EXERCISE 4

Investigating Transition Metal Complexes

INTRODUCTION

Colour is a well known property of the transition metals. The colour produced as parts of the visible spectrum are due to electron transitions between energy levels. The colour we see is due to absorption by electrons in the d orbitals therefore, if the atom has no d electrons or a full d orbital with 10 electrons, the compound will absorb outside the visible region, and appear white or colourless.

Ligands – Splitting the d orbitals

In a transition metal atom the d orbitals all have the same energy level, however ligands split the d orbitals into two groups with a difference in energy of ΔE. If the ligand of a transition metal is changed it alters the value for ΔE and therefore the colour will also change.

Factors affecting the ΔE value include:

- The type and size of the ligand.
- The bond strength between the ligand and metal ion.
- The shape and co-ordination number of the complex.
- The oxidation state.

UV-Visible Spectroscopy - Analysis of Colour

A UV-visible spectrometer can be used to measure absorption of light in the ultra-violet and visible region. A typical spectrometer has two sources, usually a deuterium lamp to provide frequencies in the ultra-violet region and a tungsten lamp to provide the visible frequencies. To run a sample the instrument needs to compare the sample solution with a reference or blank solution. This reference solution is the solvent being used for the sample solution. The blank or reference is run first for a single beam instrument or in parallel with the samples for a double beam instrument. A diffraction grating or prism is used to separate the radiation into the different frequencies producing monochromatic radiation. The spectra plotted show the absorbance of the sample at particular wavelengths.

METHOD

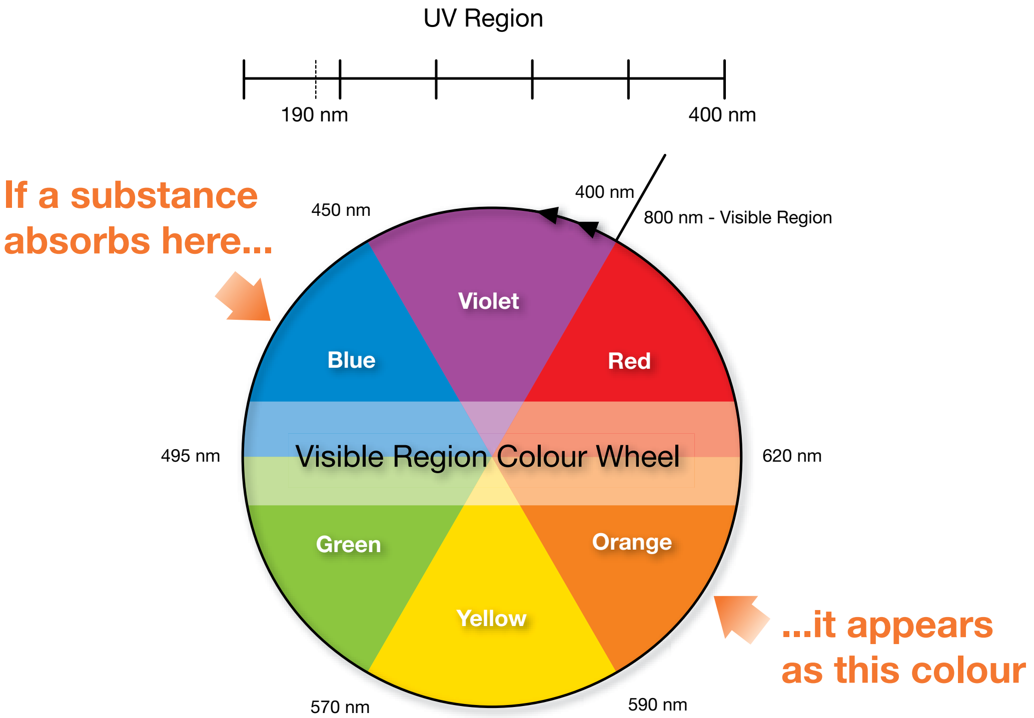
Investigation 1

The Colour in Transition Metal Complexes

1. Prepare a blank sample by filling a cuvette with the appropriate solvent and stopper with a lid.
2. The demonstrator will set up the spectrometer to scan the visible region from 350-900 nm. Run a blank and each sample as instructed. Print out the spectrum and note the wavelength for each of the absorbance peaks.
3. Investigate how the absorbance changes as the ligands are changed, record the changes in absorbance in the table provided.

Wavelengths of Different Coloured Visible Light

Red	620-750 nm
Orange	590-620 nm
Yellow	570-590 nm
Green	496-570 nm
Blue	450-495 nm
Violet	380-450 nm



Investigation 2

Beer-Lambert Law

Experiment

1. You are provided with a solution of potassium permanganate of accurately known concentration (4.0×10^{-4} mol dm^{-3}).
2. Use this solution to make up 50 cm^3 of each of the following potassium permanganate solutions using a 50 cm^3 burette to dispense your stock solution to the volumetric flasks.

Concentration	Stock solution in 50 cm^3 de-ionised water
3.2×10^{-4} mol dm^{-3}	40 cm^3
2.4×10^{-4} mol dm^{-3}	30 cm^3
1.6×10^{-4} mol dm^{-3}	20 cm^3
0.8×10^{-4} mol dm^{-3}	10 cm^3

3. Using the spectrophotometer measure the absorbance of each of your solutions in the visible region. The peak should occur at 540 nm. Also measure the absorbance of the unknown solution and record all data in the table provided.
4. Show that the Beer-Lambert Law is obeyed by plotting a graph of absorbance (y- axis) versus concentration (x-axis) using the graph paper provided. Draw the line of best fit through your points and the origin. Determine the concentration of the unknown solution from the graph.

Questions

1. How do you know if the Beer-Lambert Law has been obeyed?
2. What is the concentration of the unknown solution?

MATERIALS

Investigation 1

The Colour in Transition Metal Complexes

Chemicals

- Transition Metal compounds selection of different colours – eg copper sulphate, copper chloride, cobalt sulphate, vanadyl sulphate etc.
- Dilute ammonia solution
- 6M HCl
- Dilute sodium hydroxide
- De-ionised/distilled water

Apparatus

- Disposable plastic cuvettes and stoppers
- Wash bottles x 4
- 100 cm^3 beakers 4
- 1 box pasteur pipettes and teats
- Tissues
- Spatulas

Investigation 2

Beer Lambert Law

Chemicals

- Potassium permanganate solution 4.0×10^{-4} mol dm^{-3} (0.016 g in 250 cm^3)
- Unknown 25 cm^3 stock in 50 cm^3
- De-ionised water

Apparatus

- 5 x 50 cm^3 Volumetric Flasks
- Burette
- Funnel
- Disposable plastic cuvettes and stoppers
- Wash bottles x 4
- 1 box pasteur pipettes and teats
- Tissues
- Graph paper

Instrument

- UV-visible Spectrometer (integral printer and paper)
- Laptop (*optional*)
- Printer (*optional*)
- Connection cables x 2 (*optional)*

Set up for laptop and printer use:

- Connect UV-vis to laptop via left hand front USB port (Com 5)
- Connect printer to any USB port
- From spectrometer menu Select printer / auto print on / Computer USB / OK
- Open PVC program, set auto print to on or off depending on requirements.

RESULTS

Investigation 1

The Colour in Transition Metal Complexes

Changing Ligand

LIGAND	COLOUR	SHAPE	ABSORBANCE VALUE
$[Cu(H_2O)_6]^{2+}$	Blue	Octahedral	880 nm
$[Cu(NH_3)_4(H_2O)_2]^{2+}$	Blue-Violet	Octahedral	660 nm

Changing Co-ordination Number

LIGAND	COLOUR	SHAPE	ABSORBANCE VALUE
$[Cu(H_2O)_6]^{2+}$	Blue	Octahedral	880 nm
$[CuCl_4]^{2-}$	Yellow	Tetrahedral	880 nm and 380 nm

Changing Oxidation State

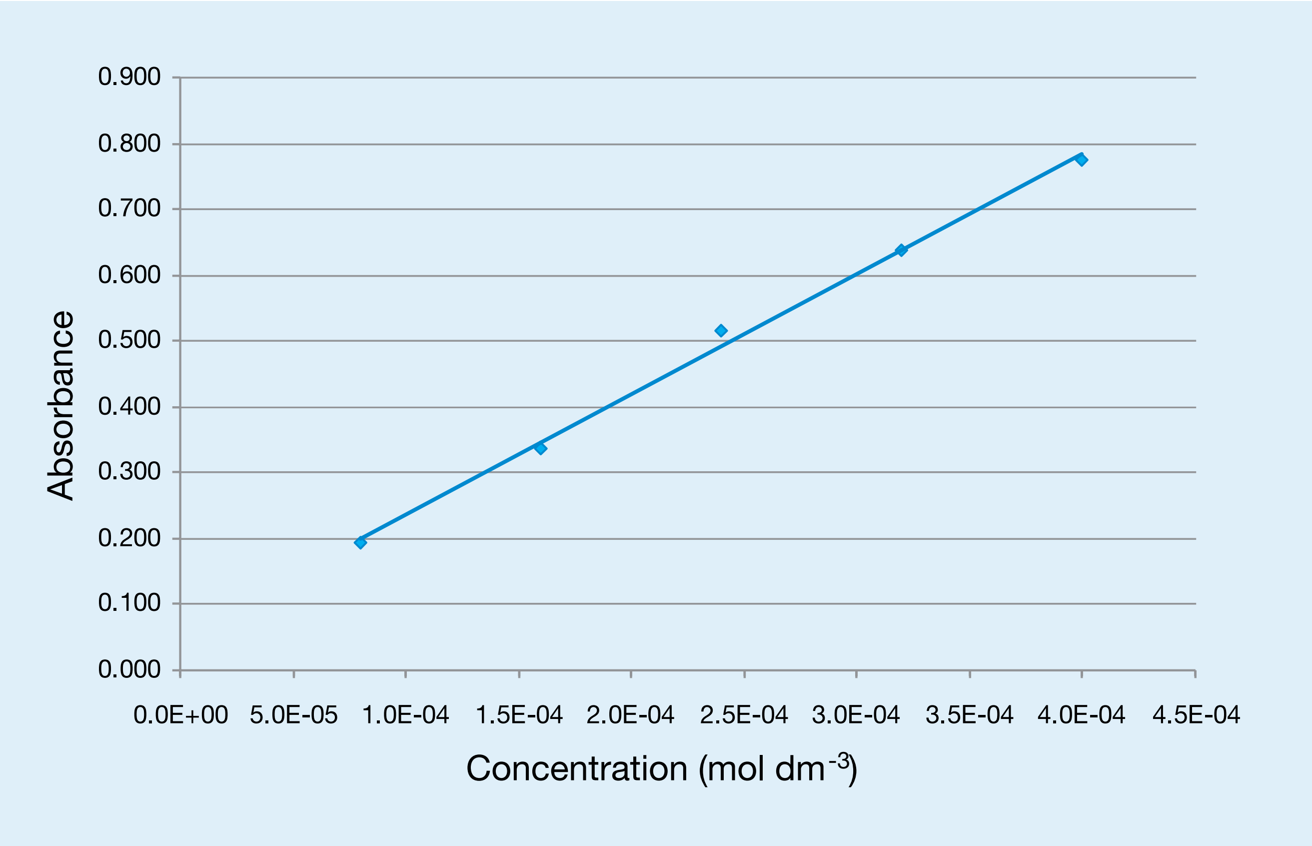
LIGAND	COLOUR	SHAPE	ABSORBANCE VALUE

Investigation 2

Beer Lambert Law

CONCENTRATION (mol dm^{-3})	ABSORBANCE
4.0×10^{-4}	0.775
3.2×10^{-4}	0.639
2.4×10^{-4}	0.516
1.6×10^{-4}	0.336
0.8×10^{-4}	0.194
Unknown	0.395

Beer-Lambert Calibration Plot – Potassium Permanganate Solution



Answers

1. The plot of absorbance versus concentration is a straight line graph.

2. From the graph the solution is 2.1×10^{-4} mol dm^{-3} the unknown solution prepared was 2.0×10^{-4} mol dm^{-3}

STUDENT WORK SHEET

Investigation 1

The Colour in Transition Metal Complexes

Changing Ligand

LIGAND	COLOUR	SHAPE	ABSORBANCE VALUE
$[Cu(H_2O)_6]^{2+}$			
$[Cu(NH_3)_4(H_2O)_2]^{2+}$			

Changing Co-ordination Number

LIGAND	COLOUR	SHAPE	ABSORBANCE VALUE
$[Cu(H_2O)_6]^{2+}$			
$[CuCl_4]^{2-}$			

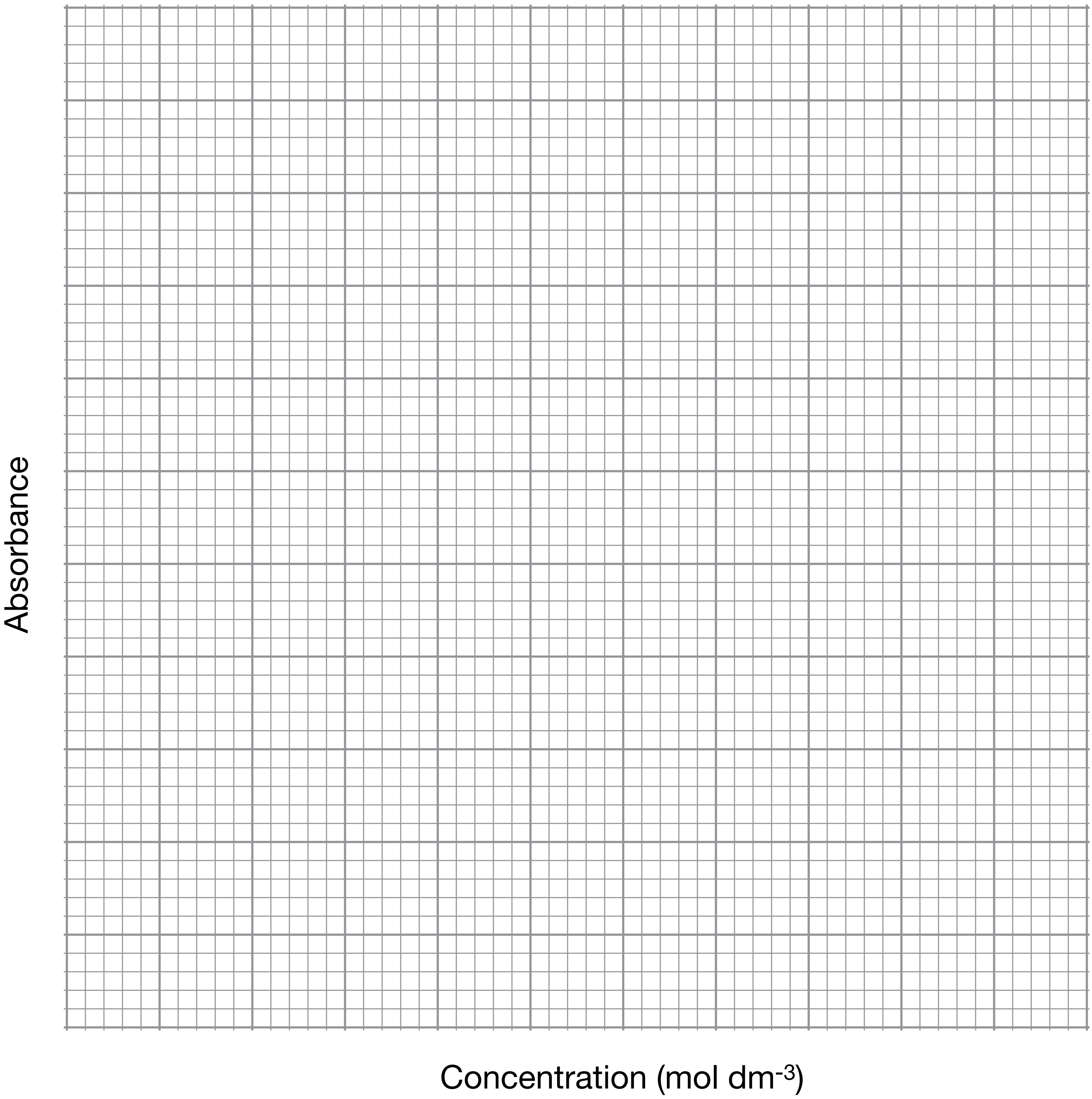
Changing Oxidation State

LIGAND	COLOUR	SHAPE	ABSORBANCE VALUE

Investigation 2

Beer Lambert Law

CONCENTRATION (mol dm^{-3})	ABSORBANCE
4.0×10^{-4}	
3.2×10^{-4}	
2.4×10^{-4}	
1.6×10^{-4}	
0.8×10^{-4}	
Unknown	



Concentration of Unknown: ______________________