

## Instrumental Analysis Lab Instructions—Chromatography Labs.

Within your group (lab day), divide into pairs.

Designate each pair as "A", "B" or "C"

Within each pair designate each person as "1" or "2"

Pay careful attention to the instructions and who is doing what. It gets complex FAST!

week conducted, report due following week	report type	GC	LC	IC
Week 1 Jan 27 - 31	wk 1 oral wk 1 written	A1 A2	B1 B2	C—both do oral (meet together, one time, instructor and both of you! ) <i>anions: 2 – 50 ppm Cl<sup>-</sup> and 2 – 10 ppm NO<sub>3</sub><sup>-</sup></i>
Week 2 Feb 3 - 7	wk 2 oral wk 2 written	A2 A1	B2 B1	C—both do written (one report, two names)
Week 3 Feb 10 - 14	wk 3 oral wk 3 written	C2 C1	A2 A1	B—both do oral <i>anions: 2 – 25 ppm SO<sub>4</sub><sup>2-</sup> and 2 – 10 ppm NO<sub>3</sub><sup>-</sup></i>
Week 4 Feb 17 - 21	wk 4 oral wk 4 written	C1 C2	A1 A2	B—both do written
Week 5 Feb 24 - 27	wk 5 oral wk 5 written	B2 B1	C1 C2	A—both do oral <i>anions: 2 – 50 ppm Cl<sup>-</sup> and 2 – 25 ppm SO<sub>4</sub><sup>2-</sup></i>
Week 6 March 9 - 13	wk 6 oral wk 6 written	B1 B2	C2 C1	A—both do written

On the odd numbered weeks, the group doing GC should arrive at my office at the scheduled time. The group doing LC should arrive 45 minutes after the scheduled time and the group doing IC will have the week off. Note: IC still has a report even though they are not in lab!!! On even numbered weeks, all students should start at the scheduled time.

**Each group (A, B, C) has a different task:**

GC—determine the concentration of two components in a mixture of unknown organic waste (probably from a 3411 lab) by GC-TCD using internal standard calibration

LC—determine the concentration of quinine in diet tonic water by LC-Fluorescence using standard addition

IC—determine the concentration of two assigned anions in a surface water sample using a calibration curve (external standard curve)

**Assignment for Week 1 (and all odd numbered weeks)**

**In Lab:** 1) Run the separation using different conditions (3-4). Determine which conditions are best for your sample. Save that method using your name. (All methods and data should have the group name as part of file name.)

IC—no lab, enjoy your “bye” week. You still need to give an oral report.

2) Determine the retention time of your analyte by spiking the mixture. GC people should identify two components of the sample (analytes), one substance not in the sample (to use as an internal standard) and one other substance to use as a solvent. LC people need to find an appropriate concentration of analyte and prep 500 mL of mobile phase. IC people need to figure out a plan for the lab the upcoming week, including how you will identify your analytes!

**Your report** (Due before starting lab the following week):

1) LC/GS oral report: make an appointment (30 min) with Dr. Myers to discuss your plan for the next week’s lab which will accomplish the purpose/task associated with your instrument. You may bring your lab notebook *only* to this meeting. Be prepared to answer specifics about instrumental conditions and why they were chosen, sample and standard preparation as well as data analysis. The point of the experiment in week 1 is to figure this out!

IC—describe in detail how you would prepare solutions that could be used to analyze for anions detailed on your schedule and how you plan to analyze your results. You do not have to report on instrument conditions.

2) written report: Have two sections (major divisions). Label the first one “DETERMINATION OF CONDITIONS” and in 1-2 paragraphs describe how you decided on the conditions that are best for your sample. Best sure to state what you tried and then why you tried something else and if that helped. Label the second division “EXPERIMENTAL SECTION” and completely and appropriately describe the experiment and instrumental conditions for week 2 of your (proposed) experiment. You do not have to describe solution preparation. See the website (under “Labs”) for examples in how to write an experimental section. Consider using the following subheadings: materials, solutions, instrumentation.

Use the professionalism rubric for general guidelines in formatting your reports. For specifics you should consult the “style guide.”

**Assignment for Week 2 (and all even numbered weeks)**

**In Lab:** Conduct the experiment approved during the oral report. The person who made the oral report is the lab manager this week and should organize the time and assign duties.

**Your report** due before the start of the next lab or one week after completing this experiment (whichever is sooner).

1) oral report: make an appointment with Dr. Myers to discuss your results. You should be prepared to answer questions what you did, why you did it that way, how you achieved your results and the quality of your results. You may bring your lab notebook and calibration curves with calculations written on the back.

2) written report: Turn in a professional report with three sections. The first section “INTRODUCTION” should contain only your purpose statement (yes, this is likely only one sentence). The second should be a complete “EXPERIMENTAL SECTION.” The experiment section refers ONLY to the experiment conducted in week “two.” Be sure to include the solutions you used, giving concentration ranges rather than individual concentrations or measured values. The third section should be “RESULTS” and may be as short as one sentence (although it can be a bit longer). Results should report the concentration of your analyte(s) with error and the retention times of the analyte(s) with error. Also attach your calibration curve(s) with handwritten calculations (used to get to your results) on the back of the graph. In the calculations, be sure to include one sample calculation for the concentration of your standards and your line equation with appropriate significant figures and error. If you feel your results require some qualification/explanation, you may take 1- 2 sentences to do so, but this is not required.