Chemistry 5210 Materials Characterization Spring 2024

1:25–3:20 pm Mondays/Wednesdays
Nicholson 315

Instructor: Andreas Stein; 219 Smith; 612-624-1802, a-stein@umn.edu, http://stein.chem.umn.edu
Office hours: Mon 3:30–4:30 pm, Tue 12:15–1:15 pm; or by appointment.

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Office hours in 455 Kolthoff: Wed 3:30–4:30 pm, Fri 11:00 am–noon

The Course: This course is scheduled as an in-person course. I intend to hold all class sessions in-person except if situational factors arise, such as personal illness of the instructor, when the class may be held synchronously via Zoom or recorded for later viewing. This course surveys a range of techniques suitable for characterization of solid-state materials, with an emphasis on techniques available at the University of Minnesota Characterization Facility, the Nanofabrication Center, and in the Department of Chemistry. The techniques discussed will include diffraction methods (powder X-ray diffraction, small-angle X-ray scattering, grazing-incidence small-angle X-ray scattering, neutron diffraction, electron diffraction), imaging techniques (scanning electron microscopy, transmission electron microscopy, atomic force microscopy and other surface probe microscopies), selected spectroscopic techniques relevant to materials (energy dispersive spectroscopy, X-ray photoelectron spectroscopy), thermal analysis (thermogravimetric analysis and differential scanning calorimetry), and pore analysis (gas sorption techniques). Throughout the course, we will address the following questions: Which technique is appropriate for what kind of material and for which property of the material? What information can you get from the technique? How does the technique work? What is the quality of the data (resolution, sensitivity, matrix effects, amounts of sample, fraction of material sampled, cost, availability) and how can it be optimized? What are the limitations of the techniques? Pitfalls? What are the sample requirements? This information will be supplemented by examples from current literature. We will also visit the Characterization Facility to see available equipment.

Readings: There is no required textbook. We will rely heavily on several excellent websites (listed below) and on current primary and secondary literature. Students should read assigned materials in advance, so that they can be discussed during the lectures.

https://myscope.training/ (also includes some simulators)
https://jascoinc.com/learning-center/theory/spectroscopy/
https://www.eag.com/techniques/
https://www.jawoollam.com/resources/ellipsometry-tutorial
https://www.doitpoms.ac.uk/tlplib/crystallography3/index.php
https://www.doitpoms.ac.uk/tlplib/diffraction/index.php
https://www.doitpoms.ac.uk/tlplib/miller_indices/index.php
https://www.doitpoms.ac.uk/tlplib/xray-diffraction/index.php
https://www.doitpoms.ac.uk/tlplib/tem/index.php
https://www.doitpoms.ac.uk/tlplib/afm/index.php
https://www.doitpoms.ac.uk/tlplib/raman/index.php
https://app-knovel-com.ezp2.lib.umn.edu/kn/resources/kpEMCSITF4/toc?kpromoter=marc

Course Web Page: The course Canvas site will be used for posting the syllabus, lecture notes, grades, and other course-related material. Please ignore any letter grades listed on the Canvas site. These are automatically generated by Canvas but will not be used during the course.

Clickers: Clickers will be used for in-class responses. The required device is the iClicker2, and I will lend these to you.

Course software: During the diffraction component of the course, we will use CrystalMaker software and the associated programs CrystalDiffract and SingleCrystal to visualize structures and their diffraction patterns. This software will also be needed for some of the homework assignments. The Chemistry Department has a software license for these programs for students in Chem 5210 (both Mac and Windows). You will need to download the program files and manuals using the following link:
https://drive.google.com/drive/folders/1zjRP7iBk7QAkbGfhPUiCaBJQnRZZXfC1?usp=sharing
You have to have to sign in with your U of M Gmail account when you access this folder on Google Drive.
The Rietveld refinement program FullProf with examples can be downloaded from the following website: https://www.ill.eu/sites/fullprof/index.html

Grading:

Quizzes and class participation:	10 %
Assignments:	20 %
Group presentation:	20 %
Midterm exam 1* (Monday Feb. 19, 1:25–3:20 p.m.):	16 %
Midterm exam 2* (Monday, April 1, 1:25–3:20 p.m.):	16 %
Final exam (Saturday, May 4, 1:30–3:30 pm):	18 %

*These exam dates are tentative and may be changed, if necessary, but they will be during class time.

During the course, all grades will be scored on a percentage basis. *Final letter grades* will be assigned based on the overall cumulative score. The following cut-offs will be used for letter grades:

A: >87%
A-: >85%
B+: >82%
B: >72%
B-: >67%
C+: >62%
C: >55%
C-: >50%

Note that at the end, I may opt to adjust these cutoffs such that it will be possible to get a particular letter grade with a score that is lower than the range indicated above. But in no case will this adjustment hurt your grade. That is to say, *any adjustments, if applied, will only be used to improve your grade, not lower it*.

If you are registered for this course on an S/N basis, a grade equivalent to C- or better on the A–F scale will be required to receive an "S". A D+ or below will receive an "N". Many programs or transfer courses do not like S/N grades or will assume that they are the minimum possible grade. Requests to change grading basis after the University deadline will not be approved. An *I* grade will be assigned only to students who have received passing grades on all work up to the final exam. If this occurs, you are responsible for contacting me before the end of finals week. Otherwise, a grade of F will be entered on the grade report.

If you need to use the Disability Resource Center (DRC, https://disability.umn.edu/), please contact DRC AS SOON AS POSSIBLE, to ensure your accommodations are in place at the beginning of the semester. You can do this by emailing drc@umn.edu or calling and leaving a message at 612-626-1333. If you already have an assigned access consultant and need modifications, please contact that person via email to address new access issues. Also let me know about any needs for accomodations at the beginning of the course.

University-wide **policies** on the following topics

- Student Conduct Code
- Use of Personal Electronic Devices in the Classroom
- Scholastic Dishonesty
- Makeup Work for Legitimate Absences
- Appropriate Student Use of Class Notes and Course Materials
- Grading and Transcripts
- Sexual Harassment
- Equity, Diversity, Equal Opportunity, and Affirmative Action
- Disability Accommodations
- Mental Health and Stress Management
- Academic Freedom and Responsibility

can be found at this webpage: https://policy.umn.edu/education/syllabusrequirements-appa

Important: In this course, you will have access to electronic material, including lecture notes, assignments, examinations, solution sets, video recordings of lectures etc. You are NOT ALLOWED to share this material with anyone who is not currently enrolled in this class and you are NOT ALLOWED to upload any of this material to any website, including but not limited to online "study sites" at any time (even after this course is finished). Violation of this rule constitutes a violation of the Student Conduct Code.

You are expected to do your own academic work and cite sources as necessary. Failing to do so is scholastic dishonesty. Scholastic dishonesty means plagiarizing; cheating on assignments or examinations; engaging in unauthorized collaboration on academic work, including the posting of student-generated coursework on online learning support and testing platforms not approved for the specific course in question; taking, acquiring, or using course materials without faculty permission, including the posting of faculty-provided course materials on online learning and testing platforms; ..."; submitting false or incomplete records of academic achievement; acting alone or in cooperation with another to falsify records or to obtain dishonestly grades, honors, awards, or professional endorsement; altering, forging, misrepresenting or misusing a University academic record; or fabricating or falsifying data, research procedures, or data analysis. If it is determined that a student has cheated, the student may be given an "F" or an "N" for the course, and may face additional sanctions from the University.

Artificial intelligence (AI) language models, such as ChatGPT, and online assignment help tools, such as Chegg®, are examples of online learning support platforms: they cannot be used for course assignments except as explicitly authorized by the instructor. The following actions are prohibited in this course:

- Submitting all or any part of an assignment statement to an online learning support platform;
- Incorporating any part of an AI generated response in an assignment;
- Using AI to brainstorm, formulate arguments, or template ideas for assignments;
- Using AI to summarize or contextualize source materials;
- Submitting your own work for this class to an online learning support platform for iteration or improvement.
- If you are in doubt as to whether you are using an online learning support platform appropriately in this course, I encourage you to discuss your situation with me.

Any assignment content composed by any resource other than you, regardless of whether that resource is human or digital, must be attributed to the source through proper citation. Unattributed use of online learning support platforms and unauthorized sharing of instructional property are forms of scholastic dishonesty and will be treated as such.

Course outline:

Course introduction
Review of crystal structure (crystal systems, Bravais lattices, close packing)
Review of Miller indices & d-spacings

Powder X-ray diffraction
Applications of powder XRD
Operation of X-ray diffractometer
X-ray diffraction patterns
Bragg's law
Features of XRD patterns
Powder diffraction files
Analyzing powder patterns
Indexing
Systematic absences
XRD patterns of mixtures
Vegard's law
Superstructures
Peak intensities
Preferred orientation
Peak widths
Scherrer equation
XRD patterns of noncrystalline materials
Rietveld refinement

Small-angle X-ray scattering (SAXS)
General applications
The experiment
Data analysis
Contrast matching
Form factor
Radius of gyration
Shape analysis
Guinier, Fourier, Porod regions
Size analysis
Specific examples

Other diffraction methods
GISAXS
Neutron Diffraction & Magnetic Structure Factor
Electron diffraction

Microscopy concepts
Optical microscopy

Scanning electron microscopy (SEM)
Structure of SEM
Detectors
Sample preparation
Controlling the various parameters
Artifacts
Specialized techniques

Energy-dispersive spectroscopy (EDS)
Overview
Generation of X-rays
Continuum X-rays
Characteristic X-rays
Energies & intensities
X-ray detection
EDS spectra
Examples of qualitative analysis
Quantitative analysis
ZAF corrections
Elemental mapping

X-ray photoelectron spectroscopy (XPS)
Overview
Operation
The instrument
Spectra
Quantification
Final state effects
Mapping
Complications
Case studies

Transmission electron microscopy (TEM)
Overview
Instrument design
Types of images and their formation
Generation of contrast
Diffraction in the TEM (selected-area electron diffraction)
Instrument alignment
STEM & HAADF
Specimen preparation
Dealing with problems
Aberration-corrected TEM
Analysis in the TEM (EDS, EELS)
Cryo-TEM
Electron crystallography

Thermal analysis
Thermogravimetric analysis (TGA)
Derivative thermogravimetry (DTG)
Differential thermal analysis (DTA)
Differential scanning calorimetry (DSC)
Applications of thermal analysis

Gas physisorption analysis
Porous materials
Sorption
The analyzer
Sample preparation
Types of isotherm & hysteresis
Langmuir & BET adsorption models
Surface areas
Pore volumes
Pore radii
Pore size distributions
Examples

Atomic force microscopy (AFM)
Overview
The instrument
Probe–sample interactions
Imaging modes
Contact mode
Tapping mode
Non-contact mode AFM
Image artifacts
Applications