

ASSIGNMENT BOOKLET

Post Graduate Diploma in Analytical Chemistry (PGDAC)

Basic Analytical Chemistry	(MCH – 001)
Separation Methods	(MCH – 002)
Spectroscopic Methods	(MCH – 003)
Electroanalytical & Other Methods	(MCH – 004)

(Valid from January 1, 2019 to December 31, 2019)

**It is compulsory to submit the assignments before filling
in the examination form.**



School of Sciences
Indira Gandhi National Open University
Maidan Garhi, New Delhi-110068
(2019)

Dear Learner,

This assignment booklet consists of the tutor marked assignments (TMAs) of MCH-001, MCH-002, MCH-003 and MCH-004 courses of the Post Graduate Diploma in Analytical Chemistry (PGDAC) programme.

We hope, you are familiar with the system of evaluation to be followed for this Programme. You may probably like to re-read the section on assignments in the Programme Guide that was sent to you earlier. As you are aware, a weightage of 30 percent has been earmarked for continuous evaluation component. For this you have to submit the responses of the enclosed tutor marked assignments to the Study Centre Coordinator. The assignments are based on the content of all the blocks of all the courses.

Before attempting the assignment, please read the following instructions carefully.

- 1 On top of the first page of your assignment response, please write the details exactly in the following format; write your answers from second page onwards.

ENROLMENT NO. :

NAME :

ADDRESS :

.....

.....

COURSE CODE :

COURSE TITLE :

STUDY CENTRE :

DATE :

(NAME AND CODE)

PLEASE FOLLOW THE ABOVE FORMAT STRICTLY TO FACILITATE EVALUATION AND TO AVOID DELAY.

- 2 Use only foolscap size paper (but not of very thin variety) for writing your answers.
- 3 Leave about 4 cm margin on the left, top and bottom of your assignment response sheet.
- 4 Your answers should be precise.
- 5 While writing answers, clearly indicate the Question No. and part of the question being solved.
- 6 Though the validity of assignment is for one year, we advise you to submit the assignment response within 12 weeks after receiving it.
- 7 **We strongly suggest that you should retain a copy of your assignment responses.**

Wishing you good luck

TUTOR MARKED ASSIGNMENT

MCH-001: Basic Analytical Chemistry

Course Code: MCH-001
Assignment Code: MCH -001/TMA/2019
Maximum Marks: 100

Note: Answer all the questions given below.

- What is meant by an analytical technique? List the characteristics of an analytical technique that need to be considered in order to ascertain the suitability of the technique for a given analytical determination. (5)
 - Define determinate errors. Briefly describe different sources of determinate errors. (5)
- Define and differentiate between accuracy and precision with the help of suitable examples. (5)
 - What is the significance of standard deviation in the analysis of the data of an analytical determination? The result (R) of an analytical determination depends on three parameters as per the relation, $R = (A+B)/C$. The values and individual absolute standard deviations (given in parentheses) of the three quantities, A, B and C obtained in an experimental determination are given below. Calculate the standard deviation in the result, R of the determination. (5)

$$A = 3.80 (\pm 0.04), B = 2.10 (\pm 0.02), C = 1.97 (\pm 0.03)$$

- Define sampling and describe sampling procedures for water. (5)
 - Explain different means of chemical exposure to the human body. (5)
- Explain the importance of compatibility of chemicals while storing them in the laboratory. (5)
 - What is the importance of enzyme catalysed reactions? (5)
- What is meant by leveling effect of a solvent? Explain with the help of a suitable example. (5)
 - Outline the procedure for the determination of hydronium ion concentration in an aqueous solution of a polyprotic acid. Determine the pH of 1.0×10^{-3} M solution of oxalic acid. The successive dissociation constants of oxalic acid are: (5)

$$K_1 = 5.9 \times 10^{-2}; K_2 = 6.4 \times 10^{-5}.$$

- Calculate the pH at different stages of a titration between 100 cm³ of 0.01 M formic acid and 0.01 M NaOH and draw the titration curve. Given: K_a (Formic acid) = 1.7×10^{-4} . Suggest a suitable indicator for the titration. (5)
 - Compute the standard electrode potential of the cell in which the following reaction takes place and write the Nernst equation for the cell. (5)



$$\text{Given } E_{\text{Sn}^{4+}/\text{Sn}^{2+}}^0 = 0.14\text{V}; \quad E_{\text{Fe}^{3+}/\text{Fe}^{2+}}^0 = 0.77\text{V}$$

- What is the importance of redox titrations in non-aqueous medium? What are the criteria for the selection of a suitable solvent for use in non-aqueous redox studies? (5)
 - Define and differentiate between stepwise and cumulative formation constants with the help of an example. (5)
- What are metallochromic indicators? Explain the principle of action of metallochromic indicators and outline the essential requirements that must be met by an indicator to be (5)

used for the visual detection of end points in complexometric titration.

- b) What is meant by a precipitation titration curve? What factors need to be considered while designing an analytical determination based on precipitation titration? (5)
9. a) What are adsorption indicators? Explain the principle of adsorption indicators taking the example of Fajan's method for the determination of chloride ions using silver nitrate. (5)
- b) Define and differentiate between coprecipitation and post precipitation. (5)
10. a) Enumerate the advantages and disadvantages of the use of organic precipitants in inorganic gravimetric analysis. (5)
- b) Briefly describe the role of computers in analytical instrumentation. (5)

TUTOR MARKED ASSIGNMENT

MCH-002: Separation Methods

Course Code: MCH-002
Assignment Code: MCH -002/TMA/2019
Maximum Marks: 100

Note: Answer all the questions given below.

1. Define separation. Explain its need and importance. (5)
2. List various methods which can be used for separating a mixture of two immiscible liquids. (5)
3. Discuss the distribution of osmium tetroxide between carbon tetrachloride and water. (5)
4. What are chelating agents? Illustrate their role in solvent extractions. (5)
5. Describe the extraction of anionic metal complexes by high molecular weight amines. (5)
6. What are masking agents? Highlight their use in the separation. What difficulties arise where such agents are used? (5)
7. Define the terms, retention time and retention factor. Also write the equations used to express them. (5)
8. What is meant by Eddy diffusion term in van Demeter equation? Discuss the factors on which this term depends. (5)
9. Briefly explain various forms of liquid-liquid chromatography. (5)
10. With the help of suitable examples, illustrate how can various substances be separated by liquid column chromatography. (5)
11. Discuss various factors which can improve column efficiency in gas chromatography. (5)
12. List the basic requirements of a liquid phase in gas chromatography. (5)
13. Briefly explain important features of stationary support used in size exclusion chromatography used for HPLC. (5)
14. What is meant by the term 'ion exchange'? List the requisites of a useful ion exchanger. (5)
15. Using suitable examples, discuss the use of hydrous oxides of polyvalent metals in ion exchange chromatography. (5)
16. Briefly explain the essential requirements of a gel which can be used for chromatography. (5)
17. Describe the unique features of size exclusion chromatography. (5)
18. What is ultra filtration? Give its use and applications. (5)
19. What is dialysis? Give its limitations. (5)
20. Illustrate electroosmotic flow. How is it useful in separations? (5)

TUTOR MARKED ASSIGNMENT

MCH-003: Spectroscopic Methods

Course Code: MCH-003
Assignment Code: MCH-003/TMA/2019
Maximum Marks: 100

Note: Answer all the questions given below.

1. a) Define and differentiate amongst the terms diffraction, refraction and reflection. (5)
b) What types of spectra are obtained by atomic absorption and molecular absorption in different species? What is the reason of different spectra in the two cases? (5)
2. a) Describe the nature of transitions involved while taking UV-Visible spectra of an organic species, inorganic species and charge transfer complex. Illustrate your answer. (5)
b) What is the difference between a single beam and a double beam UV-visible spectrometer? Explain giving schematic diagram for these types. (5)
3. a) How is Fourier Transform Infra Red spectrometer (FTIR) different from dispersive IR spectrometer? What are the advantages of (5)
b) Describe the theory of Raman spectroscopy. Name the two theories of Raman effect and briefly give the basis of these theories. (5)
4. a) Name and explain the principle that governs the activation of a molecule on absorption of a photon of suitable energy in fluorimetry and phosphorimetry. Write the implication of this principle? (5)
b) What is the effect of change of pH, solvent and temperature on fluorescence and phosphorescences? Explain. (5)
5. a) Describe the application of fluorimetry in the analysis of aquatic environment. (5)
b) What are the types of the flames used in flame photometry? How are these different from each other? Draw a labelled schematic structure of any one type. (5)
6. a) Write the merits and limitations of flame photometry. (5)
b) Write the principle of atomic fluorescence spectrometry and describe in brief the cases of interferences in this technique. (5)
7. a) Why does the plot show nonlinearity in the calibration plot-method of AAS? In what conditions this method is to be replaced by internal standard method and the standard addition method? Explain giving a schematic typical calibration plot. (5)
b) Describe the wet decomposition method of preparing a sample in AAS. What are the advantages of this method? (5)
8. a) What is plasma and how is it used in the atomic emission spectrometry? Describe the process. (5)
b) Compare AAS and AES with respect to the principle involved and give an application for each of these techniques. (5)
9. a) Describe the electron and chemical ionisation in mass spectrometry and the function of analysers in this technique. (5)
b) What is the significance of index of hydrogen deficiency in the determination of molecular formula? Give an example to explain. (5)
10. Explain the following briefly: (10)
 - i) ii) Mechanism of resonance in NMR spectroscopy
 - iii) Anisotropy of chemical bonds
 - iv) Quantitative Applications of NMR spectroscopy

TUTOR MARKED ASSIGNMENT
MCH-004: Electroanalytical and Other Methods

Course Code: MCH-004
Assignment Code: MCH-004/TMA/2019
Maximum Marks: 100

Note: Answer all the questions given below.

1.	a)	What is direct potentiometry? Drive an expression for E_{cell} for both cation and anion indicator electrode.	(5)
	b)	Calculate the potential of following electrodes:	(5)
		i) A copper electrode immersed in 0.04 M $\text{Cu}(\text{NO}_3)_2$ ii) A zinc electrode immersed in 0.05 M $\text{Zn}(\text{NO}_3)_2$	(5)
2.	a)	Discuss design and working of a calomel electrode.	(5)
	b)	Calculate solubility product of AgI at 298 K on the basis of following data: $\text{AgI(s)} + e \rightarrow \text{Ag(s)} + \text{I}^- \quad E_{\text{AgI, Ag}}^\circ = -0.15 \text{ V}$ $\text{Ag}^+ + e \rightarrow \text{Ag(s)} \quad E_{\text{Ag}^+, \text{Ag}}^\circ = +0.80 \text{ V}$	(5)
3.	a)	Describe a typical design and principle of a solid state membrane electrode. List some applications of ion selective electrodes.	(5)
	b)	Distinguish between molar conductivity and limiting molar conductivity. Explain why limiting ionic mobilities of H^+ and OH^- ions are exceptionally high.	(5)
4.	a)	At 298 K, the conductivity of pure water is $2.9 \times 10^{-6} \text{ S m}^{-1}$ and limiting molar conductivities of the H^+ and OH^- ions are 0.03498 and $0.01976 \text{ S m}^2 \text{ mol}^{-1}$, respectively. Calculate the ionic product of pure water at 298 K.	(5)
	b)	Briefly distinguish between: i) Concentration polarization and kinetic polarization. ii) Constant current electrolysis and constant potential electrolysis.	(5)
5.	a)	Describe the design and principle of hydrogen oxygen coulometer. Write three main advantages of coulometric titrations.	(5)
	b)	A constant current of 0.400 A is used to deposit copper at the cathode and oxygen at the anode of an electrolytic cell. Calculate the number of grams of each product formed in 30.4 min, assuming no other redox reaction. [Hint: Write two half-cell reactions and use Eq. 5.13]	(5)
6.	a)	What are the different pulse methods in voltammetry? Discuss any one method in some detail.	(5)
	b)	What is dropping mercury electrode? Give its advantages.	(5)
7.	a)	Derive the polarographic equation.	(5)
	b)	The polarogram of a chloramphenicol solution gives an i_d of $40.3 \mu\text{A}$. To 10.0 cm^3 of the original solution, 5.00 cm^3 of 2.5 mg/cm^3 chloramphenicol standard is added. This polarogram gives an i_d of $65.2 \mu\text{A}$. What is the concentration of the unknown solution? [Hint: $i_d \propto c$]	(5)

8.	a)	List the factors affecting TG curve. Taking a suitable example, explain the effect of furnace atmosphere on TG curve.	(5)
	b)	A mixture of CaCO_3 and CaO is analysed using TGA technique. TG curve of the sample indicates that there is a mass change from 584.2 mg to 374.5 mg between 500-900°C. Calculate the percentage of CaCO_3 in the sample.	(5)
9.	a)	Distinguish between DTA and DSC.	(5)
	b)	Draw typical enthalpograms for both exothermic and endothermic reactions. Discuss the type of information you may get from these enthalpograms.	(5)
10.	a)	Give advantages and limitations of IDA technique.	(5)
	b)	A 6.85 cm^3 solution containing $7.8 \mu \text{ Ci/cm}^3$ of ^{59}Fe was injected into the blood stream of a cow. After sometime, 1.0 cm^3 of blood was taken out and it gave 4413 count per minute. Calculate the blood volume in the body of cow.	(5)