INSTRUMENTAL ANALYSIS EXPERIMENTS

Fourth Class

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Experiment No. 1

Spectrophotometric Determination of Manganese (Mn^{7+}) and Chromium (Cr^{6+}) in admixture of potassium permanganate and potassium dichromate

Procedure:

1- Two solutions already prepared; $KMnO_4^-$ (0.05N) and $K_2Cr_2O_7$-(0.05N).
2- Transfer with the aid of graduated pipette 4 mL of $KMnO_4$ solution to a volumetric flask (50mL) dilute to the mark with distilled water (represent standard solution A).
3- Transfer with the aid of graduated pipette 4 mL of $K_2Cr_2O_7$ solution to a volumetric flask (50mL) dilute to the mark with distilled water (represent standard solution B).
4- Dilute the unknown given solution that available in the (50 mL) volumetric flask dilutes to the mark.
5- Fill the first glass cell with reference solution (i.e. distilled water), this solution used for Zero adjustment the spectrophotometer. The second glass cell is used for standard solution A. The third glass cell standard solution B; while the fourth cell is used for the unknown solution.

Notice:

- The cell is usually filled to 3/4 of total height (Never fully filled to the edge).
- The whole four side surface must be wiped carefully for plotting absorbance curve for both A&B solutions and unknown solution.
- Record and tabulate the absorbance reading each (10 nm) increments within the range (400-570 nm).

Results and Calculations

1- With the aid of absorbance table of wavelength in nanometers, plot absorbance curve versus wavelength in nanometers for solutions A, B and, unknown. Find the wavelength of maximum absorbance (λ_{max}).
2- Find absorbance for the unknown solution at wavelength of maximum absorbance (λ_{max}).
3- Calculate the concentration of $KMnO_4^-$ and $Cr_2O_7^{2-}$ in admixture using Beer's Law for mixture.

$$A_{total\ at\ \lambda\max 1} = A_{Mn} + A_{Cr} \qquad (1)\ at\ \lambda_{\max 1}$$
$$A_{total\ at\ \lambda\max 2} = A_{Mn} + A_{Cr} \qquad (2)\ at\ \lambda_{\max 2}$$

Spectrophotometric Determination of Periodate on its reaction with iodide

$$IO_4^- + 11I^- + 8H^+ \leftrightharpoons 4I_3^- + 4H_2O$$

توضع مادة البيرايودات مؤكسد قوي جداً في السحاحة ($NaIO_4$ - 100 ppm)

Procedure

1- You have ($NaIO_4$ – 100 ppm) stock solution.
2- Prepare 5 volumetric flasks with a capacity (50 mL) of the stock solution at concentrations (3, 6, 9 and 12 ppm).
3- Add to each volumetric flasks (5 mL) of citrate buffer (pH=6).
4- Then add (5 mL) of (NaI – 0.15 M) to each volumetric flask.
5- Complete the volume to the mark (50 mL) with distilled water, then mix well and record the absorbance value at (355 nm).
6- Find the concentration of the unknown solution from the graph and linear equation.

Experiment No. 3

Determination of Phosphoric acid hydrochloric acid

Procedure

1- Glass electrode, single or combined, should be combined using two buffer solutions; mainly solution having (pH = 4.01) and a solution having (pH = 9.18).
2- The unknown solution which is a mixture of H_3PO_4 and HCl. Small amount of distilled water should be added whenever it is necessary so that the electrode should be immersed inside the solution. The solution is stirred with the aid of a magnetic stirrer. **The initial reading should be taken using the pH-meter.**
3- Sodium hydroxide (NaOH – 0.1N) should be added while constant stirring is achieved the pH-reading should be read. **Addition should be inversely proportioned with the range pH changes.**
4- Base addition should be follows:

(NaOH – 0.1N)	**pH**
1 mL,1 mL,……	3.5
0.2 mL,0.2 mL,……	6.5
1 mL,1 mL,……	8.5
0.2 mL,0.2 mL,……	10.2
1 mL,1 mL,……	11.3

5- pH values should be drowned versus added base volume; then calculate from equivalence point the concentration HCl and H_3PO_4 in mM.

Experiment No. 4

Sulphate ion Determination using Turbidimetric Method

Procedure:

1- Preparation of series of solutions in a gradual increase in volume (0.1, 0.2, 0.3, 0.4, and 0.5 mL). from potassium sulphate (0.05M) in (10 mL) volumetric flask.

2- To each flask and unknown solution add of constant volume (0.5 mL) of ($BaCl_2$ - 0.05M) and (0.5 mL) of the glycerol- alcohol solution and dilute to (10 mL) with distilled water and shaken gently in order obtain a uniform particle size.

Note:

- ✓ a white precipitate will appear

$$SO_4^{2-} + Ba^{2+} \Leftrightarrow BaSO_4$$

- ✓ Each flask should be shaken at the same rate and the same number of times to reproduce the measurements.

3- Plot the calibration graph NTU versus sulphate concentration.

4- Find the concentration of the unknown NTU from the calibration graph.

Experiment No. 5

Colorimetric Determination of Iron (Fe^{3+}) using ammonium thiocyanate

Procedure

1- Prepare (100 mL) of iron(III) stock solution having the concentration of ($2 \times 10^{3-}M$) starting with ammonium ferrous sulphate following the steps:
   a) Add approximately (10 mL) of distilled water then with using graduated cylinder (5 mL) of nitric acid (1:1) to the calculated weighted ammonium ferrous sulphate.
   b) Heat the solution above (in step a) cautiously and gently for three minutes.
   c) Cool the solution then transfer it quantitatively with precision to a volumetric flask of (100 mL), then continue to the mark, close and mix well.
2- Transfer with the aid of graduated pipette (5mL) the following volumes (1,2,3,4, and 5 mL) successively to five different volumetric flasks (size 50 mL).
3- To the five volumetric flasks and the unknown solution add (5mL) with the aid of graduated pipette (size 5mL) thiocyanate solution (10% w/v) then continue to the mark with distilled water. Close and mix well.
4- Record the absorbance for the solution using (400 nm - blue filter), (500 nm - green filter), (600nm - red filter).

Results and Calculations

1- Tabulate your data according to the following table:

Volume Fe^{2+} ($2 \times 10^{3-}M$)	**$[Fe^{2+}]$M**	**Absorbance (A)**		
		λ=400 nm	**λ=500 nm**	**λ=600 nm**

2- Prepare calibration graph by plotting (A) on the y- axis and **$[Fe^{2+}]$** in mole on the x - axis for each of (λ= 400, 500n, and 600 nm).
3- Find the unknown concentration for **$[Fe^{2+}]$** from the drawn calibration curve.

Colorimetric Determination Aspirin in pharmaceutical preparation

Procedure:

1- Prepare (800 ppm) of aspirin solution in a final volume of (100 mL) as follows:
   - Add (8 mL) of sodium hydroxide solution (o.25 M) using graduated cylinder to the weighted aspirin material.
   - Heat the mixture unit boiling and leave it to boil for (2 minutes).
   - Leave the solution to cool then transfer to volumetric flask capacity (100 mL) and complete the volume to the mark by distilled water.

2- Add to a series of volumetric flasks (50 mL) capacity the volume (0, 0.5, 1, 2, 3, and 4.5 mL) of the prepared solution in the previous step.

3- add to flask including the unknown flask (5mL) of ferric chloride solution (0.025M).

4- Dilute the solution to the mark by distilled water, mix well then, record the absorbance value of the solution at (525 nm).

5- Add (4 mL) of the prepared pharmaceutical drug of (800 ppm) (prepared in step 1) to a volumetric flask capacity of (50 mL) using the steps 3 and 4 then measure the absorbance ten times.

Results and Calculations

1- Arrange your results in a table include the aspirin concentration (ppm) versus absorbance.

2- Draw the curve for the determination of aspirin.

3- Find the concentration of the unknown and the prepared pharmaceutical drug from the graph and from linear equation.

4- Calculate the relative standard deviation %RDS and the percentage of relative error %E and the percentage recovery %Recovery for the prepared pharmaceutical drug in step 5.

5- Calculate the molar absorption coefficient $\in$ for the method from the following relation:

$$\in = b \times M.wt. \times 1000$$

Where $b = slop\ of\ curve\ mL.\mu g^{1-}$

$$M.wt. = molecular\ weight\ of\ aspirin\ 180.2\ g.mol^{1-}$$

6- Calculate the necessary weight required to prepare aspirin solution at a concentration of (800 ppm) in a final volume of (100 mL).

Experiment No. 7

Estimation of the complex structure by mole ration and the continuous variation method

Procedure:

1- Prepare (100 mL) of ammonium ferrous sulphate ($5 \times 10^{4-}$M).
2- Add to a series of solutions (25 mL) capacity a constant volume of (2 mL) of prepared iron sulphate solution.
3- Add to each volumetric flask (2mL) of buffer solution (pH=5 – acetic acid), (0.1M – sodium acetate) and (1mL – hydroxylamine hydrochloride (5% w/v)).
4- Add variable volumes (1, 2, 3, 4, 6, 8, and 10 mL) of 1,10-phenanathroline ($5 \times 10^{4-}$M).
5- Dilute all the solutions to the mark by distilled water, then mix well, and leave a side for (10 minutes) for complete reaction.
6- Read the absorbance (A) at (510 nm).
7- Repeat steps 2 to 6 using variable volumes of each iron solution (1, 1.5, 2, 2.5, 3, 5, and 7 mL) and 1,10-phenanathroline (9, 8.5, 8, 7.5, 7, 5, 3 mL) with ($5 \times 10^{4-}$M). on the find volume to be constant volume of (10 mL)

Results and Calculations

1- Tabulate your results in a table include volume of each ferrous solution (V_m) and the volume of 1,10-phenanathroline (V_L) against absorbance for each method.
2- Draw the value of (V_m / V_L) versus absorbance for first method.
3- Draw the value of ($V_m / V_m + V_L$) against the value of absorbance for second method.

Experiment No. 8

Photometric Titration of Permanganate with Oxalate

Procedure:

1- Prepare a standard solution of sodium oxalate (0.01M - $Na_2C_2O_4$) from a (0.01M) standard solution in a volumetric flask in (100 mL).
2- Prepare 7 volumetric flasks of (25 mL) capacity. The first one is the reference one.
3- To each volumetric flask add (2.5 mL) of unknown concentration of $KMnO_4$ solution then (2 mL) of (2 M – H_2SO_4) is added (the catalyst). The first volumetric flask is continued to the mark with distilled water, while the rest (six volumetric flask), 0.5, 1, 1.5, 2, 2.5, and 3 mL of (0.01M – oxalate solution) is added to volumetric flask no. 2, 3, 4, 5, 6, and 7 successively, followed by the continuation of the volume to the mark with distilled water.
4- The solution transferred to large supplied tube for the purpose of heating for a while until the disappearance of the color of the one of the tubes (pink coloration of $KMnO_4$). Then all tubes are removed from heating, and cooled.
5- Absorbance is measured at (525 nm), starting from tube no.1 (reference tube).
6- A relation of absorbance (A) and the volume of $Na_2C_2O_4$ for finding the unknown concentration of $KMnO_4$.