1] **5 points** Which of the following samples would be suitable for analysis by a calibration curve technique using a potentiometric device? Which would require a standard addition type of analysis in all likelihood?

- a) Cd\(^{2+}\) in milk
- b) Cu\(^{2+}\) in distilled water
- c) Cl\(^{-}\) in blood
- d) Ag\(^{+}\) in a paint sample
- e) F\(^{-}\) in tooth paste
- f) Na\(^{+}\) in tap water

2] **5 points** What is the dynamic range of the glass pH electrode?

3] **10 points** What are the principle interferences associated with the Cl\(^{-}\) sensitive electrode? Describe the chemical property that causes these species to be interferences.

4] **15 points** The F\(^{-}\) concentration of tap water in Moscow is maintained at 1.00 mg/mL. The response of a F\(^{-}\) selective electrode for this sample was measured at 0.320 volts. A sample of tap water from Pullman was measure with the same electrode at 0.360 volts. What is the concentration of F\(^{-}\) in Pullman water? The fluoride ISE has the following response.

\[ E = \text{const} - 0.0592 \log[F^{-}] \]

5] **15 points** Calculate the standard potential for the following half-reaction, given the K\(_{sp}\) for Pd(OH)\(_2\) is 3 \times 10\(^{-28}\) and the standard potential for Pd\(^{2+}\) + 2e\(^{-}\) = Pd(s) is 0.915 V.

\[ \text{Pd(OH)}_2(s) + 2e^{-} = \text{Pd}(s) + 2\text{OH}^{-} \]

6] **20 points** Consider a solution consisting of 0.11 M Pd\(^{2+}\) and 0.23 M Ag\(^{+}\) ions. If 1.0 \times 10\(^{-6}\)M represents quantitative removal of the ions is it possible to electro-separated the two ions? If so what would be the sequence of removal. Also describe the electrode potentials require for each step.

- Ag\(^{+}\) + e\(^{-}\) = Ag(s) \hspace{1cm} E^0 = 0.799 V
- Pd\(^{2+}\) + 2e\(^{-}\) = Pd(s) \hspace{1cm} E^0 = 0.987 V

7] **10 points** Fill in the blank with a correct answer.

a) Concentration polarization is a form of ________________________________

b) Charge-transfer polarization is also known as ________________________________
c) An amperostat is a ________________________________ device

d) Electrodes of the first kind are in equilibrium with a _______________ in contact with a __________________

____________________

e) Electro-separation of metal ions are accomplished by a controlled __________________ electrolysis.
1] **10 points** As described in the lecture notes, alpha-tocopherol (vitamin E) undergoes the following reaction.

![Chemical structures](image)

Assuming water is available in the electrolyte solution, draw a cyclic voltammogram that would be consistent with this mechanism.
2] **10 points** Discuss how taste polarography enhances the signal/background ratio relative to DC polarography.

3] **10 points** What are the advantages of capillary columns relative to packed columns in GC?

4] **10 points** Describe the principles that form the operational aspects of the electron capture detector. What type of species does it detect?

5] **10 points** Why are small stationary phase packing particles advantageous in the plate height characteristics in analytical LC column? What is the major drawback to decreasing the particle size?

6] **10 points** In the separation of nonpolar organics by gradient elution HPLC is best to begin with a polar or non-polar mobile phase when using a reversed phase C-18 column? Explain why.

7] **10 points** What is the suppressor column in IEC? Be sure to include why is it usually required and how it works in your discussion.

8] **10 points** Fill in the blank with a correct answer.

   a) The capacity factor may be manipulated by ___________________________ in gas chromatography.

   b) In temperature programming a GC separation of analytes it is best to ________________ the temperature so that the most volatile species elute first.

   c) An example of a universal detector in GC is the ______________________________

   d) An example of a universal detector in HPLC is the ______________________________

   e) The FID in GC is sensitive towards ______________________________
1] When compared to HPLC, SFC has many advantages. Describe three of these in detail.

2] Describe how a transmission filter allows only a narrow bandwidth of radiation to pass. How does the destructive interference and constructive reinforcement work in this device?

3] What is a major source of the broadening of m/e peaks in magnetic sector mass analyzer? How does a double focusing mass analyzer overcome this shortcoming of the magnetic sector M.S.?

4] Describe the electro-osmotic flow action of capillary electrophoresis. What are the retention times trend for cations, anions, and neutrals?

5] Why do 4-level lasers hold an advantage over 3-level lasers?

6] Draw a sketch and describe each functional aspect of an electron impact ion source.

7] Sketch block diagrams for spectroscopic instruments involved in absorption and fluorescence. Why are there differences between the two types of instruments?

8] Fill in the blanks with a correct answer.

a) The dark current is the ______________________ of a PMT output.

b) PMT’s require liquid nitrogen temperatures due to ________________________.

c) The Raman scattering involves a ______________________ in wavelength between the _______________ and scattered radiation.

d) The excited state lifetime of phosphorescence is _________________ than fluorescence.

e) The electron multiplier is similar to the ______________________ photon detector in design.
f) MALDI requires a ______________ that absorbs__________________ in order to efficiently ionize a sample molecule.

g) An example of a gas phase soft ionization technique is _________________________.

h) An example of a desorption method for M.S. is _________________________.