Lab 2: XRD, Electron Microscopy, & Microanalysis Kelsey Lawson, 41602126

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1) What are the two openly available (free) databases you can use to look up crystal structure information and three paid databases mentioned in link 1? What is a .cif (crystallographic information file) file, what information does it contain, and what can it be used for?

The two free openly available databases are the Crystallography Open Database (COD) and the American Mineralogist Crystal Structure Database. Three paid databases are the International Crystal Structure Database (ICSD), the Cambridge Structural Database (CSD), and then Pearson's Crystal Database. Within these databases, they offer .cif files or crystallographic information files, that contain the structure of a specified material. When these files are opened with a compatible program it shows how the atoms of the crystal are structured in a 3D model. The elements atoms that are in the crystal are color-coded and size-dependent in order to distinguish what is being represented. Further, these files are mainly used for analyzing the crystal structure of a specific material which can further inform the viewer of the material properties based on bond types and unit cell orientation.

2) Identify the chemical element which generates the XRD pattern in problem 5 of HW 2 by searching the Crystallographic Open Database.

From the homework's given information as well as the lattice constant found, the chemical element is Calcium given by the database. I found this by using the cell parameters and inputting my a values: 2.22 for the minimum and 3.14 as the max while keeping the angles all at 90°. I then restricted my search to the lowest volume first then increasing higher and scrolled until I found a single element with an average parameter set which was Calcium.

3) Match! is one of many XRD data analysis programs available for phase identification of XRD data by enabling automated crystallographic data to search and match capabilities. This software is typically too expensive to purchase yourself but will always be hosted for your use by any facility where an XRD is present. For straightforward cases, search and match software like Match! are routinely used for phase identification. Based on the procedure you observe from video link 2. How would you attempt to verify that the program has identified the correct phase in your sample? How can you enhance the quality of the XRD data fit to get more accurate measurements of lattice parameters, and phase quantification in multiphase samples?

Within the program, Match!, once the file of the XRD graph has been uploaded, the phase identification is run and suggestions of matching phases in the sample are brought up in a list format where the top element is a potential match. In order to verify that the program has identified the correct phase in the sample uploaded, the program graphs the experimental data alongside a color-coded match graph so that the peaks can be compared. Looking at how close the suggested graph's peaks align with the sample's can ensure that the recommended phase is accurate. Furthermore, running the Rietveld refinement calculation using the FullProf button in

the toolbar will provide a final weighted Bragg R-factor and final reduced chi^2 value as well as updated quantity percentages of the phases identified. After, clicking the report button, also located in the toolbar, it will provide a detailed list of the phase analyses where measurements of lattice parameters and phase quantification in multiphase samples can be found.

4) Use the $\sin^2\Theta$ method described in video link 3 to:

a) Index the peaks seen in the XRD pattern below using the peak list provided.

Theta (deg)	Theta (Rad)	sin(Theta (Rad))	sin2(Theta (Rad))	Ratio 1	Ratio 2	Ratio 3	m	(hkl)
13.73	0.2396	0.2374	0.0564	1.00	2.00	3.00	3	111
15.91	0.2777	0.2742	0.0752	2.00	~~3.00~~	4.00	4	200
22.81	0.3982	0.3878	0.1504	3.00	6.00	8.00	8	220
27.035	0.4719	0.4546	0.2067	4.00	8.00	11.00	11	311
28.345	0.4948	0.4749	0.2255	4.00	8.00	12.00	12	222
33.245	0.5803	0.5483	0.3006	6.00	~~11.00~~	16.00	16	400
36.685	0.6403	0.5975	0.357	~~7.00~~	~~13.00~~	19.00	19	331
37.8	0.6598	0.613	0.3758	~~7.00~~	14.00	20.00	20	420

b) Which of the cubic crystal systems is this? (primitive cubic, bcc, or fcc)

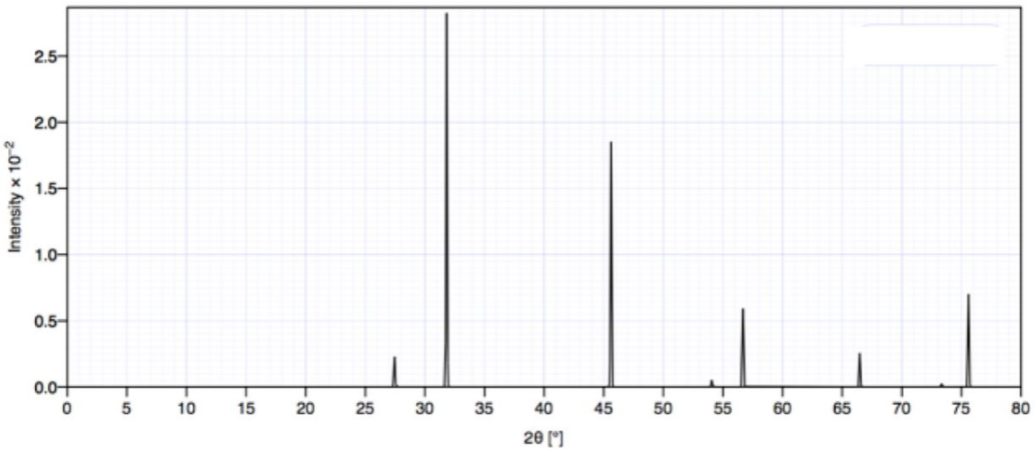
According to the XRD pattern above, the cubic crystal system is a face-centered cubic (FCC).

c) Once you have the hkl values and corresponding d-spacings, check that each entry in your table gives you the same lattice parameter. Once you find the lattice parameter use the Crystallography Open Database and search by lattice parameter to find the chemical formula and space group.

a^2	a
31.4928	5.611844617
31.5844	5.62
31.6808	5.628569978
31.4171	5.605095896
31.4928	5.611844617
31.8096	5.64
31.6179	5.622979637
31.752	5.634891303

Using the COD and entering the lattice parameter a minimum as 5.612 and maximum as 5.64, the chemical formula that matched the average of these values at 5.62 is NaCl.

Note: There will be multiple possibilities for your hkl values the higher the m value gets ($m = h^2 + k^2 + l^2$). List all the possibilities fo reach hkl and use the lower angle peaks (where m implies unique hkl) to help you determine the crystal structure based on the allowed combinations of h, k, l for the different crystal systems/. Once you have determined the crystal system, you should be able to choose the correct hkl for the higher m values.



d(hkl) [A]	2 θ (deg)	I/Imax
3.24	27.46	8.10%
2.81	31.82	100.00%
1.99	45.62	65.70%
1.69	54.07	2.00%
1.62	56.69	21.20%
1.41	66.49	9.30%
1.29	73.37	1.00%
1.26	75.60	24.80%