

ANALYTICAL & LAB INSTRUMENTS

REVISION SHEET (2) - Solved

Luminescence and Atomic Spectrometry

1. Atomic spectra are observed as sharp lines, because
 - a) Covalent bonds participate in spectral broadening.
 - b) The individual constituent atoms in the molecule produce spectral bands.
 - c) Atoms cannot vibrate or rotate as molecules do.
 - d) Energy transitions to the ground state for the individual molecular atoms are excessive compared to simple atoms.

2. Atomic spectrometry is applicable to most gas phase elements
 - a) Because of their extraordinary sensitivity (parts per trillion)
 - b) Because of the simplicity of the instrument
 - c) Because of the low cost of the instrument
 - d) Because of the reproducible results

3. In atomic spectrometry
 - a) The element to be analyzed is sequestered using an electric field
 - b) The element is isolated by heating the sample in a furnace
 - c) The element is extracted using a high power laser
 - d) The element is separated by vaporizing the sample

4. Atoms of most elements are in the ground state at temperatures below (3000 K/ 5000 K/ 7000 K/ 9000 K).

5. Atomic Absorption Spectroscopy (AAS) is a very easy and reliable technique yielding simple absorption spectra because
 - a) Spectral lines are very sharp
 - b) Absorption is almost independent of the temperature of the vapor produced
 - c) Detection sensitivities are excessively high
 - d) The method is overly selective

6. The radiation source of choice in a flame AAS is a hollow cathode lamp (HCL) because
 - a) It is cheap
 - b) Its mass production is very promising
 - c) It has a higher energy output
 - d) Its output spectrum is considerably narrow

7. In AAS, the function of the atomizer is to
- Decompose the sample into its individual constituent atoms
 - Produce as many free atoms as possible for the purpose of sensitivity
 - To free the sample from all impurities
 - Press the sample into the flame
8. What are three considerations when choosing the atomizer?
- type of sample (solid, liquid or gas)
 - concentration of the sample
 - size of the sample
9. Flame atomic absorption spectrometry (FAAS) is used primarily for **(solids/ liquids/ gases)**.
10. In AAS, the function of the nebulizer is to
- Decompose the sample into its individual constituent atoms
 - Produce as many free atoms as possible for the purpose of sensitivity
 - Convert the sample into an aerosol
 - Mix the sample with the combustible gases
11. What are the two commonly used flames in FAAS?
- air–acetylene mixture
 - nitrous oxide–acetylene mixture
27. The most common AAS discriminators are **(filters/ prisms/ diffraction gratings)**.
28. In conventional AAS the most commonly used detector is the **(PMT/ photoelectric cell/ PDA/ CCD)**, whereas in high resolution AAS the most commonly used detector is the **(PMT/ photoelectric cell/ PDA/ CCD)**.
29. In AAS the transmittance signal is converted into an absorbance one by means of **(a filter/ an amplifier/ an A/D converter/ a transducer)**.
30. In AAS qualitative information is obtained from the **(flame color/ hollow cathode lamp element/ absorbed radiation wavelength/ intensity of absorbed radiation)**. Quantitative information is obtained from the **(flame color/ hollow cathode lamp element/ absorbed radiation wavelength/ intensity of absorbed radiation)**.

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REVISION SHEET (3) - **Solved** Chromatography

1. All chromatographic techniques depend on the separation of two compounds
 - a) According to their boiling points
 - b) According to their electrostatic mobilities
 - c) **Between two immiscible phases**
 - d) According to their molecular weights
2. The stationary phase in a chromatograph is fixed in a (*beaker/ **column**/ tube/ cuvette*) or on (*a sheet/ gel/ **a solid surface**/ an aluminum foil*).
3. Define elution.
It is the appearance of the different solute compounds at the end of the stationary phase at different time instances, the compound appearing first being that with the smallest distribution into the stationary phase.
4. As the separated compounds appear at the end of the stationary phase they are
 - a. Disposed of
 - b. **Detected**
 - c. Recycled
 - d. Spectrophotometrically analyzed
5. The role of the detector is to **identify** and **quantitatively** assess the (*separated **compounds**/ impurities/ temperature of the stationary phase*).
6. In gas chromatography (GC) the sample
 - a. Is directly injected into the column
 - b. **Is vaporized and injected into the column**
 - c. Is diluted and injected into the column
 - d. Impregnates the column
7. In GC the sample is transported through the column
 - a. **By the flow of an inert gas**
 - b. By means of its inertia
 - c. By the flow of oxygen
 - d. By the flow of hydrogen
8. The rate at which the molecules progress along the column depends on their (*molecular weight/ molecular diameter/ **strength of adsorption**/ charge if any*). This in turn depends on the **type of molecule** and on the **column chosen**.
9. In a GC substances are identified by two factors. Name them
 - **the order in which they emerge from the column**
 - **the residence time of the analyte in the column**

10. In a GC temperature should be carefully controlled, because molecular adsorption and the rate of progression along the column depend on the temperature.
11. In a GC the most universally applicable detector is the
- Flame ionization
 - Thermal conductivity
 - Photoionization
 - Mass spectrometric
12. In a GC the most widely used detector is the
- Flame ionization
 - Thermal conductivity
 - Photoionization
 - Mass spectrometric
13. In a GC pressure and flow regulators may be employed. Explain.
The flow rate must be controlled and constant, hence pressure and flow regulators may be employed.
14. Name three typical carrier gases used.
- He
 - Ar
 - N₂
 - CO₂
15. The choice of the carrier gas is determined by
- The column design
 - The molecular weights of the analytes
 - The detector used
 - The sample injection method
16. Name the two injection techniques used.
- Split injection
 - Splitless injection
17. Compare between both injection techniques regarding:
- Sample concentration
 - Sample introduction into the column
 - Injector temperature
 - Sensitivity
 - Resolution

	Split Injection	Splitless Injection
Sample concentration	Concentrated	Dilute
Sample introduction into the column	Small portion of sample volume	Most of sample volume
Injector temperature	High	Low
Sensitivity	Not an issue	Most important
Resolution	Most important	Immaterial

18. Ramping/gradient control of temperature (temperature programming) during separation may be required to allow
- Elution of compounds with a wide range of molecular weights
 - Elution of compounds with a wide range of boiling points
 - Speeding up of all compounds inside the column
 - Better resolution
19. Open tubular columns may provide
- better resolution
 - more rapid separation
 - greater sensitivity
20. The three main types of open tubular columns are
- wall-coated
 - support-coated
 - porous layer
21. The problem with open tubular columns is that
- They are very fragile
 - They exhibit remarkable maintenance issues
 - They are prohibitively expensive
 - They cannot accommodate high sample amounts
22. GC capillaries may be (*1 m/ 10 m/ 100 m/ 1 km*) long. They have an internal diameter in the range 0.1 to 1 mm.
23. A flame ionization detector uses a flame to
- Ionize the organo-metallic compounds in the sample
 - Decompose the carbon-containing sample molecules
 - Melt all solid traces in the sample
 - Speed-up the detection process

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REVISION SHEET (4) - Solved Electrophoresis

4. Improvements in electrophoresis made (*AIDS vaccination/ human genome project/ cancer eradication*) possible.
5. Electrophoresis is a technique in which solutes are separated by means of
 - a) a difference in boiling points
 - b) a difference in molecular size
 - c) *an electric field*
 - d) a hydrostatic pressure
6. Analogous to the mobile phase in a chromatography system there is *a running buffer (electrolyte)* in an electrophoresis instrument.
7. The velocity at which the ions move towards the electrodes depends upon three factors. Name these factors.
 - *Their shape*
 - *Their environment*
 - *The applied electric field*
8. Strictly speaking, electrophoresis is not a chromatographic technique. Explain. *It does not have a stationary phase but it has many similarities with the separation techniques in this category.*
9. What are the types of electrophoresis?
 - *Gel electrophoresis*
 - *Capillary electrophoresis*
10. In gel electrophoresis the separation
 - a) takes place by means of adsorption
 - b) takes place by chemical reaction with the gel material
 - c) *is stopped before analytes have traveled off the support*
 - d) is evaluated at the end of the solutes trip
11. The concentration of a substance in a sample in electrophoresis is measured by
 - a) *the intensity of the analyte band*
 - b) the time of band registration
 - c) the density of the bands
 - d) the band overlap
12. In capillary electrophoresis all analytes travel (*the same distance/ diverse distances*). What is actually measured is the (*migration time/ migration distance/ migration speed*).
10. The rate of migration depends on the analytes' *size* and *charge*.

11. Write down the equation relating the travel speed to the electric field strength and mobility.

$$v = \frac{E \cdot z}{6\pi \cdot r \cdot \eta} = \mu \cdot E$$

where

E = electric field strength

z = charge of solute

r = radius of solute

η = dynamic viscosity of medium

μ = electrophoretic mobility

12. Name the components of a capillary electrophoresis system.

- power supply
- injector
- capillary
- detector

13. A high voltage is required to

- facilitate separation
- improve instrumental sensitivity
- improve instrumental specificity
- move the buffer through the capillary

14. What is the typical voltage of Question (13)?

25-30 kV

15. Write down the equation for the migration time.

$$t_m = \frac{L_d L}{\mu V} = \frac{L_d}{\mu E}$$

Where

L_d = distance from point of injection to detector

L = total capillary length

μ = mobility

V = applied voltage

E = electric field

16. Name the three factors affecting analyte migration.

- Electro-phoretic mobility
- Secondary Interactions
- Electro-osmosis

17. Electro-osmosis occurs if

- the buffer exhibits an osmotic pressure w.r.t. the surroundings
- the buffer exhibits a hydrostatic pressure w.r.t. the surroundings
- there are any fixed charges present in the system
- the buffer has a net positive or negative polarity