College of Science

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# Determination of Manganese Concentration in Fertiliser

*This method requires the use of a colorimeter*

## Safety

**Lab coats, safety glasses and enclosed footwear must be worn at all times in the laboratory.**
**Concentrated nitric acid is very dangerous: wear rubber gloves and take care when handling. It will burn your skin, and leave a yellow stain on your skin for some days if it makes contact. If you do splash some on your skin, wash your skin very well with cold running water IMMEDIATELY. Make sure your teacher is told about it.**

**When you first add nitric acid to your sample, it will likely give off some brown fumes. They are toxic, and you should therefore do this in a fume hood.**

**When you heat your solutions with nitric acid, there is a chance that the flask could break. Make sure you have water ready to clean any spills. Your teacher or laboratory supervisor should be with you at all times when you are doing this.**

**If you add extra periodide, make sure it is off the heat. Addition to boiling acid may cause it to boil out of the flask.**

## Introduction

This method uses a redox reaction to produce a coloured solution of permanganate from manganese ions. The manganese is oxidised to permanganate by reacting it with an excess of potassium periodate in acid.

$$8Mn^{2+} + 5IO_4^- + 12H_2O \rightarrow 8MnO_4^- + 5I^- + 24H^+$$

The amount of permanganate present is determined colorimetrically by comparison of the purple colour with known standards of permanganate. From this information, the concentration of manganese in the fertiliser can be calculated.

## Equipment Needed:

250 mL conical flasks

100 mL volumetric flasks

250 mL volumetric flasks

Bunsen burner or heating plate

Fertiliser sample – *make sure there is a stated amount of manganese in the sample. 'Trace' amounts of manganese are not detectable by this method.*

## Solutions Needed:

**Concentrated Nitric Acid:** (**see safety notes**).

**Potassium periodate solid:** $KIO_4$. You will need about 5g of this at most.

**Potassium permanganate solid:** This will be used to create standard solutions for colour comparison.

## Method:

### A. Sample Preparation

1. If you are using a solid pellet (or slow release fertilizer), then you will need to dissolve it. Accurately weigh 5 g of solid fertilizer into a 250 mL conical flask, and **in a fume hood** add 20 mL of concentrated nitric acid. There may be a toxic brown gas given off here  Heat this gently in the fume hood with supervision until the sample has liquefied (Note that it might not dissolve. All we need is for the fertilizer to be liquid).

   Transfer the liquid to a 250 mL volumetric flask and dilute the sample to 250 mL.

2. If you are using a liquid fertilizer, shake it well, then pipette 5 mL into a 250 mL volumetric flask and dilute.

**B. Oxidation to permanganate**

1. Pipette a 20 mL sample of your diluted fertilizer into a 250 mL conical flask. To this, add 5 mL of 6 mol$L^{-1}$ nitric acid (or concentrated, it doesn't matter, as long as it is strong) and about 2 g of potassium periodate. Both the nitric acid and periodate are added in excess, so the actual amounts should not matter.
2. Dilute the solution to about 50 mL with distilled water, and boil the solution on a Bunsen burner for 10 minutes. If no purple colour is observed, r**emove the solution from the heat,** and add another 1 g periodate, then reheat the solution to boiling (**See safety notes**). It may take more than 10 minutes for the colour to develop. Periodate should be added until no further colour develops. Allow the solution to cool.
3. Once the solution is cool, transfer all of the solution to a 100 mL volumetric flask, and dilute it with water.
4. Prepare standard permanganate solutions. First prepare a 0.005 mol $L^{-1}$ solution by accurately weighing about 0.79 g of solid $KMnO_4$ into a 1 L volumetric flask, and diluting it to the mark. Pipette 10 mL of the standard permanganate into a 200, 250, 500 mL and a 1 L volumetric flask, and fill them to the mark. This will give you permanganate solutions of 2.5, 2.0, 1.0 and 0.5 x $10^{-4}$ mol $L^{-1}$ respectively. Pipetting 30 mL of your standard solution into a 1 L volumetric flask will give you a 1.5 x $10^{-4}$ mol $L^{-1}$ solution.
5. Label each solution with the concentration, and the date you made it.

**C. Colorimetric Analysis**

This will depend on the colorimeter you have available to work with in your school. Your teacher should be able to guide you, but you need to make sure you are measuring at 530 nm. The instructions below are a good guide for what to do.

1. Fill a colorimetric tube with water (we call this a blank when you are working with colorimetry) and place it into the colorimeter. With the absorbance set to 530 nm (this is greenish light) take an absorbance reading. If there is a 'zero' adjust, or a 'blank' function on the colorimeter, use this water sample to zero the colorimeter.
2. Place your solution of lowest concentration (0.5 x $10^{-5}$ mol $L^{-1}$ from above) in the sample tube and take a reading. After recording the absorbance, wash the tube and repeat the measurement on the next most concentrated standard. Continue until all the standards have been measured.
2. Place your sample into the colorimeter tube. Take an absorbance reading and record.

## Result Calculations

1. Draw a standard curve, by plotting on a graph the absorbance of your standard solutions (y-axis) versus the concentration of the standards (x-axis). This should be a straight line. Using the absorbance measurement of your fertilizer sample, read along the graph until you reach your curve, then read off the concentration which corresponds to your absorbance. This is the concentration of permanganate in your diluted fertilizer sample.
2. Determine the number of moles of manganese in the 100 mL volumetric flask.
3. All of this sample came from a 20 mL aliquot of your fertilizer. What must the concentration of manganese be in that solution?
4. There was a total of 250 mL of that fertilizer sample. How many moles of manganese must have been in that flask?
5. All of this manganese came from your original 5 g (or 5 mL) sample of fertilizer from the bottle/jar. What weight does this manganese correspond to? What must the weight percentage of manganese be in your fertilizer?

## Contact Us

If you have any questions or comments relating to this experiment, please contact us. Please note that this service is for senior school chemistry students in New Zealand only. We regret we are unable to respond to queries from overseas.

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