

2-Hydroxy-1-acetonaphthoneoxime as analytical reagent: Gravimetric determination of nickel and palladium*

V V BALAKRISHNA, R C HUSSAIN and N APPALA RAJU

Department of Chemistry, Sri Venkateswara University, Tirupati 517 502

MS received 10 December 1976; revised 13 March 1978

Abstract. 2-Hydroxy-1-acetonaphthoneoxime is proposed as a reagent for the gravimetric determination of nickel and palladium. Nickel (10.0–40.0 mg) is quantitatively precipitated in the pH range 8.0–10.0 using a 1% (w/v) reagent solution in presence of ammonium chloride–ammonium hydroxide buffer. The greenish yellow precipitate is digested for 30 min and dried at 110–120°C to constant weight, and weighed as $\text{Ni}(\text{C}_{12}\text{H}_{10}\text{O}_2\text{N})_2$, the gravimetric factor being 0.1278. Separation and determination of copper and nickel from the same portion of the solution is described. This procedure is applied in the analysis of constantan. Palladium (5.0–35.0 mg) is quantitatively precipitated using a 0.5% (w/v) reagent solution in presence of 0.05–0.1 N HCl. The yellow precipitate is weighed as $\text{Pd}(\text{C}_{12}\text{H}_{10}\text{O}_2\text{N})\cdot\text{H}_2\text{O}$, the gravimetric factor being 0.2028.

Keywords. 2-hydroxy-1-acetonaphthoneoxime; gravimetry; nickel; palladium.

1. Introduction

2-Hydroxy-1-acetonaphthoneoxime was used in the gravimetric determination of copper by Hussain and Raju (1976). We now describe the studies made on its application to the gravimetric determination of nickel and palladium. The reagent gave a greenish yellow precipitate with nickel in the pH range 8.0–10.0 and a yellow precipitate with palladium in presence of 0.05–0.1 N HCl (pH \approx 1).

2. Experimental

Stock solutions of copper and nickel were prepared using AnalaR samples of their sulphates and were standardised (Vogel 1975). A solution of palladium chloride was prepared by dissolving 1 g of palladium chloride (Reachim, USSR) in 10 ml of concentrated hydrochloric acid and making up in a 1-l standard flask and the palladium content determined using dimethylglyoxime (Vogel 1975).

Solutions for the study of interference were prepared such that each ml contained about 10 mg of the metal. In case of gold (III) and platinum (IV), the solutions were prepared such that each ml contained about 1 mg of the metal.

A 1% reagent solution was prepared by dissolving 1 g of the oxime in 50 ml of

*Presented at the Annual Convention of Chemists, Jaipur, December 1977.

rectified spirit and then making up to 100 ml with distilled water. A 0.5% solution was used in the case of palladium.

2.1. Determination of nickel

An aliquot of standard nickel sulphate solution containing 10.0-40.0 mg of the metal was taken in a 250 ml beaker. 20 ml of ammonium chloride (1 M) were then added and the solution diluted to 10 ml. A 1% reagent solution was added slowly (1 ml per mg of nickel), followed by the dropwise addition of ammonium hydroxide (1 M) with constant stirring ($\text{pH} \approx 9.5$). The greenish yellow precipitate was digested for 30 min, cooled, filtered through a sintered glass crucible (IG-4), washed with a 1% (v/v) alcoholic solution, dried at 110–120° C and weighed as Ni ($\text{C}_{12}\text{H}_{10}\text{O}_2\text{N}_2$). The gravimetric for nickel is 0.1278.

2.2. Determination of nickel in presence of copper

Aliquots of standard solutions of copper and nickel were pipetted into a 250 ml beaker. Copper was determined at pH 4–4.5. The filtrate was concentrated to half the original volume (≈ 80 ml) and nickel was determined ($\text{pH} \approx 9.5$) by the present procedure. Copper and nickel could be accurately determined in the range 15.0–40.0 mg by this method.

2.3. Determination of copper and nickel in constantan

1.5 g of the alloy were taken in a 250 ml beaker, 40 ml of concentrated nitric acid was added and the solution evaporated. The residue was treated with 15 ml of concentrated sulphuric acid and evaporated. The cold residue was treated with 150 ml of water and heated until a clear solution was obtained; this solution was transferred to a 250 ml standard flask and made up to the mark with distilled water. The amounts of copper and nickel were determined by standard methods (Vogel 1975). Aliquots of the alloy solutions (5–15 ml) were pipetted and diluted to 150 ml. The amounts of copper and nickel were determined using 2-hydroxy-1-acetonaphthoneoxime. The results are given in table 1.

Table 1. Analysis of constantan

Copper (iodometric)	Nickel (DMG)	Copper	Found Nickel	Difference Copper	Nickel
mg	mg	mg	mg	mg	mg
14.58	12.49	14.73	12.43	+0.15	-0.06
		14.76	12.45	+0.18	-0.04
29.15	24.98	29.29	24.85	+0.14	-0.13
		29.26	24.80	+0.11	-0.18
43.73	37.47	43.82	37.65	+0.09	+0.18
		43.76	37.59	+0.03	+0.12

Table 2. Determination of palladium in presence of other metal ions (amount of palladium taken = 11.09 mg.)

Foreign metal ion added	Palladium found	Difference
mg	mg	mg
Cu (II) 60	11.19	+0.10
Ni (II) 60	11.18	+0.09
Pt (IV) 12	11.19	+0.10
Fe*(III) 50	11.18	+0.09

*masked with tartrate

2.4. Determination of palladium

Aliquots of standard palladium chloride solution (5.0–35.0 mg) were pipetted out into a 250 ml beaker and diluted to 100 ml, while adjusting the acid concentration to 0.05–0.1 N with 2N hydrochloric acid. A 0.5% reagent solution was used for precipitation. The yellow precipitate was digested for 30 min, filtered, washed, dried at 110–120°C and weighed as Pd (C₁₂H₁₀O₂N)₂ · H₂O, the gravimetric factor being 0.20280. The results are given in table 2.

2.5. Determination of the error of the method

Five determinations of 15.54 mg of nickel gave a standard deviation of 0.037 mg, the relative mean error being ±0.19%, and six determinations of 11.09 mg of palladium gave a standard deviation of 0.03₂ mg, the relative mean error being ±0.36%.

2.6. Study of interferences

The effect of metals usually associated with nickel and palladium in their ores and alloys was studied. In the case of nickel, cadmium (II), zinc (II) and chromium (III) did not interfere. The interference due to iron (III) and aluminium (III) was masked with tartrate. Copper (II), nickel (II) and platinum (IV) did not interfere in the case of palladium. Gold (III) interfered.

2.7. Determination of the metal content in the complexes

The nickel-oxime complex (~1 g) was decomposed with about 40 ml of concentrated nitric acid, the excess acid being expelled by evaporation with concentrated sulphuric acid. The residue was cooled, digested with water and filtered. The filtrate was made up to the mark in a 100 ml standard flask. Aliquots (20 ml) were taken and the nickel content was determined with dimethylglyoxime (Vogel 1975). The metal content was found to be 12.76% (calculated=12.78%).

About 200 mg of the palladium-oxime complex was decomposed with 200 ml of concentrated nitric acid followed by evaporation with 10 ml of concentrated hydro-

chloric acid. The cold residue was extracted with 6 ml of concentrated hydrochloric acid and diluted to 100 ml. Palladium content was determined with dimethylglyoxime (Vogel 1975). The metal content was found to be 20.26% (calculated = 20.28% in agreement with the proposed formula $\text{Pd}(\text{C}_{12}\text{H}_{10}\text{O}_2\text{N})_2 \cdot \text{H}_2\text{O}$).

3. Conclusions

The present study shows that 2-hydroxy-1-acetonaphthoneoxime can be used for the gravimetric determination of copper, nickel, and palladium. The reagent has better gravimetric factors than most of the oximes reported in the literature like *o*-hydroxyacetophenoneoxime (Poddar 1957), *o*-vanillin-oxime (Goel *et al* 1968), 2-hydroxy-5-methylpropiofenoneoxime (Prakash *et al* 1968), resacetophenoneoxime (Bhatki and Kabadi 1953) and 5-ethyl resacetophenoneoxime (Naik *et al* 1976) for the determination of nickel and 2-hydroxy-5-methylpropiofenoneoxime (Prakash *et al* 1966), resacetophenoneoxime (Rajareddy 1967) and 2-propionyl-1-naphtholoxime (Patkar *et al* 1973) for the determination of palladium.

Acknowledgements

The authors (VVB and RCH) are thankful to CSIR and UGC, New Delhi respectively for the award of Fellowships to them.

References

- Bhatki K S and Kabadi M B 1953 *J. Sci. Ind. Res.* **B12** 226
Goel D P, Trikha K C and Singh R P 1968 *Proc. Indian Acad. Sci.* **A68** 82
Hussain R C and Appala Raju N 1976 *Proc. Indian Acad. Sci.* **A84** 144
Naik N D, Naik H B and Desai C M 1976 *J. Indian Chem. Soc.* **53** 405
Patkar D N, Tambat N D and Merchant R D 1973 *Curr. Sci.* **42** 818
Poddar S N 1957 *Z. Anal. Chem.* **155** 327
Prakash Shri, Singh R P and Trikha K C 1966 *Talanta* **13** 1393
Prakash Shri, Dutt Yag and Singh R P 1968 *Indian J. Chem.* **6** 664
Rajareddy G 1967 *Curr. Sci.* **36** 665
Vogel A I 1975 *A text-book of quantitative inorganic analysis* (Elbs and Longman, third edition) p. 358, 479 and 512