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Analytical Chemistry II – FINAL EXAM

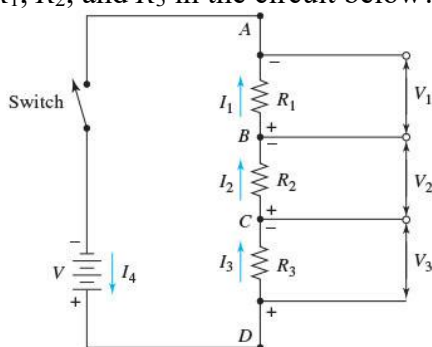
- It is not allowed to put any additional items (e.g. cell phone, calculator) on the bench.
- Sign the exam paper, and sign the attendance list at the beginning of the exam.
- You have to hand in the exam paper before leaving the classroom.
- The exam consists of two parts (I and II).
- You can answer in English or Chinese language.
- Do not use pencil; use pen.
- If you cheat, 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: $30 \times 3 = 90$ 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) $V_1 = V \left(\frac{R_1}{R_1 + R_2 + R_3} \right)$
 e) $V_1 = V \left(\frac{R_1 + R_2 + R_3}{R_2 + R_3} \right)$
2. Which electronic element conducts electric current in *forward bias* but it has low conductance in *reverse bias*?
- a) resistor
 b) potentiometer
 c) capacitor
 d) oscilloscope
 e) diode
3. Which wavelength range of electromagnetic radiation corresponds to vacuum ultraviolet light?
- a) 10-180 nm
 b) 180-400 nm
 c) 400-780 nm
 d) 0.78-300 μm
 e) 0.6-10 m

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

a) $A = \log \frac{P}{P_0}$

b) $A = \log \frac{P_0}{P}$

c) $A = \frac{P_0}{P}$

d) $A = \frac{P}{P_0}$

e) $A = P - P_0$

5. What is the correct equation for Beer's law?

(A – absorbance, ϵ – molar absorptivity, b – optical pathlength, c – analyte concentration)

a) $A = \log(\epsilon bc)$

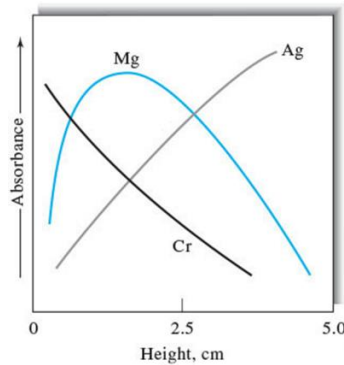
b) $A = \epsilon bc$

c) $A = \frac{\epsilon}{bc}$

d) $A = \frac{\epsilon b}{c}$

e) $A = \epsilon b \sin(c)$

6. The graph shows absorbances of three elements in relation to measurement height in flame atomization atomic absorption spectrometry. Why does the Cr absorbance decrease with increasing measurement height?



a) because of increasing dissociation of Cr compounds

b) because Cr shows high atomization efficiency

c) because Cr easily reacts with hydrogen in the flame

d) because Cr does not easily form oxides

e) because Cr forms very stable oxides

7. Which element can readily be analyzed by cold-vapor atomization atomic absorption spectrometry?

a) sodium

b) cadmium

c) mercury

d) silicon

e) uranium

8. 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) Tswett broadening

c) Fenn broadening

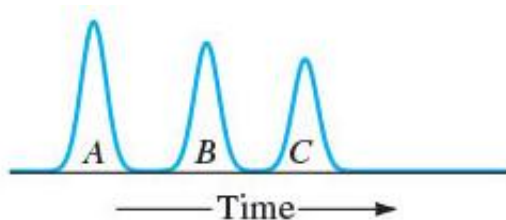
d) Doppler broadening

e) Van Deemter broadening

9. Addition of potassium to sample can improve sensitivity in analysis of strontium by flame atomization atomic absorption spectrometry. In this case, potassium is:
- a) ionization suppressor
 - b) releasing agent
 - c) protective agent
 - d) radiation buffer
 - e) solute volatilization interferent
10. 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) ionization suppressor
 - b) ionization promotor
 - c) protective agent
 - d) releasing agent
 - e) radiation buffer
11. The zone in inductively coupled plasma source used for atomic emission spectroscopic measurements has a temperature of:
- a) ~ 300 K
 - b) ~ 600 K
 - c) ~ 1000 K
 - d) ~ 6000 K
 - e) ~ 120000 K
12. Which device is used as a source of continuum UV radiation in molecular spectroscopy?
- a) hollow-cathode lamp
 - b) globar
 - c) photoconductor
 - d) deuterium lamp
 - e) phototube
13. Which of the following is the characteristic of (photo)diode array detector?
- a) It cannot be used in the visible region.
 - b) It enables recording full spectrum in a very short time.
 - c) It has very slow response.
 - d) It is much more sensitive to light than photomultiplier tube.
 - e) It emits light at one wavelength only.
14. How do we call the process, in which a molecule changes from a higher electronic state to an upper vibrational level of a lower electronic state in which the vibrational energy is great enough to rupture the bond?
- a) intersystem crossing
 - b) phosphorescence
 - c) fluorescence
 - d) predissociation
 - e) vibrational relaxation
15. Which wavelength region is called middle infrared?
- a) < 0.78 μm
 - b) 0.78-2.5 μm
 - c) 2.5-50 μm
 - d) 50-1000 μm
 - e) > 1000 μm

16. Which device is the key component in modern infrared spectrometers that enable recording infrared absorption spectra?
- a) prism
 - b) Michelson interferometer**
 - c) diffraction grating
 - d) diffraction wedge
 - e) ion source
17. What is the main function of reflectron in time-of-flight mass analyzer?
- a) to improve vacuum
 - b) to reflect laser light
 - c) to create ions
 - d) to compensate for mass dispersion
 - e) to compensate for kinetic energy dispersion**
18. Which element can normally be found in atmospheric pressure chemical ionization source?
- a) heated filament
 - b) corona electrode**
 - c) Taylor cone
 - d) laser
 - e) ion trap
19. Multiple charging of proteins is most prominent in
- a) electron ionization
 - b) electrospray ionization**
 - c) chemical ionization
 - d) matrix-assisted laser desorption/ionization
 - e) inductively coupled plasma ionization
20. Which technique would you apply to study distribution of iron on the surface of silicon?
- a) HPLC
 - b) FIA-ICP-MS
 - c) LA-ICP-MS**
 - d) ESI-MS
 - e) UV-Vis absorption spectrometry
21. Why do we need chromatographic separations?
- a) to decrease selectivity
 - b) to increase the required sample volume
 - c) to speed up analysis
 - d) to miniaturize analytical systems
 - e) to increase selectivity and reduce interferences**
22. Which technique is particularly useful in analysis of samples that contain low-molecular-weight compounds whose boiling points differ significantly?
- a) thin-layer chromatography
 - b) GC with temperature programming**
 - c) ion-exchange liquid chromatography
 - d) reversed-phase liquid chromatography
 - e) UV-Vis absorption spectrometry

23. What is the typical inside diameter of fused silica wall-coated open tubular column used in gas chromatography?
- < 0.1 μm
 - $\sim 0.5\text{-}5\ \mu\text{m}$
 - $\sim 10\text{-}50\ \mu\text{m}$
 - $\sim 0.1\text{-}0.3\ \text{mm}$
 - > 1 mm
24. Which mobile phase, commonly used in gas chromatography, gives the lowest theoretical plate height at high average linear velocities?
- N_2
 - H_2
 - He
 - H_2O
 - CH_3OH
25. Which statement about flame ionization detector, used in gas chromatography, is true?
- It is non-destructive.
 - It selectively responds to halogen-containing compounds.
 - It responds to the number of carbon atoms entering the detector per unit of time.
 - Analytes have to be in ionic form before they enter the detector (*i.e.* already in the column).
 - It relies on measurement of electrical resistance of a heated wire.
26. Which transducer/detector uses radioactive β emitter?
- electron capture detector
 - flame ionization detector
 - atomic emission detector
 - bolometer
 - thermocouple
27. 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)?



- $A > B > C$
 - $C > B > A$
 - $A > C > B$
 - $B > A > C$
 - $A = B = C$
28. Stationary phase in HILIC chromatography has to contain
- polar moieties
 - non-polar moieties
 - polystyrene
 - polyimide
 - plate height

29. Which element is often placed between ion-exchange chromatographic column and conductometric detector?
- ion source
 - ion trap
 - micromembrane suppressor
 - reversed-phase chromatographic column
 - ion-exclusion chromatographic column
30. Why can some anions be analyzed by conventional capillary zone electrophoresis method, in which an on-capillary detector is positioned at the capillary outlet located in the proximity of cathode?
- because anions are attracted by cathode
 - because anions are not affected by cathode
 - because anions are pushed by hydrodynamic flow toward cathode
 - because anions are pushed by parabolic flow toward cathode
 - because anions are pushed by electroosmotic flow toward cathode

II. Answer the following questions: (maximum: $2 \times 5 = 10$ points)

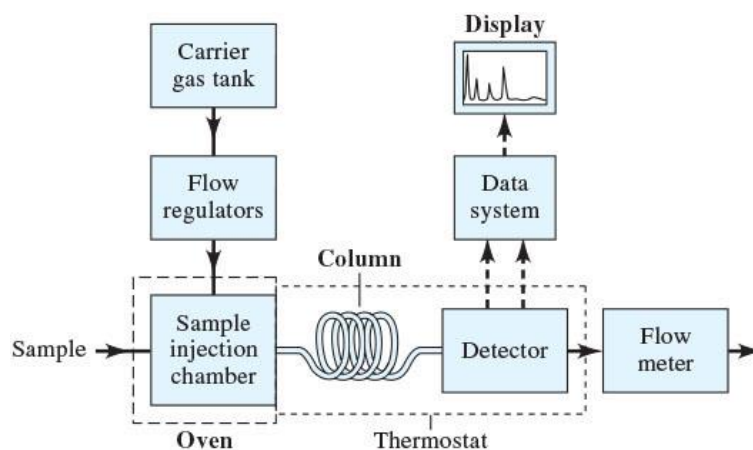
- Indicate the question letter before answering.
- Provide a complete, accurate, clear, high-quality answer to every part of each task.
- Adhere to the answer length limits. Up to 3 points may be deducted per question for exceeding the length limits.
- Handwriting must be clear.
- Schemes and graphs must be labeled.

A. Draw detailed block diagram of gas chromatograph. Explain the operational principle of this instrument.
- answer length limit: 1 figure with labels, 150 words

B. Draw detailed scheme of (photo)diode array UV-Vis absorption detector for HPLC. Explain the operational principle of this device.
- answer length limit: 1 figure with labels, 150 words

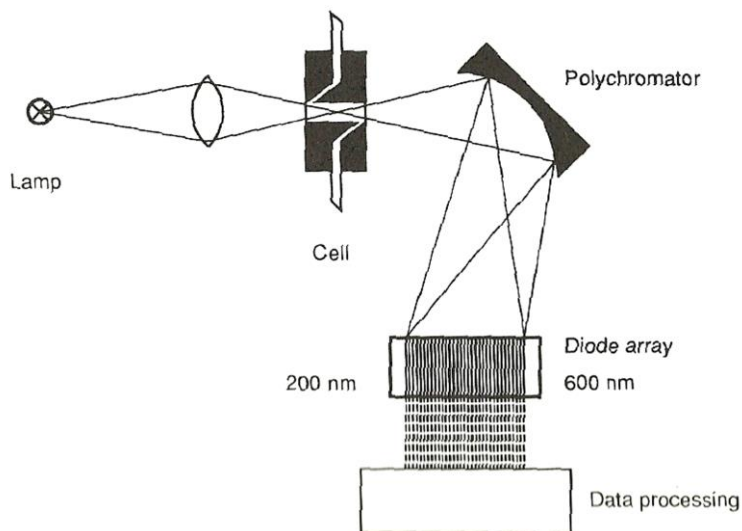
ANSWERS:

A.



- In gas chromatograph, a chemically inert carrier gas (helium, argon, nitrogen, hydrogen) is applied at a pressure of 10-50 psi to move gaseous analytes along the column.
- The flow of the gas is regulated by a flow regulator. Typical flow rates: 25-150 mL/min (packed columns); 1-25 mL/min (capillary columns).
- Sample is injected to the column inlet.
- Sample injection chamber and column are thermostatted.
- Separated analytes are detected by a detector at the end of the column.
- Signal from the detector is recorded by data system, processed, and chromatogram is displayed.

B.



- Eluent is delivered to a channel (flow-through cuvette) with two windows.
- Light (from a light source) passes through the channel.
- The polychromatic light is split to constituent wavelengths.
- The light beams with different wavelengths are relayed onto (photo)diode array.
- Typically, a Z-shaped flow-through cuvette is used to maximize optical pathlength.
- The volume of the cell should be as small as possible to minimize extracolumn band broadening.