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ESTIMATION OF NICKEL (II), USING DIMETHYLGLYOXIME (GRAVIMETRIC ANALYSIS)

AIM

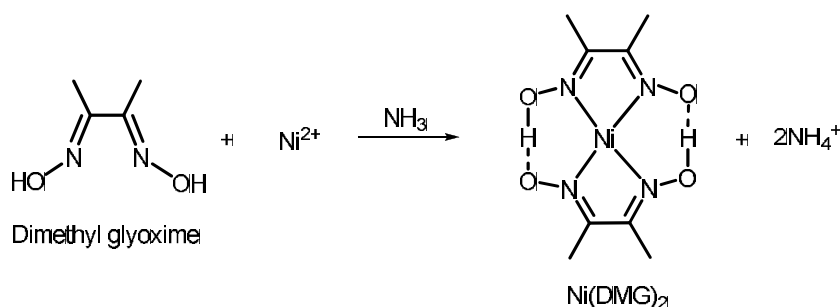
To estimate the nickel (II) using dimethylglyoxime, a gravimetric analysis.

THEORY

Nickel dimethyl glyoximate is soluble in hot water in mineral acids and in solution containing more than 50 % ethanol by volume, but it is insoluble in dilute ammonia. So, faintly acidic or hot nickel salt solution on treatment with slight excess of ethanolic solution of dimethyl glyoxime and then rendering the mixture faintly alkaline with NH_4OH , precipitates rose-red colored Nickel dimethyl glyoximate quantitatively.

Precipitation from hot and weakly acidic solutions gives more easily filterable precipitate. When a large excess of precipitant is used, some of the precipitate may dissolve in the alcohol present in the and so only a slight excess of the reagent should be added. Fe(III), Cr(III), Al(III) and large amounts of Co(II), Zn(II) and Cu(II) may interfere, by adding a soluble tartarate the interference due to Fe(III), Cr(III) and Al(III) can be prevented.

CHEMICAL EQUATION



MATERIALS REQUIRED

Ammonium nickel (II) sulfate, dimethyl glyoxime, HCl, ethanol, ammonia, distilled water, beaker, Gooch crucible, glass rod, dessicator, measuring cylinder, vacuum filtration setup, hot plate, steam bath, hot air oven etc.

PROCEDURE

Step-1: Determination of Constant weight of Gooch Crucible

1. Take a Gooch crucible or sintered glass and heat at $110\text{-}120^\circ\text{C}$ in a hot air oven for 20-30 min.
2. Remove and cool it in a dessicator for 15 min.
3. Then, weigh it on an electronic balance and note the reading.
4. Repeat this process for 2-3 times to obtain constant weight of crucible.

Table 1:

S.No.	Weight of Gooch Crucible (W_1) gm	Weight of Gooch Crucible + Ni ($C_4H_7O_2N_2$) ₂ (W_2) gm
1 st Weight		
2 nd Weight		
3 rd Weight		

Step-II: Precipitation of Nickel dimethyl glyoximate

1. Weigh accurately 0.3-0.4 gm of $(NH_4)_2SO_4 \cdot NiSO_4 \cdot 6H_2O$, transfer into a 500 mL beaker and dissolve it into 50 mL of distilled water.
2. Add 5 mL of HCl- H_2O (1:1) by stirring with a glass rod.
3. Then the solution is diluted to 150 mL with distilled water.
4. Heat it at 70-80°C on a hot plate and add 20 mL of 1% ethanolic solution of dimethyl glyoxime followed by adding dilute ammonia with constant stirring until the smell of ammonia persists.
5. Then the solution is allowed to stand on a steam bath for about 20 min. When the precipitate has settled down, a drop of dimethyl glyoxime is added to test the complete precipitation.
6. The beaker is allowed to stand for cooling and then filter through a weighed Gooch crucible.
7. The precipitate is washed with cold water until it free from chloride.
8. Then dry it in an oven at 110-120°C for 30 min.
9. Cool it in a dessicator for 15 min and take the weight.
10. Repeat this process 2-3 times to obtain a constant weight.

OBSERVATIONS AND CALCULATIONS

1. Weight of Gooch Crucible, $W_1 =$ _____ gm
2. Weight of Gooch Crucible + Ni ($C_4H_7O_2N_2$)₂, $W_2 =$ _____ gm
3. Weight of Ni ($C_4H_7O_2N_2$)₂, $(W_2 - W_1) =$ _____ gm
4. Calculation of amount of Nickel.

RESULTS

Weight of Ni (II) = _____

Old Handout:

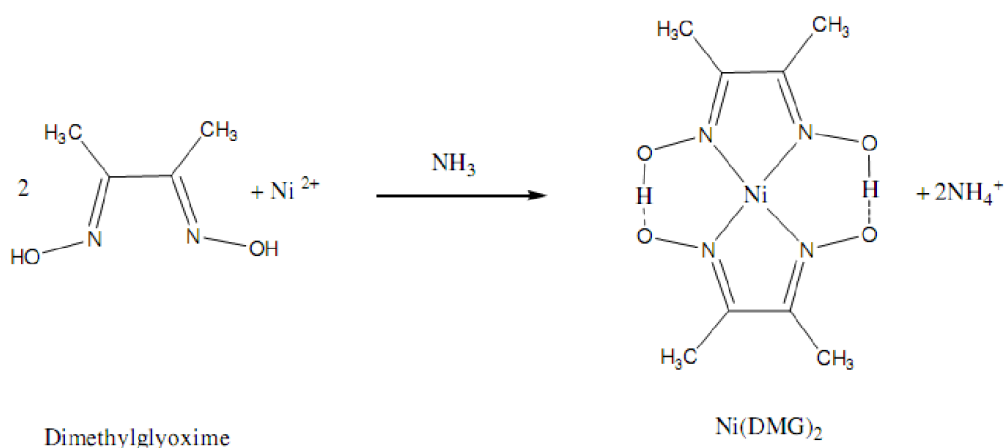
Experiment No.3

Gravimetric estimation of Nickel (II), using dimethylglyoxime

Materials Required	Equipments and glasswares
$(\text{NH}_4)_2\text{SO}_4$, Ni $\text{SO}_4 \cdot 6\text{H}_2\text{O}$	Gooch crucible (G4)
HCl	air oven
Ethanol	desiccator
dimethyl glyoxime	500 mL beaker
NH_4OH	glass rod
Time required: 4 hours	

Purpose: Gravimetric analysis of nickel.

Theory: Nickel dimethyl glyoximate is soluble in hot water (0.6 mg in 100 ml), in mineral acids and in solution containing more than 50% ethanol by volume, but it is insoluble in dilute ammonia. So, faintly acidic or hot nickel salt solution on treatment with slight excess of ethanolic solution of dimethyl glyoxime and then rendering the mixture faintly alkaline with NH_4OH , precipitates rose-red colored Nickel dimethyl glyoximate quantitatively.



Precipitation from hot and weakly acidic solutions gives more easily filterable precipitate. When a large excess of precipitant is used, some of the precipitate may dissolve in the

alcohol present in the precipitant and so only a slight excess of the reagent should be added. Fe(III), Cr(III), Al(III) and large amounts of Co(II), Zn(II) and Cu(II) may interfere; by adding a soluble tartarate the interference due to Fe(III), Cr(III) and Al(III) can be prevented.

Procedure:

- (a) **Determination of crucible constant weight** of a gooch or sintered glass after heating at 110°-120°C in an air oven for 45 minutes, cooling in a desiccator for 20 minutes and weighing and repeating the process.
- (b) **Precipitation and weighing of Nickel dimethyl glyoximate:** 0.3-0.4 g of $(\text{NH}_4)_2\text{SO}_4 \cdot \text{Ni SO}_4 \cdot 6\text{H}_2\text{O}$ is accurately weighed out into a 500 mL beaker, dissolved in 50 mL of water and 5 mL of 1:1 HCl by stirring with a glass rod. The solution is diluted to 150 ml with distilled water, and heated to 70°-80°C. 20 ml of 1% ethanolic solution of dimethyl glyoxime is added, followed by dilute ammonia with constant stirring until the smell of ammonia persists. The solution is allowed to stand on a steam bath for about 30 minutes. When the precipitate has settled down, a drop of dimethyl glyoxime is added to test complete precipitation. The beaker is allowed to stand for cooling and then filtered through a weighed Gooch crucible. The precipitate is washed with cold water until free from the chloride, dried in an oven at 110°-120°C for 45 minutes, cooled in a desiccator for 20 minutes and then weighed. This process of heating, cooling and weighing is repeated until constant weight is obtained.

Results:

Table 1

Weight of the Gooch Crucible (W_1)g		Weight of the Gooch Crucible + Ni ($\text{C}_4\text{H}_7\text{O}_2\text{N}_2$) ₂ (W_2)g	
1 st weight		1 st weight	
2 nd weight		2 nd weight	
3 rd weight		3 rd weight	

Table 2

Weight of the Gooch Crucible (W_1)g	Weight of the Gooch Crucible + Ni ($C_4H_7O_2N_2$) ₂ (W_2)g	Weight of Ni ($C_4H_7O_2N_2$) ₂ ($W_2 - W_1$)g

Calculation: Ni ($C_4H_7O_2N_2$)₂ \equiv Ni

288.69 58.69

Weight of Nickel = [$\{(W_2 - W_1) \times 58.69\} / 288.69$] g