CIV ENV 365: Environmental Laboratory (Winter 2023)

Syllabus

January 9, 2023

Tuesday between 1:00 and 5:00 pm in Tech Room A230 &Tech Room A147/A151
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Website: CANVAS

Abstract: The focus of this course is to perform a suite of chemical measurements to document the quality of environmental samples and therefore assess the fate of contaminants. This course builds on concepts covered in previous courses – e.g., 260, 364 & 367 – with an emphasis on characterizing the distribution and evolution of chemicals in the natural environment or within engineered systems. It provides hands on experiments focused on making measurements using state of the art and conventional instrumentation.

1 Course Objectives

Most of environmental issues and assessments rest on the determination of the concentrations of chemical species in environmental systems. These analytical methods must be able to characterize contaminants and the chemical matrices of air/atmospheric, soils/sediments, and water samples. Other aspects are related to the presence of microbes – e.g., pathogens, viruses, – but in this case also, detection methods rely on analytical methods that have been developed following concepts and principles derived from chemistry.
Hence analytical chemistry is really a core discipline upon which environmental engineers rely to make decision but are often poorly trained about. The overarching objective of this laboratory-based course is introduce you to some of these analytical methods that are used to analyze chemicals in the environment.
This year, the course is structured around the characterization of a solid waste material that is produced during the first stage of drinking water treatment: a drinking water treatment residual. You will be using bulk solid analyses methods as well as wet chemical methods to assess the interactions of this material with a natural water.

Specific objectives are:

1. to work on a team to plan, conduct and interpret the results of experiments (ABET #5 & #6)
2. to be familiar with scientific instrumentation for measuring the concentrations of contaminants (ABET #1 & #7)
3. to learn and improve during the quarter basic laboratory skills
4. to be able to effectively report, analyze and interpret laboratory results (ABET #6)
5. to use statistical methods in order to report analytical errors/uncertainties.

2 Textbooks & Online Resources

- **Reading**: Analytical Chemistry 2.1 by David Harvey (2016)
  It is an e-book, that is made available free of charge by the author after being sold as an academic book. It is a good resource, please download your copy from Canvas, you will find it in the first module. For each of the experiments, you will find a chapter of this book that provides basic information. Under the sponsorship of the NSF websites dedicated to teaching chemical methods and principles have been put together. These are great resources for getting a deeper understanding of the fundamentals upon which analytical methods are based.
- **Additional Recommended Readings**: There are many analytical books that cover the basic principles and the instrumentation that will be used in this class. Below is a list of additional reading material that you can get access to online, and books that are reported here because they are located in the teaching lab. However, this year please only use the online material listed. The textbooks listed below are excellent references for more detail coverage of the subject matter and are reported for your perusal.

  1. Visit `https://chem.libretexts.org/Bookshelves/Analytical Chemistry` – a repository for various analytical methods and details about some core principles upon which chemical instrumentation is based.
  2. **Analytical Chemistry** (2004) – A Modern Approach to Analytical Science - edited by: R. Kellner, J.-M. Mermet, M. Otto, M. Valcárcel, and H. M. Widmer, publisher Wiley-VCH. (note: a very complete treatment of analytical methods, a must have if you go in this field).
  3. **Principles of Instrumental Analysis** (1998 - Fifth Edition) by D.A. Skoog, F.J. Holler, and T.A. Nieman, publisher Saunders College Publishing. *(note: a more advanced text that builds upon the foundation of the text above, in particular it provides detailed information about electronic components entering the design of instrumentation)*.

3 Prerequisites

- Chemical Processes in Aquatic Systems: CIV ENV 367
- Computer Programming/Use: Understanding how to use ChemEql, basic knowledge of Python Jupyter notebook to write reports.

4 Grading

Final Grade = Laboratory reports (60%) + Final report (40%)

5 Additional Information

- **Computer Use:** Most of the calculations and the reports will be prepared as notebooks – Jupyter Notebooks using Python as a programming language. Examples will be provided on CANVAS and are also present in the textbook.
- **Class Participation:** Participation to this class is mandatory as you will be performing hands-on experiments. The expectation is that you will all be able to perform experiments using the suite of instruments present in the environmental laboratory as well as some instruments that are housed in IMSERC – Integrated Molecular Structure Education and Research Center. A timetable will be constructed on the first week for the quarter.
  This year we will visit region 5 US-EPA environmental laboratory located in Chicago, so that you can have further information about the challenges related to the analyses of trace contaminants in the environment.

Laboratory coats, gloves, and face shields will be provided. If you develop any sign related to COVID-19, or have tested positive to the virus, please inform us and ask about guidance for resuming lab participation. In any case, the instructors & the TA will complete the experiments if needed and the raw data will be sent to you for analysis and interpretation.

6 Weekly Schedule & List of Experiments

The schedule and the list of experiments that will be performed will be decided during the first 2 weeks of the course. Each laboratory team will then rotate through the suite of instrument used to characterize the outcome of a simulated release of chemicals species from the solid waste material studied.
The list of instruments and analytical methods at your disposal in the Environmental Laboratory is:

1. **"pH-meters":** These are high entry impedance voltmeters to measure the difference of potential produced by a combination pH electrode. NIST buffer solutions for calibration will be prepared and measurements will be made on the milli-volt scale.
2. **Turbidity-meter/Nephelometer:** to measure the concentration of suspended particles in solution
3. **Conductivity-meter:** to measure the overall ability of the solution to conduct an electric current. This measurement provides a proxy for the ionic strength of the solution.
4. **Titration Alkalinity**: this will be determined by means of a computerized titration system.
5. **TOTCa & TOTMg**: the concentration of these cations – as also other elements in solution – will be determined by Flame Atomic Absorption Spectroscopy – FAAS.
6. **TOTNa, TOTK, and TOTLi** : the concentration of these akali metals – i.e., elements in the first left row of the periodic table – are best measured by Flame Atomic Emission Spectroscopy – FAES –
7. **Major and Minor Anions**: are measured by Ion Chromatography – IC –
8. **Dissolved Organic Carbon**: Measurement of DOC is performed by high temperature catalytic oxidation followed by infra-red detection of the CO_2 evolved in the process
9. **Trace metal analysis**: Analyses of dissolved metals – such as transition metals Co, Cr, Cu, Ni, and Zn as well as "heavy metals" such as Cd and Pb - are analyzed by Graphite Furnace Atomic Absorption Spectroscopy using a high resolution monochromator and a continuous light source – HR-CS-GFAAS. This method also works well for some oxyanions such as As and Se, and is also used to measure contaminants such as PFAS by using molecular absorption.
10. **Nutrients**: $TOTPO_4$, $TOTNH_4$, $TOTNO_2$, and $TOTSiO_2$: are analyzed by spectrophotometry after forming colored complexes and measuring their molecular absorption. Colorimetric methods work also well for some metals, such as for dissolved Fe and can be specific of a given oxidation state.
11. **Nutrients**: $TOTNO_2$ and $TOTNO_3$ can also be analyzed by differential molecular absorption spectroscopy measurements.
12. **Portable Air Sensor**: Use of a Temptop portable sensor to analyze for temperature; atmospheric particles: total count as well as PM 2-5 & 10; gases: CO_2 and formaldehyde (CHOH).

The instruments located in IMSERC that will be used for solid analyses are:

1. **"TGA":** Thermogravimetric analysis to document the weight loss of a solid sample (waste/soil) as a function of temperature. A more detailed LOI – loss on ignition – measurement.
2. **"XRF":** X-ray fluorescence provides a way to determine the presence of inorganic contaminants, such as metals – Cu, Zn, Pb, ... –