Chem 454 Name:_____
Instrumental Analysis Exam I
February 5, 1999 80 possible points

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.

$$E = const - 0.0592 \log[F^-]$$

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

$$Pd(OH)_2(s) + 2e^- = Pd(s) + 2OH^-$$

6] **20 points** Consider a solution consisting of 0.11 M Pd^{2+} and 0.23 M Ag^+ ions. If 1.0×10^{-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.

$Ag^+ + e^- = Ag(s)$ $E^0 = 0.799$ V
$Pd^{2+} + 2e^- = Pd(s)$ $E^0 = 0.987$ 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

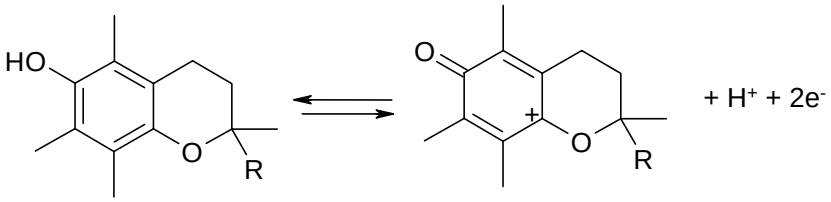
d) Electrodes of the first kind are in equilibrium with a _____ in contact with a

_____

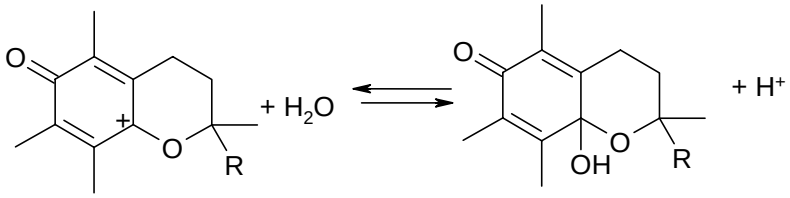
e) Electro-separation of metal ions are accomplished by a controlled _____ electrolysis.

Chem 454 Name:_____
Instrumental Analysis Exam 2
March 10, 1999 80 possible points

1] **10 points** As described in the lecture notes, alpha-tocopherol (vitamin E) undergoes the following reaction.

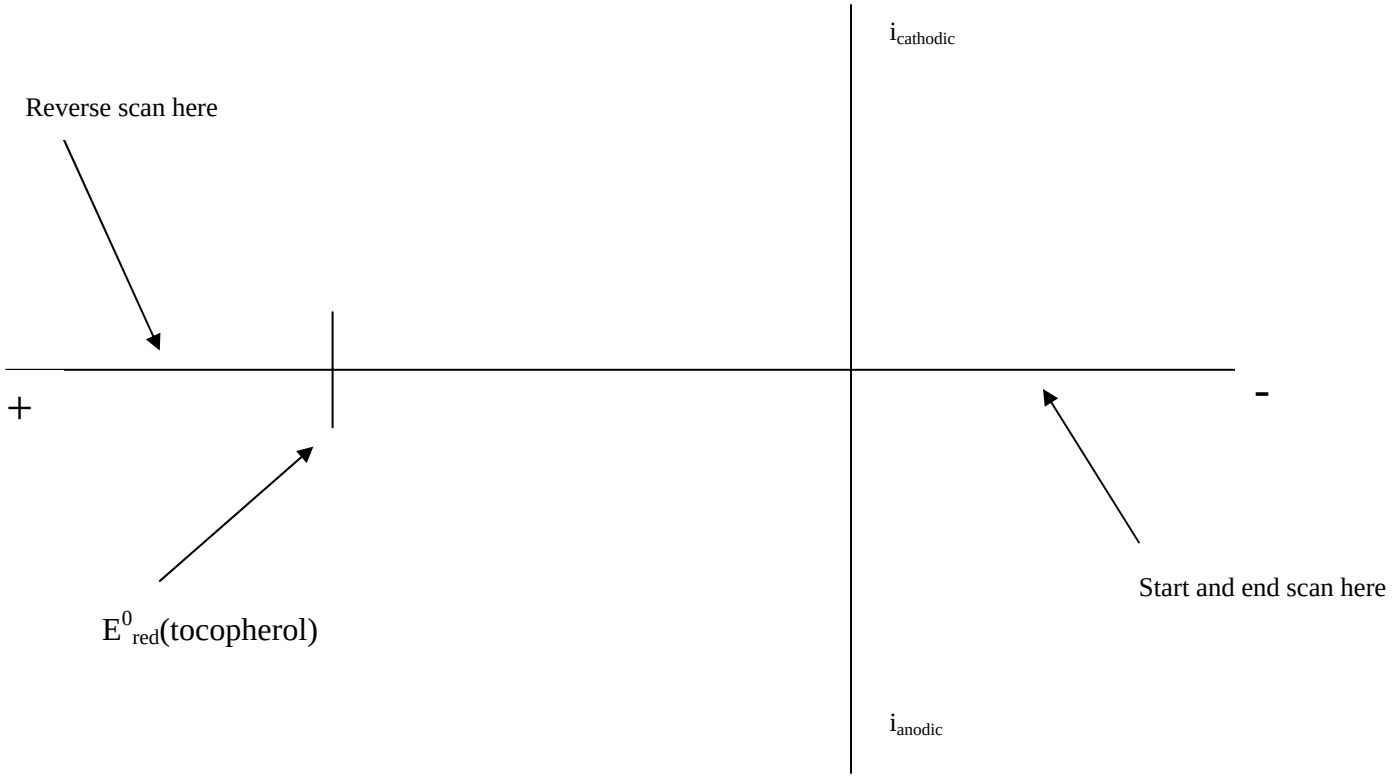


Compound 1 Compound 2



Compound 2 Compound 3

Assuming water is available in the electrolyte solution, draw a cyclic voltammogram that would be consistent with this mechanism.



2] **10 points** Discuss how tast 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 _____

Chem 454 Name:_____

Instrumental Analysis

Exam 3

April 19, 1999

Each Question is worth 10 points for a total of 80 possible points

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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 _____.