BIRLA INSTITUTE OF TECHNOLOGY & SCIENCE, PILANI
First Semester 2022-2023
Comprehensive Exam (OPEN Book)

Course Name: Pharmaceutical Analysis	**Course No: PHA F211**
Total Marks: 20 **Date: 27-12-2022**	**Duration: 2.5 h (Max)**

Instructions: a) All questions are compulsory; b) Marks will be deducted if calculations are not accompanied by proper explanation; c) Handwriting should be legible; d) Give the answers for all sub-parts together in one place; e) draw diagrams/equations wherever needed

1) Pharmaceutical preparation of ferrous fumarate ($C_4H_2FeO_4$; MW = 169.9) is determined using Cerimetry. The 0.1487 g of sample of the product was dissolved in 7.5 mL of dilute sulfuric acid and the solution was dissolved in 25 mL water. The titration with 0.0996 M Cerium sulfate resulted in the visual end point at 8.51 mL. Calculate the percentage purity of the product. [3]

2) The concentration of methotrexate drug was determined by HPLC using a pure standard. The 10.1 mg of standard drug was dissolved in the 100 mL mobile phase and ran through C18 column resulting in the peak area of 12006 in the chromatogram. The 10.1 mg of the test sample was then diluted and analyzed in the identical manner resulting the peak area of 11605. Find out the purity of the methotrexate sample. [3]

3) During Karl-Fischer (KF) analysis, a sample of 20 mg of pure water was titrated resulting in consumption of 5.13 mL of the KF reagent. A 2.1016 g of a powered drug was then analyzed in an identical manner resulting in the amperometrically determined end point at 0.72 mL. Determine the moisture content (%w/w) in the drug. [3]

4) The iodide in a sample of KI (MW = 166.00) was converted to iodate by treatment with an excess of bromine. The unused bromine was removed by boiling; an excess of barium ion was then added to precipitate the iodate. In the analysis of a 1.59 g sample of KI, 0.0538 g of barium iodate (MW = 487.13) was recovered. Express the results of this analysis as percent potassium iodide. [4]

$$H_2O + Br_2 + I^{\ominus} \longrightarrow Br^{\ominus} + IO_3^{\ominus} + H^{\oplus}$$
$$Ba^{2+} + IO_3^{\ominus} \longrightarrow Ba(IO_3)_2$$

5) The calcium amount in a tootpaste can be determined using complexometry. A 5.613 g sample of the dried paste was taken and transferred to a 250-mL beaker and dissolved in 25 mL of 6 M HCl. After filtering, the solution is diluted to 250 mL in a volumetric flask. A 10.00 mL aliquot is placed in a 125-mL Erlenmeyer flask and buffered to a pH of 10. Titrating with 0.04988 M EDTA requires 44.11 mL to reach the end point. Determine the amount of calcium in the paste as %w/w $CaCO_3$ (MW =100.09). [3]

6) The concentration of F^- in a water sample is determined by a F^- ion-selective electrode. A 25.00-mL sample is placed in a beaker, and a potential of +0.102 V is measured. A 1.00-mL aliquot of a 200.0 ppm standard solution of F^- is added, after which the potential is found to be +0.089 V. Report the concentration of F^- in parts per million. [4]

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1) Draw the qualitatively accurate titration curves with properly labelled axis, for the following titrations and provide brief explanation.   [4]
A) amperometric titration where both the titrand and titrant are reducible,
B) conductometric titration of acetic acid with NaOH (titrant).

2) Complexometric titration of Ca^{2+} was performed using EDTA as titrant at pH 10 and at pH 3. Compare the titration curves at these two pH conditions and provide explanation for the difference.   [3]

3) A chromatogram shown on the right was obtained using a column of 2 m length. Define symbols k', N, and H and determine their values (use w = 22 s, t_m = 25 s) using the chromatogram.   [3]
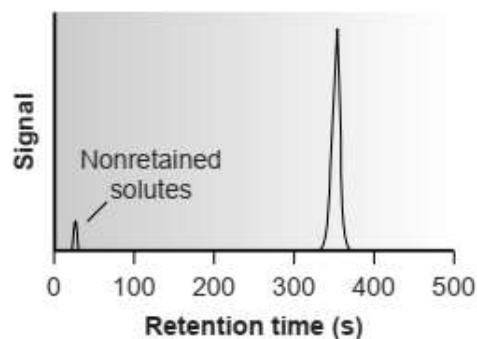
4) With the help of a diagram explain how occlusions are formed and how to remove such impurities.   [2]

5) Briefly describe the principle of Mohr's method with the help of reactions involved.   [3]

6) With an example describe the application of limit tests for
A) soluble matter, B) ash values.   [3]

7) Name four factors that determine the selection of an analytical method.   [2]