# **CHE 347** Physical and Analytical Laboratory

### Syllabus – updated 10/12/20

Fall 2020 Professors Chaiken and Franck	CV-19 version
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Physical Chemistry is focused on finding quantitative relationships between measurements of macroscopic physical and chemical properties of matter and energy that constitute microscopic theories of nature. Centuries of careful and well-documented experimentation form the empirical basis for microscopic theories. One long-term goal is to be able to predict what would happen when conducting experiments that have *not* been done before. Another, driven by the constantly evolving challenge of extending and improving the quality of life for all, is to be able to design new and more sensitive methodologies that illuminate different facets of chemical processes at the molecular scale.

We concentrate on measurements that directly reveal either the quantized nature of microscopic matter, such as spectroscopy, or, in keeping with CHE 346, thermodynamic quantities, which are explicitly macroscopic and need no explicit connection with the microscopic properties of matter, or even the existence of atoms. We are especially interested in the kinds of information that yield to quantitative theoretical interpretation. The exposure you receive in this class will not be exhaustive, but should serve as a basis and practice for you to utilize while progressing into new areas on your own.

As a practical matter, **all analytical chemistry techniques originate as physics or physical chemistry experiments in the search for new knowledge**. During the search, physical chemists build new apparatus to study basic properties of energy and matter, and these may become analytical instruments, as engineers use existing knowledge to solve problems. Ultimately, analytical chemists use these instruments to study literally every aspect of nature. As such, some of our exercises will focus on the measurement process itself. A general introduction to measurement strategy and technology combined with a continuing emphasis on proper treatment of experimental error will underpin all planned activities of the semester.

In addition to learning about the scientific substance of these chemical and physical measurements, we will also gain experience in the handling and presentation of data and results. It is imperative that we all become as adept as possible in our ability to communicate our science either orally or via the written word. The lab reports of this course and your interactions with your TAs and Professors Chaiken and Franck give opportunities for students to gain experience in these areas.

We all need to become as proficient as possible in the searching of the scientific literature in general. This does not stop at Wikipedia or Google Scholar. Students should become proficient and interested in SciFinder, the search engine run by the ACS Chemical Abstracting Service (CAS), or perhaps PubMed, Web of Science, etc., etc. The literature of science and technology is vast and growing very rapidly and SU has given you access to plenty. You will have the opportunity to search, read and try to understand the primary scientific literature. Learning is a life-long process so join the club!

We all need to become disciplined at always working safely for our own sake and in consideration of others. This includes working in an environmentally responsible manner. The usual rules and procedures for working safely in PChem Lab are described later but are always foremost in our minds. As in all past years, on the first day, if you are in lab in person, you will be required to sign a waiver for all the usual risks associated with working/being in a laboratory. This year is not like past years in many ways and so we have instituted an entirely new set of protocols and procedures that usually would not be required.

# **CV-19**

In these CV-19 times our scheduling will reflect our collective need to 1) suppress the transmission of the virus and 2) to mitigate the effects of our on-campus activities being interrupted by a CV-19 uptick/spike, possibly with very little advance notice.

Constant use of masks, 6 ft distancing, gloves, and/or handwashing/disinfectant are the rule, and are necessary to protect those around you. If you expose your nose and/or mouth for any reason in the lab or classroom, even temporarily, you will need to leave, and will not be able to make up the required work.

We have chosen a format that will have time slots that allow **two students** to enter lab at a specific time and collect data for a set period e.g. 20-30 minutes. **One TA** will be present. In this manner we can accommodate at most **12 students in a section** since there would be 6 slots per section (3 hr) and 2 students per slot. You will need to appear at lab promptly, ready to go forward with a notebook set up to allow expeditious data collection, or this model will fail. If you arrive at most a few minutes early, you can ask questions of the TAs before you enter the lab room itself. Note that your TA will have to cut you off **potentially abruptly** when your time slot ends – so be sure to be prepared!!

We will provide specific guidance, but, students will sign up on blackboard for these slot times on a week by week basis. Any two students who want to be lab partners should contact us as soon as possible (post a message on the Blackboard  $\rightarrow$  Discussions  $\rightarrow$  "Labmates Wanted") and they are responsible for making the logistics work. We will assign partners to the extent necessary. To facilitate this process, please visit the Blackboard  $\rightarrow$  Discussions  $\rightarrow$  "Labmates Wanted" discussion ASAP, where you can post your availability in order to find a labmate who matches well with your schedule.

Planned activities and experiments are listed in Table 1 below. In general, you will have 2 weeks to collect your data and generate your lab report, with the lab report Keeping CV-19 in mind, the initial labs are more focused on data acquisition and the later labs on data analysis. It is imperative that students maintain a good notebook in order to return to early experiments later in the semester to provide written assignments based on the data collected. Your capacity to do this will be part of your grade.

Note that all data acquisition needs to be completed on or before 11-13-2020. In particular, experiment 3, "Heat of Combustion", will be a full lab report that Professor Chaiken will grade by hand. This experience will help you produce your Final Project which will be in the form of an "Executive Summary" or possibly a "White Paper". The following week i.e. the week before Thanksgiving week (beginning Monday November 16), we will present a Final Project that also utilizes the same data you already collected, possibly together with additional data we will provide. The Final Project will be due on the last day of instruction: December 4.

Solutions or samples will be already prepared by the TAs and instruments e.g. spectrometer will be warmed up. Training videos for many instruments will be identified to you so you can look at them on-line before entering the lab. Of course, as for any "normal" semester, there will be other materials as well for preparation and all will be found on Blackboard.

We may need to redistribute some students into different time slots, be proactive/flexible in scheduling, and some students may welcome same. Our schedule prioritizes students beginning to accumulate data as soon as possible after the first day of classes from working hands-on, in-lab. We believe this affords the best chance of 1) all students being able to get hands-on experience with instrumentation and procedures and 2) have data to use for all required lab reports and such in the event of a CV-19 uptick/spike and dismemberment of our on-campus semester.

It is the students', TAs' and the Professors' individual choices as to whether to risk face to face contact for the completion of this course. We shall maintain a parallel presentation on Blackboard as we complete this semester. It will be possible to finish CHE 347 in a virtual format only if bad things happen. At the time of this writing planning is in real time as information from students and administration is coming in.

One might ask whether there is any reason to incur any risk whatsoever and allowing that each person's own decision is weighted in the context of their own pros and cons. The pros i.e. the reasons to incur any risk at all would seem to emanate from the perception that a person placing their eyes and hands on a specific instrument or apparatus or combination thereof gains some familiarity beyond that accessible by looking at a picture or video. The Internet has introductory/training videos available, originating mostly with the manufacturers of instruments, that are intended to be followed by hands-on practice, to certify competence in their technology.

Certification for any skill usually requires hand on expertise in addition to demonstrations of knowledge. While there must be similarities between instrumentation that performs the same function from different manufacturers, there can be important differences. Skill set depends on experience with different types of instruments and measurements. Many kinds of research require very special precautions for safety and performance of potentially hazardous lab procedures whether CV-19 is active or not. And so, in our profession, we require proficiency at utilizing the recommended precautions to safely perform any experiment we choose. Having used science to mitigate our collective risk, we will provide as much experience with actual laboratory instrumentation as possible safely.

#### If you Contract CV-19

Notify both your instructor and TA's ASAP. Partly, this is to help protect your classmates, TA, and labmate. We can also make allowances for assignments, but only with a written note from a doctor and a positive test.

### **Locations and times**

Lab lectures are by Zoom and (if the pandemic allows) in-person on Mondays, originating live from CST 1-019 from 2:15 to 3:10 pm. In any case these lectures will be recorded and made available on Blackboard. Labs are from 9:00– 11:50 am Tuesdays, and from 2:15 – 5:05 pm Wednesdays, Thursdays, and Fridays. Some experiments will be in LSB 301 and some in 0-016 CST.

#### **Alternating In-Person Attendence**

In order to maintain social distancing, the class is divided into a *green* and *yellow* group as follows:

Yellow Group	Green Group
Paige O Adebo	Sana Fathell Alamarie
Athena Andrea Basdekis	Steven Matthew Axelsen
Marcia Ann Cal	Lilly Basgall
Sandy Ynhu Cao	Wilasinee Daloonpet
Anthony James Defrancesco	Sophia Elizabeth Figueroa
Alexis Jean Eckhart	Brent Gosselin
David Anthony Fikhman	Alyssa J Grzesiowski
Conal Gallagher	Oduduabasi James Isaiah
Thomas Joseph Jensen	Laxmi Khatiwada
Stanley Jimenez	Emily Nicole Krueger
Cheyenne Michelle Kersavage	Simran Dharmendra Lakhani
Jessica Ngoc Minh Khuc	Angela L Martinez
Caroline J Leduc	Cheyene L Muenzel
Diarra Mack	Lucia Cortina Rinaldi
Daniel J Pelkey	Ryan Gordon Ryersen
Natasha Prince	Brinley Nicole Walawender
Jacob Tyler Shoen	Elizabeth M Wall
Morgan Storino	Erin K Wisard
Jack M Wenner	James Richard Zackey
	Dylan Joseph Gray

For office hours and for the first lecture, you are **required** to turn on your video during Zoom, unless you contact us ahead of time documenting a particular hardship that prevents this.

# **Contact information**

Dr. Chaiken, Dr. Franck, and TAs will meet with students by appointment. Email addresses are:

Dr. Chaiken: jchaiken@syr.edu

Dr. Franck: jmfranck@syr.edu

Minh Quan Hoang Dinh: midinh@syr.edu

Alec Beaton: aabeaton@syr.edu

Nicole Spanneda:<u>nspanedd@syr.edu</u>

It is your responsibility to contact your TA(s)/Professors with questions in a timely fashion. Please note that your TA's do have many time commitments, and we encourage them **not** to reply to requests in less than 24 hrs.

Office hours:

- Thursdays [11am-12pm] Nicole S. zoom link
- Fridays [1pm-2pm] Alec B. zoom link
- Prof Franck [open] zoom link

# Schedule

Note that the labs start *on the first Tuesday, the 25<sup>th</sup>*! Because we will begin lab the first week, you should read the material ASAP and complete your prelab before you appear in lab. The official due-date for the prelab will be on the second Monday, at the same time as Exp 2 prelab.

Description of the numbered lab experiments is given in Table 2

The schedule and list of experiments in **Table 1** is **tentative**: *It is your responsibility to watch for updates to this document (can be announced in lecture or on Blackboard) throughout the semester!* 

Please note that post-labs are generally due on midnight, either 1 or 2 weeks after your lab section.

Table 1

Schedule	
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Dates	Lab #	Additional Lecture Info	Mon Lab Lecture Format	Due
Aug 24 – Aug 28	Exp 1	TA: Intro and Safety	All Online	<i>Day of Section:</i> Exp 1 Data
Aug 31 – Sep 4	Exp 2	Notes on Final Project Data	Green group in-person	<i>Day of Section:</i> Exp 2 Data
Sep 7 - Sep 11	Exp 3	(Labor Day): Spectroscopy of HOD	Pre- Recorded	<i>Day of Section:</i> Exp 3 Data
Sep 14 - Sep 18	Exp 4	Discussion of Lab Reports Pre-recorded tutorial on Python/Excel posted	Yellow group in-person	<i>Day of Section:</i> (1) Exp 4 Data (2) Exp 3 Lab Report
Sep 21 - Sep 25	Exp 5		Green group in-person	<i>Day of Section:</i> Exp 5 Data
Sep 28 - Oct 2	Exp 6		Yellow group in-person	<i>Day of Section:</i> (1) Exp 6 Data (2) Exp 4 postlab ( <i>midterm</i> )
Oct 5 – Oct 9	Exp 7		Green group in-person	Day of Section: (1) Exp 7 Data (2) Exp 5 postlab Oct 11: Complete Python / Excel tutorial recording
Oct 12 - Oct 16	Exp 8		Yellow group in-person	<i>10/16</i> Exp 6 Postlab
Oct 19 - Oct 23		No Lecture – Office Hours		<i>10/23</i> Exp 7 Postlab
Oct 26 - Oct 30		Discussion of Final Project	Green group in-person	<i>10/30</i> Exp 8 Postlab
Nov 2 - Nov 6		Further discussion of Final Project	Yellow group in-person	
Nov 9		No Lecture – Office		

- Nov 13	Hours	
Nov 16 - Nov 20	Thanksgiving Week	
Nov 23 – Dec 4	Class is virtual for remainder of semester	
Dec 4		Final Project Due

### Table 2

#### **Order of Experiments**

#	Торіс	Instructor/TA
1	Absorption and Emission Spectroscopy, Calibration Curves	Chaiken/Minh
2	Final Project Data collection (Chaiken/Minh part only)	Chaiken/Minh
3	NIR spectroscopy of HOD, PCA, $\Delta G_{mixing}$	Chaiken/Minh
4	Heat of Combustion	Chaiken/Minh
5	Enthalpy of Reaction	Franck/Alec
6	Adsorption Isotherms (ATR)	Franck/Nicole
7	Calculate Heat of Combustion	Franck/Nicole
8	Proton exchange of pyruvic acid via NMR	Franck/Alec
	Final Project	All

Completing the pre-lab work for the wrong experiment will result in you not being allowed to attend that week's lab or not be given data for the lab if attending virtually, and you will receive a grade of 0% for the experiment you were meant to perform that week.

### **Due dates for pre-lab work**

Pre-lab readings for each lab must be completed before that week's lab lecture. A set of prelab questions for each experiment will be made available to students on Blackboard on the Monday before the week of the experiment (so for example, the questions for Exp 2 will be available the first day of class). Answers to each question must be submitted on Blackboard no later than 2:00 p.m. on the day of the lab lecture for that week's experiment. (This would mean submitting answers for the pre-lab questions for Heat of Combustion by Monday, September 21 by 2:00 pm).

If you do not submit the answers on time you will not be allowed to attend that week's lab and will receive a grade of 0% for that experiment. If you receive a score of less than 50% on the pre-lab questions you must write the correct answers for **each** question in your lab book/Word document (even the questions you answered correctly online) and present/email it for inspection by your TA at the beginning of your lab. Failure to do this will result in your not being allowed to participate in that week's lab and receiving a grade of 0% for that experiment.

**You must be prepared for each lab** - *even if you are doing the course entirely on-line.* Students who are not prepared slow down the class and can pose safety risks to themselves and others. This is particularly true in CV-19 times. Even if you are doing the course entirely on-line the *process* of becoming prepared is worth repeating again and again. All kinds of experiments are different and require thought to be prepared. What are you looking for? What do you need to do once you have the data? There is much to gain when doing experiments *if you know what you are doing at the time you are doing it.* 

You must prepare an experimental procedure for each experiment. It should be brief, functional in that it enables (or would in the case of an entirely on-line format) the student to carefully, expeditiously and accurately execute the lab procedure, and understandable to anyone reading who is familiar with CHE 347. This procedure must be written in your lab notebook in ink, **not** pencil. If prepared on a computer and lab attended in person, the procedure must be pasted or taped securely into your lab book. If attending completely virtually, then the procedure must be submitted as a word document.

For some labs, you will be required to look up specific pieces of information (e.g. spectra, literature values). This will be made clear in the pre-lab instructions. These values must be written clearly in your lab book/word document by the beginning of the lab.

If attending in person, you must have your lab book open to the procedure for the current experiment by 2:15 p.m. the day of the lab (by 9:00 a.m. for the Tuesday section) **or in analogous time frame for your time slot**. The TA will check each lab book to ensure that all pre-lab work has been properly performed (procedure, requested literature values, and, if necessary, answers to pre-lab questions). If you do not have the pre-lab work ready at the beginning of the lab, you will be dismissed and will receive a grade of zero for that experiment. If you arrive more than ten minutes late to the lab, the TA will not check your lab book and you will be dismissed from the lab (and will receive a grade of 0% for that experiment). If attending the class only virtually, then your word document/prelab must be submitted to the appropriate TA at the same times as for in person, except because virtual attendance requires no time slot, the submission must occur at least 15 minutes before the start of the Monday lecture for that week.

### **Due Dates for Post-Lab**

Because we want to complete data collection for all lab exercises as early in the semester as possible in case we have a CV-19 spike, your valid data must be handed in/emailed to appropriate TA by End of Business (EOB=5pm prevailing US Eastern Time) for all labs.

Post-lab work that is handed in late will be penalized at a rate of 10% per day. Thus, Work handed in at e.g. 5:01 p.m. on the due date **will be penalized 10%**. Weekends count as one day. The full lab report (Heat of Combustion Lab) is due EOB on the lab day of the week of Oct 9 in lab. Penalization for late lab reports is the same as for regular post-lab work

(described above). The Final Project post-lab work is due the last day of the semester, December 4.

# **Digital Turn-In**

To turn in hand-written assignments, you must use the smartphone app TurboScan (iOS + android) or Scannable (iOS). There may be a small fee associated with these apps. It is possible other apps produce acceptable output (small size PDF files with good background removal), but you must send us a test example before utilizing them. (For example, Microsoft OneLens produces unsuitable output as do many other free scanning apps, and raw photographs are also unacceptable).

# Grading

Grades will be assigned as follows:

Weekly post-lab work	64%
Full lab report	5%
Final project	15%
Pre-lab questions	6%
Lab lecture participation	4%
In-Lab Performance	6%

There will be no make-up labs. The experiment with the lowest total grade (sum of pre-lab questions and post-lab work) will be dropped at the end of the semester. If you do not attend a lab for any reason, the grade of zero assigned for that experiment will count as your lowest grade and will be dropped. If you miss more than one lab, only one grade will be dropped, and the other lab(s) you missed will be assigned a grade of zero.

We have yet to agree internally with possible ways for students taking the class entirely online to gain points for participation if they are unable to view the lecture concurrently. Most likely all your participation points will be shifted to your prelab grade. (Suggestions are always welcome BUT there is no guarantee that your suggestions will be implemented.)

### **Tentative Grading Scheme**

Provided the class average for total points earned at the end of the semester exceeds 55% of the maximum possible point total, the following grading scale will prevail.

### **Total Points Earned**

Points EarnedLetter GradeTop 10-15% of classANext 5%A-

Next 5%	B+
Next 10%	В
Next 10%	B-
Next 20%	C+
Next 20%	С
Below	TBD

Note that these boundaries are flexible because e.g. situations arise when multiple students earn almost the same number of points and they must all get the same grade. If the class average proves to be less than in previous years, then the percentages of students earning better grades will slip and those for less good grades will increase.

#### **Bonus Points**

Given these uncertain times it is conceivable that there *may* be some (very few, if any) opportunities to increase your overall grade by performing additional work. These opportunities for bonus points will be announced during the lab lecture or by email/Bb announcement. They may not be announced on Blackboard or via email: It is the individual student's responsibility to make themselves aware of these opportunities.

# **Dress code**

Close-toed shoes must be worn in the laboratory, and long hair must be tied back. Safety glasses, lab coats, and gloves will be provided for some experiments. You must wear this protective gear if requested to do so by your Professor or TA. Failure to adhere to these rules will result in dismissal from the laboratory and a grade of 0% assigned for that experiment.

# **Blackboard Site**

Course information/registration is available through the course Blackboard site: blackboard.syr.edu

Important course information can be accessed there. Tutorials and required readings for labs can also be accessed there. Pre-lab questions must be answered on Blackboard.

#### **Religious Observances Policy**

#### http://supolicies.syr.edu/emp\_ben/religious\_observance.htm

SU recognizes the diversity of faiths represented among the campus community and protects the rights of students, faculty, and staff to observe religious holy days according to their tradition. Under the policy, students are provided an opportunity to make up any examination, study, or work requirements that may be missed due to a religious observance provided they notify their instructors before the end of the second week of

classes. For fall and spring semesters, an online notification process is available through MySlice/Student Services/Enrollment/My Religious Observances from the first day of class until the end of the second week of class. The religious observances policy requires accommodation for the religious holiday itself, not for travel days if a student will be observing the holiday elsewhere.

**Academic Integrity:** The Syracuse University Academic Integrity Policy holds students accountable for the integrity of the work they submit. Students should be familiar with the Policy and know that it is their responsibility to learn about instructor and general academic expectations with regard to proper citation of sources in written work. The policy also governs the integrity of work submitted in exams and assignments as well as the veracity of signatures on attendance sheets and other verifications of participation in class activities. Serious sanctions can result from academic dishonesty of any sort. For more information and the complete policy, see:

#### http://supolicies.syr.edu/ethics/acad\_integrity.htm.

**Special Accommodations:** If you believe that you need accommodations for a disability, please contact the Office of Disability Services (ODS), http://disabilityservices.syr.edu, located at 804 University Avenue, room 309, or call (315) 443-4498 for an appointment to discuss your needs and the process for requesting accommodations. ODS is responsible for coordinating disability-related accommodations and will issue students with documented disabilities "Accommodation Authorization Letters." as appropriate. As accommodations may require early planning and generally are not provided retroactively, please contact ODS as soon as possible.