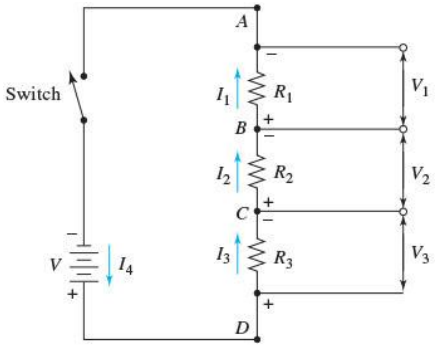
Analytical Chemistry II – FINAL EXAM

- **It is not allowed to put any additional items (*e.g.* cell phone, calculator) on the bench.**
- **Sign the exam paper, and sign the attendance list at the beginning of the exam.**
- **You have to hand in the exam paper before leaving the classroom.**
- **The exam consists of two parts (I and II).**
- **You can answer in English or Chinese language.**
- **Do not use pencil; use pen.**
- **If you cheat (*e.g.* use cell phone), you will get 0 points from this exam.**

I. Choose the most accurate answer:

Circle the letter corresponding to your choice, or write the answer letter next to the question.
(maximum: 15 × 4 = 60 points)

1. Which formula relates V_1 with V, R_1, R_2, and R_3 in the circuit below?



a) $V_1 = V(R_1 + R_2 + R_3)$
b) $V_1 = V\left(\frac{R_2+R_3}{R_1+R_2+R_3}\right)$
c) $V_1 = V\left(\frac{R_1+R_2+R_3}{R_1}\right)$
d) $V_1 = V\left(\frac{R_1+R_2+R_3}{R_2+R_3}\right)$
e) $V_1 = V\left(\frac{R_1}{R_1+R_2+R_3}\right)$

2. What is the definition of absorbance (A)? (P_0 – power of incident light, P – power of transmitted light)

a) $A = \log\frac{P_0}{P}$
b) $A = \log\frac{P}{P_0}$
c) $A = \frac{P_0}{P}$
d) $A = \frac{P}{P_0}$
e) $A = P - P_0$

3. What is the correct equation for Beer's law?
(*A* – absorbance, ε – molar absorptivity, *b* – optical pathlength, *c* – analyte concentration)
   a) $A = \log(\varepsilon bc)$
   b) $A = \frac{\varepsilon}{bc}$
   c) $A = \frac{\varepsilon b}{c}$
   d) $A = \varepsilon bc$
   e) $A = \varepsilon b \sin(c)$

4. What factors contribute to the broadening of spectral line widths in atomic spectrometry? Line broadening due to uncertainty effect, pressure (collisional) broadening, and...
   a) impedance broadening
   b) Doppler broadening
   c) Tswett broadening
   d) Fenn broadening
   e) Van Deemter broadening

5. In flame atomization atomic absorption spectrometry, how do we call a cation that preferentially reacts with a species that would otherwise react with the analyte to cause a chemical interference?
   a) protective agent
   b) releasing agent
   c) radiation buffer
   d) ionization suppressor
   e) ionization promotor

6. Which technique would you apply to study distribution of iron on the surface of silicon?
   a) HPLC
   b) LA-ICP-MS
   c) FIA-ICP-MS
   d) ESI-MS
   e) UV-Vis absorption spectrometry

7. Which element can normally be found in atmospheric pressure chemical ionization source?
   a) heated filament
   b) Taylor cone
   c) laser
   d) corona electrode
   e) ion trap

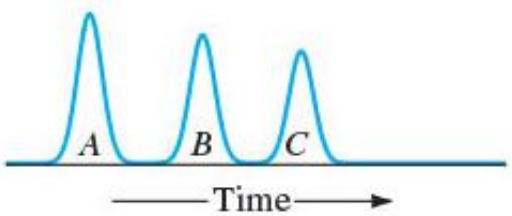
8. What is the characteristic feature of nanoelectrospray ionization?
   a) very high limits of detection (in terms of total analyte mass)
   b) very high ionization efficiencies
   c) very long desolvation times
   d) use of very high pressures (several atmospheres)
   e) compatibility with inductively coupled plasma source

9. Which technique is particularly useful in analysis of samples that contain low-molecular-weight compounds whose boiling points differ significantly?
   a) GC with temperature programming
   b) thin-layer chromatography
   c) ion-exchange liquid chromatography
   d) reversed-phase liquid chromatography
   e) UV-Vis absorption spectrometry

10. Which mobile phase, commonly used in gas chromatography, gives the lowest theoretical plate height at high average linear velocities?
    a) He
    b) N_2
    c) H_2
    d) H_2O
    e) CH_3OH

11. In gradient elution,
    a) temperature of the column is decreased
    b) the ratio of solvents is varied in a preprogrammed way
    c) electric field gradient is applied to the column
    d) total flow rate of the mobile phase is decreased
    e) all the compounds have the same retention time

12. The chromatogram shows elution of compounds *A*, *B*, and *C* from a reversed-phase chromatographic column using a high-polarity mobile phase. What is the polarity order of the three compounds (from high to low)?



    a) $A > C > B$
    b) $B > A > C$
    c) $A = B = C$
    d) $A > B > C$
    e) $C > B > A$

13. Some UV-Vis detectors for high-performance liquid chromatography incorporate Z-shaped flow through cuvette. Why?
    a) to improve chromatographic resolution
    b) to improve flow profile
    c) to decrease chromatographic peak width
    d) to enable coupling of high-performance liquid chromatography with mass spectrometry
    e) to maintain large optical pathlength

14. What is the characteristic feature of differential refractive index detector for high-performance liquid chromatography?
    a) It is unaffected by flow rate.
    b) It is unaffected by temperature changes.
    c) It has high sensitivity.
    d) It is compatible with gradient elution methods.
    e) It is only applicable to detection of proteins with different charge states.

15. Carbon dioxide is a common component of mobile phases in supercritical fluid chromatography. What is its critical temperature?
    a) 31.3 °C
    b) 132.5 °C
    c) 152.0 °C
    d) 201.2 °C
    e) 310.5 °C

II. Answer the following questions: (maximum: 4 × 10 = 40 points)
- Indicate the question letter before answering.
- Provide a complete, accurate, clear, high-quality answer to every part of each task.
- Adhere to the answer length limits. Up to 3 points may be deducted per question for exceeding the length limits.
- Handwriting must be clear.
- Schemes and graphs must be labeled.

A. Explain the concept of electron capture detector. Draw detailed scheme of electron capture detector.
- answer length limit: 150 words, 1 figure with labels

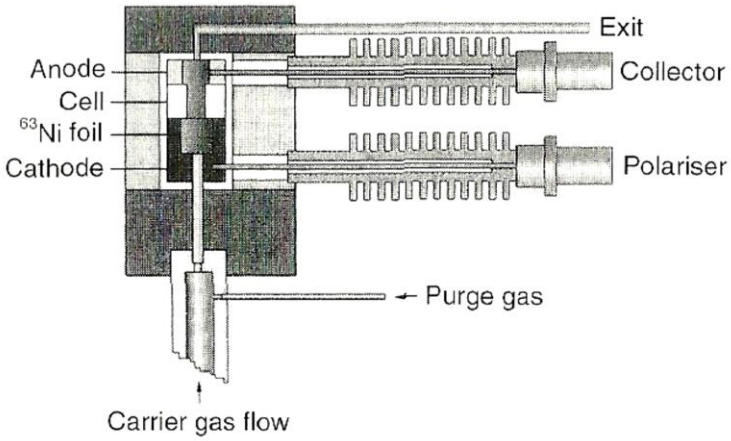
B. Draw detailed scheme of typical apparatus for high-performance liquid chromatography.
- answer length limit: 1 figure with labels

C. Explain the concept of supercritical fluid chromatography. Draw detailed scheme of supercritical fluid chromatography system.
- answer length limit: 150 words, 1 figure with labels

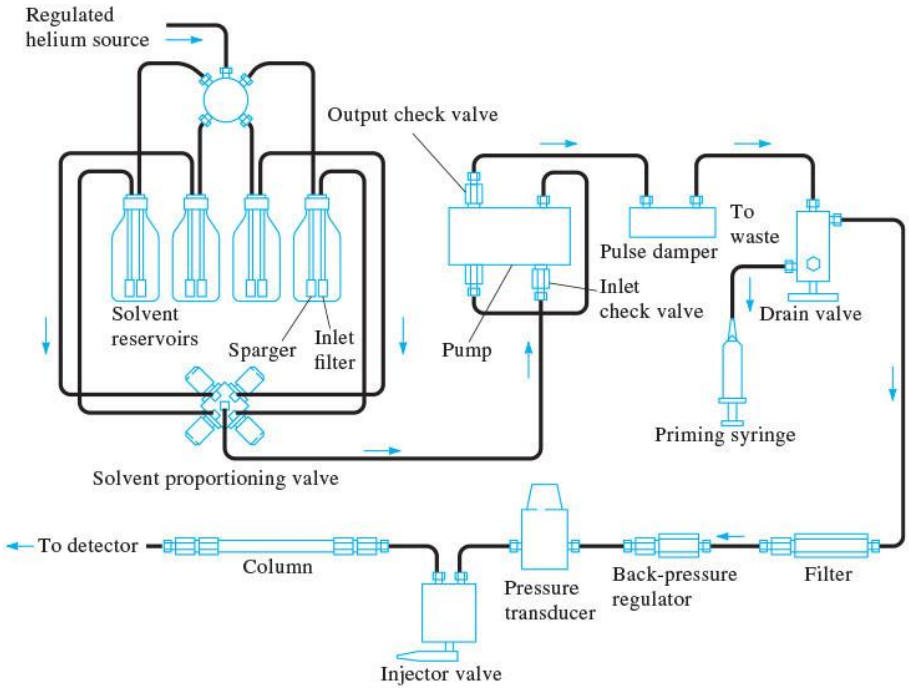
D. Explain the concept of capillary electrophoresis. Draw detailed scheme of capillary electrophoresis system.
- answer length limit: 150 words, 1 figure with labels

ANSWERS: (You can also use the reverse sides.)

A.
- Electron capture detector selectively responds to halogen-containing compounds (pesticides, PCBs).
- The sample eluate from a column is passed over a radioactive β emitter.
- An electron causes ionization of the carrier gas, and the production of a burst of electrons.
- The electric current decreases in the presence of organic molecules containing electronegative functional groups that tend to capture electrons.
- Sensitive to: halogens, peroxides, nitro groups.
- Insensitive to: amines, alcohols, hydrocarbons.

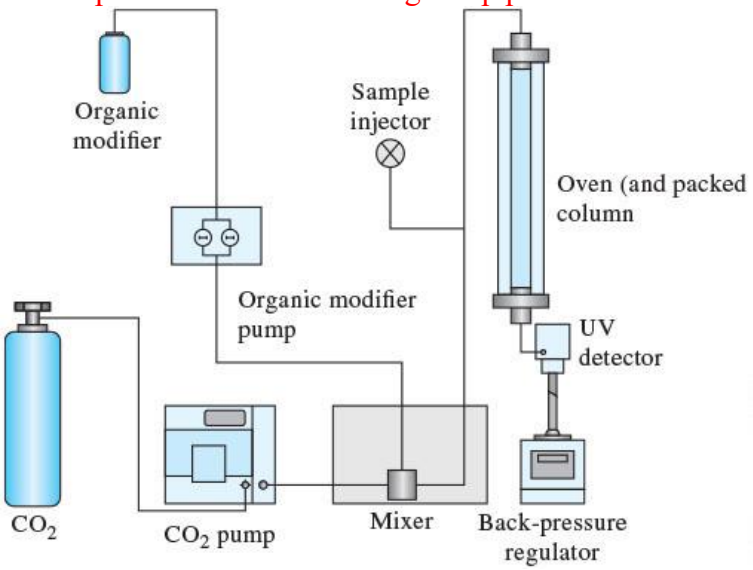


B.



C.

- Supercritical fluid chromatography is a hybrid of gas chromatography and liquid chromatography.
- Packed columns and open tubular columns can be used.
- Nonvolatile or thermally unstable compounds can be separated.
- Compared to high-performance liquid chromatography, the supercritical fluid chromatography system needs to contain additional elements: oven, back-pressure regulator.
- Organic modifiers are added to the mobile phase.
- Various detectors can be implemented.
- Low consumption of organic solvents (friendly to environment).
- When a molecule dissolves in supercritical medium, the process resembles volatilization but at a much lower temperature than in gas chromatography.
- At a high average linear velocity of mobile phase, plate height is lower in supercritical fluid chromatography than in high-performance liquid chromatography.
- Thus, one can perform fast separations while obtaining sharp peaks.



D.

- In capillary electrophoresis, separations occur in an electrolyte-filled open tubular capillary ($L \leq 100$ cm, ID 20-100 μm) under the influence of an electric field.
- A small (~ nL) sample plug is first injected into the capillary (inlet end).
- Electrolyte vessels are placed at the inlet and outlet of the capillary.
- High voltage (5-30 kV) is applied across the capillary.
- Solutes migrate from the inlet toward the outlet due to their own electrophoretic mobilities as well as electroosmotic flow.
- They are detected near the outlet of the capillary.