# Z4 - Analysis of Structures and Quantitative Analysis of Crystalline Substances by X-ray Diffraction Method

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The experiment uses the method of X-ray diffractometry for testing polycrystalline single- and multi-phase samples. The exercise is performed on an Empyrean diffractometer (PANalytical), working in Bragg-Brentano geometry (vertical), equipped with a ceramic X-ray tube with a Cu anode. The Ni filter is used for monochromisation of the incident (or dispersed) beam. Due to the fact that both Polish and English literature, at various levels, is easily accessible, this exercise also gives students the opportunity to take their first steps or develop their skills in quickly and efficiently gathering information on a given topic. In the basic version, a polycrystalline sample of a regular crystal is tested, and a powder diffractogram is the basis for determining the type of elementary cell and determining the value of the crystalline lattice parameter of the substance being tested. Exercise can be carried out not only in the basic version. To ambitious students, it gives almost unlimited possibilities to create their own research programs or to implement modified programs proposed by the tutor. Although the exercise is performed individually, and each student receives only two tasks, it is possible to plan a larger research project carried out by a group of cooperating students. The implementation of student projects is possible after obtaining the consent of the tutor.

## Preparatory questions

1. Crystals; the concept of a direct and reciprocal lattice.
   - Knowledge of definitions and understanding of terms is necessary: basic translation vectors (base vectors), spatial lattice, base (motive), crystal lattice, elementary cell, Miller pointers, crystalline surfaces and directions in the crystal, interplanar distances, single crystals, polycrystals..
   - The structure of sodium chloride (NaCl).
2. Receiving and characteristics of X-rays.
   - X-ray tubes for structural investigations.
   - X-ray tube radiation spectrum (continuous spectrum, characteristic spectrum).
3. The interaction of X-rays with matter.
4. X-ray diffraction on crystalline bodies.
   - X-ray diffraction geometry (Lauego equation, Bragg law, Ewald's construction).
   - Intensity of X-ray diffraction reflections, rules of extinction for regular crystal lattice.
5. Selected experimental diffraction methods.
   - Powder method: theoretical basis, geometry of the measurement system and methodology of measurement using a polycrystalline diffractometer in Bragg-Brentano geometry, extrapolative methodology for determining the exact value of the crystalline lattice parameter (including measurement uncertainty).
   - Laue method: theoretical basis, geometry of the measurement system in the geometry of the reflected beam and the beam passing.
   - Elements of dosimetry and health and safety regulations applicable when working with X-rays.

Note: Basic information necessary to pass the initial test is contained in chapters 1 and 2 of the textbook "Introduction to solid state physics", C. Kittel and in notebooks 1 and 3 of the textbook "Materials for X-ray structural analysis", Z.Bojarski, E .Łągiewka. To take the full advantage of time spent in the Laboratory, you should also familiarize yourself with the issues written in italics here.

## Computational assignments

1. The lattice line goes through the points with the following coordinates: 0 1/2 1 and 1 2 1/2. Provide direction indicators for this simple lattice.
2. Calculate the value of the short-wavelength limit of the continuous spectrum of X-ray tube radiation supplied with voltage U = 40 kV.

More interesting tasks and test questions with varying degrees of difficulty can be found on the page of the 2nd National Crystallographic Olympiad: http://www.komkryst.pan.pl/index.php/en/olimpiada-2016.

## Apparatus and materials

The exercise is performed on an Empyrean X-ray diffractometer (PANalytical) working in the pseudo-

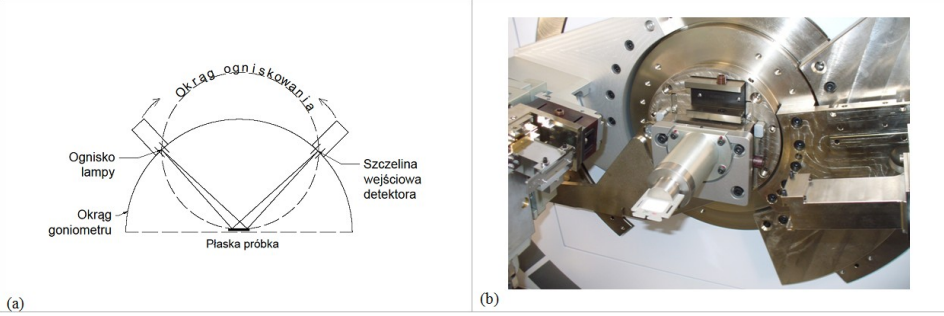


Figure 1: Empyrean diffractometer goniometer in Bragg-Brentano configuration.

focal Bragg-Brentano geometry (Fig. Ref Goniometer). In the basic version, a polycrystalline NaCl sample and a two or three-phase sample provided by the tutor are tested.

## Experiment

The laboratory block usually consists of Z4 and Z22 experiments. We usually start with the task no I, but it is best to contact the tutor (eg by email) to set the details.

**Task I. Studying a single-phase polycrystalline sample by the powder diffractometry method in Bragg-Brentano geometry**

1. Sample preparation
   - apply a small amount of the test substance in a mortar to make a fine powder (to the consistency of the powder); in the basic version, the test substance is usually NaCl,
   - put the prepared powder in the measuring cuvette by forming a flat, well-pressed and possibly smooth surface.
2. In the presence of the tutor, place the cuvette with the powder in the goniometer holder of the diffractometer.
3. Following the instructions of the tutor and the detailed instructions of the measuring device perform the registration of the sample diffraction pattern.

**Task II. Sydying a multiphase polycrystalline sample by the powder diffractometry method in Bragg-Brentano geometry**

In the way described in Task no I, prepare a sample take the measurement for the selected multi-phase substance.

While working, keep detailed notes in the laboratory notebook so that you can reproduce all the steps and values of all the parameters of the experiments carried out.

Remember to make the appropriate entry in the apparatus work log.

## Data analysis

1. Use any method to determine the location of diffraction lines with an accuracy of up to $0.1^o$.
2. Indicate diffraction maxima (the diffractometer is equipped with a lamp with a Cu anode).
3. Using the method of fitting the line profile determine the exact values of the diffraction maxima for $2\Theta > 60°$ and their uncertainties - attach the relevant graphs to the report. Note: It is best to use the Winplotr program, which should then be shown in the report in the Bibliography section.
4. Using the extrapolative method, determine the exact value of the lattice constant of the tested sample and estimate the uncertainty.
5. For a multiphase sample, perform a qualitative composition analysis using available databases and/or previous measurements of standard substances.

## Safety rules

Detailed health and safety regulations can be found on the board in the X-ray diffractometry laboratory.

## References

[1] D. Halliday, R. Resnick, J. Walker, *Podstawy fizyki*, PWN, Warszawa, 1996, tom 5, rozdziały 41.9-41.10.

[2] C. Kittel, *Wstęp do fizyki ciała stałego*, PWN, 1970, rozdziały 1 i 2, str. 15-91.

[3] Z. Bojarski, E. Łągiewka, *Materiały do ćwiczeń z rentgenowskiej analizy strukturalnej*, (do wypożyczenia w IIPF, z szfki przy zestawie eksperymentalnym): Zeszyt 1 - całość (szczególnie rozdział 4 o

BHP); Zeszyt 2, str. 7-36 i 45-49; Zeszyt 3, str. 6-23 i 61-95; Zeszyt 4, str. 5-28; Zeszyt 5, str. 50-72.

[4] A. Zięba, *Analiza danych w naukach ścisłych i technice*, PWN, Warszawa, 2013.

and attached materials available after logging in to the laboratory website or any other textbooks of crystallography and solid state physics as well as information and materials available on the Internet.