Oxalic acid as a primary standard

Education in Chemistry
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This experiment provides experience in preparing a primary standard, using a balance and standardising solutions using titrations.

Standard solutions are solutions of accurately known concentration. Many compounds are unsuitable for making up standard solutions directly, as they will not produce accurate concentrations. This can be for several reasons, for example sodium hydroxide is unsuitable as it absorbs water and carbon dioxide from the atmosphere. However, there are some compounds from which standard solutions can be directly prepared – known as primary standards.

These compounds must have a high state of purity, be stable in air and in solution, be soluble and have a reasonably high formula mass.

Oxalic acid is suitable for use as a primary standard and can then be used to standardise other solutions.

Preparing a standard solution of 0.1 mol l^{-1} oxalic acid

Chemicals and apparatus

Oxalic acid dehydrate
Deionised water
Balance (accurate to 0.01 g)
Wash bottle
250 cm^3 volumetric flask
250 cm^3 beaker

Hazards

Oxalic acid is harmful and severely irritating if it is swallowed or comes into contact with the skin or eyes. Oxalic acid dust irritates the respiratory system. Oxalic acid solutions can have serious systemic effects if ingested. Wear eye protection. SSERC recommends that splashes should be washed off the skin immediately and eye protection should be worn.

Method

1. Calculate the mass of one mole of oxalic acid, then work out the mass required to make up 250 cm^3 of 0.1 mol l^{-1} solution.
2. Put the 250 cm^3 beaker on the balance. Measure out approximately the mass of oxalic acid calculated in step 1 into the cm^3 beaker. Record the accurate mass in your book.
3. Add about 50 cm^3 deionised water and stir to dissolve.
4. Pour the solution carefully into the 250 cm^3 volumetric flask.
5. Rinse out the beaker with deionised water at least twice, adding the rinsings to the flask.
6. Add deionised water to the volumetric flask until the bottom of the meniscus touches the line.
7. Stopper the flask and invert to mix.
8. Work out the accurate concentration of the oxalic acid solution.

Standardising 0.1 mol l^{-1} sodium hydroxide solution

Chemicals and apparatus
Approximately 0.1 mol l^{-1} standard oxalic acid solution
Approximately 0.1 mol l^{-1} sodium hydroxide solution
Phenolphthalein indicator
20 cm^3 pipette and filler
50 cm^3 burette and stand
Conical flasks and beakers
White tile

Hazards
Solutions of oxalic acid can have serious systemic effects if ingested and irritate the eyes and skin. Sodium hydroxide solution is irritating to the eyes at this concentration. Wear eye protection and wash any splashes off skin immediately. Phenolphthalein solution is flammable due to being dissolved in alcohol.

Method
1. Rinse the pipette with the oxalic acid solution, and pipette 20 cm^3 oxalic acid into a conical flask.
2. Add a few drops of phenolphthalein solution.
3. Rinse and fill the burette with the sodium hydroxide solution. Note the initial reading on the burette in the table below:

Titration number	Initial reading on burette / cm^3	Final reading on burette / cm^3	Volume of NaOH added / cm^3
1			
2			
3			

4. Titrate the oxalic acid until a permanent colour change is just observed.
5. Repeat until two concordant results (within 0.2 cm^3) are obtained (note: more than three attempts may be needed).
6. Calculate the average titre, and so calculate the accurate concentration of the sodium hydroxide.

Ways to extend the practical
1. Use the standardised sodium hydroxide to calculate the concentration of ethanoic acid in commercial vinegar solution by titrating using phenolphthalein as an indicator.
2. Use the standardised sodium hydroxide to calculate the percentage of acid in marble via a back titration by reacting a known mass of marble with excess standard hydrochloric acid, then titrating the excess acid with the standard sodium hydroxide.