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| Chemical Investigations |
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| Teacher/Technician Guide |



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Manganese in Steel

*UNIT 1 PPA 2*

**Introduction**

Colorimetry is an analytical technique used to determine the concentrations of coloured substances in solution. It relies on the fact that a coloured substance absorbs light of a colour complementary to its own and furthermore, the amount of light it absorbs (absorbance) is proportional to its concentration.

Colorimetry is particularly suited to the determination of manganese in steel because the manganese can be converted into permanganate ions which are coloured. The conversion is achieved in two stages. Using nitric acid, the manganese is first oxidised to manganese(II) ions which are then oxidised to permanganate ions by the more powerful oxidising agent, potassium periodate.

**Health & Safety**

Wear eye protection and if any chemical splashes on your skin wash it off immediately.

The acidiﬁed 0.0010 mol l-1 potassium permanganate is a skin/eye irritant (due to the acid). Consider gloves.

Both 2 mol l-1 nitric acid and its vapour are corrosive and toxic causing severe burns to the eyes, digestive and respiratory systems and in contact with the skin. But at this concentration a well-ventilated room is sufficient to control the risk from the fumes. Wear goggles (BS EN1663) and consider gloves if splashes are likely.

85 % phosphoric acid is corrosive; it burns and irritates the eyes and skin. A systemic irritant if inhaled and if swallowed, causes serious internal injury. Wear gloves and goggles (BS EN1663).

Acidiﬁed potassium periodate solution has no hazards other than of the 2 mol l-1 nitric acid it is made up in. Corrosive to the eyes, skin and respiratory system. Wear goggles (BS EN1663) and consider gloves if splashes are likely.

Potassium persulphate irritates the eyes, skin and respiratory system (causing dermatitis and possible allergic reactions for regular users). Avoid raising dust. Wear gloves.

Propanone is volatile and highly ﬂammable and is harmful if swallowed. The vapour irritates the eyes, skin and lungs and is narcotic in high concentrations. Work in a well-ventilated lab or use a fume cupboard. Consider gloves if splashes are likely.

**Requirements**

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| 0.0010 mol l-1 acidiﬁed potassium permanganate | steel paper clips |
| 2 mol l-1 nitric acid | 85 % phosphoric acid |
| acidified potassium periodate solution | potassium persulphate |
| propanone | deionised water |
| anti-bumping granules |  |
| standard ﬂasks (50 cm3 and 100 cm3) | 50 cm3 burette  |
| colorimeter  | 540 nm filter |
| optically matched cuvettes | balance (accurate to 0.001 g) |
| glass beakers (50 cm3 and 250 cm3)  | Bunsen burner, heating mat and tripod |
| measuring cylinders (50 cm3 and 10 cm3)  | dropper |
| watch glass | filter funnel |
| tweezers | wash bottle |
| wire cutters |  |

**Procedure**

**Part A - Calibration graph**

Rinse the burette, including the tip, with 0.0010 mol l-1 acidiﬁed potassium permanganate and ﬁll it with the same solution.

Run 2 cm3 of the permanganate solution into a 50 cm3 standard ﬂask and make up to the graduation mark with deionised water. Use a dropper for the final centimetre.

Stopper the ﬂask and invert it several times to ensure the contents are completely mixed.

Rinse a cuvette with some of this solution and fill it.

Using the colorimeter (ﬁtted with a 540 nm ﬁlter) measure the absorbance of the solution in the cuvette.

Repeat steps two to five with 4, 6, 8, 10, 12 and 14 cm3 of the permanganate stock solution in the burette.

Plot a calibration graph of ‘absorbance‘ against ‘concentration of potassium permanganate’.

Your teacher/lecturer will provide you with the accurate concentration of the potassium permanganate stock solution.

**Part B - Conversion of manganese to permanganate**

Carry out the following procedure in duplicate.

Degrease a steel paper clip by swirling it with a little propanone in a small beaker. Using tweezers, remove the paper clip and leave it to dry for about a minute or so on a paper towel.

Cut up the paper clip into small pieces.

Weigh accurately about 0.2g of the paper clip pieces and transfer them to a 250 cm3 glass beaker.

Add approximately 40 cm3 of 2 mol l-1 nitric acid to the beaker and cover it with a watch glass.

Heat the mixture cautiously, in a fume cupboard, until the reaction begins. Continue heating gently to maintain the reaction, but remove the source of heat if it becomes too vigorous.

Once the steel has dissolved, allow the solution to cool a little. Add a couple of anti-bumping granules and then boil the solution until no more brown fumes are given off.

After the solution has cooled considerably - no more than ‘hand hot’ - add about 5 cm3 of 85 % phosphoric acid, approximately 0.2g of potassium persulphate and a couple of fresh anti-bumping granules. Boil the mixture for about 5 minutes.

To this solution, add approximately 15 cm3 of acidified potassium periodate solution plus a couple of fresh anti-bumping granules and then gently boil the mixture. The solution should start to tum pink. Continue gentle boiling until the intensity of the pink colour remains constant. This should take about 5 minutes.

Allow the pink solution to cool to room temperature and then transfer it to a 100 cm3 standard ﬂask leaving the anti-bumping granules in the beaker.

Rinse the beaker with a little deionised water and add the rinsings (but not the anti-bumping granules) to the ﬂask. Repeat this procedure until you are within about a centimetre of the graduation mark on the ﬂask.

Using a dropper, make up the solution to the graduation mark with deionised water.

Stopper the ﬂask and invert it several times to ensure the contents are completely mixed.

Using a colorimeter fitted with a 540 nm filter, measure the absorbance of the solution.

Use your calibration graph to convert the absorbance to a permanganate concentration and then calculate the percentage by mass of manganese in the steel paper clip.

**Notes**

The acidiﬁed potassium permanganate solution will require to be standardised. This can be done by titrating it against a standard solution of sodium oxalate. It will be necessary to heat the sodium oxalate solution to about 60°C prior to titration since potassium permanganate does not oxidise oxalates in cold solution.

Alternatively, the potassium permanganate solution could be prepared from a commercial volumetric standard.

The presence of the phosphoric acid speeds up the periodate oxidation of the manganese(ll) ions to permanganate ions. It also forms colourless complexes with iron ions and allows the solution to be boiled without hydrolysis taking place.

The potassium persulphate oxidises any carbide and ensures that all species are in their highest oxidation states before adding the periodate. In practice, the oxidation of the manganese(ll) ions to permanganate ions occurs quite rapidly with or without the additional potassium persulphate.

**Technician Guide**

Requirements per student (or group)

**Reagents**

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| steel paper clips (2) | 0.0010 mol l-1 acidiﬁed potassium permanganate (~60 cm3) (0.158 g potassium permanganate per litre of 1 mol l-1 sulphuric acid) |
| 2 mol l-1 nitric acid (~80 cm3) (128 cm3 concentrated nitric acid per litre) | 85 % phosphoric acid (~10 cm3) |
| 5 % w/v acidiﬁed potassium periodate (~30 cm3)(5 g potassium periodate per 100 cm3 of 2 mol l-1 nitric acid) | potassium persulphate (~0.4 g) |
| deionised water | propanone (~ l0 cm3) |
| Anti-bumping granules |  |

**Apparatus**

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| --- | --- |
| 50 cm3 standard ﬂask (1) | 100 cm3 standard ﬂasks (2) |
| 50 cm3 burette (1) | access to colorimeter fitted with 540 nm ﬁlter |
| optically matched cuvettes (2) | access to balance (accurate to 0.001 g) |
| 50 cm3 glass beaker (1) | 250 cm3 glass beakers (2) |
| Bunsen bumers (2) | heating mats (2) |
| tripods (2) | 10 cm3 measuring cylinder (1) |
| 50 cm3 measuring cylinder (1) | clock glasses (2) |
| ﬁlter funnel (1) | tweezers (1) |
| wash bottle (1) | dropper (1) |
| wire cutters (1) |  |

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