

Complexometric determination of nickel using edta essay sample



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Introduction:

The Aim of the experiment is to determine the percentage of nickel salt using a complexometric technique. Ethylenediaminetetraacetic acid (EDTA) is a hexadentate ligand which forms stable complexes with most metal ions, thus is widely used to determine metals in complexometric titrations. EDTA can be represented as H_4Y and in alkaline conditions, it exists as Y^{4-} ions:

The Y^{4-} ions form 1: 1 complexes with metal ions. For example, Ni^{2+} ions bind with them to form a stable octahedral complex NiY^{2-} . An ordinary indicator cannot be used since the reaction does not involve a simple acid - alkali neutralisation so 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 the metal ions than does EDTA. Procedure:

Firstly, the theoretical percentage by mass of nickel in $NiSO_4 \cdot 6H_2O$ was calculated. Approximately 2.6g of hydrated nickel(II) sulphate was transferred to a weighing bottle and both the contents and bottle were weighed. About 25cm³ of deionised water was added to a 100cm³ beaker and the bulk of the nickel salt was transferred to the water. The weighing bottle was then reweighed with the remaining salt. The solution in the beaker was stirred until the solid dissolved and the solution was then transferred to a 100cm³ standard flask. The beaker was then rinsed with deionised water and the rinsings were added to the standard flask. This procedure was repeated until the solution reached within a centimetre of the graduation mark on the standard flask. Using a dropper, the solution was made up to

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the graduation mark with deionised water. The flask was stoppered and inverted several times to ensure the contents were thoroughly mixed.

The burette was then rinsed with 0.10 mol l⁻¹ EDTA and filled with the same solution. The pipette was rinsed with a little of the nickel salt solution and 20 cm³ of this was pipetted into a conical flask. The solution was then diluted to about 100 cm³ with deionised water. Approximately 0.05 g of murexide indicator was added to the diluted nickel salt solution together with approximately 10 cm³ of ammonium chloride solution. The mixture was then titrated with the EDTA solution and after the addition of about 15 cm³ EDTA solution, the solution was made alkaline by the addition of approximately 10 cm³ of 0.88 aqueous ammonia.

The titration was then continued to the end-point which was shown by the first appearance of a blue-violet colour. This titration was used as a trial run, as detection of the end point is very difficult. The titrated solution was kept to help detect the end-points in the subsequent titrations. The titration was repeated until two concordant results were obtained. The percentage by mass of nickel in the sample of hydrated nickel(II) sulphate was calculated using the accurate concentration of the EDTA solution. For one determination, the percentage error was calculated and then the absolute error in the percentage of nickel in NiSO₄ · 6H₂O.

Results:

Theoretical Calculation:

GFM of hydrated nickel(II) sulphate, NiSO₄ · 6H₂O = 262.8

X 100 = 22.3%

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$$\%Ni = 58.7$$

$$262.8$$

Experimental Results:

Mass of hydrated nickel(II) sulphate used = 2.6g

| Rough titre| First titre| Second titre|

Initial burette reading (cm³)| 0| 19.5| 0|

Final burette reading (cm³)| 19.5| 40.8| 21.5|

Volume of EDTA added (cm³)| 19.5| 21.3| 21.5|

Average of concordant results:

$$= 21.4 \text{ cm}^3$$

$$21.3 + 21.5$$

$$2$$

Concentration of EDTA solution = 0.10 mol l⁻¹

The number of moles of EDTA used to react with 20cm³ of nickel(II) solution

$$= V \times C$$

$$= 0.0214 \times 0.10 = 0.00214 \text{ moles}$$

Therefore, number of moles of Ni²⁺ ions in 20cm³

$$= 0.00214 \text{ moles}$$

Number of Ni²⁺ ions in the total volume of 100cm³

$$= 0.00214 \times 5 = 0.0107 \text{ moles}$$

Therefore, mass of nickel in salt

$$= N \times F_m$$

$$= 0.0107 \times 58.7$$

$$= 0.62809\text{g}$$

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X 100

From these results the % mass of nickel in the salt

= 0. 62809

2. 6

= 24. 2%

Conclusion:

The percentage by mass of nickel in hydrated nickel(II) sulphate was shown by experiment to be 24. 2%. This compares very well with the theoretical value of 22. 3%.

Evaluation:

The experimental result fits in very well with the theoretical result. This suggests that the hydrated nickel(II) sulphate used was very pure. Impurities such as other metal ions would have reacted with EDTA to give higher titre values and, therefore, a higher experimental value for the percentage of nickel present. During the first titration, it was quite difficult to determine when the end-point had been reached, but the appearance of the blue-violet colour was sharp. The ammonia-ammonium chloride solutions are used as a buffer to keep the pH constant. Possible uncertainty values in the measurements include:

* Burette readings $\pm 0. 05 \text{ cm}^3$

* Pipette volumes $\pm 0. 06 \text{ cm}^3$

* Balance readings $\pm 0. 01\text{g}$ ($\pm 0. 02\text{g}$ for tared mass)

* Volumetric flasks $\pm 0. 2 \text{ cm}^3$

* The concentration of the EDTA solution.

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Uncertainty Calculations:

Uncertainty in mass of NiSO₄ · 6H₂O

$$= 0.02\text{g}$$

$$\times 100 = 0.77\%$$

% uncertainty in mass of NiSO₄ · 6H₂O

$$= 0.02$$

$$2.6$$

Uncertainty in volume of NiSO₄ (aq)

$$= 0.2\text{cm}^3$$

$$\times 100 = 0.20\%$$

% uncertainty in volume of NiSO₄ (aq)

$$= 0.2$$

$$100$$

Uncertainty in pipetted volume of NiSO₄ (aq)

$$= 0.06\text{ cm}^3$$

$$\times 100 = 0.3\%$$

% uncertainty in pipetted volume of NiSO₄ (aq)

$$= 0.06$$

$$20$$

Uncertainty in concentration of EDTA = 0.0002 mol l⁻¹

$$\times 100 = 0.2\%$$

% uncertainty in concentration of EDTA

$$= 0.0002$$

$$0.1$$

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Uncertainty in titre volume of EDTA = 0.1 cm³

$$\times 100 = 0.5\%$$

% uncertainty in titre volume of EDTA

$$= 0.1$$

$$21.4$$

% uncertainty in percentage of Ni

$$= 0.77 + 0.2 + 0.3 + 0.2 + 0.5$$

$$= 1.97\%$$

$$\times 24.2 = 0.48\%$$

Absolute uncertainty in percentage of Ni

$$= 1.97$$

$$100$$

Hence, percentage of Ni in NiSO₄ · 6H₂O

$$= 24.2 \pm 0.5\%$$