Powder X-ray diffraction (PXRD)

**Instructions for the Advanced Lab Course (B.Sc., L3)
and the Research Lab Course (M.Sc.)
at Physikalisches Institut of the Physics Department
of the Goethe-Universität Frankfurt**

Supervisor: Sarah Krebber
E-Mail: sarah.krebber@web.de
Telephone: 069 798 47260
Location: 0.409 (Bauteil 4, UG1, AG Krellner)
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The whole course on one page

What is it about?

- Preparing and recording of X-Ray Powder Diffractograms
- Identifying simple crystal structures by means of diffractograms
- Qualitative phase analysis

What will be done?

An unknown compound will be identified by indicating a recorded X-Ray Powder Diffractogram. A mixture of unknown compounds will be analysed for contained compounds. Using a third sample, influences of texture in X-Ray diffractometry.

What will be learned?

- Preparing samples for X-Ray Powder Diffractometry
- Recording of X-Ray Powder Diffractograms
- Generating and dealing with X-Rays
- Applying the Bragg equation in order to index diffractograms
- Determining simple crystal structures by their diffraction patterns

Why is this interesting?

- Structural characterization of crystalline samples
- Phase analysis and structure resolution of unknown samples
- Connecting structural transitions to electronic phase transitions
- Industrial screening of material properties
- Crystalline order in thin films

1 Vorzubereitende Inhalte

The following key words should be familiar to a degree, that knowledge of their contents can be applied at work in the lab and for interpreting the measurements. The central key words are marked with an asterisk (*) and should be discussed in the literature part of the lab report.

- Crystallography
  - Crystal systems*
  - Bravais lattices*
  - Reciprocal space and reciprocal lattice*
  - Miller indices*
  - Symmetry elements in solids, space groups
  - Types of bindings, constituent interactions in the crystal
- Structural determination
  - Bragg and Laue condition*
  - their equivalence
  - Structure factor*
  - Extinction of reflexes*
  - Atomic scattering factor*
  - Phase problem
  - Informations from location, intensity and form of reflexes*
  - Texture*
- Structural resolution (background only)
  - Patterson technique
  - Rietveld refinement
- Crystal quality*
  - Defects
  - Pair Distribution Functions (PDF)
  - Crystallite sizes, Scherrer equation
  - Noise

- Procedures of X-Ray Powder Diffractometry
  - Single Crystal versus Powder Diffractometry
  - Transmissive and refractive measurements
    - Bragg-Brentano geometry*
    - Debye-Scherrer geometry
    - Guinier geometry
  - Sample requirements of the methods
  - Generating X-Rays in the lab
  - Monochromators: Types, setup and function
  - Detection of X-Rays
- Applying the procedures*

2 Technical remarks

During lab course, you will work with a D8 diffractometer by Bruker. Commissioning is allowed only after closely consulting the lab assistant. The diffractometer is a full-protection device. If handled conforming to the standards of commission, harmful exposure to ionizing radiation is impossible.
During lab course, you will also possibly work with mildly hazardous materials (e.g. solvents). If handled conforming to the standards of commission, dangers to your personal health are highly improbable.

3 Conducting the lab course

1. Comment on the connections between binding types, crystal structure and material properties, assuming two compounds exhibiting the same distribution of elements. Exemplify this discussing the different modifications of Carbon. What is the relevance of phase analysis complementing analysis of the elements?

2. Unknown sample FP1

   - Prepare sample „FP1“.
   - Conduct a XRPD measurement on FP1.
   - Index the measured diffractogram and conclude the crystal structure and lattice parameter(s). Identify the sample using the Hanawalt index.

3. Unknown sample FP2

    - Prepare sample „FP2“.
    - Conduct a XRPD measurement on FP2.
    - FP2 contains the elements Oxygen (O), Chlorine (Cl), Potassium (K), Titanium (Ti), Copper (Cu) and Strontium (Sr). Identify the compounds in the sample consulting a database.

4. Unknown sample FP3

    - A poorly prepared sample „FP3-1“ will be given by the supervisor.
    - prepare sample „FP3-2“to the best of your abilities.
    - Record a diffractogram for both samples.
    - FP3 contains the elements Hydrogen (H) and Carbon (C). Identify the compound consulting a database.
    - Compare the diffractograms. What do you conclude regarding the habitus of the crystals?

4 Literature

Preparing this lab course, please rely on specialized literature rather than on internet sources, whenever possible (exception: citation of pictures). Literature usually presents the matter of interest in a more coherent and comprehensive way, and properness is reassured via an editing process. Internet sources on the other hand are mostly unreliable and not citable (with very few exceptions)!
This literature might be useful:

- W. Massa: Kristallstrukturbestimmung, Teubner-Verlag. (Compact introduction to X-Ray methods and structural resolution, german)
- W. Borchardt-Ott: Kristallographie, Springer Spektrum (Introduction with focus on symmetries, german)
- V.K. Pecharsky und P.Y. Zavalij: Fundamentals of powder diffraction and structural characterization of materials, Springer-Verlag. (Focus on Powder Diffractometry and refinement, english)
- W. Kleber: Einführung in die Kristallographie, De Gruyter-Verlag. (Extensive introduction to crystallography and structural resolution, german)
- Every introduction into solid state physics also contains a chapter on crystal structures and their resolution with X-Ray methods. This will be less detailed, but can provide a first overview to the topic.

5 Software

Crystal structures can be depicted using different crystallographic programs. Two freeware programs are:

- Mercury:
  `https://www.ccdc.cam.ac.uk/Community/csd-community/freemercury/`
- VESTA: `http://jp-minerals.org/vesta/en/`