

Transition elements and complex compounds microscale experiment

Learning objectives

- 1 Develop an understanding of sustainable practices by carrying out a microscale experiment to minimise the use and disposal of toxic substances.
- 2 Relate experimental observations to the oxidation state, ligand type and coordination number of transition element compounds.

Introduction

During this microscale experiment you will look for evidence of complex formation and changes in oxidation state – two important characteristics of transition elements.

Instructions

1. Read the **Health, safety and technical notes** and **Chemical hazards table** before you begin.
2. Cover page 3 with a clear plastic sheet (if it is not laminated).
3. Draw a suitable table to record your observations from each experiment.
4. Place a drop of each solution from vanadium to zinc in the appropriate boxes in the **Microscale table**. (Note: one drop will act as a reference).
5. For each transition element solution, add the reactants described in the **Experimental methods** table to the appropriate box in the experiment row of the **Microscale table**. Observe each box carefully and compare it to the reference box, noting down any colour changes for each reaction in your results table.

[Note: if your microscale experiment does not clearly show results, ask your teacher if you can scale it up into a test tube reaction.]

Lab notes

Once you have noted initial observations in your table:

- Write possible explanations for each experimental observation you have made.
- Discuss your findings with your neighbour and then your teacher (they will provide further detail for each experiment).
- Complete your experimental notes by adding any final conclusions.

Health, safety and technical notes

Personal protective equipment (PPE)

Wear eye protection throughout (safety goggles). Your teacher may provide gloves.

Equipment

- Safety goggles
- Student worksheet (page 3 laminated)
- Clear plastic sheet (eg, acetate sheet overlay if you are not using a laminated worksheet)
- Magnifying glass

Chemical reagents

Solutions should be in plastic dropper bottles or plastic pipettes.

- Acidified potassium dichromate, 0.2 mol dm^{-3}
- Potassium manganate(VII), 0.2 mol dm^{-3}
- Cobalt(II) nitrate, 0.5 mol dm^{-3}
- Ammonia solution, 2 mol dm^{-3}
- Ammonium vanadate(V), 0.1 mol dm^{-3}
- Hydrochloric acid, 1 mol dm^{-3}
- Sulfuric acid, 1 mol dm^{-3} (to acidify reactions where necessary)
- Hydrogen peroxide, 5% solution
- Sodium hydroxide, 1 mol dm^{-3}
- Copper(II) sulfate, 0.2 mol dm^{-3}
- Iron(II) sulfate, 0.2 mol dm^{-3} (ensure solution is acidified with sulfuric acid for manganate reaction)
- Iron(III) nitrate, 0.2 mol dm^{-3}
- Potassium iodide, 0.2 mol dm^{-3}
- Starch solution (freshly made)
- Zinc metal granules (or metal foil pieces)
- Zinc(II) sulfate solution, 0.2 mol dm^{-3}

Experimental methods

Place a drop of each solution from vanadium to zinc in the appropriate reference and experiment boxes in the **Microscale table** below. Only add the reactants described to the experiment box of the appropriate transition metal solution. Compare your experimental observations to the reference box.

Solution Transition metal	Experiment instruction
Ammonium vanadate(V) Vanadium (V^{5+})	Add one drop of dilute hydrochloric acid and a small piece of zinc.
Acidified potassium dichromate(VI) Chromium (Cr^{6+})	Add one drop of 5% hydrogen peroxide solution.
Potassium manganate(VII) Manganese (Mn^{7+})	Add one drop of the acidified iron(II) sulfate solution.
Iron(III) nitrate Iron (Fe^{3+})	Add one drop of potassium iodide solution. After one minute, add one drop of starch solution.
Cobalt(II) nitrate Cobalt (Co^{2+})	Add two drops of ammonia solution.
Copper(II) sulfate Copper (Cu^{2+})	Add two drops of ammonia solution.
Zinc(II) sulfate Zinc (Zn^{2+})	Add two drops of sodium hydroxide solution.

Microscale table

Transition metal ion	V^{5+}	Cr^{6+}	Mn^{7+}	Fe^{3+}	Co^{2+}	Cu^{2+}	Zn^{2+}
Reference							
Experiment							

Chemical hazards

Carefully read the hazards table below before starting the experiment. Wear safety goggles and take care not to get the solutions on your skin.

<ul style="list-style-type: none"> Acidified potassium dichromate(VI), $\text{K}_2\text{Cr}_2\text{O}_7$, 0.2 mol dm^{-3} is corrosive (skin, eyes), harmful (ingestion), an irritant (respiratory), a sensitiser (skin, respiratory), serious health hazard (RE) and serious health hazard (CMR). Avoid skin contact.
<ul style="list-style-type: none"> Potassium manganate(VII), KMnO_4, 0.2 mol dm^{-3} is an irritant (skin, eyes), suspected of damaging the unborn child, stains skin and clothes. Avoid skin contact.
<ul style="list-style-type: none"> Cobalt(II) nitrate, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 0.5 mol dm^{-3} is a sensitiser (skin, respiratory) and a serious health hazard (CMR – hazard to fertility and/or the unborn child).
<ul style="list-style-type: none"> Ammonia solution, $\text{NH}_3(\text{aq})$, 2 mol dm^{-3} is corrosive (eyes) and an irritant (skin).
<ul style="list-style-type: none"> Ammonium vanadate(V), NH_4VO_3, 0.1 mol dm^{-3} (acidified with sulfuric acid) is an irritant (skin, eyes).
<ul style="list-style-type: none"> Sulfuric acid, H_2SO_4, 1 mol dm^{-3} is an irritant (skin, eyes).
<ul style="list-style-type: none"> Hydrogen peroxide, 5% solution $\text{H}_2\text{O}_2(\text{aq})$ is an irritant (eyes).
<ul style="list-style-type: none"> Sodium hydroxide solution, $\text{NaOH}(\text{aq})$, 1 mol dm^{-3} is corrosive (skin, eyes).
<ul style="list-style-type: none"> Copper(II) sulfate solution, $\text{CuSO}_4(\text{aq})$, 0.2 mol dm^{-3} is corrosive (eyes) and an irritant (skin).
<ul style="list-style-type: none"> Iron(II) sulfate, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}(\text{aq})$, 0.2 mol dm^{-3} is an irritant (skin, eyes).
<ul style="list-style-type: none"> Iron(III) nitrate, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}(\text{aq})$, 0.2 mol dm^{-3} can be an irritant (skin, eyes).
<ul style="list-style-type: none"> Zinc(II) sulfate, $\text{ZnSO}_4(\text{aq})$, 0.2 mol dm^{-3} is corrosive (eyes).
<p>The following are of low hazard:</p> <ul style="list-style-type: none"> Hydrochloric acid, $\text{HCl}(\text{aq})$, 1 mol dm^{-3} Potassium iodide, KI, 0.2 mol dm^{-3} Starch solution Zinc metal, $\text{Zn}(\text{s})$, granules (or metal foil pieces – take care of sharp edges).

Questions

- Which solution(s) did not give the experimental results you might have expected for a transition element?
- Explain why this is. (Hint: consider the electron configuration of this metal.)
- Explain how the changing oxidation states of the transition metals result in the colour changes observed.
- Suggest any other factor(s) that may be responsible for changing the colour of a transition metal compound.
- Extension question:** The colours of transition element compounds are usually explained in terms of d-d transitions. However, this is not always the case. In the manganese experiment, the oxidation state was 7+. Deduce why this means that d-d electron transitions are not possible in this case.