1. (10 group + 10 individual) Group question (on separate page).

2. In class we discussed several fundamental sources of noise.
   a. (10) What gives rise to Johnson noise in electronic circuits?
   b. (5) What things can instrument designers and experimenters do to minimize this source of noise?

3. (10) Distinguish between sensitivity, selectivity, and detection limit.

4. Dual slope analog to digital converters (ADC’s) are used extensively.
   a. (10) Explain how this ADC works.
   b. (5) What are the advantages and disadvantages of this ADC?

5. (10) What problem can arise when using an amplifier with a low input impedance?

6. (15 pts) When linear sweep voltammetry is run using a Pt electrode, the resulting voltammogram has a peak shape. When linear sweep voltammetry is run on the same solution using a dropping mercury electrode, the resulting voltammogram is sigmoidal (s-shaped like a titration curve). What gives rise to the different shapes in the two voltammograms run under the same conditions?

7. (15 pts) Thermistors are temperature sensing devices whose resistance changes with temperature. In the circuit below, R₁ is a thermistor with a resistance of 10 KΩ at 100° C and 100 KΩ at 0° C. Assume all the resistors including the thermistor have a ±1% tolerance. Calculate $E_{out}$ for the following temperatures. Don’t forget significant figures.
   a. 100° C
   b. 0.0° C

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I PLEDGE ON MY HONOR THAT DURING THE EXAM I HAVE NEITHER GIVEN NOR RECEIVED ASSISTANCE NOR HAVE I SEEN ANY DISHONEST WORK.

If you feel you can’t sign this, contact the instructor (e-mail or in person)
1. (10 group + 10 individual) Group question (on separate page).
2. (12 pts) What is the difference between a diffusion layer and a double layer?
3. (12 pts) What are the advantages of using a three electrode system over using a two electrode system for voltammetry?
4. (12 pts) In electrochemistry a current follower is often used to measure the current produced at the working electrode. Draw the schematic for a current follower that will produce 1 mV/1 µA.
5. (7 pts each) For each pair of conditions, which will produce the smaller (better) H in a chromatographic separation? Explain your reasoning.
   a. A 46 mm diameter column packed with 5µm particles or packed with 3 µm particles of the same type of packing material?
   b. 15 cm or 25 m column length packed with identical particles?
6. (15 pts) Draw a schematic of an FID detector and explain how it works.
7. (15 pts) Using the chromatogram below and the ruler supplied, calculate the number of theoretical plates (N) generated for peak 4. Show your measurements on the figure and your work for the calculation. Express your answers to the proper number of significant figures.

**GROUP QUESTION**

Each group (3-4 people) will have 10-20 minutes to discuss this problem TOGETHER and arrive at a solution. Then INDIVIDUALLY each member will explain the reasoning for that answer. Answer the question on this page. (There are 6 other questions on this exam).

\[
\begin{align*}
    \text{Sn}^{4+} + 2 \text{e}^- &\rightarrow \text{Sn}^{2+} \quad E^\circ = +0.136 \text{ V} \\
    \text{Fe}^{3+} + \text{e}^- &\rightarrow \text{Fe}^{2+} \quad E^\circ = +0.771 \text{ V} \\
    \text{Ce}^{4+} + \text{e}^- &\rightarrow \text{Ce}^{3+} \quad E^\circ = +1.61 \text{ V}
\end{align*}
\]

1. A solution contains Sn(II) and Fe(II) at approximately equal but unknown concentrations and also contains 0.10 M KNO₃ as an inert supporting electrolyte. A Pt electrode and an NHE reference electrode are introduced into the solution.
   a. Sketch the theoretical cyclic voltammogram expected when the voltage is cycled from -0.2 V to +1.0 V and back again. Label the E°’s on your sketch.
   b. This same solution is titrated with Ce⁴⁺, and the titration is followed potentiometrically using the same electrodes. Sketch the titration curve and label the Sn and Fe E°’s on your sketch.

Exact calculations are not necessary for the sketches, but clearly indicate the shape and position of the curves.
Exam 3

1. (10 group + 10 individual) Group question (on separate page).

2. (14 pts) Optics in a spectrometer can introduce aberration in the focus of the light beam. Briefly describe the two types of aberration encountered in spectrometers.

3. (15 pts) Calmagite (an organic sulfonic acid) forms a 1:1 complex with Ca$^{2+}$ that has a wine-red color in a pH 10 solution. A 100 ppm Ca(Calmaigite)$^{-1}$ solution is analyzed for Ca by flame atomic absorption spectroscopy (AA) and by UV-VIS absorption spectroscopy (Spec 20 or Ocean Optics type instrument). Both techniques utilize radiation in the UV region. The absorption bands (lines) observed in the AA are very narrow (<0.5 nm) but the bands in UV-VIS are very broad (>100 nm). What are the reasons for the big differences in the band widths?

4. (10 pts) XPS and Auger spectroscopy both give information about the elemental composition of a surface. In both methods the kinetic energy of an ejected electron is measured. Describe the process that generates the signal electron in each technique.

5. (15 pts) Draw the block diagram of a dispersive IR (not FT) and briefly explain how each block works.

6. (10 pts) What advantages does an FTIR have over a dispersive IR instrument?

7. (16 pts) From your extensive knowledge of chemistry and chemical instrumentation, identify three techniques that could be used to determine the level of Zn$^{2+}$ in plant tissue. Choose the method you think would be the best approach and explain why you prefer it.

**GROUP QUESTION**

Each group (3-4 people) will have 10-20 minutes to discuss this problem TOGETHER and arrive at a solution. Then INDIVIDUALLY each member will explain the reasoning for that answer. Answer the question on this page. (There are 6 other questions on this exam).

(10 group + 10 individual) In a wide variety of samples, [Fe$^{2+}$] can be determined by titration with permanganate utilizing the reaction:

\[ 5 \text{Fe}^{2+} + \text{MnO}_4^{-1} + 8 \text{H}^+ \rightarrow 5 \text{Fe}^{3+} + \text{Mn}^{2+} + 4 \text{H}_2\text{O} \]

Permanganate is a dark purple color, Fe$^{2+}$ is a light green, Fe$^{3+}$ is a pale yellow, and Mn$^{2+}$ is almost colorless.

In a manual titration, the permanganate functions as its own indicator, and your eyeball signals you when to stop the titration. However, in an automated titration you need a substitute for your eye to follow and record the titration curve. Identify three methods that could be used to follow this titration. Draw a block diagram of the complete experimental setup required for the automated titration and briefly explain what each block does. Make sure you identify the specific requirements for each of your three detection methods.