Powder Diffraction – "Thought Exercises"

24th National School on Neutron and X-ray Scattering

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Thought Exercises

- Consider the following scenarios based on what you learned about powder diffraction!
- Ask yourself: How would I solve this problem? What tools or techniques are available to me, and what are the pros and cons of each? What do I actually need to answer the question that I am trying to address?
- There are of course way more scenarios than what could be included in this handout – especially for challenging/non-standard/in-operando experiments: *Talk to the beamline scientists before you prepare and submit your proposal!!! They are the experts, and they are happy to help you!*

Thought Exercise 1

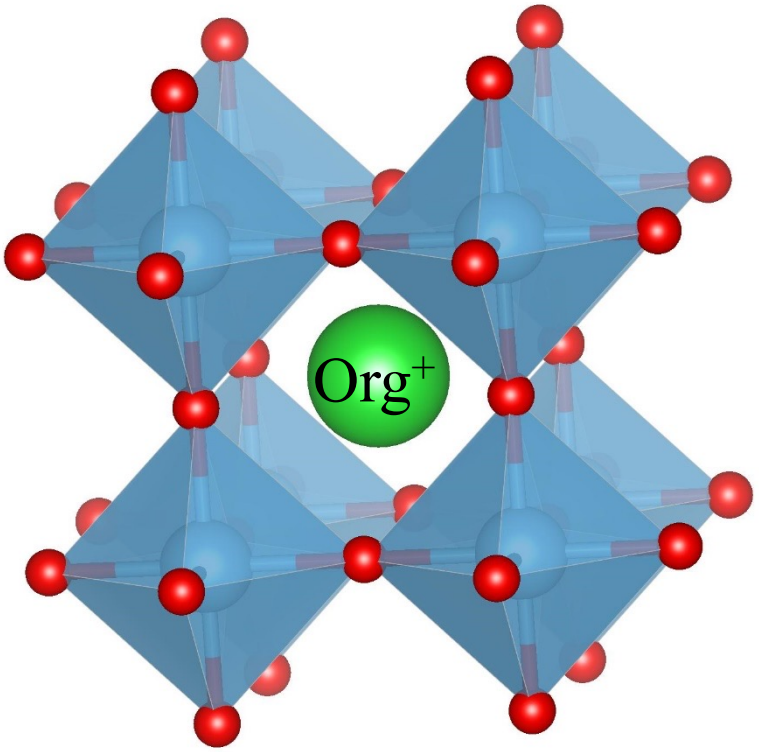
- You attempted to prepare a metal oxide sample in the lab by heating a carbonate precursor in air at 1000 °C. How do you confirm whether your experiment was successful?

Thought Exercise 2

- Your collaborator gives you 20 mg of a pharmaceutical powder sample and asks that you carry out a Rietveld refinement to address how much of polymorph 1 and polymorph 2 of this compound is present in the sample. How do you proceed?

Thought Exercise 3

- You synthesized a novel lead halide perovskite, $OrgPbI_3$, with an organic counterion that we will call "Org^+". This cation can adopt several different isomers (isomer = different arrangement of the atoms), and you have found that your solvent choice affects the properties of your products. What do you do to characterize them?



Thought Exercise 4

- You are spending the summer as a visiting researcher at University X to do electrochemical studies on your materials with an expert. After preparing a sample and collecting a powder pattern, you notice that the pattern does not look the way you expected.

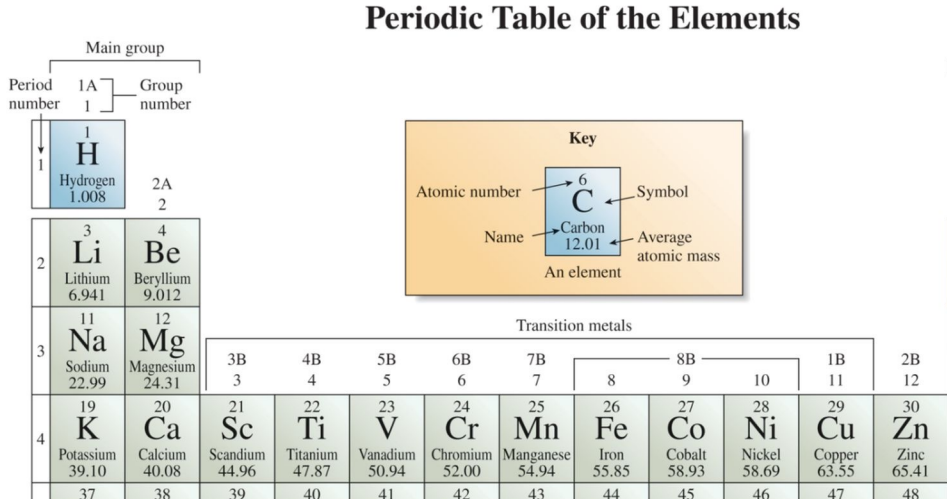
  1) What are some potential “unexpected differences”?

  2) What could cause these differences?

  3) Are these differences a problem or not? If they are, how can you prove that these issues are present?

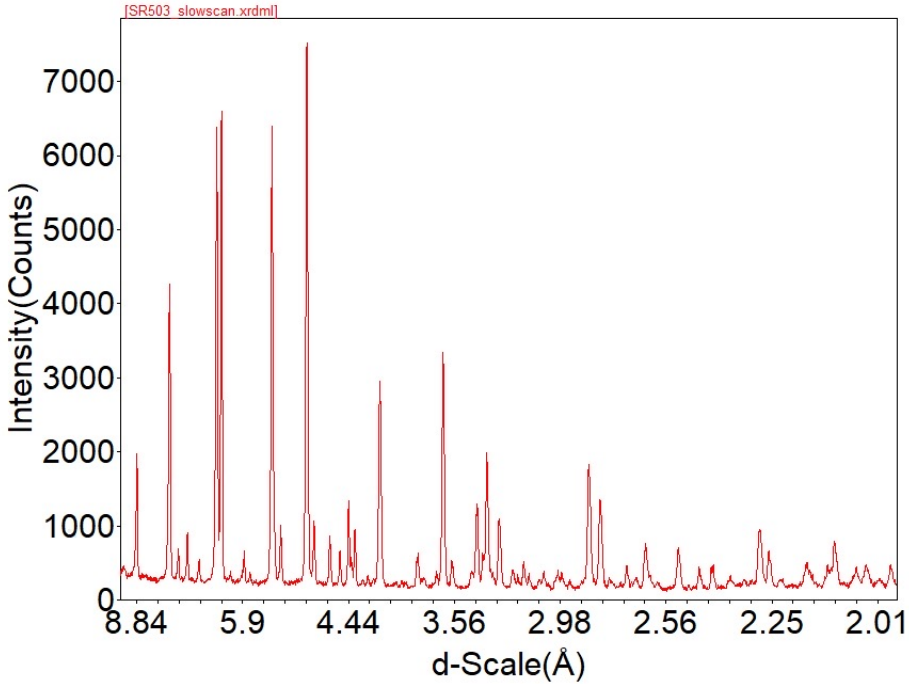
Thought Exercise 5

- You are investigating $Mn_xFe_{1-x}PSe_3$ compositions. How can you determine whether the metal cations are statistically mixed on all sites, or whether there is a preference for specific sites?



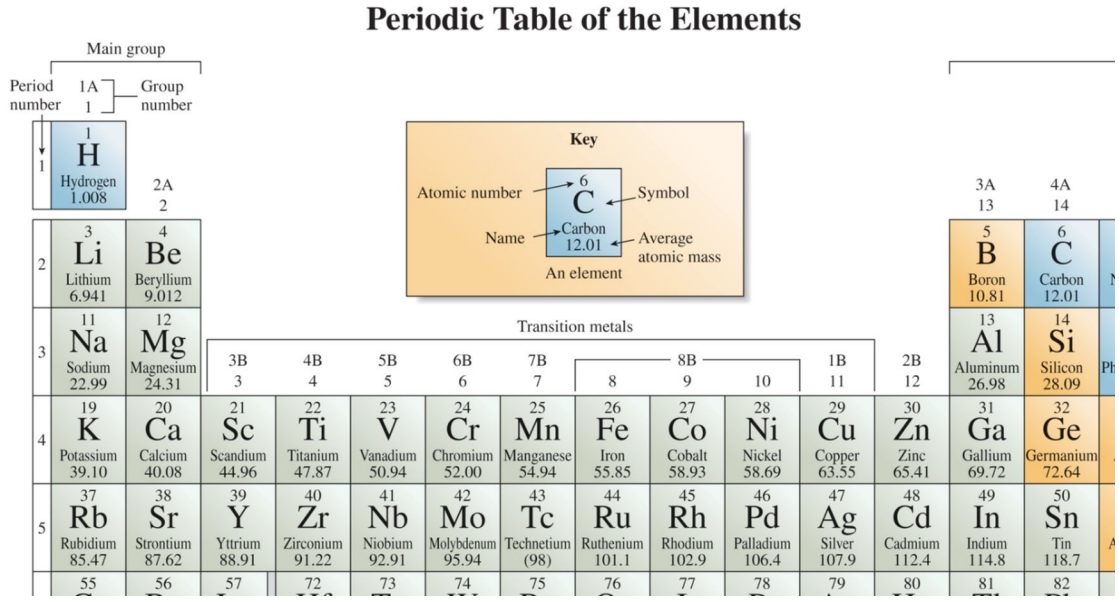
Thought Exercise 6

- You collect the following PXRD pattern on your lab diffractometer, and fail at indexing it. What could you do to overcome this?



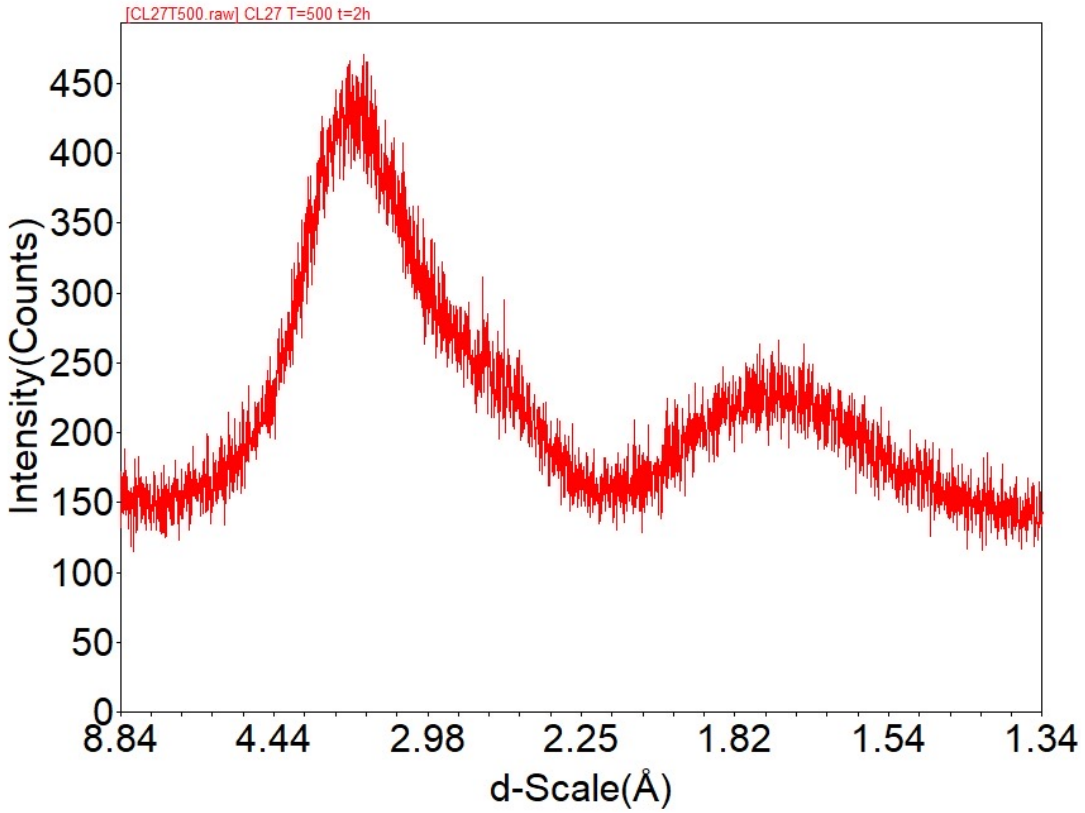
Thought Exercise 7

- You want to analyze a clathrate with the formula $Cs_8Cd_4Sn_{42}$, and answer the question whether Cd and Sn have a preference for specific framework sites. What approach should you take?



Thought Exercise 8

- You collect the following diffraction pattern. What experiments could you do to gain structural knowledge of your material?



Thought Exercise 9

- You need to collect diffraction data on a strongly absorbing sample. What are your options for overcoming this problem for (i) neutron and (ii) synchrotron experiments?