



## Separation Methods

The University of Toledo  
Department of Chemistry and Biochemistry  
CHEM 4310/6310/8310

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<b>Instructor:</b>	Emanuela Gionfriddo	<b>Term:</b>	Spring 2021
<b>Email:</b>	emanuela.gionfriddo@utoledo.edu	<b>Class Location:</b>	BO 2059
<b>Office Hours:</b>	online by appointment	<b>Class Day/Time:</b>	M,W,F 1-1:55 p.m.
<b>Office Location:</b>	-	<b>Credit Hours:</b>	3
<b>Office Phone:</b>	419-530-1508	<b>Course Website:</b>	Blackboard (blackboard.utdl.edu)

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### SPECIAL UNIVERSITY COURSE EXPECTATIONS DURING COVID-19

#### ATTENDANCE

The University of Toledo has a missed class policy. Students must perform a daily health assessment, based on [CDC guidelines](#), before coming to campus each day, which includes taking their temperature. Students who are symptomatic/sick should not come to class and should contact the Main Campus Health Center at 419-530-3451. Absences due to COVID-19 quarantine or isolation requirements are considered excused absences. Students should notify the instructor as soon as possible. These absences may not require written notice.

#### FACE COVERINGS

All students must wear face coverings while on campus, except while eating, alone in an enclosed space, or outdoors practicing social distancing. **No students will be permitted in class without a face covering.** If you have a medical reason that prevents you from wearing a face-covering due to a health condition deemed high-risk for COVID-19 by the Centers for Disease Control and Prevention (CDC), you should submit a request for an accommodation through the Student Disability Services Office (SDS) by completing the [online application](#). Students will need to provide documentation that verifies their health condition or disability and supports the need for accommodations. If a student is already affiliated with SDS and would like to request additional accommodations due to the impact of COVID-19, they should contact their accessibility specialist to discuss their specific needs.

#### SOCIAL DISTANCING

Students should practice social distancing inside and outside the classroom. Please follow signage and pay attention to the seating arrangements. Do not remove stickers or tape from seats and/or tables; these items are there to provide guidance on the appropriate classroom capacity based on the recommended six (6) feet of social distancing between individuals. Please be conscious of your personal space and respectful of others. Also be cognizant of how you enter and exit the room; always try to maintain at least six (6) feet of distance between yourself and others.

#### DESKS AND WORK SPACES

Students will need to sanitize their desks and/or work space before class with the University provided sanitizing spray and paper towels.



## **SPECIAL NOTES**

It is important to note that based on the unpredictability of the COVID-19 virus things can change at any time so please be patient and understanding as we move through the semester. Please keep the instructor informed of concerns you may have about class, completing course work/assignments timely and/or health concerns related to COVID.

## **COURSE SPECIFIC INFORMATION**

### **COURSE/CATALOG DESCRIPTION**

*The course offers a comprehensive survey of separation methods used for chromatography, extraction and various approaches to chemical analysis. Among the topic included in the course outline, major emphasis is given to the three main separation platforms used for instrumental analysis, namely, gas- and liquid-chromatography and capillary electrophoresis. The fundamentals of extraction together with classical and modern extraction techniques are also discussed.*

### **COURSE OVERVIEW**

The purpose of this course is to present the fundamentals and practical aspects of the most commonly used chemical separations, to describe instrumentation used and provide examples of separation methods used for real case scenarios of chemical analysis. The course is divided into various sections describing separations involving chromatography, ion exchange resins, electric fields, extraction and phase changes.

### **STUDENT LEARNING OUTCOMES**

*The "Separation Methods" course is devoted to the students' deep understanding of both fundamental and practical aspects of the separation processes that govern chromatography, extraction and other aspects of chemical analysis. After completion of the course the student will be able to:*

- *Classify and describe principles of operation of the separation techniques discussed during the course.*
- *Critically select and/or couple separation strategies that fit the purpose of different analytical needs.*
- *Evaluate the separation quality of a chromatogram and learn strategies and factors to improve the chromatographic separation.*

### **TEACHING STRATEGIES**

This course consists of face-to-face lectures. Additional literature and links to online learning tools and videos will be provided to support the course material. Discussion during the in-class lectures is highly encouraged. Lecture notes and additional learning material will be available on Blackboard.

### **PREREQUISITES AND COREQUISITES**

*For undergraduate students: Instrumental analysis CHEM 4300-001, 091 (CRN: 42011, 42021)*

### **REQUIRED TEXTS AND ANCILLARY MATERIALS**

**"Chromatography: Principles and Instrumentation"**, Mark F. Vitha, 2016, Wiley, ISBN: 978-1-119-27088-1



Additional material will be provided by the instructor during in-class lectures.

### **TECHNOLOGY REQUIREMENTS**

*A laptop computer is recommended for access to online videos and tutorials that support in-class lectures. For tests and final exam, the use of a scientific calculator is required. Moreover, one of the tests will consist of a PowerPoint presentation on at least two of the topics covered during the course, it is students' responsibility to learn the use of said software.*

This course is designated as a face-to-face class for Fall 2020. Computer access to the internet will be necessary to monitor Blackboard or in the chance, the university, and thus the class, revert to an on-line format. Resources for on-line based learning can be found at [www.utoledo.edu/dl/students/required-info-online-learners.html](http://www.utoledo.edu/dl/students/required-info-online-learners.html)

### **UNIVERSITY POLICIES**

*The University is an equal opportunity educational institution. Please read [The University's Policy Statement on Nondiscrimination on the Basis of Disability Americans with Disability Act Compliance](#).*

### **ACADEMIC ACCOMMODATIONS**

*The University of Toledo is committed to providing equal access to education for all students. If you have a documented disability or you believe you have a disability and would like information regarding academic accommodations/adjustments in this course please contact the [Student Disability Services Office](#).*

### **ACADEMIC POLICIES**

*Students are expected to follow the guidelines of student conduct as outlined in the Student Handbook ([http://www.utoledo.edu/student affairs/pdfs/studenthandbook.pdf](http://www.utoledo.edu/student%20affairs/pdfs/studenthandbook.pdf))*

***Academic Dishonesty:** The academic honesty policies, as stated in the 2017-2018 UT Catalog will be strictly enforced. Any student found violating the UT academic honesty policies will be penalized in accordance with these policies.*



## COURSE EXPECTATIONS

- Attendance is expected, if you are absent, you are responsible for any material covered in the lecture including (but not limited to) assignments, policy changes, or schedule changes.
- Exams cannot be made up unless the student is missing for a university sanctioned event, illness, or death of a loved one. Documentation will be required. Please notify the instructor as soon as possible for any of these reasons.
- Cell phone use is not permitted during lectures or exams.
- During the in-class class lectures, it is not allowed texting, use of social media or Skype, use of cameras, recorders and mobile phones (unless specified by the instructor for interactive learning activities).
- Specific seating will be determined for the entire semester. Students are expected to use the same seat each class period. If you need to arrive late or leave early please do not disrupt the class upon entering. Students are highly advised to arrive on time!
- Each student found to disturb or interrupt the lecture will be gently invited to leave the classroom with the only purpose of guaranteeing a proper learning experience for other students attending the lecture.
- Students should keep in mind that the course notes (powerpoints) have the sole purpose of facilitating the in-class lectures and are not meant to be the sole source of learning material. The textbook and additional material provided by the instructor should be considered as the reference learning material to prepare for exams in this course.

## GRADING

*Course points: please refer to the following table for the distribution of points that will contribute to the final grade for the course*

<b>Test #1</b>	<i>100 points</i>
<b>Test #2</b>	<i>100 points</i>
<b>Test #3</b>	<i>100 points</i>
<b>Final Exam</b>	<i>200 points</i>
<b>TOTAL</b>	<i>500 points</i>

### **Final Grading**

*The final grading will be calculated based on the percentage of total points acquired from tests and the final exam. Please refer to the following table to relate the calculated percentages to the final grades.*

	<b>A ≥ 92%</b>	<b>A- ≥ 89%</b>
<b>B+ ≥ 85%</b>	<b>B ≥ 80%</b>	<b>B- ≥ 75%</b>
<b>C+ ≥ 71%</b>	<b>C ≥ 67%</b>	<b>C- ≥ 63%</b>
<b>D+ ≥ 59%</b>	<b>D ≥ 55%</b>	<b>D- ≥ 50%</b>
<b>F &lt; 50%</b>		



## **COMMUNICATION GUIDELINES**

*The students are welcome to communicate with the instructor about any aspect of the course with concerns you have or any item that might limit your success, both by email and/or during office hours. All email communications need to be addressed to Dr. Gionfriddo ([emanuela.gionfriddo@utoledo.edu](mailto:emanuela.gionfriddo@utoledo.edu)) and contain the course name and your name in the email object. Emails will generally be answered within 24 hours.*

## **ACADEMIC AND SUPPORT SERVICES**

Please follow this link to view a comprehensive list of [Student Academic and Support Services](http://www.utoledo.edu/studentaffairs/departments.html) (<http://www.utoledo.edu/studentaffairs/departments.html>) available to you as a student

## **SAFETY AND HEALTH SERVICES FOR UT STUDENTS\***

Please use the following link to view a comprehensive list [Campus Health and Safety Services](#) available to you as a student.

## **INCLUSIVE CLASSROOM STATEMENT**

In this class, we will work together to develop a learning community that is inclusive and respectful. Our diversity may be reflected by differences in race, culture, age, religion, sexual orientation, gender identity/expression, socioeconomic background, and a myriad of other social identities and life experiences. We will encourage and appreciate expressions of different ideas, opinions, and beliefs so that conversations and interactions that could potentially be divisive turn, instead, into opportunities for intellectual and personal development.

## COURSE SCHEDULE

	<b>Class schedule</b>	<b>Topics</b>	<b>Learning outcomes</b>
<b>Week #1 and #2</b>	Jan 20, 2020	<b>Introduction to the Separation Methods course</b>	- familiarize with syllabus and student learning assessment methods
	Jan 22, 25, 27, 29 2019	<b>Fundamentals of Chromatography:</b> <ul style="list-style-type: none"> <li>• Component separation</li> <li>• Retention time</li> <li>• Separation factor</li> <li>• Resolution and theoretical plates</li> <li>• Band broadening</li> <li>• Van Deemter's equation</li> </ul>	<ul style="list-style-type: none"> <li>- How chemicals are separated on a column</li> <li>- Chromatographic nomenclature</li> <li>- Theory of column efficiency</li> <li>- Chromatographic resolution</li> </ul>
<b>Week #3</b>	Feb 1, 3, 5 2020	<b>Gas Chromatography (GC):</b> <ul style="list-style-type: none"> <li>• Fundamentals of Gas Chromatographic separations</li> <li>• Stationary phases and mobile phases</li> <li>• Key operating variables controlling retention</li> <li>• Kovats indices</li> <li>• Rohrschneider's and McReynolds' phase constants</li> </ul>	<ul style="list-style-type: none"> <li>- Fundamentals of separation by gas chromatography</li> <li>- Understanding the key parameters controlling retention</li> <li>- Predict the chromatographic behavior of molecules</li> <li>- Understanding interactions between stationary phase and analytes</li> </ul>
<b>Week #4</b>	Feb 8, 17, 19, 2020	<b>Gas Chromatography instrumentation:</b> <ul style="list-style-type: none"> <li>• GC columns</li> <li>• Injection port</li> <li>• Oven/Column compartment</li> <li>• Detectors for GC</li> </ul> <b>Introduction to two-dimensional gas chromatography (2D-GC)</b>	<ul style="list-style-type: none"> <li>- Understanding how basic components of a gas chromatograph function</li> <li>- Understanding fundamentals of 2D GC</li> </ul>

<p style="text-align: center;">Week #5</p>	<p style="text-align: center;">Feb 15, 17, 19, 2020</p>	<p><b>High-Performance Liquid Chromatography (HPLC):</b></p> <ul style="list-style-type: none"> <li>• Fundamentals of liquid chromatography separations</li> <li>• Modes of liquid chromatography:             <ul style="list-style-type: none"> <li>○ Normal Phase LC (NPLC)</li> <li>○ Reverse Phase LC (RPLC)</li> <li>○ Ion exchange chromatography (IEC)</li> <li>○ Hydrophilic interaction chromatography (HILIC)</li> </ul> </li> </ul> <p><b>HPLC instrumentation:</b></p> <ul style="list-style-type: none"> <li>• Columns and guard columns for HPLC</li> <li>• Pumps</li> <li>• Detectors for HPLC</li> </ul>	<ul style="list-style-type: none"> <li>- Understanding interactions between mobile phase, stationary phase and analytes in LC</li> <li>- Understand different chromatographic modes based on mechanisms of interactions</li> <li>- Predict retention behavior of analytes</li> <li>- Understanding how basic components of a liquid chromatograph function</li> </ul>
<p style="text-align: center;">Week #6</p>	<p style="text-align: center;">Feb 22, 24, 26 2020</p>	<p><b>Specific uses and advances in liquid chromatography:</b></p> <ul style="list-style-type: none"> <li>• Preparative-scale Chromatography</li> <li>• Ultra-high Performance Liquid Chromatography (UPLC)</li> <li>• Supercritical fluid chromatography (SFC)</li> <li>• Tandem column Liquid Chromatography</li> <li>• Two-dimensional Liquid Chromatography (2D-LC)</li> </ul>	<ul style="list-style-type: none"> <li>- Assess the difference between preparative and analytical LC</li> <li>- Understanding fundamentals of 2D LC</li> </ul>
<p style="text-align: center;">Week #7</p>	<p style="text-align: center;">March 1 2020</p>	<p><b>Test #1</b></p>	<p>Assessment of learning progress on:</p> <ul style="list-style-type: none"> <li>• Fundamentals of chromatography</li> <li>• Gas Chromatography</li> <li>• Liquid Chromatography</li> </ul>
	<p style="text-align: center;">March 3,4 2020</p>	<p><b>Other separations involving chromatography I:</b></p> <ul style="list-style-type: none"> <li>• Thin layer chromatography (TLC)</li> <li>• Size Exclusion Chromatography/Gel Permeation chromatography (SEC, GPC)</li> </ul>	<ul style="list-style-type: none"> <li>- Understanding different chromatographic modes</li> </ul>
<p style="text-align: center;">Week #8</p>	<p style="text-align: center;">March 8, 12 2020*</p> <p style="text-align: center;">*March 10,2020 Institutional break</p>	<p><b>Other separations involving chromatography II:</b></p> <ul style="list-style-type: none"> <li>• Affinity chromatography</li> <li>• Chiral Chromatography</li> </ul>	<ul style="list-style-type: none"> <li>- Understanding different chromatographic modes</li> </ul>

Week #9	March 16, 17, 19, 2020	<b>Separations involving ion exchange resins:</b> <ul style="list-style-type: none"> <li>• Ion exchange</li> <li>• Ion chromatography</li> <li>• Ion retardation, Ion exclusion, Ligand exchange</li> </ul>	- Understand the mechanisms of separation based on the use of membranes
Week #10	March 23, 24, 26, 2020	<b>Separations involving electric fields I:</b> <ul style="list-style-type: none"> <li>• Electrophoresis- General concepts</li> <li>• Immunoelectrophoresis</li> <li>• Capillary Electrophoresis (CE)</li> <li>• Capillary electrochromatography (CEC)</li> </ul>	<ul style="list-style-type: none"> <li>- Understanding the effect of electric fields on separations</li> <li>- Fundamentals of capillary electrophoresis and related instrumentation</li> </ul>
Week #11	March 31, 2020* <small>*March 29,2020 Institutional break</small>	<b>Separations involving electric fields II:</b> <ul style="list-style-type: none"> <li>• Field Flow Fractionation (FFF)</li> </ul>	- Fundamentals of Field Flow Fractionation
	April 2, 2020	<b>Test #2</b>	Assessment of learning progress on: <ul style="list-style-type: none"> <li>• Separations involving ion exchange resins and electric fields</li> </ul>
Week #12	April 5,7,9, 2020	<b>Separations involving extraction I:</b> <ul style="list-style-type: none"> <li>• Extraction: general concepts</li> <li>• Continuous extraction: Soxhlet extraction</li> <li>• Countercurrent (extraction) Chromatography (CCC)</li> <li>• Liquid-liquid extraction (LLE)</li> <li>• Solid-phase extraction (SPE)</li> <li>• Solid-phase microextraction (SPME)</li> <li>• Liquid-phase microextraction (LPME)</li> <li>• Supercritical fluid extraction (SFE)</li> </ul>	<ul style="list-style-type: none"> <li>- Fundamentals of extraction methodologies</li> <li>- Liquid based extraction methods</li> <li>- Sorbent based extraction methods</li> <li>- Exhaustive versus not exhaustive extraction methods</li> </ul>
Week #13	April 12,14,16 2020	<b>Separations involving membranes:</b> <ul style="list-style-type: none"> <li>• Osmosis and reverse osmosis</li> <li>• Dialysis and Electrodialysis</li> <li>• Filtering and Sieving</li> </ul>	- Principles of separation mechanisms by osmosis and dialysis
Week #14	April 19,21,23, 2018	<b>Separations involving flotation:</b> <ul style="list-style-type: none"> <li>• Purge and Trap</li> <li>• Dynamic Headspace Analysis</li> </ul>	- Principles of headspace sampling



Week #15	April 24,26, 2020	<b>Test #3</b>	<i>Powerpoint presentation on a peer-reviewed research article involving at least two of the separation strategies discussed during the course</i>
Week #16	May 7, 2020	<b>Final Exam</b> <i>12:30-2:30 p.m. location and mode TBD</i>	<b><i>The final exam will cover all the topics discussed during the course</i></b>