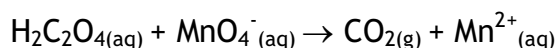


The redox titration will be done between potassium permanganate and ethanedioic acid. This reaction requires acid catalyst because ethanedioic acid is too weak an acid to make the solution acidic enough to react at a reasonable rate. Sulphuric acid is in the mixture and provides the acid catalyst. The ethanedioic acid in the mixture will reduce the manganate(VII) ions (MnO_4^-) into manganese(II) ions (Mn^{2+}). The potassium permanganate will oxidise the oxalic acid into carbon dioxide.



Reactants

$\text{H}_2\text{C}_2\text{O}_4$: Carbon +3
Manganese +7

Products

CO_2 : Carbon +4 MnO_4^- :
 Mn^{2+} : Manganese +2

To work out the ionic equation, balanced electron-half equations for both the potassium permanganate and the ethanedioic acid have to be worked out. Adding electrons (e^-), water (H_2O) and hydrogen/hydroxide ions (H^+/OH^-) depending on the conditions, they can be created.

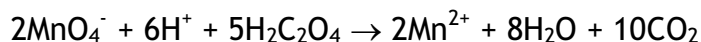
Manganate(VII) ions are reduced to form manganese(II) ions.



Ethanedioic acid is oxidised to form carbon dioxide.



The two equations combine and are balanced to create the overall ionic equation.



Equipment list:

- ☐ Potassium permanganate (0.02mol dm^{-3})
- ☐ Mixture of oxalic acid and sulphuric acid
- ☐ Conical flask
- ☐ 25ml pipette
- ☐ Pipette filler
- ☐ Funnel
- ☐ Burette
- ☐ Retort stand
- ☐ Clamp

Method:

- 1) Use the pipette and the pipette filler to collect 25ml of the mixture of oxalic acid and sulphuric acid and put it in the conical flask.
- 2) Use the clamp to connect the burette with the retort stand.
- 3) Use the funnel to fill the burette with the potassium permanganate until the meniscus reaches zero.
- 4) Place the conical flask with the mixture underneath the point of the burette.
- 5) Using the stopcock on the burette, enter the potassium permanganate into the conical flask until there is a colour change to produce a rough estimate. Record this in a table in cm^3 .
- 6) Carry out the same experiment again slowly to record a more accurate answer. Record in the table.
- 7) Repeat step 7) until two concordant results have been collected.

Diagram:

Example of table:

| | START | FINISH | TITRE |
|-------------|-------|--------|-------|
| ROUGH | | | |
| ACCURATE(1) | | | |
| ACCURATE(2) | | | |

The mean titre will be the volume of potassium permanganate used and the concentration of the oxalic acid can now be found. For example, if the volume of potassium permanganate used was 12.30cm³;

$$n = cv \quad [\text{KMnO}_4] = 0.02 \text{mol dm}^{-3}; v = 0.0123 \text{dm}^3$$

$$n = 0.000246 \text{mol}$$

Ratio of MnO₄⁻:H₂C₂O₄ is 2:5

Therefore, n(H₂C₂O₄) = 0.000615mol

$$\text{H}_2\text{C}_2\text{O}_4; c = n/v; n = 0.000615 \text{mol}; v = 0.025 \text{dm}^3$$

$$[\text{H}_2\text{C}_2\text{O}_4] = 0.0246 \text{mol dm}^{-3}$$

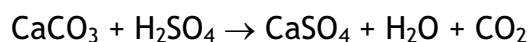
Risk assessment:

- ☐ Potassium permanganate - Harmful; gloves should be worn. Contact with combustible materials may cause fire.
- ☐ Sulphuric acid - Corrosive; gloves must be worn.
- ☐ Goggles should be worn for eye protection.

Other points to consider:

- ☐ The concentration of the potassium permanganate is 0.02mol dm⁻³. This is because it is the concentration it is stored in labs and can be purchased. Potassium permanganate at a high concentration is poisonous.
- ☐ The results recorded in the table will be to two decimal places.
- ☐ The pipette is more accurate than the burette with ±0.05cm³ compared to ±0.04cm³.

The second experiment is a gas collection. Calcium carbonate is an example of a substance which can be used in this experiment. The equation for a reaction between calcium carbonate and sulphuric acid is;



A 100cm³ measuring cylinder will collect the carbon dioxide. To ensure the amount of gas released isn't too much, the maximum amount of calcium carbonate added must be established.

One mole of gas occupies 24000cm^3
 $1/240\text{mol}$ occupies 100cm^3
 Ratio of $\text{CO}_2:\text{CaCO}_3$ is 1:1
 Therefore, maximum no. of moles of CaCO_3 is $1/240$
 $n \times \text{Mr} = \text{mass}$; $n = 1/240$; $\text{Mr}(\text{CaCO}_3) = 100$
 Maximum mass of $\text{CaCO}_3 = 0.417\text{g}$ to 3sf.

Equipment list:

- ☐ Calcium carbonate (mass<0.417g)
- ☐ Mixture of oxalic acid and sulphuric acid
- ☐ Tub
- ☐ Water
- ☐ 100cm^3 measuring cylinder x2
- ☐ Conical flask (w/rubber tubing)
- ☐ Block of wood
- ☐ Cork
- ☐ Weighing scales
- ☐ Weighing boat

Method:

- 1) Fill the tub with water.
- 2) Measure 25cm^3 of the mixture with a measuring cylinder and put it in the conical flask with the rubber tubing.
- 3) Fill the other measuring cylinder with water from the tub and invert it in the tub, with aim to avoid any air entering it.
- 4) Place the conical flask on a block of wood.
- 5) The other end of the rubber tubing should be placed underneath the inverted measuring cylinder.
- 6) Using the weighing scales, measure any mass of calcium carbonate below 0.416g via a weighing boat.
- 7) Pour the calcium carbonate from the weighing boat into the conical flask and immediately put the cork on the conical flask.
- 8) Record the amount of gas released in the measuring cylinder.

Diagram:

The concentration of the acid can now be worked out. If, for example, 88cm^3 of carbon dioxide was released;

One mole of gas occupies 24000cm^3
 $(1/24000) \times 88 = n(\text{CO}_2) = 0.00367\text{mol}$
 Ratio of $\text{CO}_2:\text{H}_2\text{SO}_4$ is 1:1
 Therefore, $n(\text{H}_2\text{SO}_4) = 0.00367\text{mol}$

$$c=n/v; n = 0.00367; v = 0.025\text{dm}^3$$
$$[\text{H}_2\text{SO}_4] = 0.147\text{mol dm}^{-3}$$

Risk assessment:

- Sulphuric acid - Corrosive; gloves must be worn.
- Goggles should be worn for eye protection.

Other points to consider:

- We can see in the example that if 88cm^3 is released, the concentration is 0.147mol dm^{-3} . This is not approximately 0.2mol dm^{-3} . If 100cm^3 of CO_2 gas was released, $[\text{H}_2\text{SO}_4]$ would be 0.167mol dm^{-3} . But if $[\text{H}_2\text{SO}_4]$ was nearer the approximated value, not all of the acidic molecules would have reacted. To solve this problem, a 250cm^3 measuring cylinder could be used. This can collect more gas and also leads to a larger maximum amount of calcium carbonate which can be used, which increases the chance of all the acidic molecules reacting. However, with this change, the accuracy decreases. The 250cm^3 measuring cylinder has an accuracy of $\pm 0.5\text{cm}^3$ compared to the 100cm^3 measuring cylinder with $\pm 0.25\text{cm}^3$.
- It is difficult to put the cork on the conical flask directly after entering the calcium carbonate into it. Also, carbon dioxide is soluble in water. Both decrease the chance of 100% yield.

Bibliography;

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