**Chem/MatS/ChEn 4223W**
*Polymer Laboratory*
Spring 2013
M 3:35 pm – 4:25 pm, Smith 231
*and*
T 8:00 am – 11:50 am, Smith 249A *or*
Th 8:00 am – 11:50 am, Smith 249A


**Instructor:** Professor T. Andrew Taton
425 Smith Hall, 6-4681
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*Office hours by appointment.*

**Lab TAs:** Chandru Ramasubramanian
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**Prerequisites:** Chem/MatS/ChEn 4221/5221 (Synthetic Polymer Chemistry) ***or*** MatS/ChEn 4214 (Polymers)

**Recommended:** Chem 2311 (Organic Lab)

**Required Materials:** Bound laboratory notebook (available at Univ. bookstore)
Safety goggles (bring your own, or buy at Smith 249 General Chemistry stockroom)

**Recommended Text:** Anne M. Coghill, Lorrin R. Garson, eds., *The ACS Style Guide* (3rd ed.; Oxford University Press, New York, 2006).

**Class Website:** http://www.chem.umn.edu/groups/taton/chem4223/. In general, lab instructions and assignments will be posted on the website in PDF format rather than handed out, so you will need to be able to view and print PDF files for this course. (Adobe Acrobat Reader can be used for this, available at http://www.adobe.com/products/reader.html.) In addition, you will need to use the Moodle discussion forum (http://moodle2.umn.edu/) for this class to exchange lab data when that is called for.

**Schedule for 2012:**

| Labs: | Weeks | New concepts learned |
|---|---|---|
| Lab 1: Free-Radical Bulk Polymerization | 1-3 | *What makes polymers special?*<br>*General principles of polymer synthesis, solubility, purification*<br>*Polymerization kinetics* |
| Lab 2: Statistical Copolymers via Free-Radical Copolymerization | 3-4 | *NMR analysis of polymers*<br>*Copolymer reactivity ratios*<br>*Qualitative physical properties of polymers and copolymers* |
| Lab 3: Microemulsion Polymerization | 4-5 | *Emulsion polymerization and chain transfer*<br>*GPC analysis of $M_n$ and $M_w$*<br>*Particle sizing by DLS* |
| Lab 4: Anionic, Living Polymerization and Block Copolymers | 6-8 | *Anionic polymerization*<br>*Endgroup analysis, $M_n$ by NMR*<br>*Molding*<br>*Plastics and elastomers—mechanical testing, stress-strain behavior*<br>*Block copolymer structure-function relationships* |
| Lab 5: Controlled Radical Polymerization | 9-12 | *Controlled/living radical polymerization* |
| Lab 6: Polylactide: Synthesis, Tacticity and Properties | 10-12 | *Polymer tacticity by NMR*<br>*Stereodefects: Polarimetry, polarized optical microscopy*<br>*Polymer crystallization, kinetics*<br>*Thermal characterization by DSC* |
| Lab 7: Crosslinked Thermosets via Ring-Opening Metathesis Polymerization | 13-14 | *Thermoplastics into thermosets; polymer crosslinking*<br>*Manipulation of phase transitions in polymers*<br>*Memory materials* |

Underlined labs require full lab reports.

**Reference Texts:**

*(All available through Walter Library Reserve unless noted.)*

Joel Fried, *Polymer Science and Technology* (2nd ed.; Prentice Hall, Englewood Cliffs, N.J., 2003). Introductory text on polymer science and applications.

Paul Hiemenz and Timothy Lodge, *Polymer Chemistry* (2nd ed.; CRC Press, 2007) A more detailed text on both polymer science and polymer physics.

George Odian, *Principles of Polymerization* (4th ed.; John Wiley & Sons, New York, 2004). Very detailed text on polymerization chemistry and kinetics.

Stanley Sander *et al.*, *Polymer Synthesis and Characterization: A Laboratory Manual* (Academic Press, San Diego, 1998). Good introductions to some polymer techniques. None of our labs come from this manual.

*Wiley Database of Polymer Properties* (Wiley, New York, 1999-present). Compendium of physical properties of polymer materials. Chock full of data. *Available online at http://tinyurl.com/wileypolyprops.*

James Mark, *Polymer Data Handbook* (2nd ed.; Oxford University Press, New York, 2009). Less data than Brandrup above, but indexed by polymer rather than by data type.
*Available online at http://tinyurl.com/polydatahandbk.*

Herman Mark, *Encyclopedia of Polymer Science and Technology* (Wiley-Interscience, Hoboken, NJ, 2003). Contains chapters on general topics in polymer science. *Available online at http://tinyurl.com/encyclopolyscitech.*

Quang Tho Pham *et al.*, *Proton and Carbon NMR Spectra of Polymers* (5th ed.; Wiley, New York, 2003). Contains NMR spectra of many reference polymers; helpful for assigning your own spectra. *Available in Walter Reference, 2nd floor (Quarto QC463.P5 P76 2003).*

Henry Hsieh and Roderic Quirk, *Anionic Polymerization: Principles and Practical Applications* (Marcel Dekker, New York, 1996). Great detailed resource on methods in anionic chain polymerization, and particularly on block copolymer synthesis. *Available online at http://tinyurl.com/anionpoly.*

Nikos Hadjichristidis, Stergios Pispas and George Floudas, *Block Copolymers: Synthetic Strategies, Physical Properties, and Applications* (Wiley-Interscience, Hoboken, NJ, 2003). Graduate-level text on block copolymer synthesis and properties.

Geoffrey Holden, *Understanding Thermoplastic Elastomers* (Hanser Gardner, Cincinnati, 2000). Addresses physical properties of block copolymer rubbers. Great, simple introduction.

Ian W. Hamley, *Block Copolymers in Solution* (Wiley, Chichester, UK, 2005). Explains behavior of block-copolymer surfactants and micelles.

Ken J. Ivin and Hans (J. C.) Mol, *Olefin Metathesis and Metathesis Polymerization* (Academic Press, San Diego, 1997). Very clear reference on ring-opening metathesis polymerization (ROMP).

William D. Callister, Jr., *Materials Science and Engineering: An Introduction* ($8^{th}$ ed.; John Wiley & Sons, New York, 2010). Good reference on the mechanical properties of materials.

Leslie H. Sperling, *Introduction to Physical Polymer Science* ($4^{th}$ ed.; Wiley-Interscience, New York, 2005). Complete text on physical polymer properties.

Jack Avery, *Injection Molding Alternatives* (Hanser/Gardner, Cincinnati, 1998). Despite its specific title, an excellent general reference on polymer processing techniques. In particular, compares and contrasts different processing approaches for making different plastic products.

Jerold M. Schultz, *Polymer Crystallization: The Development of Crystalline Order in Thermoplastic Polymers* (Oxford University Press, New York, 2001). Theoretical and experimental details of polymer crystallization.

Anne M. Coghill, Lorrin R. Garson, eds., *The ACS Style Guide* ($3^{rd}$ ed.; Oxford University Press, New York, 2006). Important reference for formatting theses and papers in ACS journals. *Available in Walter Reference, $2^{nd}$ floor (QD8.5 .A25 2006).*

*Other, online reference materials are described on the "Links" page of the course website.*

**Grading:**

| | |
|---|---|
| 17% | In-lab performance |
| 33% | Assignments |
| 50% | Lab reports |

**Attendance:** It is in your best interest to attend the lectures; you will learn things that will be necessary to complete pre-lab assignments and to conduct and write up the labs properly.

Lab attendance is mandatory. You can only be excused from participating in a lab if you notify the instructor in advance. You can be excused from participating in a lab only due to jury duty, subpoenas, military service, religious holidays, and participation in school sports events, and only if the instructor is notified two weeks in advance. In case of illness (as verified by a doctor's note) or death in the immediate family (be prepared to verify), notify the instructor within 24 hours of missing the lab. Unexcused absence from lab will cause you to lose all of that day's credit for lab performance, assignments and that day's portion of the lab writeup. So please don't miss lab. If you miss a lab due to an excused absence, we will make every attempt to help you do your lab at another time and/or date, as long as you give us notice (as described above).

**Lab Performance:** You will be evaluated on your level of preparation, lab etiquette, and notebook keeping. Every day you are in lab, you will earn up to 10 points based on your performance. You are expected to show up to the lab on time (at 8 am). If you arrive late, you will receive a maximum of 5 points for that lab period.

**Assignments:** You will have a quite a few short assignments due throughout the semester. Some of these will be pre-lab questions intended to prepare you for that day's lab; these will be due at the beginning of your lab section. Others will be data work-ups, intended to make sure your data analysis makes sense before you include it in a major lab report; these might be posted on the WebCT discussion board for sharing with classmates, or e-mailed to me, depending on the instructions. Late assignments will not be graded.

**Lab reports:** Much of your grade in this class will be determined by three written lab reports. Even though you will do your lab work in pairs, you will be expected to turn in your own independent reports. (Rules on plagiarism will apply to these; see Academic Integrity below.) While you are permitted, even encouraged, to prepare your assignments together, the work you turn in should be your own. We encourage you to consult with writing tutors at the Center for Writing (http://writing.umn.edu/sws/) to improve your written work before turning it in.

Your lab reports will take the format of research papers in a scientific journal such as *Macromolecules*. At the beginning of the course, you will be revising your reports, with our help, to develop your scientific writing style. By the end of the course, you will be writing these reports on your own. You will need access to a word processing program (e.g., Microsoft Word), a graphing program (e.g., Microsoft Excel) and a chemical structure drawing program (ChemDraw, available to U of M students for free download

at http://tinyurl.com/umnchemdraw). You will also be using a number of other software packages in the Microcomputer Lab, that will be described in the individual lab instructions..

Late lab reports will be accepted, but will receive a penalty of 10 points for each 24-hour period they are late, starting immediately. (So, 10 minutes late is still 10 points off.) In cases where you can revise reports to regain lost points, late penalty points *cannot* be recovered.

**Lab Groups:** You will work with a labmate that you choose, though we reserve the right to switch you. Make sure that you know how to contact your labmate (phone, email, etc.).

**Notebook:** You are expected to maintain a neat organized notebook with your data, observations and comments. You will need to keep your own data; please do not rely on your partner to take down the data for an experiment. Your notebooks will be checked periodically as part of your in-lab performance evaluation.

**Safety:** You will be given a hand-out on laboratory safety in lab; please read it. As in all laboratory settings, practice the "safety first" method. You will be required to wear full-coverage safety goggles in the lab at all times. (It's not just a good idea; it's Minnesota State law.)

**E-mail:** All students and instructors should be reachable at their University-wide e-mail accounts. If you normally use a departmental or private e-mail account, you should set up your University-wide account to automatically forward messages to this other address. You can do this most easily through the account settings page, http://www.umn.edu/validate.

**Reference Texts:** Although the instructor will lecture on various topics related to the experiments you do in this lab, it will often be your responsibility to find appropriate reference materials. This means polymer textbooks, handbooks, encyclopedias, and original literature (i.e. journal articles). *SciFinder Online* (http://scifinder.cas.org/), the search tool for the American Chemical Society's Chemical Abstracts Service, is an excellent tool for searching for journal articles on a particular topic. To use SciFinder Online, you will need to register (http://tinyurl.com/scifinderumn) with your umn.edu e-mail address. You can also usually access journal articles directly from a SciFinder search by clicking "Get Full Text". To read the articles online, you will either need to be using a computer with a UMN IP address, access them via the U of M Libraries website (http://www.lib.umn.edu/#journals to search), or use a virtual private network (VPN) tunnel. (Go to http://www.oit.umn.edu/vpn/ to download the software needed to do this, and to learn more. It's easy, I promise.)

**FERPA Compliance:** In this class, our use of technology will make students' names visible within the course WebCT website, but only to other students in this class. Since we are using a secure, password-protected course website, this will not increase the

risk of identity theft or spamming for anyone in the class. If you have concerns about the visibility of your name, please contact Andy for further information.

**Academic Integrity** is essential to a positive teaching and learning environment. All students enrolled in University courses are expected to complete coursework responsibilities with fairness and honesty. Failure to do so by seeking unfair advantage over others or misrepresenting someone else's work as your own can result in disciplinary action. More information on the definition and consequences of academic dishonesty can be found at the Office for Student Academic Integrity website (http://www.osai.umn.edu/). In this course, direct copying of assignments or fabricating lab data will be considered dishonest. Any student responsible for scholastic dishonesty can be assigned a penalty up to and including an "F" or "N" for the course. If you have any questions regarding the expectations for a specific assignment, please ask.

Our goal is to be as available and as accommodating as we can be. If you feel you are having troubles or concerns about the class, please feel free to contact Andy or the TA's; we want you to have a positive learning experience in this course. If you feel for any reason that we are not taking your concerns seriously, and that this is negatively impacting your learning, the Student Dispute Resolution Center (http://www.sos.umn.edu/) makes both informal (ombudsman) and formal (advocate) representatives within the University available for students to share their concerns with.