Analytical Instrumental Laboratory
CHEM 314W

Introductions:
1. Syllabus and Course Structure
2. Course website
3. Course projects
4. Overview of Techniques
5. Paper Format Activity

Dr. Sarah Hayes
Asst. Professor
Preparing for class

Experiments

CHEM 314 Experimental requests: Required for Experiments 2-6

Quantifying Red Dye #40 by Standard Addition
Standard Additions Myth and Reality (Ellison 2008)
Instrument Instructions

**Experiment 1: Elemental Analysis

1-15 Introduction
Before Class: Bring two copies of your favorite 3-5 page peer reviewed article to class.
We will review papers in class for format and content.
Activity: Paper format class activity

Techniques Overview
Experimental Techniques powerpoint

1-20 Instrumental Measurements, Noise, Error
Reading: Skoog ‘n’ Holler Chapter 1, 5
Error Reading
Before Class: Read the following book section and fill out the first page of the class notes before coming to class.

CHEM 314- https://instrumentalanalysis.community.uaf.edu
How will we address each question?
Which technique answers which Q?
Techniques for elemental analysis

**Handheld XRF**
- Iron
- Not detectable?
- Aluminum
- Other?

**Dissolve metal**
- HNO₃
- HCl
- ???

**Major element**
- Flame AA
- SEM, XRF

**Trace metals**
- ICP-MS
- ICP-MS
Plastics Identification

Probes vibrations of functional groups within sample

- FT-IR
- Flame test
- Others?

Spectral interpretation
The UV-Vis analysis of the soda confirmed the presence of red 40 in the soda. The concentration of blue 1 in the soda was below the detection limit of the instrument and therefore could not be confirmed.
Calorimeter- energy per mass

https://people.chem.umass.edu/botch/Chem122S08/Chapters/Ch6/CalorimBomb.jpg
High Performance Liquid Chromatography (HPLC)

Step 1: samples adjusted for concentration
Step 2: samples separated in column
Step 3: samples detected using UV-Vis or EDLS

Identified on the basis of:
Retention time  UV-Vis absorption
Light scattering  Mass Spectrometry
Gas Chromatography- Mass Spectrometry (GC-MS)

Step 1: sample injection: liquid or headspace
Step 2: samples separated in column
Step 3: totals molecules counted
Step 4: molecules fragmented
Step 5: ion fragments detected

Headspace or direct liquid injection
Identified on the basis of:
Retention time
Mass spectrum
Electron Microscopy

Step 1: preparation of samples
Step 2: carbon or gold coat samples
Step 3: Electron microscopy
Choose your own adventure (CYOA)

You must propose three ideas in the project proposal

Potential ideas:
Identification/quantification or molecules using **NMR**
X-ray diffraction (**XRD**) to identify mineral species (or some organics)
X-ray fluorescence (**XRF**) to quantify elements
Additional Separations
Additional color examination
Paper format exercise

What goes where in a paper?
Next Tuesday (Jan 20)

8am Meet in Runcorn Room
Lecture- Introduction to Measurements
Experiment- Standard Addition

The Schedule

<table>
<thead>
<tr>
<th>Date</th>
<th>Monday Lecture</th>
<th>Tuesday Lab</th>
<th>Thursday Lab</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-12</td>
<td>No class</td>
<td>No class</td>
<td>Introduction</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Paper format: writing assessment</td>
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<tr>
<td>1-19</td>
<td>No class</td>
<td><em>Introduction to Measurements (Ch 1)</em></td>
<td>Planning safe experiments</td>
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<td></td>
<td></td>
<td><em>Noise and Error (Ch 5)</em></td>
<td>Standard Addition</td>
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<tr>
<td>1-26</td>
<td><em>Interactions b/w light and matter (Ch 6)</em></td>
<td>Standard Addition</td>
<td>Metals: sample destruction, XRF</td>
</tr>
</tbody>
</table>

* Project overview requirements
Preparing for class

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