

# Powder Diffraction – “Thought Exercises”

---

*24<sup>th</sup> National School on Neutron and X-ray Scattering*

Cora Lind-Kovacs  
Department of Chemistry & Biochemistry  
The University of Toledo, Toledo, OH  
[Cora.lind@utoledo.edu](mailto:Cora.lind@utoledo.edu)

# 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

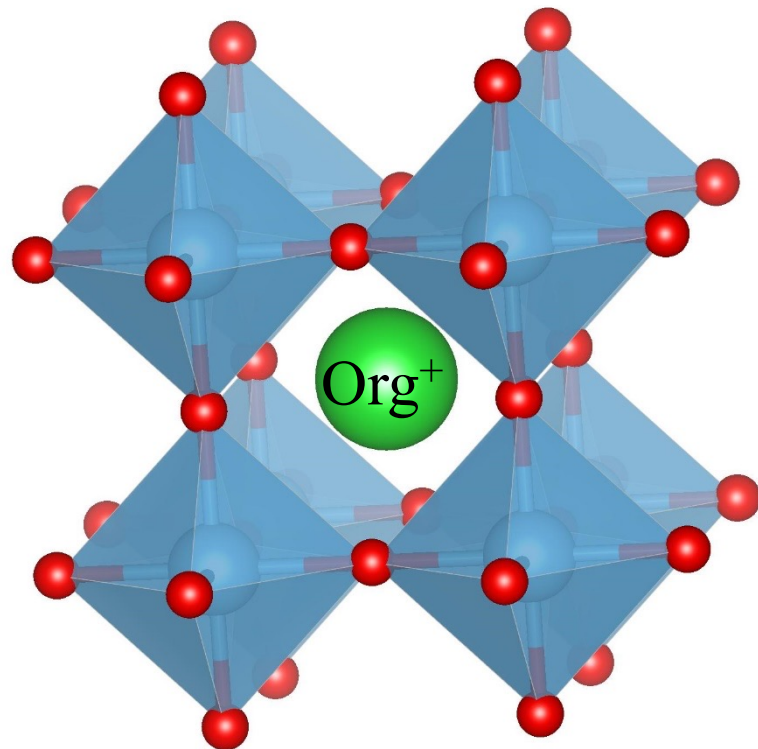
---

- ◆ 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,  $\text{OrgPbI}_3$ , with an organic counterion that we will call “ $\text{Org}^+$ ”. 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  $\text{Mn}_x\text{Fe}_{1-x}\text{PSe}_3$  compositions. How can you determine whether the metal cations are statistically mixed on all sites, or whether there is a preference for specific sites?

## Periodic Table of the Elements

The diagram illustrates the periodic table with a focus on element information. It includes a 'Main group' section on the left, a 'Key' section in the center, and a 'Transition metals' section on the right.

**Main group:** This section shows the first two columns of the periodic table. It includes the following elements:

Period number	1A	2A
1	H (Hydrogen, 1.008)	
2	Li (Lithium, 6.941)	Be (Beryllium, 9.012)
3	Na (Sodium, 22.99)	Mg (Magnesium, 24.31)
4	K (Potassium, 39.10)	Ca (Calcium, 40.08)

**Key:** This section provides a template for how to read an element's information from the periodic table. It uses the element Carbon (C) as an example:

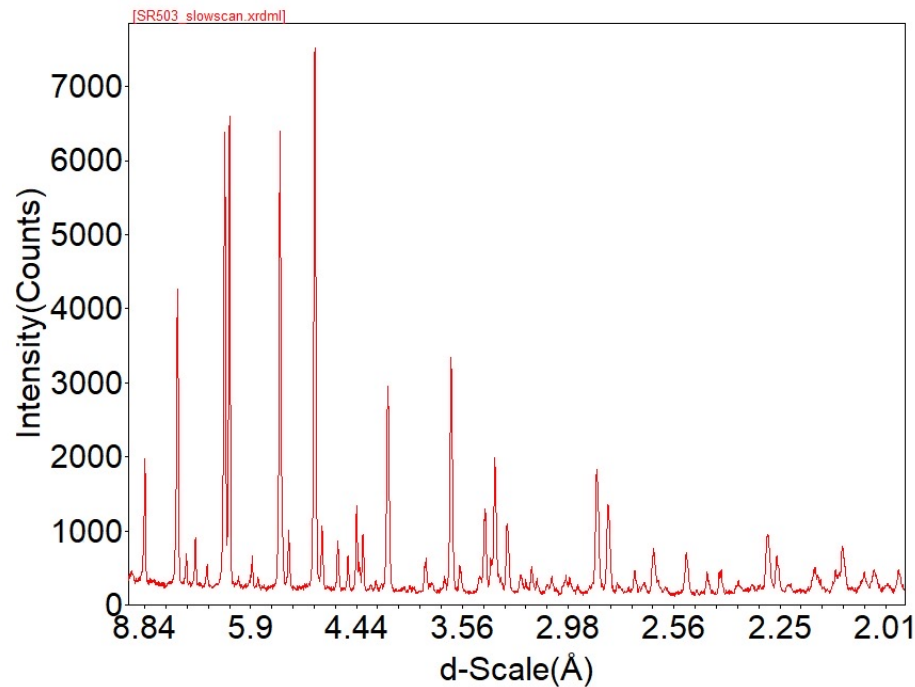
- Atomic number:** 6
- Symbol:** C
- Name:** Carbon
- Average atomic mass:** 12.01
- An element:** Carbon

**Transition metals:** This section shows the elements in the d-block of the periodic table, from Scandium (Sc) to Zinc (Zn). It includes the following elements:

3B	4B	5B	6B	7B	8B	1B	2B
3	4	5	6	7	8	9	10
Scandium 21 44.96	Titanium 22 47.87	Vanadium 23 50.94	Chromium 24 52.00	Manganese 25 54.94	Iron 26 55.85	Cobalt 27 58.93	Nickel 28 58.69
Copper 29 63.55	Zinc 30 65.41						

# 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  $\text{Cs}_8\text{Cd}_4\text{Sn}_{42}$ , and answer the question whether Cd and Sn have a preference for specific framework sites. What approach should you take?

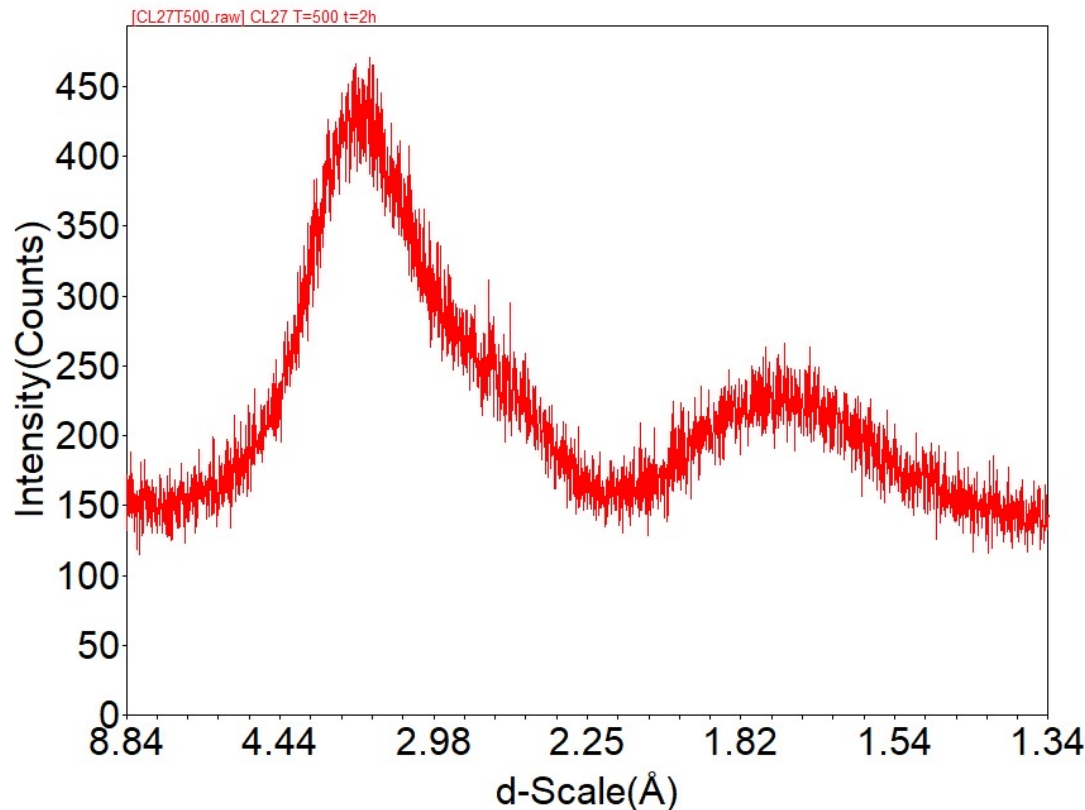
Periodic Table of the Elements

Main group																	
Period number	1A															3A	4A
	1															13	14
1	1															5	6
	1															B	C
	1															10.81	12.01
	1																
2	2															3	4
	2															Li	Be
	2															6.941	9.012
	2																
3	3															11	12
	3															Na	Mg
	3															22.99	24.31
	3																
4	4															19	20
	4															K	Ca
	4															39.10	40.08
	4																
5	5															37	38
	5															Rb	Sr
	5															85.47	87.62
	5																
	5															55	56
	5																
	5															72	73
	5																
	5															74	75
	5																
	5															76	77
	5																
	5															78	79
	5																
	5															80	81
	5																
	5															82	83
	5																

Key			
Atomic number	6	Symbol	C
Name	Carbon	Average atomic mass	12.01
An element			

# 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?