

NOTE

**Spectrophotometric Determination of Palladium(II)
with 3-Phenylazohydroxylamine**

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3-Phenylazohydroxylamine has been suggested as photometric reagent for determination of palladium(II). The complex shows constant absorbance to the pH range of 5.0 at 425 nm and Beer's law is obeyed upto 8.5 ppm. The sensitivity of the reagent is 0.0054 g/cm⁻² and molar absorptivity 5.69 × 10⁴ l mol⁻¹ cm⁻¹ at 425 nm.

3-Phenylazohydroxylamine acts as a non-selective ligand towards Pd(II). The complexation between this reagent and Pd(II) is attributed to presence of oxime and azo groups. 3-Phenylazohydroxylamine is a versatile ligand for the determination of various metal ions¹.

3-Phenylazohydroxylamine was prepared by coupling hydroxylamine hydrochloride (3.6 gm) with benzene diazonium chloride (from 9.3 ml aniline) at 0.5°C. 3-Phenylazohydroxylamine hydrochloride thus obtained is neutralised with sodium carbonate to get the desired compound. It was then recrystallised with aqueous ethanol. 0.5% Reagent solution was prepared in 70% ethanol.

To an aliquot containing Pd(II), add sufficient excess of 3-phenylazohydroxylamine, adjust the pH of the solution in the range 5.0, heat contents for 10 mins. on water-bath, make the volume to 10 ml and measure absorbance at 425 nm against reagent blank. The complex shows constant absorbance in the pH range 5.0 at 425 nm. The composition of the complex was found to be 1 : 2 (metal : ligand) by different methods. The log k was found to be 7.46 at 25 ± 0.5°, pH 5.0. Beer's law is obeyed upto 8.5 ppm. The sensitivity of the reagent is 0.0054 g/cm² and molar absorptivity is 5.69 × 10⁴ l mol⁻¹ cm⁻¹ at 425 nm.

Synthetic solution containing 2.45 ppm of Pd(II) and varying amounts of diverse ions were prepared for the purpose. Tolerance limits (error ± 2%) for metal ions are stated in brackets below:

Pd-3-Phenylazohydroxylamine, perchlorate, tartarate, chloride, phosphate, borate (4000 ppm each), iodide, nitrate, flouride, acetate (1500 ppm each); bromide, Cd(II) (800 ppm each); Zn(II), Co(II), Ni(II) (400 ppm each); thiosulphate, oralate, EDTA, Pb(II) (40 ppm each); thiourea, Ir(III) Os(VIII) (5 ppm each).

The reagent was successfully used for the determination of palladium in trace amount. Synthetic mixture containing palladium (5 ppm) mixed

with ruthenium (10 ppm) or gold (7 ppm) was prepared and palladium (II) was determined by above procedure.

REFERENCES

1. S. P. Mathur, R. S. Thakur and R. D. Jaiman, *Revue Roumaine de Chimie*, **26** 1033 (1981).
2. S. Singh, V. Pradhan, S. Sharma and S. P. Mathur, *J. Inst. Chemists (India)*, **58**, 197, 1986.

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