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Nickel Titration

*UNIT 2 PPA 1*

**Introduction**

Since ethylenediaminetetraacetic acid - commonly abbreviated to EDTA - forms stable complexes with most metal ions, it is widely used to determine metals in what are known as complexometric titrations. EDTA is a tetracarboxylic acid and can be represented as H4Y. In alkaline conditions,

EDTA exists as Y4+ ions:



The Y4+ ions form l:l complexes with metal ions. For example, Ni2+ ions bind with them to form the complex, NiY2- , which has the octahedral structure shown opposite.

The end-point of an EDTA complexometric titration can be detected by means of a metal ion indicator - an organic dye which changes colour when it binds with metal ions. For it to be suitable in an EDTA titration, the indicator must bind less strongly with metal ions than does EDTA.

**Health & Safety**

Wear eye protection and if any chemical splashes on your skin wash it off immediately.

Hydrated nickel(II) sulphate is carcinogenic if inhaled and harmful by ingestion and inhalation. It irritates the eyes and skin. Continued skin contact can cause dermatitis. Avoid raising a dust or aerosol. Wear gloves.

EDTA is a skin/eye irritant but the solution is of no significant hazard..

0.88 aqueous ammonia is toxic if inhaled in high concentrations or if swallowed. The solution and vapour irritate the eyes. The solution burns the skin and swallowing causes internal damage. Wear goggles and gloves and handle it in a fume cupboard.

1 mol l-1 ammonium chloride is irritating to skin and eyes.

Murexide is a skin/eye/respiratory irritant and may be a mutagen. The quantities are very small but avoid raising dust.

Nickel(EDTA) complex - limited information is available on its toxicity. its high stability and water solubility will almost certainly give rise to low toxicity.

**Requirements**

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| 100 cm3 standard flask | 250 cm3 conical ﬂasks |
| 20 cm3 pipette | 50 cm3 burette |
| weighing bottle | balance (accurate to 0.01 g) |
| 25 cm3 measuring cylinder | pipette ﬁller |
| ﬁlter funnel | white tile |
| wash bottle | glass stirring rod |
| dropper |  |
| hydrated nickel(II) sulphate (NiSO4.6H2O) | 0.10 mol l-1 EDTA solution |
| 1 mol l-1 ammonium chloride | 0.88 aqueous ammonia |
| murexide indicator | deionised water |

**Procedure**

Part

1. Calculate the theoretical percentage by mass of nickel in NiSO4.6H2O.

Part B

1. Carry out the following procedure in duplicate.
2. Transfer approximately 2.6 g of hydrated nickel(II) sulphate to a weighing bottle and weigh the bottle and contents.
3. Add about 25 cm3 of deionised water to a 100 cm’ beaker and transfer the bulk of the nickel salt to the water.
4. Reweigh the bottle with any remaining salt.
5. Stir the solution in the beaker until the solid has dissolved.
6. Transfer the solution to a 100 cm3 standard ﬂask.
7. Rinse the beaker with a little deionised water and add the rinsings to the ﬂask. Repeat this procedure until you are within about a centimetre of the graduation mark on the ﬂask.
8. Using a dropper, make up the solution to the graduation mark with deionised water.
9. Stopper the ﬂask and invert it several times to ensure the contents are thoroughly mixed.
10. Rinse the burette, including the tip, with 0.10 mol l-1 EDTA and fill it with the same solution.
11. Rinse the 20 cm3 pipette with a little of the nickel salt solution and pipette 20 cm3 of it into a conical ﬂask. Dilute the solution to about 100cm3 with deionised water.
12. Add murexide indicator (approximately 0.05 g) to the diluted nickel salt solution together with approximately 10 cm3 of ammonium chloride solution.
13. Titrate the mixture with the EDTA solution and after the addition of about 15 cm3 of EDTA make the solution alkaline by adding approximately 10 cm] of 0.88 aqueous ammonia.
14. Continue the titration to the end-point which is shown by the first appearance of a blue-violet colour. Detection of the end-point can be very difficult, so use this as a trial run. Keep the titrated solution to help you detect the end-points in subsequent titrations.
15. Repeat the titrations until two concordant results are obtained.
16. Calculate the percentage by mass of nickel in your sample of hydrated nickel(II) sulphate using the accurate concentration of the EDTA solution provided by your teacher/lecturer.
17. For one of your determinations, calculate the percentage error and hence the absolute error in the percentage of nickel in NiSO4.6H2O. Your teacher/lecturer will provide you with the error in the concentration of the EDTA solution.

**Note**

It is worthwhile reminding the students to keep the solution from their trial titration to help them detect the end-points in subsequent titrations.

**Technician Guide**

Requirements per student (or group)

**Reagents**

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| AnalaR nickel(II) sulphate 6-hydrate (~5.2 g)  | 0.10 mol l-1 (-120 cm3) (37.22 g AnalaR ethylenediaminetetraacetic acid disodium salt 2-hydrate per litre) |
| 1 mol l-1 ammonium chloride (~60 cm3) (53.5 g ammonium chloride per litre) | murexide indicator (~0.3 g) (grind murexide with AnalaR sodium chloride in the mass ratio 1:100) |
| 0.88 aqueous ammonia (~60 cm3) | deionised water |

**Apparatus**

|  |  |
| --- | --- |
| 100 cm3 standard ﬂasks (2)  | 25 cm3 measuring cylinder (1) |
| 250 cm3 conical ﬂasks (2)  | pipette filler (1) |
| 20 cm3 pipette (1)  | ﬁlter funnel (1) |
| 50 cm3 burette (1)  | white tile (1) |
| weighing bottle (1)  | wash bottle (1) |
| access to balance (accurate to 0.01 g)  | glass stirring rod (1) |
| dropper (1) |  |

**Notes**

The AnalaR disodium salt of EDTA can be regarded as a primary standard and so can be used directly to prepare the standard solution.

Alternatively, the EDTA solution could be prepared from a commercial volumetric standard.