NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY

FACULTY OF APPLIED SCIENCES

DEPARTMENT OF APPLIED CHEMISTRY

CLASSICAL METHODS AND CHEMOMETRICS
SCH 5110

FIRST SEMESTER EXAMINATION QUESTION PAPER

2024

This examination paper consists of 3 pages

Time Allowed:	3 hours
Total Marks:	100
Special Requirements:	Periodic Table
Internal Examiner:	Dr. E. Dube
External Examiner:	Prof. G. Mehlana

INSTRUCTIONS

1. Answer all questions from Section A and **ANY THREE** questions from Section B.
2. Section A carries **40 marks** and each question in Section B carries **20 marks**.
3. Use of calculators is permissible

Mark Allocation

Questions	Marks
1	40
2	20
3	20
4	20
5	20
Total Possible Marks	**100**

Section A

Question 1.

a) Below is an abstract by Rahul Bhochiya, Pradip Kumar Mondal and Shah Jaimin Balkrishna, entitled **A novel pH-sensitive method for the quantification of Ca^{2+} ions by complexometric titration with Na_3HEDTA**, ***Anal. Methods***, 2024,**16**, 5628-5632

Abstract: A simple and accurate complexometric titration has been developed by the reaction between Ca^{2+} ions (pH 7) and Na_3HEDTA (pH 11) solutions. Formation of a Ca-EDTA complex and release of H^+ ions in the system were traced by a decrease in the pH. At the equivalence point of the titration, a sharp increase in the pH was observed. Looking at the working pH range (pH 6.2–7.5) of this method, another complexometric titration method has been developed by using bromothymol blue as a pH sensitive indicator. This is the first report of a complexometric titration by using an acid base indicator.

i) The work by Bhochiya *et al* in ***Anal. Methods***, 2024,**16**, 5628-5632; show that classical methods of analysis are still preferred, explain why? **[3 marks]**

ii) Explain the principle of complexometric titration as applied to the reaction between Ca^{2+} ions and Na_3HEDTA **[4 marks]**

iii) Describe the role of pH in this complexometric titration and explain why monitoring pH is essential in this method. **[4 marks]**

iv) The authors observed a sharp increase in pH at the equivalence point of the titration. Illustrate this titration curve with a diagram, providing a clear explanation of the observation and its significance. **[4 marks]**

v) Why are multidentate ligands such EDTA mostly used in this method instead of monodentate ligands. **[2 marks]**

vi) Based on the abstract, propose improvements or modifications that could enhance the accuracy or application range of this complexometric titration method. **[2 marks]**

b) Describe the EDTA titration method for the determination of aluminum in samples **[10 marks]**

Section B

Question 2

You are assigned the following tasks:

a) Analysis of a Mixture Containing Dipyrone and Paracetamol in Pharmaceutical Preparations

Dipyrone is soluble in aqueous solutions, while paracetamol can be dissolved in hydrochloric acid by heating in a boiling water-bath for 30 minutes. Both dipyrone and paracetamol react with potassium hexacyanoferrate(III). **[10 marks]**

b) Determination of Diclofenac in Pharmaceutical Preparations

Diclofenac is precipitated from aqueous solution using copper(II) acetate at pH 5.3. **[10 marks]**

For each of the above tasks, design a classical method that you will use clearly describing its:

i) Principle or mechanism
ii) The assumptions made during the analysis
iii) The procedure for the method

Note: Pharmaceutical preparations may contain excipients such as binders, colors, etc.

Question 3.

a) i. Define nonaqueous titration. **[2 marks]**

ii. Explain why nonaqueous media are used for the titration of weak acids and weak bases. **[3 marks]**

b) Describe an experimental procedure to standardize a hydrochloric acid solution using sodium carbonate as a primary standard. Include the reaction equation. **[6 marks]**

c) You are provided with an impure sample of sodium hydroxide. Explain how you would determine its concentration using oxalic acid as a primary standard. **[6 marks]**

d) If a primary standard is found to absorb moisture from the air during weighing, what steps can be taken to mitigate errors in the titration process? **[3 marks]**

Question 4.

a) Design a combustion analysis experiment to determine the carbon and hydrogen content in a coal sample. Outline the equipment, reagents, and steps required. **[7 marks]**

b) A 0.500 g sample of an organic compound is combusted, producing 1.10 g of CO_2 and 0.450 g of H_2O. Calculate the mass percentage of carbon and hydrogen in the compound. **[8 marks]**

c) Identify potential sources of error in gravimetric combustion analysis and discuss how they can affect the accuracy of results. **[5 marks]**

Question 5

a) Define the terms 'equivalence point' and 'end point' **[1 marks]**

b) i) Describe the mechanism of a neutralization reaction between a strong acid and a strong base. **[5 marks]**

ii) How does the reaction differ when a weak acid is titrated with a strong base? **[4 marks]**

c) Fajans titration uses an adsorption indicator. Describe how this indicator works. **[5 marks]**

d) Describe a Volhard titrations method. **[5 marks]**

Question 6

A 1.22 g sample of impure lead nitrate was dissolved in 40.0 mL of distilled water and analyzed using gravimetric methods to determine the mass percentage of lead. Sodium sulfate solution was added dropwise from a burette to precipitate the lead ions as lead sulfate. Addition continued until no further precipitate formed, after which a few extra drops of sodium sulfate were added to ensure complete precipitation. The resulting precipitate was filtered, thoroughly washed, dried, and weighed. The dried precipitate had a mass of 1.44 g.

a) Explain the importance of thoroughly washing the precipitate during this analysis. **[2 marks]**
b) Identify and explain the key assumptions made in this analytical method. **[3 marks]**
c) Calculate the percentage composition of lead in the original sample based on the given data. **[8 marks]**
d) Discuss why the concentration and volume of the sodium sulfate solution used are not critical for this calculation. **[3 marks]**
e) If the precipitate in this analysis exhibits poor filterability, identify potential causes and propose solutions to improve the filtering process. **[4 marks]**