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, 2022 to December 31, 2022)

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
(2022)

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   **:** ………………………...

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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

Basic Analytical Chemistry

Note: * Answer all the questions given below. The marks are indicated in the brackets.

* Please answer these in your own words; answer copied from the course material will fetch poor marks.

Q.1 a) 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)

b) Define determinate errors. Briefly describe different sources of determinate errors. (5)

Q.2 a) Define and differentiate between accuracy and precision with the help of suitable examples. (5)

b) 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.
A = 3.80 (± 0.04), B = 2.10 (±0.02), C = 1.97 (±0.03) (5)

Q.3 a) Define sampling and describe sampling procedures for water. (5)

b) Explain different means of chemical exposure to the human body. (5)

Q.4 a) Explain the importance of compatibility of chemicals while storing them in the laboratory. (5)

b) What is the importance of enzyme catalysed reactions? (5)

Q.5 a) What is meant by leveling effect of a solvent? Explain with the help of a suitable example. (5)

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

Q.6 a) Calculate the pH at different stages of a titration between 100 cm^3 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)

b) Compute the standard electrode potential of the cell in which the following reaction takes place and write the Nernst equation for the cell.

$$Sn^{4+}(aq) + 2Fe^{2+}(aq) \rightleftharpoons Sn^{2+}(aq) + 2Fe^{3+}(aq)$$
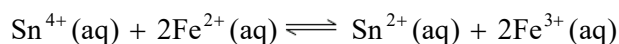
Given $E^0_{Sn^{4}/Sn^{2+}} = 0.14V$; $E^0_{Fe^{3+}Fe^{2+}} = 0.77v$ (5)

Q.7 a) 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)

b) Define and differentiate between stepwise and cumulative formation constants with the help of an example. (5)

Q.8 a) 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 used for the visual detection of end points in complexometric titration. (5)

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)

Q.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)

Q.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
Separation Methods

Answer all the questions given below.

1. How is volatility important for the separation of components of a mixture? Also briefly explain different forms of distillation. (5)

2. Describe different criteria which can be used for the selection of separation methods. (5)

3. Discuss the role of salting out agents in extraction giving suitable examples. (5)

4. What is a diluent? Give different reasons for the use of diluents. Also list important characteristics of diluents. (5)

5. With the help of a suitable example, explain the extraction equilibrium involving formation of metal chelates. (5)

6. Briefly explain the following:
   i) Extraction by synergism
   ii) Stripping (5)

7. Briefly discuss liquid chromatography and its various forms. (5)

8. With the help of suitable diagrams, explain different stages of the elution process in column chromatography. (5)

9. What are different stationery phases which can be used in liquid-solid chromatography? Give their general characteristics and parameters associated with them. (5)

10. What is HPLC? Give its characteristic features and advantages. (5)

11. What are different interaction forces which aid in GC separation? What is the major advantage of GC over distillation? (5)

12. List the basic requirements of a liquid phase used in GC. (5)

13. Give a schematic representation and a list of various components of HPLC instrument. (5)

14. List various advantages of HPLC. (5)

15. How are ion exchangers classified? Give two examples each of natural and synthetic ion exchangers along with their uses. (5)

16. How does the moisture content and particle size affect the rasin properties? (5)

17. Discuss important properties of gels which are used for chromatographic purposes. (5)

18. Briefly explain the preparative applications of size exclusion chromatography. (5)

19. Briefly explain the following:

    i)   Reverse Osmosis

    ii) Nanofiltration

    iii) Ultrafiltration

    iv) Microfiltration

    v)   Dialysis   (5)

20. Discuss the experimental aspects of DNA Gel electrophoresis including instruments and reagents used.   (5)

Tutor Marked Assignment

Spectroscopic Methods

Note: * Answer all the questions given below. The marks are indicated in the brackets.

* Please answer these in your own words; answer copied from the course material will fetch poor marks.

1. a) Define electromagnetic radiation in terms of the wave mechanical model. (2)

   b) The Gyan Vani transmission of IGNOU is broadcast at 105.6 MHz in the Delhi region. Compute the energy of a photon corresponding to this frequency. (3)

   c) The transmittance of a solution, containing 7.3 mg of an potassium dichromate per 100 cm^3, taken in a cuvette of path length of 1 cm is measured at 455 nm. If the percentage transmittance found to be 6, calculate the molar absorptivity of the oxidizing agent. (3)

   d) State the necessary condition for a molecule to show IR spectrum. Will HCl show IR spectrum? (2)

2. a) What are monochromators? Describe the working of a grating monochromator. (3)

   b) In what way is the standard addition method of calibration in UV-VIS spectrophotometry better than the standard solution method? (2)

   c) Explain the origin of Raman spectrum in terms of quantum theory of radiation. (3)

   d) State the 'Rule of mutual exclusion'. What is its significance? (2)

3. a) Explain the origin of fluorescence and phosphorescence spectra in terms of Jablonski diagram. (3)

   b) What is meant by fluorescence quenching? How does it affect the quantum yield of a fluorescence emission? (2)

   c) The experimental set up for the fluorescence spectral measurement is different from that for the absorption measurement. Comment (2)

   d) Why do we need to modify the instrumental set up for the fluorescence spectrometer to make phosphorescence measurements? Describe the modification. (3)

4. a) What is chemiluminescence? Describe an analytical application of chemiluminescence in the area of environmental pollution measurement. (4)

   b) Describe the application of fluorescence measurements in medical diagnosis by taking the example of blood glucose determination. (3)

   c) What is meant by room temperature phosphorescence? How is it achieved? (3)

5. a) Briefly explain the origin of atomic spectrum. (2)

   b) Explain why the atomic spectra are line spectra whereas the molecular spectra are band spectra. (2)

   c) Discuss the fate of analyte sample in the flame of a flame photometer. (3)

   d) What is internal standard method? Under what conditions is it used? (3)

6. a) Briefly explain the different types of spectral interferences observed in flame photometry. (3)

   b) Explain different pathways of atomic fluorescence emission. (5)

   c) In what way is halogen cathode lamp (HCL) better than a continuous source for atomic fluorescence measurement? (2)

7. a) Explain the principles of atomic absorption spectrophotometry and atomic emission spectrometry. (3)

   b) Why line sources are preferred over continuum sources for AAS? (2)

   c) Enlist the advantages of GFAAS over FAAS. (3)

   d) What are nebulisers? Explain the principle of ultrasonic nebuliser. (2)

8. a) Enlist different components of an ICP torch. (2)

   b) What makes argon a good choice for the plasma gas? (2)

   c) Describe the acid digestion method of solution preparation in AES. (2)

   d) What are different types of instruments used for ICP-AES? Which of these is better and why? (4)

9. a) Explain the following terms in the context of NMR spectrometry.

   - Chemical shift

   - Spin-spin splitting

   - Larmor precession (6)

   b) FT – NMR is better than CW – NMR. Comment on the statement. (2)

   c) What is the principle of mass spectrometry? In what way is it different from other spectrometric methods? (2)

10. a) Explain McLafferty rearrangement with the help of a suitable example. (3)

    b) Calculate IHD for the molecule having the molecular formula, $C_3H_6O_2$. (2)

    c) What kind of structural information is available from UV, IR, NMR and Mass spectra of an organic compound? (5)

Tutor Marked Assignment

Electroanalytical and Other Methods

Note: * Answer all the questions given below. The marks are indicated in the brackets.

1. a) Derive an expression for the determination of an equilibrium constant using electrode potential measurement. (5)

   b) Calculate the potential of the cell given below

   $$Cu \left| Cu^{2+} \left(0.010 \ M\right) \right\| Ag^{+} \left| Ag \left(0.10 \ M\right) \right.$$ (5)

   Hint: use Appendix I for the standard electrode potential values.

2. a) How is the conductance varied with concentration? Explain with the help of suitable examples. (5)

   b) At 298 K, the limited molar conductivities at infinite dilution of sodium benzoate, hydrochloric acid and sodium chloride are 8.24×10^{-3} S m^2 mol^{-1}, 4.26×10^{-2} S m^2 mol^{-1} and 1.26×10^{-2} S m^2 mol^{-1}, respectively. Calculate the limiting molar conductivity of aqueous benzoic acid. (5)

3. a) Discuss the factors which limit the accuracy of pH measurements. (5)

   b) Give some applications of ion selective electrodes. (5)

4. a) Describe concentration polarisation. Give the importance of overvoltage. (5)

   b) A solution of Zn is electrolysed for 30 s using a current of 1.0 mA. Calculate the mass of Zn plated on the electrode for the electrochemical reaction:

   $$Zn^{2+} + 2e \ \rightarrow \ Zn$$ (5)

5. a) Write the advantages of coulometric titrations. (5)

   b) Calculate the amount of Co (II) deposited on the surface of a cathode, if a constant current of 0.96 A for 28.4 minutes is passed through solution of Co (II). Assume that current efficiency is 90%. (5)

6. a) Conductivity of 1.6×10^{-2} mol dm^3 acetic acid is 0.0215 S m^{-1}. Calculate the degrees of dissociation and the dissociation constant of acetic acid. You may consult Table 4.2 for the limiting ionic conductivities of the hydrogen and acetate ion. (5)

   b) How would you analyse a mixture of Ca, Sr, and Ba oxalates using thermogravimetry? Draw a thermogram for the decomposition of Ca, Sr and Ba oxalates. (5)

7. a) What are the advantages of DTA and DSC over TGA? (5)

   b) An impure sample of $CaC_2O_4.H_2O$ is analysed using TGA technique. TG Cure of the sample indicates the total mass change from 85 mg to 30.7 mg when this sample was healed upto 900° C. Calculate the % purity of the sample. (5)

8. a) Draw a DTA Curve for $CaC_2O_4.H_2O$. Give the interpretations of different peaks exist in the thermogram. (5)

    b) At 291 K, the conductivity of pure water is 4.3×10^{-6} S m^{-1}. Calculate the ionic product of pure water at 291 K. (5)
        Hint: consult Table 4.2 for the limiting ionic conductivity of H$^+$ and OH$^-$ ions.

9. a) Give advantages and limitations of NAA technique. (5)

    b) Discuss the working of a Scintillation Detector. (5)

10. a) Draw and label the three electrode cell for hydrodynamic voltammetry. (5)

    b) Write the Ilkovic equation and mention the meaning of each of the terms used in it. (5)