Microscale titration

These technician notes are part of a collection of microscale chemistry resources at: rsc.li/4iiljbl. Integrated instructions for both gravimetric and volumetric titration are available from rsc.li/4iclogx.

Introduction

Introduce learners aged 11–16 to the principle of titration without the cognitive load of a full-scale set up.

Microscale titrations can be gravimetric (measuring the mass of the reaction vessel before and after) or volumetric (measuring the volume by counting the drops of solution added).

Equipment (per group)

- 1 x beaker, 10 cm^3 or small vial
- 1 x white tile
- 1 x clamp stand
- 1 x plastic pipette (preferably a fine-tipped pipette)
- 1 x measuring cylinder (10 cm^3)
- 1 x mass balance (to 0.01 g)
- Vinegar solution (shop-bought, diluted by a factor of 4), 1 cm^3
- Phenolphthalein solution (0.05%), 1 drop
- Sodium hydroxide solution (0.2 mol dm^{-3}), 10 cm^3

Safety equipment

Eye protection: safety glasses to EN166F

Equipment set-up

Dilute shop-bought vinegar by a factor of four (by taking 25 cm^3 of vinegar and making up to 100 cm^3 with water).

Method

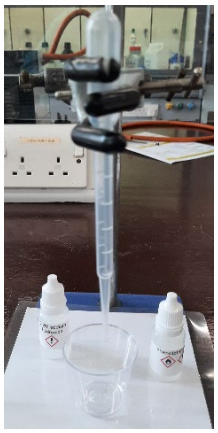
Gravimetric titration

1. Add 1 drop of phenolphthalein to the small beaker and record its mass.

2. Using a measuring cylinder, measure out 1 cm^3 of diluted vinegar and add it to the beaker. Record its mass.
3. Fill the plastic pipette with $NaOH$ solution and clamp it gently.
4. Place the beaker below the pipette.
5. Carefully tighten the clamp to add one drop of $NaOH$ solution to the beaker at a time.
6. Swirl the beaker.
7. Add $NaOH$ solution dropwise until a permanent pink colour is formed.
8. Record the mass of the beaker.

Volumetric titration

1. Add 1 drop of phenolphthalein to a small beaker or vial.
2. Add ~1 cm^3 of vinegar to the same beaker.
3. Fill a dropping pipette with sodium hydroxide solution (around 0.2 M).
4. Clamp the pipette gently.
5. Tighten the clamp carefully until 1 drop of $NaOH$ is added to the beaker/vial.
6. Swirl the beaker/vial.
7. Repeat steps 5 and 6 (counting the number of drops added) until a pale permanent pink colour is seen.

Safety

- Read our standard health and safety guidance and carry out a risk assessment before running any live practical.
- Refer to SSERC/CLEAPSS Hazcards and recipe sheets.
- Hazard classification may vary depending on supplier.

Chemical supplied for the practical	Preparation
Vinegar Not classified as hazardous.	Vinegar Dilute shop-bought vinegar by a factor of four (by taking 25 cm^3 of vinegar and making up to 100 cm^3 with water).

Chemical supplied for the practical	Preparation
Sodium hydroxide, 0.2 mol dm^{-3} $NaOH(aq)$  WARNING Irritant (skin, eyes). CLEAPSS Hazcard 091A.	Sodium hydroxide $NaOH(s)$ (40.0 g mol^{-1}) This substance is dangerous in contact with WATER (a vigorous exothermic reaction occurs), ZINC, ALUMINIUM (hydrogen evolved).  Five-fold dilution of the 1 M solution. CLEAPSS recipe sheet RB085.
Phenolphthalein 0.05% solution  WARNING Flammable CLEAPSS Hazcard 032.	Phenolphthalein, solid DANGER  May include a risk of one or more hazard statements including, but not limited to: May be fatal/toxic if inhaled or in contact with skin or eyes. May cause breathing difficulties if inhaled. May damage fertility or the unborn child. Check CLEAPSS Hazcard 032 for most up to date guidance. Ethanol (and IDA) WARNING Flammable  CLEAPSS Hazcard 040A. Prepare solution following CLEAPSS recipe sheet RB046.

Disposal

Rinse the aqueous solutions down a foul-water drain with plenty of water.

Acknowledgements

This resource is based on a method developed by CLEAPSS, PP019 Analysis of vinegar (small scale), available at science.cleapss.org.uk.