

Name \_\_\_\_\_

Chemistry 426  
Instrumental Analysis

Exam 2

2002 June 13

Norm Dovichi

Exams are closed-book.

You are allowed to use a sheet of paper during the exam that you have marked as you wish beforehand.

This exam has 6 pages.

1. (5 points each for a total of 25 points)
  - i. Consider the analysis of arctic air samples for parts-per-billion of Freon 11 ( $\text{CCl}_3\text{F}$ ), a chemical that can damage the earth's ozone layer. If you had a gas chromatograph, which of the following detectors would be most useful for the analysis of the air samples?
    - a. electron capture detector
    - b. thermal conductivity detector
    - c. flame ionization detector
    - f. all of the above could be used for analysis of parts-per-billion of  $\text{CCl}_3\text{F}$  in air
    - e. it is not possible to detect parts-per-billion of  $\text{CCl}_3\text{F}$  with any of these detectors
  - ii. What is the primary advantage of temperature programmed gas chromatography?
    - a. It allows separation of materials of substances with similar vapor pressures that otherwise would elute at the same time.
    - b. It facilitates the analysis of mixtures of substances with widely differing vapor pressures
    - c. It increases the number of theoretical plates for the column
    - d. It facilitates the use of the electron capture detector
    - e. None of the above
  - iii. Consider the effect of mass diffusion on band broadening in a gas chromatographic column. How does this effect depend upon the flow rate of the mobile phase?
    - a. The height equivalent to a theoretical plate decreases with an increase in the flow rate of the mobile phase.
    - b. The height equivalent to a theoretical plate increases with an increase in the flow rate of the mobile phase.
    - c. The height equivalent to a theoretical plate is independent of the flow rate of the mobile phase.
    - d. There is insufficient information to answer the question
  - iv. Which of the following detectors is commonly used in gas chromatography?
    - a. Fluorescence
    - b. Refractive index
    - c. Atomic absorbance
    - d. Flame ionization
    - e. All of the above are commonly used in gas chromatography
    - f. None of the above is commonly used in gas chromatography
  - v. Consider the effect of eddy diffusion on band-broadening in a chromatographic column. How does this effect depend upon the flow rate of the mobile phase?
    - a. the height equivalent to a theoretical plate increases with an increase in the flow rate of the mobile phase.
    - b. the height equivalent to a theoretical plate decreases with an increase in the flow rate of the mobile phase.
    - c. the height equivalent to a theoretical plate is independent of flow rate of the mobile phase.
    - d. There is insufficient information to answer the question

2. (25 points) A mixture of organic acids was separated by reversed-phase liquid chromatography. The data obtained for the chart recording were as follows: For acid A, the peak appeared 22 cm after injection with a full width at baseline of 8 cm. For acid B, the peak appeared 34 cm after injection with a full width at baseline of 6 cm. A non-retained compound generated a peak that appeared 4 cm after injection. The mobile phase flow rate in the column was 20 mL/min. The recorder chart speed was 5 cm per minute. Last, the column was 32 cm in length. Calculate:
- The adjusted retention time for peak A
  - The capacity factor for peak A
  - The resolution of compounds A and B.
  - The number of theoretical plates in the column for acid B.
  - The height equivalent to a theoretical plate for acid B

3. a. (5 points) Anodic stripping voltammetry is used for analysis of trace metals. Briefly explain how the mercury working electrode is produced for anodic stripping voltammetry.

b. (5 points) In anodic stripping voltammetry, what electrical quantity is varied during the measurement?

c. (5 points) In anodic stripping voltammetry, what electrical quantity is measured?

d. (5 points) What electrochemical processes occur with an analyte at the mercury working electrode during preconcentration?

e. (5 points) What electrochemical processes occur with an analyte at the mercury working electrode during stripping?

4. Draw a block diagram for the following instruments with the correct orientation of the components (continued on next page).

(5 points). The main components in a magnetic sector mass spectrometer.

(5 points) Draw a diagram and label the components in a saturated silver-silver chloride reference electrode.

4.(continued)

(5 points) A flow injection analysis system to automate the Konig reaction, which produces a highly colored product from cyanide.

(5 points) A split injector used in gas chromatography.

(5 points) A liquid chromatography injector with an injection loop

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  - ii. What is the primary advantage of temperature programmed gas chromatography?
    - a. It allows separation of materials of substances with similar vapor pressures that otherwise would elute at the same time.
    - b. **It facilitates the analysis of mixtures of substances with widely differing vapor pressures**
    - c. It increases the number of theoretical plates for the column
    - d. It facilitates the use of the electron capture detector
    - e. None of the above
  - iii. Consider the effect of mass diffusion on band broadening in a gas chromatographic column. How does this effect depend upon the flow rate of the mobile phase?
    - a. **The height equivalent to a theoretical plate decreases with an increase in the flow rate of the mobile phase.**
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2. (25 points) A mixture of organic acids was separated by reversed-phase liquid chromatography. The data obtained for the chart recording were as follows: For acid A, the peak appeared 22 cm after injection with a full width at baseline of 8 cm. For acid B, the peak appeared 34 cm after injection with a full width at baseline of 6 cm. A non-retained compound generated a peak that appeared 4 cm after injection. The mobile phase flow rate in the column was 20 mL/min. The recorder chart speed was 5 cm per minute. Last, the column was 32 cm in length. Calculate:
- The adjusted retention time for peak A  
 **$t' = (22 - 4) \text{ cm} \times 1 \text{ min} / 5 \text{ cm} = 3.6 \text{ min}$**
  - The capacity factor for peak A  
 **$\text{Capacity factor} = k' = (V_R - V_M) / V_M = (22 - 4) / 4 = 4.5$**
  - The resolution of compounds A and B.  
 **$\text{Resolution} = R = (V_2 - V_1) / [0.5 * (W_2 + W_1)] = (34 - 22) / [0.5 * (8 + 6)] = 1.7$**
  - The number of theoretical plates in the column for acid B.  
 **$\text{Number of theoretical plates} = n = 16 (V_R / W)^2 = 514$**
  - The height equivalent to a theoretical plate for acid B  
 **$h = 32 \text{ cm} / 514 = 0.062 \text{ mm}$**

3. a. (5 points) Anodic stripping voltammetry is used for analysis of trace metals. Briefly explain how the mercury working electrode is produced for anodic stripping voltammetry.

**Hg<sup>2+</sup> in the aqueous solution is reduced to Hg<sup>0</sup> metal, coating the working electrode.**

b. (5 points) In anodic stripping voltammetry, what electrical quantity is varied during the measurement?

**Voltage vs SCE**

c. (5 points) In anodic stripping voltammetry, what electrical quantity is measured?

**Current**

d. (5 points) What electrochemical processes occur with an analyte at the mercury working electrode during preconcentration?

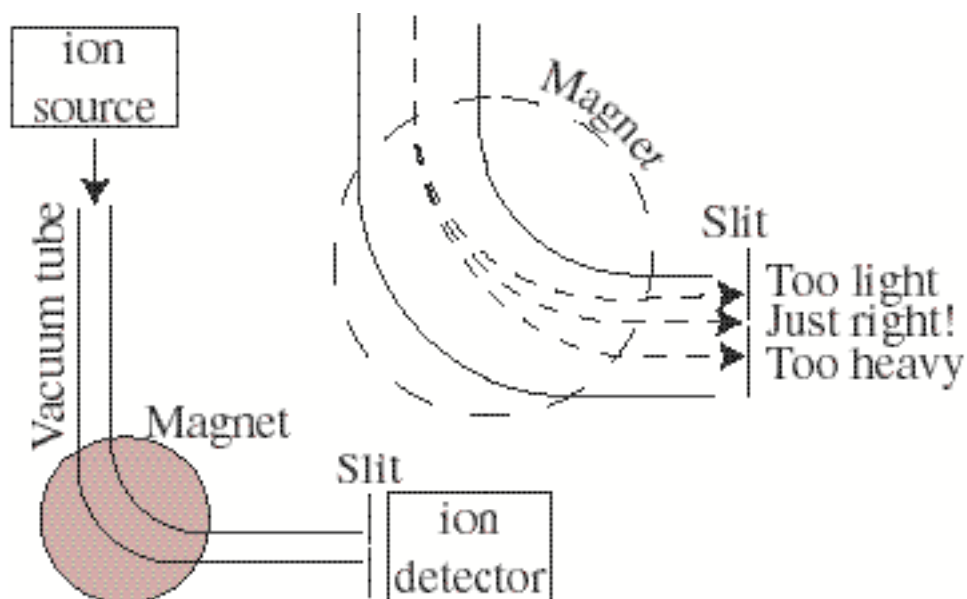
**Cation metal analytes are reduced to neutral metal and absorbed into the Hg<sup>0</sup>.**

e. (5 points) What electrochemical processes occur with an analyte at the mercury working electrode during stripping?

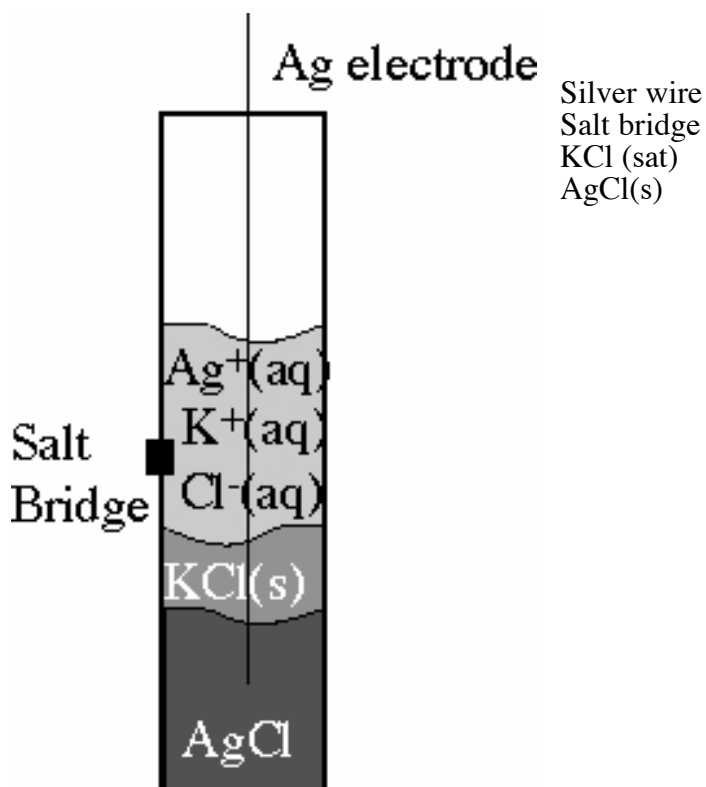
**Neutral metals are oxidized, producing current.**

4. Draw a block diagram for the following instruments with the correct orientation of the components (continued on next page).

(5 points). The main components in a magnetic sector mass spectrometer.

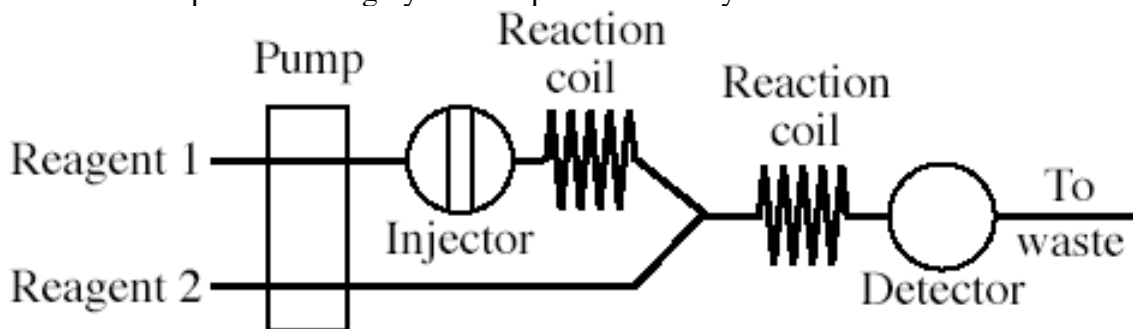


(5 points) 20. (8 points) Draw a diagram and label the components in a saturated silver-silver chloride reference electrode.

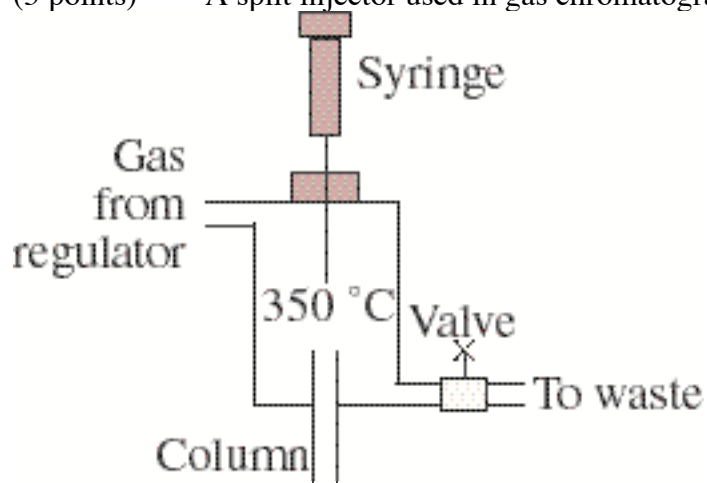


4.(continued)

(5 points) A flow injection analysis system to automate the Konig reaction, which produces a highly colored product from cyanide.



(5 points) A split injector used in gas chromatography.



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