

Board of Studies: Chemistry

Class: TYBSc (Chemistry)

Subject Code: CHEM3504

Subject: Analytical Chemistry

Semester: V

Question Bank;

GRAVIMETRIC ANALYSIS AND THERMOGRAVIMETRIC ANALYSIS.

MCQ-

1. Which sentence is false about preparation of the solution ?

- A. It has required PH.
- B. It is diluted solution.
- C. It is concentrated solution.
- D. All of the above

2. What is a supersaturation ?

- A. Solution containing low amount of solute
- B. Solution containing equilibrium amount of solute
- C. Solution containing more amount of solute than normal circumstances.
- D. None of this.

3. In which step, ions or element are aggregated in Gravimetric analysis?

- A. Supersaturation
- B. Nucleation
- C. Particle growth
- D. None of the above

4. For gravimetric analysis Rate of Nucleation is fast in?

A. Small ppt

B. Large ppt

C. A and B

D. None of the above

5. When more than two ions in solution and simultaneously second ion is also precipitated it is called a

A. Co precipitation

B. Post precipitation

C. A and B

D. None of the above

6. OSTWALD RIPENING is -----

A. Re precipitation

B. Dissolved small ppt

C. Produced larger ppt

D. All of the above

Answer key

1. C 2. C. 3.B. 4. A 5.A 6. D

Q. Describe in brief -

1. What is gravimetric analysis ? What are the conditions for good precipitation ?
2. State and Explain 'Common ion effect'
3. How is common ion effect used in lowering the solubility of a compound?
4. Define the terms solubility and solubility product.
5. Explain the term solubility product with suitable example.
6. How can you relate the solubility product with the phenomenon of precipitation?
7. 'The decrease in solubility by the common ion effect is of great importance in gravimetric analysis'. Explain.
8. What is the effect of complex-ion formation on the solubility of a compound?
How is this phenomenon used in gravimetric analysis?
9. Write a note on supersaturation and precipitate formation.

10. What are the conditions to get a good precipitate in gravimetric analysis
11. What is co-precipitation? What is its origin? Explain with suitable examples
12. What is post-precipitation? Explain it with suitable examples.
13. What precautions can be taken to minimize co-precipitation and post-precipitation?
14. Explain the role of digestion in purification of a precipitate. Give its advantages.
15. Discuss the factors affecting solubility of precipitate.
16. Why is the precipitate in gravimetric analysis washed?
17. How can you classify the wash liquids? Explain.
18. Washing more effective when carried out by many small portions than by few portions of the wash liquid." Explain.
19. What is thermo-gravimetric analysis? What are the different types of thermogravimetry?
20. What information do you get from TG curves? Illustrate it with suitable example.
21. What is filtration? How is it brought about? What are different media used for filtration?
22. How will you select and use a filter paper for filtering the given precipitate?
23. What is a Gooch crucible? How is it used for filtration?
24. Explain in short the type of crucible fitted with permanent porous plates.
25. What are the advantages in using a sintered glass crucible?
26. What are the conditions an ideal wash liquid should satisfy?
27. 'Water is not always a good wash liquid'. Explain.
28. Explain the technique washing by decantation
29. What is the difference between drying and ignition?
30. Explain the incineration of the filter paper
 - (1) in the presence of a precipitate (ii) apart from the precipitate.
31. Explain the role and technique of digestion in purification of a precipitate.
32. Explain the term 'co-precipitation' and 'post-precipitation'. Give at least three points of distinction between them.
33. What is the purpose behind digestion? Give the changes occurring during digestion.
34. Explain why BaSO_4 is digested following precipitation but this procedure is not used for silver chloride or ferric hydroxide.
35. Describe precipitation from homogeneous solution. What are its advantages?
36. Write a note on thermogravimetry.
37. Write a note on types of T.G.A.
38. What is the common ion effect? Discuss the importance of common ion effect in separation of IIIA group radicals from IIIB group radicals.
39. What is thermogravimetric analysis? Give its types. Explain the factors that affect the TGA curve.
40. Explain the effect of acid on solubility of a precipitate with suitable example

Q. Write short notes on –

- 1) Co precipitation
- 2) Post Precipitation
- 3) Common ion effect

ELECTROGRAVIMETRIC ANALYSIS

MCQ-

1. Which of the following measurements involves electrolysis:
 - a) The measurement of pH with a pH electrode
 - b) A redox titration using a redox indicator
 - @ A stripping analysis of uranium in water
 - d) A titration in which the analyte solution potential is measured as a function of added titrant relative to the standard hydrogen electrode.
 - e) All of the above.
2. Which of the following electrochemical methods requires the formation of an insoluble form of the analyte?
 - a) Electrogravimetry
 - b) Coulometry
 - c) Potentiometry
 - d) Voltammetry
 - e) Polarography
3. Which of the following phenomena is not a factor that affects polarization at an electrode?
 - a) Diffusion of the analyte to the electrode surface
 - b) Diffusion of the product from the electrode surface
 - c) Convection
 - d) The standard cell potential for the redox couple

- e) A significant activation barrier for the reaction
4. The limiting current in a linear sweep voltammogram is related to:
- a) The standard reduction potential for the redox couple under investigation
 - b) The reduction potential of the reference electrode
 - c) The point at which concentration polarization begins
 - d) The concentration of the analyte of interest
 - e) The reduction potential of water
5. Which of the following forms of electrochemistry seeks to obtain the condition of full polarization?
- a) Potentiometry
 - b) Voltammetry
 - c) Coulometry
 - d) Electrogravimetry
 - e) Ohmetry

Q. Discribe in brief ---

- 1 Explain the terms used in electrogravimetric analysis
2. Explain the term decomposition potential and its importance in electroanalytical methods of analysis.
3. Explain the factors which influence the over voltage at cathode and anode
4. Discuss the technique of electrolytic separation of metals with controlled Cathode potential taking a suitable example. Draw the labeled diagram for electrolysis.
5. How will you bring about the separation and determination of copper and nickel from an alloy?
6. What apparatus is used in electrolysis? Explain each one of them by taking a suitable example.
7. Define the term electrochemical equivalent.
8. State and explain Faraday's laws of electrolysis.
9. State Faraday's second law of electrolysis.
10. Give the statement of Faraday's First law of electricity.

Problems

1. The cell $\text{Cu} | \text{Cu}^{2+} (7.5 \times 10^{-5} \text{ M}) || \text{Ag} (1.26 \times 10^{-2} \text{ M}) | \text{Ag}$ has a resistance of 4.25 ohm. Calculate the initial potential if this cell is to be operated with a current 0.028 A.

(Ans. 0.2945 V)

2. Consider a cell consisting of Cu electrode in contact with Cu^{2+} ions (1 M), a Cd electrode in contact with Cd ions (1 M) and a connecting salt bridge. The cell has a resistance of 4 ohms.

SPECTROPHOTOGRAPHY

MCQ-

1) The measurement $\lambda = 4000 \text{ \AA}$ can also be expressed as

(1) 40 m, (ii) $4.0 \times 10^5 \text{ cm}$, (iii) $4.0 \times 10^{-5} \text{ m}$,

2. Colorimeters are used in the region

(i) 200 nm-600 nm, (ii) 400 nm-800 nm (iii) 400 nm (iv) 0.9

3. For detection of electromagnetic radiation following device is used:

- i) Photomultiplier tube,
- (ii) Diffracting grating.
- (iii) Interference filter.
- (iv) mercury arc

4. A diffracting grating is a suitable dispersing element that can isolate a selected band of wavelengths by

- (i) scattering
- (iii) constructive interference,
- (ii) refraction,
- (iv) absorption

5. Mathematically, the absorbance is defined as

- (1) $A = \log P/P$.
- (ii) $A = \log T$.
- (iii) $A = -\log PP$
- (iv) $A = \log P/P$.

- 6 . According to Hartley, compounds of similar structures have
- (i) same absorbance reading
 - (ii) analogous absorption spectra
 - (iii) different transmittance readings
 - (iv) same molar absorptivity

Q.Describe in brief.

- 1.What is spectrophotometer ?
- 2 Describe essential parts of a colorimeter. Explain how it can be used in quantitative analysis.
3. Describe the essential components of a spectrophotometer with block diagram.
Give the functions of each component.
4. Explain the differences between absorbance and absorptivity.
5. Describe the various applications of spectrophotometry.
- 6.Explain the following terms: Explain terms
 - a) Transmittance
 - b) Radiant power
 - c) Absorbance
7. Write Notes on
 1. Simultaneous spectrophotometric determination
 - 2) Monochromator
 2. Additivity of absorbance
 - 3.Types of filters in colorimetry.
 4. Job 's continuous variation method.
 5. What are the conditions for simultaneous spectrophotometric determination?
 6. Discuss the construction and working of a simple colorimeter.
 7. What are light filters? Explain absorption filters and interference filters
Comment on the choice of a suitable filter.
 8. Write a note on types of filters used in colorimetry. a
 9. Discuss the construction and working of single beam colorimeter.
 10. What are filters in colorimeter? Explain its type.
 11. Discuss the mole ratio method used to study the co-ordination compounds.
 12. Describe the essential components of spectrophotometer with block diagram.
Give the function of each component.
 13. Write notes on: Photomultiplier tube, Photovoltaic cell
 14. Difference between colorimeter and spectrophotometer.
 15. Explain Mole ratio method for determination of molar composition of complex.
 16. What is spectrophotometer? How does it differ from colorimeter?
 17. Explain: Working of photovoltaic cell.

18. Write note on: Use of photomultiplier tube in spectrophotometer.
19. Describe the essential components with its block diagram.
21. Define the terms: absorbance, transmittance, absorptivity and molar absorptivity.
22. How the pK value of an indicator is determined by spectrophotometrically?
25. Explain: Difference between colorimeter and spectrophotometer.
26. Describe single beam spectrophotometer with neat labelled diagram.
27. Write note on: Interference filters.
28. Describe simultaneous determination of a binary mixture by spectrophotometry
With suitable example.
29. Explain mole ratio method and Job's continuous variation method for determination
of molar composition of complex.
30. Describe in detail spectrophotometric titrations. What are their advantages over
normal titrations
31. What is spectrophotometric titration? Describe various types of Spectrophotometric
titration curves with suitable example.

Q. Short answer questions

Define the following terms:

1. Radiant power,
2. Transmittance
3. Absorbance
4. Absorptivity
5. Molar absorptivity
6. Monochromators
7. Absorption spectrum
8. Frequency of radiation.
9. Wave number of radiation
10. Wavelength of radiation.
- 11 λ_{\max} .
12. Molar extinction coefficient.
13. Optical density 14. Auxochrome.
15. Hypochromic shift.
16. Bathochromic shift.

Q. Answer the following

1. State Beer's law
- 2 State Lambert's law
3. State Lambert's - Beer's law
4. What is molar extinction coefficient?
5. If transmittance of a solution is 0.58. What is its absorbance ?

6. Distinguish between turbidimetry and colorimetry.
7. What do you know about hypochromic shift?
8. A solution of CuSO_4 shows absorbance of 0.76 at 620 nm. Calculate its transmittance.

POLAROGRAPHY

4. Explain in details the terms: i) Half wave potential, ii) Diffusion current
5. Give Ilkovic equation and explain the various terms involved in the equation.
6. Explain the importance of i) supporting electrolyte, ii) gelatine and iii) nitrogen gas in polarography.
7. Give reasons: In polarographic technique -
 - i) concentrated solution of KCl is added in test solution,
 - ii) small amount of solution of gelatine is added in test solution..
 - iii) nitrogen gas is flushed through the test solution.
8. Explain: Current maximum in polarogram.
9. What is the principle of qualitative analysis by polarography? Describe the applications of polarography with respect to qualitative analysis.
10. What is the principle of quantitative analysis by polarography? Describe the applications of polarography with respect to quantitative analysis.
11. What are the merits of dropping mercury electrode? What precautions are necessary before conducting polarographic analysis?
12. Draw a schematic diagram of an apparatus used in polarography and label different parts. Give applications of polarography.
13. Explain the principle of polarography. What are the advantages of using DME in this technique.
14. Write notes on :
 - 1) DME
 2. Diffusion Current
 3. Current maximum in polarogram
 4. Maximum suppressor
 5. Qualitative applications of polarography
 6. Quantitative applications of polarography
 7. Ilkovic equation
 8. Oxygen interference
 9. Polarography
 10. Polarogram
 11. Half wave potential
 12. Supporting electrolyte
 13. Oxygen interference in polarographic analysis.
15. What is the principle of polarography? Describe construction and working of polarograph
16. Explain the qualitative applications of polarography.

17. Draw schematic diagram of an apparatus used in polarography. Explain its working giving polarographic curve.
18. Explain the factor's affecting polarographic wave.
19. Sketch ideal polarographic wave. Explain the terms migration current and limiting current.
20. What is a current maximum in polarography? How is it suppressed?
21. Sketch ideal polarographic wave and explain migration current.
22. Give construction and working of Dropping mercury Electrode.
23. Sketch ideal polarographic wave. Explain residual current and limiting current

Q. Short answer type Questions.

1. Draw the schematic diagram of polarograph and label different parts. Discuss applications of polarography.
2. What are merits of DME ?
3. What are the limitations of DME?
4. Sketch and label the ideal polarographic wave. 5. Give Ilkovic equation and explain the terms involved in it.
6. Give the importance of gelatin in polarographic measurements.]
7. Give the importance of nitrogen gas in polarographic measurements.
8. KCl acts as a best supporting electrolyte in polarography technique. Why?
9. What are the reasons for developing current maximum in Polarogram?
10. What is the principle of qualitative analysis by polarography ?
11. What is the principle of quantitative analysis by polarography ?
12. What is supporting electrolyte in polarography.
13. What is oxygen interfering nuisance in polarographic technique?
14. Give the condition under which the limiting current is attained.
15. What are the reasons for generating residual current?
16. What is limiting current in polarography?
17. What is diffusion current in polarography?
18. Explain the term residual current in polarography.
19. What is limiting current in polarography ?
20. Define the term half wave potential.
21. Define the term diffusion current.
22. What is residual current in polarography?
23. What is decomposition potential in polarography ?
24. What is polarogram?
25. Define the term migration current in polarography. 26. State the Ilkovic equation.
26. What is indicator electrode in polarography?

Q. Define the following terms:

- | | | |
|---------------------|------------------------|---------------------------|
| 1. Polarogram | 2. Half wave potential | 3. Diffusion current |
| 4. Limiting current | 5. Ilkovic equation | 6. Supporting electrolyte |

MCQ

1. Polarography is a branch of
 - a) voltammetry
 - b) Potentiometry
 - c) chronopotentiometry
 - d) amperometry
2. DME in polarography works as
 - (a) reference electrode
 - (b) macro electrode
 - (c) potentiometry
 - (d) amperometry
3. The life time of a mercury drop of DME during its working should be
 - (a) 1 to 15 seconds
 - (b) 3 to 6 seconds
 - (c) less than 3 seconds
 - (d) more than 30 seconds
4. In polarographic measurements when reference electrode acts as an anode then cations present in the solution experience
 - (a) oxidation
 - b) oxidation and formation of amalgam
 - (c) loss of electrons from them
 - d) Reduction
5. Half wave potential is a tool for.....
 - (a) identification of electroactive species
 - (b) determination of concentration of electroactive species
 - (c) quantitative analysis
 - (d) study of half life of electroactive species.
6. Residual current generates during polarographic measurements due to
 - (a) condenser current + migration current
 - (b) condenser current + faradic current
 - (c) migration current + faradic current
 - (d) diffusion current + limiting current
7. Capillary parameters in Ilkovic equation are
 - (a) n and D
 - (b) D and m

(c) m and t

(d) t and D

Q.Describ in brief –

1. What is polroography? Give construction of polarographic cell.
2. Describe the construction and working of DME What are its merits and demerits?
3. Sketch the ideal polar graphic wave and explain the terms- Residual current, limiting current, diffusion current.

ATOMIC ABSORPTION SPECTROSCOPY –

Q.Explain in brief the following.

1. Distinguish between AAS and FES.
2. What are the advantages of AAS over FES ?
3. What are the limitations OR disadvantages of AAS method ?
4. What are the advantages and disadvantages of premixed and total consumption burners?
5. Define the terms sensitivity and detection limit.
6. How will you determine lead in petrol by AAS?
7. What factors are responsible for precision of AAS measurements?
8. What is sputting?
9. A low temperature flame is used for the analysis of alkali and alkaline earth metals why?
10. What is role of monochromatic in AAS? 11. Why is hallow cathode lamp used in AAS?
12. State any two advantages of AAS.
13. What is the principle of AAS?
14. Define the term 'detection limit' in AAS.
15. What is meant by chemical interference in AAS.
16. What is meant by spectral interference in AAS.
17. Define nebulization in AAS.
18. Give the principle of AAS. Give the construction and working of hollow cathode lamp(HCL) in AAS.
19. What is the function of flame in AAS.

Q. Define the following terms.

- a. Atomic absorption spectroscopy b. Hollow cathode lamp c. Atomizer,
d. A premix burner, e. Monochromator f. Amplifier,
g. Detection limit, h. Interference I. Spectral interference
j. Atomic absorption spectra, k. Total consumption burner, l. Chopper
m. Nebulisation n. Detector, o. Sensitivity
p. Chemical interference

Q. Describe in brief-

1. What do you mean by the term AAS. Give its principle, advantages & disadvantages
2. With the help of a block diagram describe instrumentation of AAS.
3. What are the basic components of AA spectrometer?
Discuss: (i) Radiation source, (ii) Atomizers, (iii) Monochromator, (iv) Read-out device.
4. How are (i) spectral and (ii) chemical interferences prevented or corrected for in AA determinations?
5. Write a note on the applications of AAS. At least give one example.
6. Discuss the points of differences between AAS and FES.
7. Define sensitivity and detection limit of an atomic absorption spectroscopy.
8. Give construction and working of total consumption burner and premix burner used in AAS.

Q Short Answer Type Exercises

1. In atomic absorption the sample is..... and excited by heat energy generated by
 - a. vaporized, flame
 - b. Atomized burner
 - c. quantized, lamp
 - d. Ionized furnace
2. Resonance detectors permit the isolation of these lines which those
 - a. lowest
 - b. medium
 - c. Poor
 - d. highest

3. The AAS issensitive than flame photometry
 - a. more
 - b. less
 - c. medium
 - d. high
4. In AAS..... is obeyed over a wide range of concentrations
 - a. Lambert's law
 - b. Nernst law
 - c. Beers law
 - d. Bouget's law
5. For alkali metals, laboratory type..... lamps can be used as a source instead of Hollow cathode lamp
 - a. Discharge
 - b. Tungsten
 - c. Hydrogen
 - d. Incandescent lamp
6. InAAS, high concentration of gaseous atoms cause.....of absorption lines
 - a. Narrowing
 - b. Broadening
 - c.. resonance
 - d. Sputtering
- 7.....is the most widely used flame in AAS
 - a.nitrous oxide
 - b.acetylene
 - c.air-acetylene
 - d.coal gas

FLAME EMISSION SPECTROSCOPY

Q. Describe in brief-

1. What do you mean by the term 'Flame photometry'? OR Define flame photometry,
2. What are the advantages and disadvantages of flame photometry over other Spectrochemical emission methods ?
3. What is the principle of flame photometry ?
4. Draw a block diagram of flame photometer.
5. What are the advantages and disadvantages (drawbacks) of internal standard photometer?
6. Discuss the interferences in flame photometry. How they are eliminated.
7. What are the various events that occur when a solution containing anion is atomized through a flame?

8. How the flame photometry is used in quantitative analysis ?
9. Mention different types of fuels used in flame photometry.
10. Give the expression to calculate the number of excited molecules in FES.
11. Give the Boltzmann's equation in FES.
12. Give the limitations of FES.
13. Mention different types of fuels used in FES.
14. What is the role of monochromatic used in FES?
15. Give the principle of FES.
16. Describe the application of FES.
17. Name the factors which affect the intensity of light emission in FES.
18. What is spectral interference in FES?

Q. Define the following terms:

- | | | |
|--------------------------------|------------------|-----------------|
| 1. Flame emission spectroscopy | 2. Monochromator | 3. Amplifier |
| 4.) Anion interference | 5 Detector | 6. Interference |
| 7. Cation-cation interference | | |

Q. Describe in brief-

1. What are the various steps involved in flame photometry?
2. What are the different types of burners used in flame photometer to produce analytical flame?
3. Draw an optical diagram of a flame spectro-photometer? Discuss the various steps involved in it. Mention its advantages and disadvantages.
4. Discuss the various applications of flame photometry.
5. Discuss the principle of flame photometry. What are masking agents. What are the different components of a flame photometer and What are their uses.
6. Discusses total consumption burner used in FES.
7. Explain with suitable diagram total consumption burner used in FES.
8. Explain with suitable diagrams premixed burners used in flame emission spectroscopy
9. Give the construction and working of total consumption burner and laminar flow burner used in FES.
10. Explain the principle of FES and the steps involved in FES. Give the series of events in flame.

Q. Short-Answer Type questions MCQ-

1. Flame emission spectroscopy is a special type of emission spectroscopy in which is..... Used for excitation of atoms.
 - a) Flame
 - b) Burner
 - c) Lamp
 - d) Furnace

2. Flame photometers use..... as detectors.

- a) Phototubes
- b) Photovoltaic cells
- c) Photomultiplier tubes
- d) Photoconductors.

3. The intensity of spectral lines..... in different parts of the flame.

- (a) Varies
- (b) Changes
- (c) remains same
- (d) Remain unchanged.

4. In flame most of the lines emitted are from the

- (a) Neutral atom
- (b) Gaseous atom
- (c) gaseous ion
- (d) ion

5. Spectral interference is caused especially when..... are used to isolate the desired radiant energy.

- (a) Gratings
- (b) Prisms
- (c) Furnace
- (d) Monochromator

6. Spectral interference is much..... when Monochromator are used.

- (a) less
- (b) High
- (c) Very high
- (d) Low