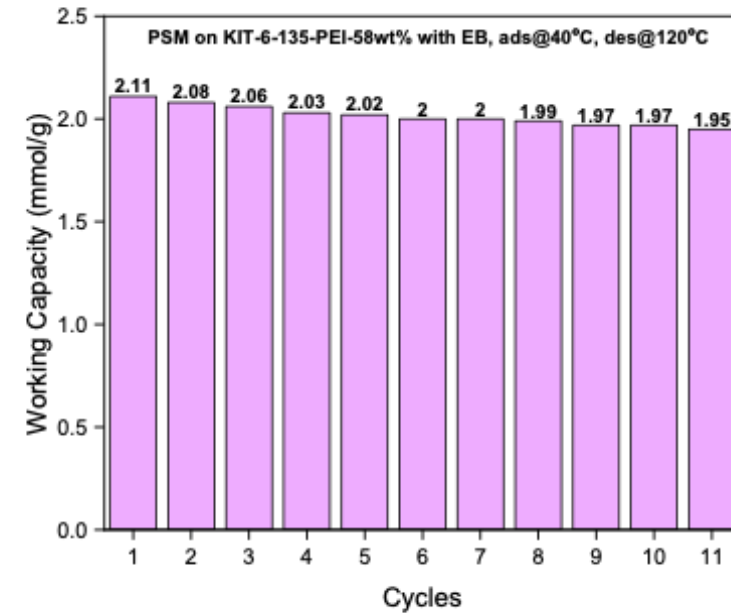
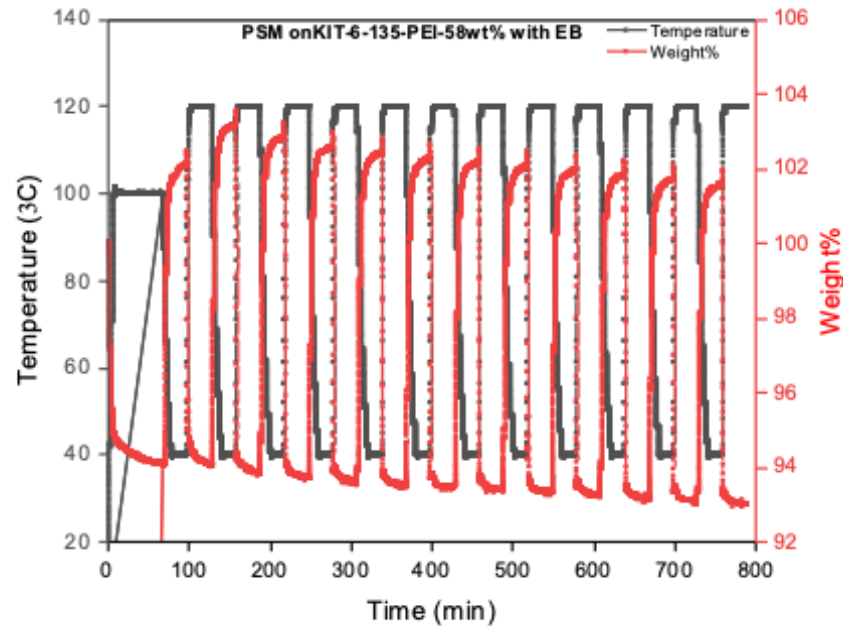
TGA - calcium oxlate



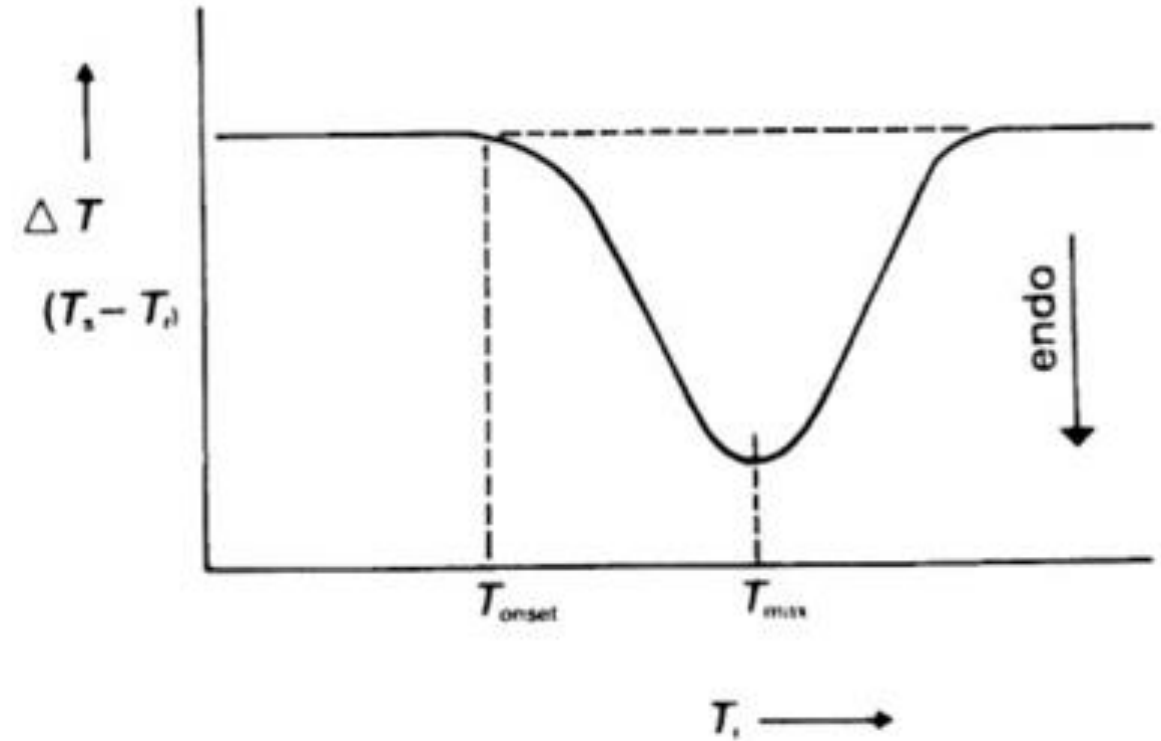
Reaction	Measured Weight Loss (%)
1	12.34
2	18.70
3	30.09



TGA – CO₂ adsorption



DTA Differential Thermal Analysis



DSC Differential Scanning Calorimetry

