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Chemistry
Unit A2 2: Practical Manual

Questions Solutions

Practical 7.1

Titrate iodine with sodium thiosulfate using starch indicator and hence estimate oxidising agents by their reaction with excess acidified potassium iodide (spec ref 5.3.1)

1. Moles $S_2O_3^{2-}$ = 0.200 × 28.6/1000 = 0.00572

   Moles I_2 = 0.00572/2 = 0.00286 = moles ClO^-

   Conc ClO^- = 0.00286/0.0300 = 0.0953 mol dm^{-3}

Practical 7.2

Titrate acidified potassium manganate(VII) with reducing agents (spec ref: 5.3.2)

1. Calculated amount will be less than 200 mg due to: not all of the iron(II) in the tablets dissolved in the sulfuric acid, possible loss of solution during transfer, inaccurate measurements during titration.

2. A lower titre would be obtained resulting in a lower calculated mass of iron(II) sulfate in the tablet.

3. Chloride ions in hydrochloric acid can reduce manganate(VII) ions to Mn^{2+}.

Practical 7.3

Determine the purity of a Group II metal oxide or carbonate by back titration (spec ref: 5.3.4)

1. Bubble the gas through limewater which will change from colourless to milky.

2. The titration is between a strong acid and strong base.

Practical 8.1

Carry out paper and thin-layer chromatography and measure the R_f values of the components and interpret chromatograms (spec ref: 5.4.1/5/4/3)

1. The pencil line will not dissolve in the mobile phase.

2. The components in each indicator would have a different affinity for the mobile phase (compared to the stationary phase) and so would move up the stationary phase by different amounts.

3. The R_f values for a pure sample will be equal to the book values for a given mobile and stationary phase.

4. Iodine and ninhydrin act as developing agents and allow the spots to be observed on the chromatogram.

5. The amino acids produced often have similar R_f values.

6. Draw a base line using a pencil close to the bottom of the paper/thin-layer plate.
   Spot the samples onto the paper/thin-layer plate and allow to dry.
   Place the paper in the tank and allow to run in first solvent.
   Mark the solvent front and allow to dry.
   Rotate the paper/thin-layer plate through 90° and run in the second solvent.
   Mark the solvent front and allow to dry.
   Measure the distance travelled by the solvent front, and the distance moved by each spot (to centre If the substances to be separated are colourless, the spots are made visible by spraying with a locating agent e.g. ninhydrin.

Practical 9.1

Use ethylene diamine (1,2-diaminoethane), phenylamine and aqueous ammonia to demonstrate ligand replacement based on lone pair availability (spec ref: 5.5.8)

1. $[Cu(H_2O)_6]^{2+}$ + $2C_6H_5NH_2$ → $[Cu(H_2O)_4(C_6H_5NH_2)_2]^{2+}$ + $2H_2O$

2. $[Cu(H_2O)_4(C_6H_5NH_2)_2]^{2+}$ + $4NH_3$ → $[Cu(H_2O)_2(NH_3)_4]^{2+}$ + $2C_6H_5NH_2$ + $2H_2O$

3. $[Cu(H_2O)_2(NH_3)_4]^{2+}$ + $2H_2NCH_2CH_2NH_2$ → $[Cu(H_2O)_2(H_2NCH_2CH_2NH_2)_2]^{2+}$ + $4NH_3$

4. The ammonia or phenylamine would not be able to replace the ethylene diamine ligands, so the only colour change corresponds to hexaaquacopper(II) ions reacting with ethylene diamine.

Practical 9.2

Demonstrate the relative strengths of ligands using hydrated copper(II) ions and hydrochloric acid (spec ref: 5.5.9)

1. Blue to yellow.

2. $[Cu(H_2O)_6]^{2+}$ + $4Cl^-$ → $[CuCl_4]^{2-}$ + $6H_2O$

3. The chloride ion is large so only four will fit around and bond with the central copper(II) ion.

4. There is an increase in entropy/the free energy change is negative.
   The concentration of chloride ions is high.

Practical 9.3

Carry out qualitative tests for the formation of transition metal hydroxides with sodium hydroxide and aqueous ammonia (spec ref: 5.5.12)

1. $[Co(H_2O)_6]^{2+}$ + $2OH^-$ → $[Co(H_2O)_4(OH)_2]$ + $2H_2O$

   $[Cu(H_2O)_6]^{2+}$ + $2OH^-$ → $[Cu(H_2O)_4(OH)_2]$ + $2H_2O$

   $[Fe(H_2O)_6]^{2+}$ + $2OH^-$ → $[Fe(H_2O)_4(OH)_2]$ + $2H_2O$

   $[Cr(H_2O)_6]^{3+}$ + $3OH^-$ → $[Cr(H_2O)_3(OH)_3]$ + $3H_2O$

   $[Fe(H_2O)_6]^{3+}$ + $3OH^-$ → $[Fe(H_2O)_3(OH)_3]$ + $3H_2O$

   $[Mn(H_2O)_6]^{2+}$ + $2OH^-$ → $[Mn(H_2O)_4(OH)_2]$ + $2H_2O$

2. $[Cr(H_2O)_3(OH)_3]$ + $3OH^-$ → $[Cr(OH)_6]^{3-}$ + $3H_2O$

3. $[Co(H_2O)_4(OH)_2]$ + $6NH_3$ → $[Co(NH_3)_6]^{2+}$ + $4H_2O$ + $2OH^-$

   $[Cr(H_2O)_3(OH)_3]$ + $6NH_3$ → $[Cr(NH_3)_6]^{3+}$ + $3H_2O$ + $3OH^-$ *

   *reaction is concentration dependant

4. $[Ni(H_2O)_6]^{2+}$ + $2OH^-$ → $[Ni(H_2O)_4(OH)_2]$ + $2H_2O$

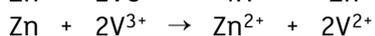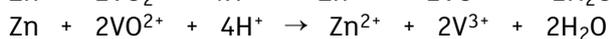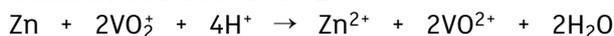 $[Ni(H_2O)_4(OH)_2]$ + $6NH_3$ → $[Ni(NH_3)_6]^{2+}$ + $4H_2O$ + $2OH^-$

Practical 9.4

Carry out the reduction of acidified ammonium metavanadate with zinc and observe the sequence of colours (spec ref: 5.5.13)

1. VO_2^+ +5   yellow
   VO^{2+} +4   blue
   V^{3+} +3   green
   V^{2+} +2   violet

2. The Zn/Zn^{2+} standard electrode potential is more negative than any of the vanadium ion standard electrode potentials. Zinc can therefore reduce vanadium from an oxidation state of +5 to +2.
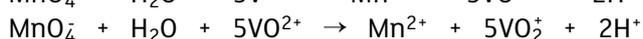 $Zn + 2VO_2^+ + 4H^+ \rightarrow Zn^{2+} + 2VO^{2+} + 2H_2O$
   $Zn + 2VO^{2+} + 4H^+ \rightarrow Zn^{2+} + 2V^{3+} + 2H_2O$
   $Zn + 2V^{3+} \rightarrow Zn^{2+} + 2V^{2+}$

3. Zinc reacts with the sulfuric acid.
   $Zn + H_2SO_4 \rightarrow ZnSO_4 + H_2$

1. The violet solutions turns green, blue and eventually yellow.

2. The MnO_4^-/Mn^{2+} standard electrode potential is more positive than any of the vanadium ion standard electrode potentials. Manganate(VII) can therefore oxidise vanadium from an oxidation state of +2 to +5.
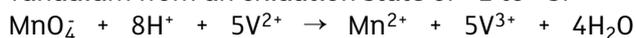 $MnO_4^- + 8H^+ + 5V^{2+} \rightarrow Mn^{2+} + 5V^{3+} + 4H_2O$
   $MnO_4^- + H_2O + 5V^{3+} \rightarrow Mn^{2+} + 5VO^{2+} + 2H^+$
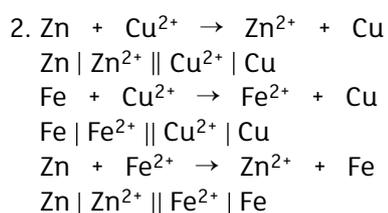 $MnO_4^- + H_2O + 5VO^{2+} \rightarrow Mn^{2+} + 5VO_2^+ + 2H^+$

Practical 10.1

Determine the electrode potentials of a series of cells and predict their values using standard electrode potentials (spec ref: 5.6.2)

1.

Cell	Reduction half-cell	Oxidation half-cell	Calculated cell potential difference / V	Measured cell potential difference / V
Zinc/copper	copper	zinc	+1.10	
Iron/copper	copper	iron	+0.78	
Iron/zinc	iron	zinc	+0.32	

2. $Zn + Cu^{2+} \rightarrow Zn^{2+} + Cu$
   $Zn \mid Zn^{2+} \parallel Cu^{2+} \mid Cu$
   $Fe + Cu^{2+} \rightarrow Fe^{2+} + Cu$
   $Fe \mid Fe^{2+} \parallel Cu^{2+} \mid Cu$
   $Zn + Fe^{2+} \rightarrow Zn^{2+} + Fe$
   $Zn \mid Zn^{2+} \parallel Fe^{2+} \mid Fe$

3. The calculated value should compare favourably with the accepted value of -0.13 V.

4. $Zn + Pb^{2+} \rightarrow Zn^{2+} + Pb$
   $Zn \mid Zn^{2+} \parallel Pb^{2+} \mid Pb$
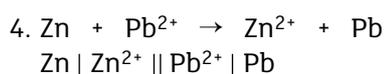
Practical 11.2

Prepare aspirin using salicylic acid and ethanoic anhydride (spec ref: 5.11.7)

1. The yield will probably be no more than 50%. Reasons include: reaction does not go to completion, side reactions, loss in transfer.
   Theoretical yield = (2.00/138) x 180 = 2.61 g
   % yield = (actual yield/2.61) x 100

2. The melting point of the pure product is 139 °C. A pure sample will have a sharp melting point within this +/- 1 °C of this value. An impure sample will begin melting lower than this temperature and melt over a broader range.

3. If a purple colour is observed this indicates the presence of a phenol group ie -OH group attached to a benzene ring. This suggests the presence of an impurity, the reactant salicylic acid.

4. Ethanoic acid: the reaction is slow and does not go to completion.
   Ethanoyl chloride: the reaction produces the by-product hydrogen chloride, which is a toxic gas.

5. Addition of cold water converts any unreacted ethanoic anhydride to ethanoic acid.
   $(CH_3CO)_2O + H_2O \rightarrow 2CH_3COOH$

6. The crude solid should have a low solubility in cold solvent and a high solubility in the solvent when warm. A minimum volume should be used as the solid may not crystallise out of solution as the solution is cooled if too much solvent is used.

Practical 11.3
Use chromatography to compare the purity of laboratory-made aspirin with commercial tablets (spec ref: 5.11.8)

1. R_f values are calculated by: distance travelled by spot/distanced travelled by solvent.

2. The crude sample will show a minimum of two spots, the recrystallised sample should have one spot and the commercial sample should have one spot with the same R_f value as the recrystallised sample and may show additonal spots.