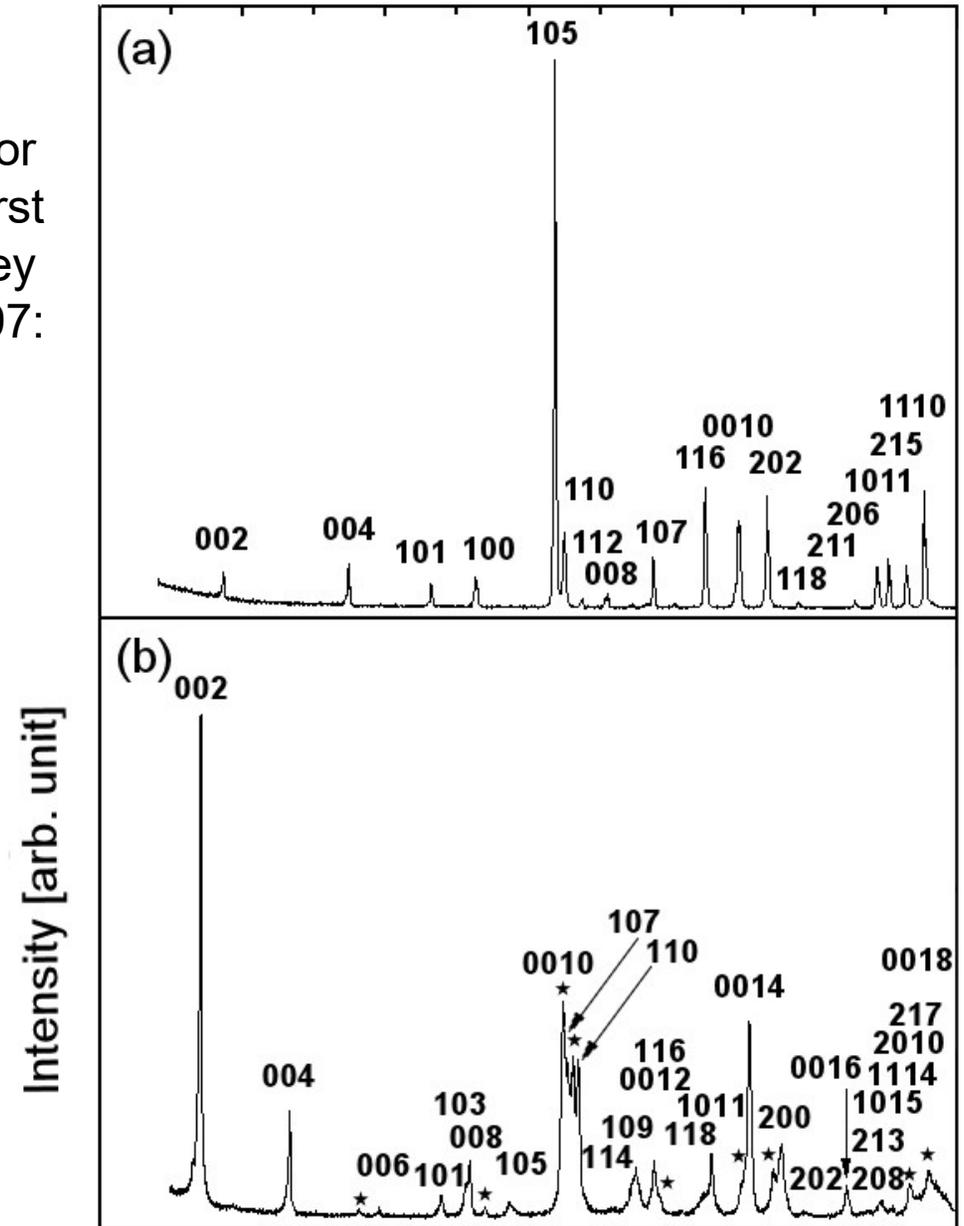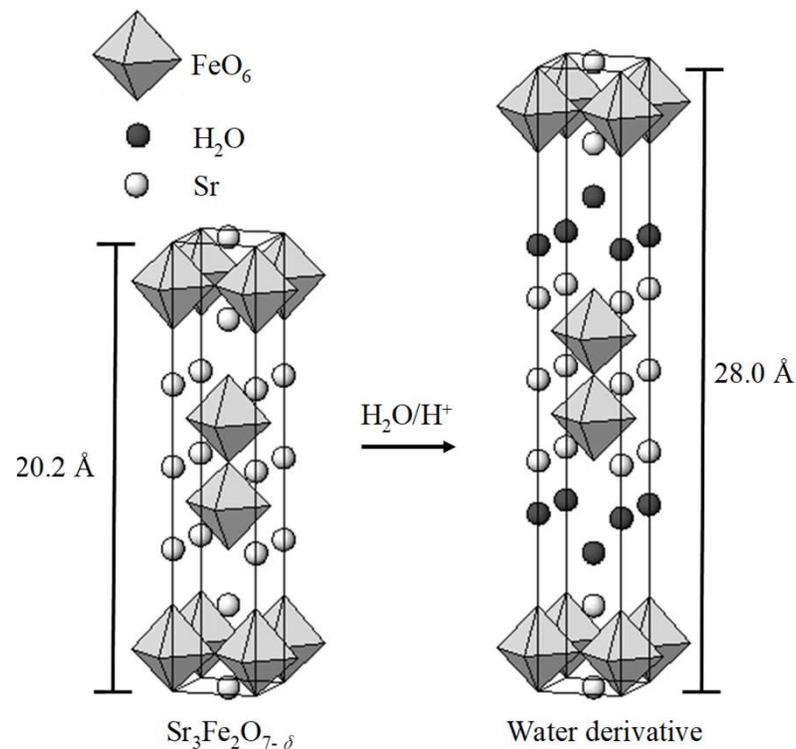# EXERCISE 3.1.

Please calculate the c lattice parameter for the water-derivative phase (b) from the first three (00$l$) reflections (002, 004, 006); they are at the 2θ angles 6.31, 12.65 and 18.97: tetragonal I4/$mmm$, CuK$_\alpha$ = 1.5406 Å



Lehtimäki, Hirasa, Matvejeff, Yamauchi & Karppinen, *J. Solid State Chem.* **180**, 3247 (2007).

# EXERCISE 3.2.

Below are the lattice parameters a and c calculated for one sample (tetragonal *P4/mmm*; Cu-K$_\alpha$ = 1.5406 Å) from the 002, 004, 100 and 200 reflections.

Now, please use the 110, 102 and 112 reflections to calculate the lattice parameters, for verification:

$$\sin^2\theta = (h^2 + k^2) \cdot \frac{\lambda^2}{4a^2} + l^2 \cdot \frac{\lambda^2}{4c^2}$$

| 2θ | hkl |
|---|---|
| 22.766 | 1 0 0 |
| 23.026 | 0 0 2 |
| 32.411 | 1 1 0 |
| 32.603 | 1 0 2 |
| 40.136 | 1 1 2 |
| 46.499 | 2 0 0 |
| 47.054 | 0 0 4 |

| Miller | 2θ | sinθ | sin²θ | Parameter (Å) |
|---|---|---|---|---|
| 002 | 23.03 | 0.1996 | 0.0398 | 7.724 |
| 004 | 47.05 | 0.3991 | 0.1593 | 7.725 |
| 100 | 22.77 | 0.1947 | 0.0390 | 3.905 |
| 200 | 46.50 | 0.3947 | 0.1558 | 3.906 |

# EXERCISE 3.3.

**Please index the diffraction pattern below; the unit cell is cubic. What are the lattice parameters and what is the centering? $\lambda$=1.5406 Å (Cu Kα)**
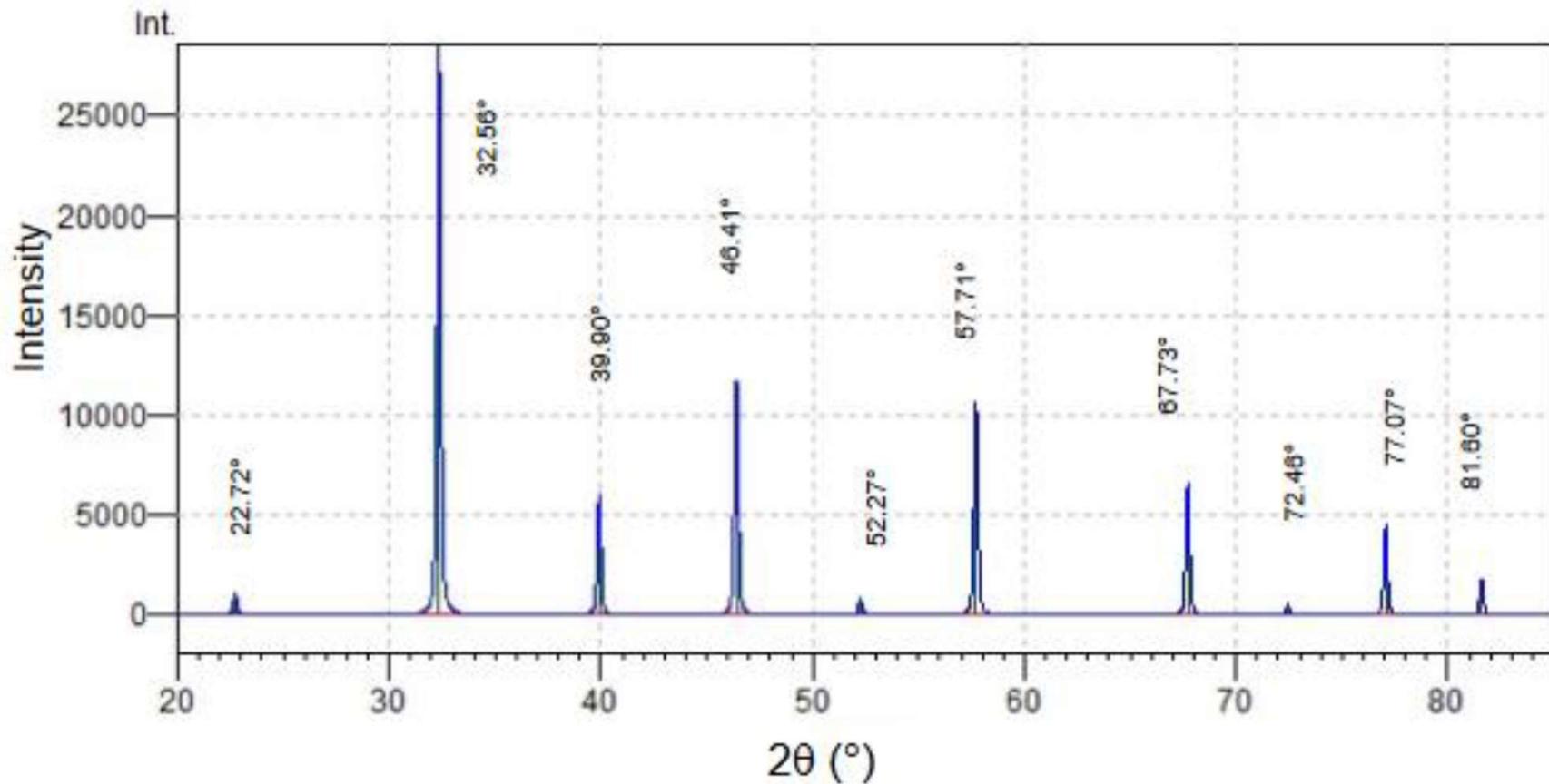
# EXERCISE 3.4.

For an unknown crystalline material the following analysis data are available:

- Elemental analysis: Ba 89.57 p-%, O 10.43 p-%
  (atomic weights: Ba 137.33; O 15.9994)

- Density: 5.922 g/cm³ ($N_A$ = 6.022 x $10^{23}$)

- Peaks in powder XRD pattern (Cu$K_\alpha$: $\lambda$ = 1.5406 Å) at $2\theta$ angles:

  27.88, 32.30, 46.32, 54.92, 57.60, 67.60, 74.62, 76.90, 85.88, 92.54

  a. Assign Miller indices for the diffraction peaks assuming cubic NaCl type unit cell.

  b. Calculate lattice parameter *a*.

  c. Draw the unit cell.

  d. What is Z?

  e. Calculate Ba-O bond length.

## EXERCISE 3.5.

Please shortly explain the important observations (up to four!) you can make from the figure below.
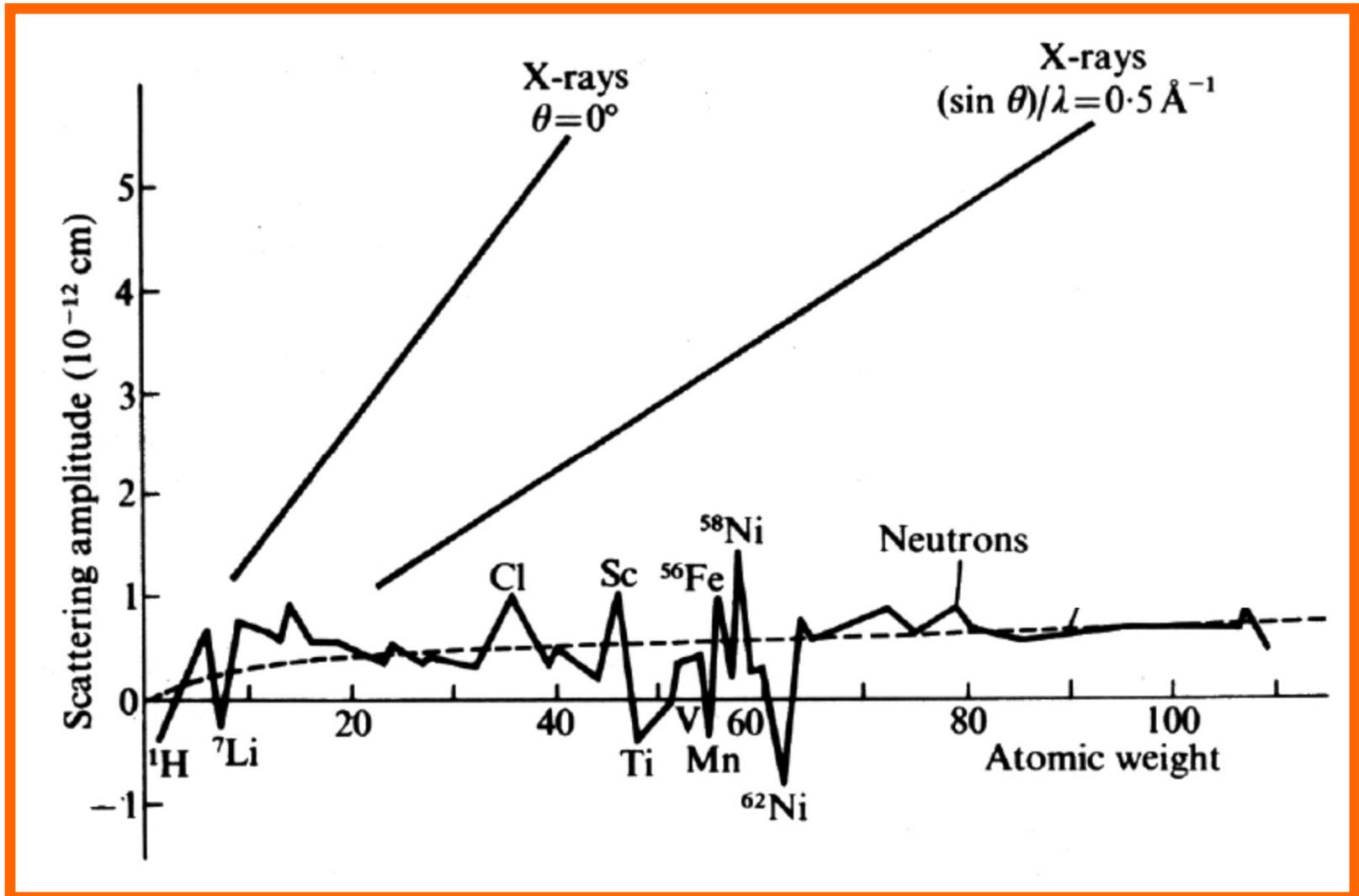
# EXERCISE 3.6.

On the lecture slides (Lec 6) there is a sentence:

For ND, no "bonding effects" in atomic positions → Important when hydrogen-bonded structures are studied → ND reveals typically ~0.2 Å longer O-H bonds than XRD.

Please try to explain this in more detail.

HINT: consider the electron densities in hydrogen bonds



Hydrogen bond

$\delta-$  $\delta+$  $\delta-$

low electron density