

Moderne Methoden der Strukturbestimmung

Modern Methods of Structure Determination

Dr. Günther Thiele

What you will learn..

- Overview of available methods at FU Berlin
- Detailed knowledge on the most important methods

- Measurement – Data Analysis – Refinement of Single Crystal Structures
- Measurement – Data Analysis – Evaluation of Powder Patterns

- Discussion, Presentation and Publication of Structure Results

Lectures

Hands-On
Sessions

Exam

When and what and where?

Lectures

Tuesdays, 2:15 pm – 4pm, Wednesdays, 2:15 pm – 4 pm
Either HS B or A (check which one on homepage)

Hands-On Sessions

29.10 – 22.11 Groups of three, each group one evening (5:15 pm)
Online signup (link: homepage AG Thiele)
@basement Fabeckstr., meet in front of the elevator

13.11 – 04.12 Instead of the Lecture: Refinement (Bring Laptop!)

Exam

17.02 – 21.02 Online signup
Groups of three, 45 minutes per group
5 Min Presentation of the Refinement
10 Min Discussion of suitable methods

Time Schedule

October

| | M | T | W | T | F | S | S |
|----|----|----|----|----|----|----|----|
| 40 | 30 | 1 | 2 | 3 | 4 | 5 | 6 |
| 41 | 7 | 8 | 9 | 10 | 11 | 12 | 13 |
| 42 | 14 | 15 | 16 | 17 | 18 | 19 | 20 |
| 43 | 21 | 22 | 23 | 24 | 25 | 26 | 27 |
| 44 | 28 | 29 | 30 | 31 | 1 | 2 | 3 |
| 45 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |

November

| | M | T | W | T | F | S | S |
|----|----|----|----|----|----|----|----|
| 44 | 28 | 29 | 30 | 31 | 1 | 2 | 3 |
| 45 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| 46 | 11 | 12 | 13 | 14 | 15 | 16 | 17 |
| 47 | 18 | 19 | 20 | 21 | 22 | 23 | 24 |
| 48 | 25 | 26 | 27 | 28 | 29 | 30 | 1 |
| 49 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |

December

| | M | T | W | T | F | S | S |
|----|----|----|----|----|----|----|----|
| 48 | 25 | 26 | 27 | 28 | 29 | 30 | 1 |
| 49 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| 50 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| 51 | 16 | 17 | 18 | 19 | 20 | 21 | 22 |
| 52 | 23 | 24 | 25 | 26 | 27 | 28 | 29 |
| 1 | 30 | 31 | 1 | 2 | 3 | 4 | 5 |

January

| | M | T | W | T | F | S | S |
|---|----|----|----|----|----|----|----|
| 1 | 30 | 31 | 1 | 2 | 3 | 4 | 5 |
| 2 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
| 3 | 13 | 14 | 15 | 16 | 17 | 18 | 19 |
| 4 | 20 | 21 | 22 | 23 | 24 | 25 | 26 |
| 5 | 27 | 28 | 29 | 30 | 31 | 1 | 2 |
| 6 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

February

| | M | T | W | T | F | S | S |
|----|----|----|----|----|----|----|----|
| 5 | 27 | 28 | 29 | 30 | 31 | 1 | 2 |
| 6 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| 7 | 10 | 11 | 12 | 13 | 14 | 15 | 16 |
| 8 | 17 | 18 | 19 | 20 | 21 | 22 | 23 |
| 9 | 24 | 25 | 26 | 27 | 28 | 29 | 1 |
| 10 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |

March

| | M | T | W | T | F | S | S |
|----|----|----|----|----|----|----|----|
| 9 | 24 | 25 | 26 | 27 | 28 | 29 | 1 |
| 10 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| 11 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| 12 | 16 | 17 | 18 | 19 | 20 | 21 | 22 |
| 13 | 23 | 24 | 25 | 26 | 27 | 28 | 29 |
| 14 | 30 | 31 | 1 | 2 | 3 | 4 | 5 |



Lecture



Diffraction Time



Exam



Refinement – Bring your Laptop

Lectures: Tuesdays, 1pm – 5pm

| | | | | | |
|------------|--------------------|--------------|------------|--------------------|------------|
| 15.10.2019 | Introduction | | 16.10.2019 | Introduction | |
| 22.10.2019 | Diffraction Theory | | 23.10.2019 | Diffraction Theory | |
| 29.10.2019 | Diffraction Theory | | 30.10.2019 | Diffraction Theory | |
| 05.11.2019 | Diffraction Theory | | 12.11.2019 | Diffraction Theory | |
| 12.11.2019 | Diffraction Theory | | 13.11.2019 | Diffraction Theory | |
| 19.11.2019 | Hands On | | 20.11.2019 | Hands On | |
| 26.11.2019 | Hands On | | 27.11.2019 | Hands On | |
| 03.12.2019 | Hands On | | 04.12.2019 | Hands On | |
| 10.12.2019 | Powder | | 11.12.2019 | Powder Hands On | |
| 17.12.2019 | SS NMR | Steinhauer | 18.12.2019 | SS NMR | Steinhauer |
| 07.01.2020 | SS NMR | Steinhauer | 08.01.2020 | SS NMR | Steinhauer |
| 14.01.2020 | Vibrations | Beckers | 15.01.2020 | Vibrations | Tsegaw |
| 21.01.2020 | Publication | | 22.01.2020 | EPR | Risse |
| 28.01.2020 | SQUID | Malischewski | 29.01.2020 | Mass Spectrometry | Springer |
| 04.02.2020 | Mass Spectrometry | Springer | 05.02.2020 | Practices | |
| 11.02.2020 | Practices | | 12.02.2020 | Q&A | |

Installation Session

max. 30 Minutes

Laptops!

Single Crystal Structure Determination

- 1) Synthesis of Compounds
- 2) Crystallization
- 3) Sample Preparation
- 4) Sample Measurement
- 5) Data Integration
- 6) Structure Refinement
- 7) Structure Analysis, Presentation
- 8) Structure Publication

Do it Yourself

@basement

First evening 5:45 pm

Second evening 5:15 pm

How to crystallize?

- Solvent evaporation
- Solvent diffusion
- Temperature gradient
- Concentration gradient

Ideal Crystal Size for the
IPDS 2T: 1 mm each side

**The slower the
Crystallization
Process, the better the
Crystal Quality!**

Information & Slides & Online SignUp

Homepage FU Berlin > BCP > Chemie > AG Thiele > Lehre > Strukturkurs

Your To-Do-List

- 1) Crystallize something
- 2) Set up your laptop
- 3) Find your group (three people)
- 4) SignUp Online for Diffraction time
- 5) SignUp @ BlackboardFU
or on paper (“alte Prüfungsordnung“)

For the diffraction time:

At the sign-up date: be there at 5:45 pm with your crystals

The next day: be there at 5:15 pm with your crystals (PXRD)

The screenshot shows the website for the 'Moderne Methoden der Strukturaufklärung WS19/20' course. The header includes the logo of the Free University of Berlin and navigation links like 'Startseite', 'Mitarbeiter/innen', 'Standorte', 'Sitemap', 'Impressum', and 'Datenschutz'. A search bar is also present. The main navigation bar lists 'DR. GÜNTHER THIELE', 'NEWS', 'TEAM', 'FORSCHUNG', 'LEHRE', 'PUBLIKATIONEN', 'FORSCHUNGSPRAKTIKA', and 'SEMINARE'. The breadcrumb trail reads: 'Startseite > Chemie/Biochemie > Chemie > Forschung > Anorganische Chemie > Nachwuchsgruppe Thiele > Lehre > Moderne Methoden der Strukturaufklärung WS19/20'. The main content area is titled 'Moderne Methoden der Strukturaufklärung WS19/20' and 'Moderne Methoden der Strukturbestimmung // Modern Methods of Structure Determination'. It lists 'Lectures' on Tuesdays and Wednesdays, and 'Practical Sessions' involving X-ray and SS-NMR measurements. A 'NEWS' sidebar on the right contains three items: 'Neue Bachelorarbeit zu Oxomerkuraten gestartet' (12.10.2019), 'Neues Forschungspraktikum zum Elektrospeining gestartet' (16.09.2019), and 'Neue Promotionsstelle zu Sulfidoferraten verfügbar' (15.08.2019). A pagination indicator shows '1 / 4'.

Questions ?

This Modul will be evaluated (January?). Please participate to give your input on how to improve it.

Single Crystal Diffraction

1. Hard Core Theory

1. Crystal Lattice
2. X-Ray Diffraction
3. Reciprocal Space
4. Structure factors
5. Symmetry

2. Practical Aspects

1. Experimental Methods
2. Structure Solution
3. Structure Refinement
4. Special Effects
5. Databases