Roll No. .....

E–761

M. Sc. (Third Semester)
EXAMINATION, Dec.-Jan., 2020-21

CHEMISTRY

Paper Fourth (CH–16)

(Analytical Techniques and Data Analysis)

Time : Three Hours] [*Maximum Marks : 80*

[*Minimum Pass Marks : 16*

Note : Attempt all Sections as directed.

Section—A 1 each

(Objective/Multiple Choice Questions)

Note : Attempt all questions.

Choose the correct alternative :

1. Reagent blanks are always prepared in analytical methods to eliminate the :
   (a) instrumental error
   (b) operative error
   (c) error of the method
   (d) All of the above

2. The indeterminate error associated in any analysis can be minimized by :
   (a) performing Q-test

(b) calculating correlation coefficient

(c) making large number of observations

(d) performing F-test

3. The normal Gaussian curve indicates that _____ of the individual data fall within the range $\overline{X} \pm 2s$, where $\overline{X}$ is mean and *s* is standard deviation.

(a) 90%

(b) 95%

(c) 99%

(d) 99.9%

4. Perchloric acid should never be added directly to organic or biological material during wet digestion process because :

(a) it may cause explosion

(b) it may destroy the chemical structure of the analyte

(c) it is highly corrosive

(d) All of the above

5. The numerical answer to the mathematical operation, $\frac{13.67 \times 0.08476}{4.623}$, comes out to be 1.1586692 when a calculator is used. Among the options given below what would be the final answer for the above mathematical operation as per rules of significant figures ?

(a) 1.1586

(b) 1.158

(c) 1.159

(d) 1.1587

6. The commonly used chromatographic methods for qualitative drug analysis are :
   (a) HPLC
   (b) TLC
   (c) GLC
   (d) All of the above

7. The purity of a solute collected between two times t_1 and t_2 during chromatographic separation is :
   (a) Amount of solute eluted – amount of impurity eluted
   (b) Amount of solute eluted / amount of impurity eluted
   (c) Amount of solvent eluted + amount of impurity eluted
   (d) Amount of solvent eluted / amount of impurity eluted

8. Which of the following is not an important property that governs the extent of separation ?
   (a) Polarizability
   (b) Vapor pressure
   (c) Temperature
   (d) Radius of gyration

9. Which of the following statements is correct for paper chromatography ?
   (a) A sheet or strip of paper acts as the adsorbents.
   (b) It is based on the principle which is partly partition and partly adsorption.
   (c) It is a sheet method.
   (d) All of the above are correct.

10. Solvent extraction is governed by which law ?
    (a) Boyle's law
    (b) Ostwald's dilution law
    (c) Nernst's distribution law
    (d) Beer's law

11. Which of the thermal procedures is not destructive in nature ?
    (a) DSC
    (b) DTA
    (c) TGA
    (d) None of the above

12. The property measured in TGA is :
    (a) Rate of change in weight
    (b) Change in weight
    (c) Change in temperature
    (d) Heat involved or absorbed

13. A rapid TGA method is used for which of the following process ?
    (a) Decomposition of crystals endothermally
    (b) Decomposition of polymers exothermally
    (c) Decomposition of enzymes exothermally
    (d) Decomposition of reactions isothermally

14. The detection device of DTA depends on :
    (a) Maximum temperature required
    (b) Chemical reactivity of the sample
    (c) Sensitivity of the D.C. amplifier
    (d) All of the above

15. Which of the following forms of electrochemistry seeks to obtain the condition of full polarization ?

    (a) potentiometry

    (b) voltammetry

    (c) coulometry

    (d) electrogravimetry

16. For coulometric titration of chloride ions which of the following statements is not correct ?

    (a) Ag^+ ions are generated coulometrically.

    (b) The end point is detected by amperometry when a sudden decrease in current occurred due to generation of excess Ag^+ ions.

    (c) Generation of excess Ag^+ ions occurs as the total Cl^- ions present in the sample become exhausted due to formation of AgCl.

    (d) All of the above statements are incorrect.

17. Second derivative curve for potentiometric titration is the relation between :

    (a) E & V

    (b) ΔE & V

    (c) $\Delta^2 E$ & ΔV^2

    (d) ΔE & V^2

18. The reasons for using dropping mercury electrode are :

    (a) The surface area of the electrode can be calculated from the weight of the drops.

(b) The diffusion current assumes a steady value immediately and is reproducible.

(c) Mercury forms amalgams with many metals.

(d) All of these above

19. Polarography can be used for the analysis of :

(a) Oxidizing substances

(b) Reducing substances

(c) Both (a) and (b)

(d) None of the above

20. Diffusion current is due to _____.

(a) Applied electric field over a given distance

(b) Random motion of holes

(c) Recombination of holes and electrons

(d) Variation in carrier concentration

Section—B 2 each

(Very Short Answer Type Questions)

Note : Attempt all questions. Write answer in 2-3 sentences.

1. How is error propagated during operation of addition and subtraction steps ?
2. What is wet digestion techniques used for ?
3. What is solvent extraction ?
4. Define the term 'Chromatography'.
5. What are the special features of reference material used in DTA ?
6 What are the factors that can affect TG curve ?
7. What is voltammetry ? How does it differ from potentiometry ?
8. What are residual and migration current ?

Section—C 3 each

(Short Answer Type Questions)

Note : Attempt all questions. Write answer in 75 words.

1. Discuss sample collection procedures for aerosol samples.
2. What would be the pH of a solution of 2.0×10^{-3} M HCl to the correct number of significant figures ?
3. Explain in detail how does ion exchange column chromatography works in (a) separation of lanthanides and (b) softening of water.
4. Explain percolation and immersion principles of solvent extraction along with their merits and demerits.
5. What is thermogravimetric analysis ? Give its types. Explain the factors that affect the TGA curve.
6. What are the physical/chemical phenomena responsible for exothermic and endothermic reactions in DTA ? Give examples.
7. Draw neat and labelled diagram on differential pulse polarography or square wave polarography techniques.
8. Give a brief outlined procedure for the determination of chloride ion concentration in tap water by using potentiometry.

Section—D 5 each

(Long Answer Type Questions)

Note : Attempt all questions. Write answer in 150 words.

1. You have received three shipments of Monazite sand of equal weight that contain traces of europium. Analyse of the three ores provided europium concentrations of 397.8 ± 0.4, 253.6 ± 0.3 and 368.0 ± 0.3 ppm respectively. What is the

average europium content of the ores and what are the absolute and relative uncertainties ?

Or

The sulphate content in a water samples was measured by ion-chromatographic technique for five times and the following values are obtained 0.1010 ppm, 0.1020 ppm, 0.1005 ppm, 0.1030 ppm and 0.1015 ppm. Calculate standard deviation, relative standard devation and the variance for the analyses.

2. Derive a mathematical expression for amount of solute remaining unextracted in the aqueous solution after multiple extractions with an organic solvent.
3. What is automated method ? Explain the principle, instrumentation and application of flow injection analysis (FIA).
4. The molar conductivity of 0.1 M CH_3COOH solution is 4.6 s cm^2 mol^{-1}. Calculate the conductivity and resistivity of the solution. Explain instrumentation of a conductometer.

Or

Write brief notes on any *two* of the following :

(i) Dropping mercury electrode

(ii) Diffusion current

(iii) Cyclic voltammetry