

Determination of manganese in steel by permanganate colorimetry

Use this method if you have access to a colorimeter

Introduction

Steel is mainly composed of iron; however, most kinds of steel contain a small amount (up to 1%) of manganese (with chemical symbol Mn). This can be detected by oxidising it to the permanganate ion (MnO_4^-), which has an intensely purple colour. The concentration of manganese can then be determined by measuring the intensity of the purple colour, and comparing it with the colour of known permanganate solutions.

Equipment and Materials Required:

- steel sample (eg: steel wool, paper clips, nails, etc.)
- potassium permanganate
- 6 mol L⁻¹ nitric acid
- concentrated phosphoric acid
- potassium periodate
- 10 mL and either 5 or 15 mL pipettes
- 200, 250, 500, 1000 mL volumetric flasks
- 250 mL conical flask
- 10 and 100 mL measuring cylinders
- bunsen burner, tripod and gauze mat
- at least 6 boiling tubes (or large test tubes)
- distilled water

Method

Preparation of MnO_4^- standard solutions

First prepare a 0.005 mol L⁻¹ standard solution of potassium permanganate by accurately weighing out about 0.79 g of solid KMnO_4 and dissolving it in 1 L of distilled water in a volumetric flask. The concentration of permanganate ions solution may be calculated as follows:

$$[\text{MnO}_4^-] \text{ (mol L}^{-1}\text{)} = \text{mass of KMnO}_4 \text{ used (g)} \div 158 \text{ (g mol}^{-1}\text{)} \div 1 \text{ (L)}$$

Pipette 10 mL of the above permanganate solution into separate 200, 250, 500 and 1000 mL volumetric flasks and make each one up to the mark with distilled water, giving you standard permanganate solutions

with concentrations of 2.5, 2.0, 1.0 and 0.5 x 10⁻⁵ mol L⁻¹ respectively. Also pipette 15 mL of your 0.005 mol L⁻¹ permanganate solution into a 1000 mL volumetric flask and make up to the mark with distilled water, giving a standard with concentration of 1.5 x 10⁻⁵ mol L⁻¹.

Clearly label each standard solution you have prepared.

Preparation of steel sample for analysis:

1. Accurately measure the mass of about 0.2 g of steel. Good steel samples to use include steel wool, paper clips, or small nails.
2. Place your sample in a 250 mL conical flask and add 20 mL of 6 mol L⁻¹ nitric acid. (Caution: Reaction of steel with nitric acid produces toxic nitrogen oxides. Do this in a fume hood. Concentrated acids are highly corrosive – wear safety glasses and rubber gloves and take care when handling.) Once the reaction has stopped, warm the flask gently over a Bunsen burner (using tripod and gauze mat) for a few minutes to drive out all remaining nitrogen oxide gases. The steel has now dissolved, mostly to form Fe^{3+} ; any manganese has been converted to Mn^{2+} ions.
3. Add 80 mL of distilled water followed by 5 mL of concentrated phosphoric acid (Take care with concentrated acid), and swirl to mix.
4. Add about 0.5 g of solid potassium periodate. This contains the IO_4^- ion which oxidises Mn^{2+} to MnO_4^- .
5. Heat the mixture over a Bunsen burner to nearly boiling for 5 minutes, and then keep heating it (without boiling) until the solution begins to develop a purple colour. Continue heating until there is no further colour change – this may take a further 15 – 30 minutes. Figures 1 – 4 illustrate the sort of colour change that you can expect to see during this time.
6. Let your solution cool for 5 minutes. Pour it carefully into a 250 mL volumetric flask, rinsing any remaining solution into the flask with distilled water. Finally, make up to the mark with distilled water and shake to mix.



Figures 1 – 4. Development of purple colour due to formation of MnO_4^- in steel sample solution. Figure 1 shows the nearly colourless solution containing dissolved steel, before any oxidation of manganese has occurred. As the solution is heated over a period of about 20 minutes the purple colour of MnO_4^- gradually becomes stronger and stronger, as shown in Figures 2, 3 and 4.

Colorimetric analysis

1. Accurately measure 10 mL of your steel sample solution into a clean, dry boiling tube. (NB: a boiling tube, or large test tube, is ideal; however any small vessel – eg: a beaker, flask or glass vial may also be used). This measurement is most accurately made using a 10 mL pipette; however, it is possible to do this accurately enough (and with less hassle) using a clean 10 mL measuring cylinder if you measure carefully.
 2. Next, measure 10 mL of each MnO_4^- standard solution into separate boiling tubes (one standard per tube) in order of increasing concentration, beginning with the $0.5 \times 10^{-4} \text{ mol L}^{-1}$ standard. It is a good idea to first rinse your pipette or measuring cylinder with a few mLs of the $0.5 \times 10^{-4} \text{ mol L}^{-1}$ standard. NB: Make sure you label each boiling tube appropriately with the name of the sample or standard it contains. A test tube rack is very useful for holding and transporting your tubes (see Figure 5). Alternatively you can use a large beaker to hold them.
 3. Measure the absorbance at wavelength of 530 nm for each coloured solution using your colorimeter. The measured absorbance of light is a direct measure of the intensity of the solution's colour. Figures 5 and 6 illustrate the range of colour intensities that you can expect from your set of MnO_4^- standards.
2. Draw a line of best fit for your data points that goes through the origin (because absorbance must be zero when MnO_4^- concentration is zero.
 3. Now identify the point on your line of best fit which corresponds to the absorbance measured for your unknown steel sample. By drawing a vertical line to the horizontal axis you will be able to determine the concentration of MnO_4^- in your unknown solution.
 4. Use this concentration to calculate the mass of manganese (in g) in your original steel sample, remembering that the number of moles of MnO_4^- produced is equal to the original number of moles of Mn in the sample. The molecular weight of Mn is 54.9 g mol^{-1} .
 5. Using the mass of your original steel sample calculate the percentage of manganese in the steel.
 6. If the absorbance value you measured for your unknown steel sample is greater than the absorbance value for your highest concentration MnO_4^- standard, you will need to modify the above procedure. Try diluting your purple steel sample solution so that its absorbance value lies within the range of your standards. (NB: Any dilution must be taken into account in your calculations.)



Figure 5. Purple coloured permanganate standard solutions, corresponding to the following MnO_4^- concentrations: $0.5, 1.0, 1.5, 2.0, 2.5 \times 10^{-4} \text{ mol L}^{-1}$.

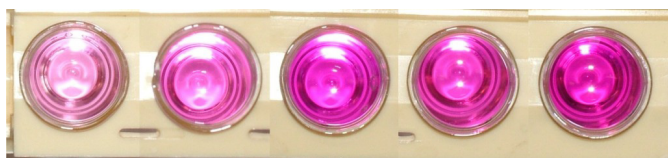


Figure 6. The same boiling tubes as in Figure 5 (MnO_4^- standard solutions with concentrations of $0.5 - 2.5 \times 10^{-4} \text{ mol L}^{-1}$) viewed from above, looking directly down the length of the tube. This view provides the most accurate "eyeball" comparison of the solutions' colour intensities.

Calculations

1. Using only the absorbance results obtained for your MnO_4^- standard solutions (not your unknown steel sample), prepare a graph with $[\text{MnO}_4^-]$ (in mol L^{-1}) as

the horizontal axis and absorbance (at 530 nm) as the vertical axis.

2. Draw a line of best fit for your data points that goes through the origin (because absorbance must be zero when MnO_4^- concentration is zero.
3. Now identify the point on your line of best fit which corresponds to the absorbance measured for your unknown steel sample. By drawing a vertical line to the horizontal axis you will be able to determine the concentration of MnO_4^- in your unknown solution.
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6. If the absorbance value you measured for your unknown steel sample is greater than the absorbance value for your highest concentration MnO_4^- standard, you will need to modify the above procedure. Try diluting your purple steel sample solution so that its absorbance value lies within the range of your standards. (NB: Any dilution must be taken into account in your calculations.)

Additional Notes

1. The phosphoric acid added to the dissolved steel solution, forms a colourless complex with Fe^{3+} . This makes the permanganate colour easier to see.

Contact Us

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