

Transition elements and complex compounds microscale experiment

This resource accompanies the article **Teaching transition metals and complex ions at post-16** in *Education in Chemistry* which can be viewed at: rsc.li/3szf11y. The article provides classroom tips, activities and ideas to prevent misconceptions.

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.

Overview

In this practical activity, learners conduct a series of microscale experiments to model sustainable practices in the laboratory as they investigate the chemistry of the transition elements. Learners will use experimental evidence to identify reactions involving complex formation and changes in metal oxidation state. See **Teaching notes and expected observations** for each reaction. The experiment will take approximately 20 minutes.

Core concepts: transition elements, variable oxidation states, redox and precipitation reactions, complex compounds, chemistry and colour.

Safety and hazards

Read our standard health and safety guidance (rsc.li/3vDwEuC) and carry out a risk assessment before running any practical. See the accompanying **Technician notes** download for preparation notes, CLEAPSS hazcards, recipe books and disposal.

Equipment

- Safety goggles
- Chemical resistant gloves (optional – none of the chemicals require gloves but learners should avoid skin contact with some of the solutions)
- Student worksheet (page 3 laminated)
- Clear plastic sheet (eg, acetate sheet overlay if you are not using a laminated worksheet)
- Magnifying glass

Chemical reagents

Provide solutions in plastic dropper bottles or in plastic pipettes. See the accompanying guidance on apparatus and techniques for microscale chemistry (rsc.li/3nRRXpz), including instructions for preparing a variety of solutions.

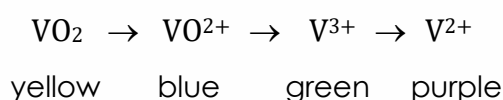
- Acidified potassium dichromate, 0.2 mol dm⁻³ (if not acidified, add excess sulfuric acid 1 mol dm⁻³ to the stock solution bottle)
- Potassium manganate(VII), 0.2 mol dm⁻³
- Cobalt nitrate, 0.5 mol dm⁻³
- Ammonia solution, 2 mol dm⁻³
- Ammonium vanadate(V), 0.1 mol dm⁻³
- Hydrochloric acid, 1 mol dm⁻³
- 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

Teaching notes and expected observations

Vanadium

Colour change = change in oxidation state

Bubbles (of hydrogen) are seen. The yellow colour of the ammonium vanadate's vanadium(V) ions (VO₂⁺) gradually changes (as the vanadium is reduced) to blue due to the formation of vanadium (IV) ions (VO²⁺). The colour changes to green due to vanadium(III) ions (V³⁺) and possibly to lilac due to vanadium(II) ions (V²⁺) (although this species is a strong reducing agent and is very air-sensitive).

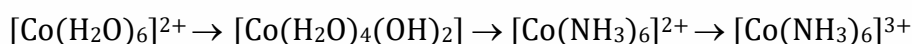


See related resource: rsc.li/43AT8wZ

Cobalt

Colour change = ligand exchange and change in oxidation state

For the cobalt nitrate solution, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, the addition of one drop of ammonia acts as a base to remove hydrogen ions from the hexaaqua cobalt(II) ion, $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$, to give the green hydroxide precipitate $[\text{Co}(\text{H}_2\text{O})_4(\text{OH})_2]$. Addition of further ammonia dissolves the precipitate and the ammonia acts as a ligand to replace water (ligand exchange) to give hexaamminecobalt(II) ions, $[\text{Co}(\text{NH}_3)_6]^{2+}$, (greenish brown) which will further oxidise in air to produce $[\text{Co}(\text{NH}_3)_6]^{3+}$ (darker brown).

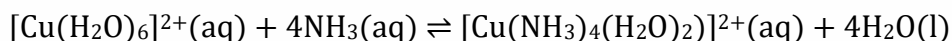


Consider doing the cobalt equilibrium demonstration (rsc.li/3QeUESe) to show change in ligand type and cobalt complex coordination numbers.

Copper

Colour change = ligand exchange

The copper sulfate solution provides the hexaaqua copper(II) ions, $[\text{Cu}(\text{H}_2\text{O})_6]^{2+}$, which upon addition of ammonia solution forms a light blue precipitate of tetraaquadihydroxocopper(II), $[\text{Cu}(\text{H}_2\text{O})_4(\text{OH})_2](\text{s})$, together with the developing deep blue solution colour change due to formation of tetraamminediaquacopper(II) ions, $[\text{Cu}(\text{NH}_3)_4(\text{H}_2\text{O})_2](\text{aq})$.



See related experiment: rsc.li/471ypVX

Zinc

Colour change = not observed

A gelatinous white precipitate of zinc hydroxide, $\text{Zn}(\text{OH})_2$, is observed. Zinc is not a transition metal because it only has one oxidation state in its compounds and the Zn^{2+} ion has a full d sub-shell.

Answers

1. Zinc solution.
2. Zinc is not a transition element. Its electronic configuration is $1s^2 2s^2 2p^6 3s^2 3p^6 3d^{10} 4s^2$ or $[\text{Ar}] 3d^{10} 4s^2$. It has a full d sub-shell – it does not have an incomplete or partially filled d sub-shell which is a requirement to be classified as a transition element.
3. For a given transition metal ion, a change in oxidation number can change the degree of d-orbital splitting caused by the coordinated ligands. This will result in different d-orbital energy level differences and therefore different frequencies/wavelengths of visible light being absorbed and likewise complementary colour observed.
4. For a given transition metal complex shape (coordination number), changing the ligand will result in differing degrees of d orbital splitting. Strong field ligands (eg, CN^-) cause a greater d orbital split (greater energy difference) and weak field ligands (eg, I^-) will cause a smaller split (small energy difference). Therefore, the wavelengths/frequencies absorbed and complementary complex colour observed will vary depending upon the particular ligand.

Another factor that can cause a change in colour is the coordination number. The d orbitals split differently depending on the complex geometry (coordination number) which gives rise to different energy level differences and therefore different d-d electron transitions possible. For example, octahedral (coordination number 6) complexes have greater d orbital splitting and therefore a greater energy level difference between d orbitals for electron transitions to occur than tetrahedral (coordination number 4) complexes have. Therefore, for a given transition element, an octahedral complex might be expected to absorb visible light with higher frequency/shorter wavelength and thus the complementary colour observed will be of lower frequency/longer wavelength (more towards red end of the visible range) than in a corresponding tetrahedral complex.

(Note: a change in coordination number also involves a change in the ligand type itself, so differences in the observed colours of such transition metal complexes will be due to both of these changes (complex shape and coordination number).

Extension

5. The electronic configuration of elemental manganese is $[\text{Ar}] 3d^5 4s^2$. The manganese ion therefore has the electronic configuration $[\text{Ar}]$ or $1s^2 2s^2 2p^6 3s^2 3p^6$. Thus, manganese in the oxidation state (VII) has no d electrons, so no d-d transitions are possible.