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Research Paper

Studies on Palladium-N-Thiobenzyl Hydroxylamine complexes

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Abstract: The pink colored complexes of Pd (II) and N-thiobenzoyl hydroxylamine are extractable into chloroform. The spectrophotometric studies following Job's and molar ratio methods have shown that 1:2 (metal-ligand) complex predominate in the acidity range of 3.5-8 N HCl. The stability constant of 1:2 metal ligand complex system have been evaluated spectrophotometrically following Ledeon's Yatisimirrkii's and Harvey manning method.

Keywords: N-thiobenzoyl hydroxylamine, palladium-N-thiobenzoyl hydroylamine complexes

Introduction:

N-thiobenzoyl hydroxylamine is found to be an interesting analytical reagent. The reagent is bindentate ligand and forms complexes with metal-ligand ratio of 1:2 in acidity range of 3.5-8 N HCl. Prior, the chelating ability of hydroxamic acid has extensively been investigated (Mathur et. al., 1978 a & b; Sigel and Mecormick, 1970; Khanna *et. al*., 1978; Mathur *et. al*., 1978). Presence of sulphur in the compound as important donor atom and being able to form σ- and π-bonds may lead some important conclusions. The present communication deals with spectrophotometric determination of stepwise formation constants of palladium-N-thiobenzoyl hydroylamine complexes. The values of stability constants, stepwise and over all have been calculated following extended Leden's Yatsimiirski (Yalsimirski, 1956), and Harvey-manning method (Harvey and Manning, 1950).

Experimental:

The reagent was prepared following the method of Shetty. A standard solution of palladium was prepared by dissolving palladium chloride in distilled water containing hydrochloric acid. The palladium content in the solution was determined using dimethyl glyoxime. All chemicals used were of spectro-grade quality. A Beckmann Model-DU quartz spectrophotometer was used for absorbance measurements.

Procedure:

An aliquot of palladium (II) solution (0.85×10^{-3} mole/litre) was taken in a 100 ml separatory funnel and the acid

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concentration adjusted at 5 N Hydrochloric acid. The reagent solution in chloroform was added in such a way that total volume of non aqueous phase was always kept 10 ml. at each step of titration. The ratio of aqueous and non aqueous phase was 1:1 and mixture was shaken for 15 minutes to acquire equilibrium. The brown chloroform layer was allowed to settle, separated and dried over anhydrous sodium sulphate. The extract after drying over sodium sulphate was diluted to 25 ml. with chloroform. The complex shows an absorption maximum at 530 nm. when measured against solvent blank. Equimolar solution of palladium and reagent was used to find out metal to ligand ratio by Job's (Harvey and Manning,1950; Shetty and Companella 1970; Job, 1928) method of continuous variation and mole-ratio method. The photometric measurement made at 530 nm from 5 N Hydrochloric acid indicated a metal-ligand ratio 1:2. The complex in extractable into chloroform from 3.5 to 8 N Hydrochloric acid with no appreciable change in sensitivity value.

Determination of Stepwise formation constants:

The formation constants of K_1 and K_2 of palladium complexes ML_1 and ML2 respectively were obtained following graphical extrapolation method of Leden and Yatsmirskii. A number of subsidiary functions were constructed and appropriate limiting values at zero ligand concentrations were utilized to arrive at the result. The value of log K_1 and log K_2 obtained by above methods are given in Table 1.

Table 1: Stepwise stability constants of palladium complex (1:2 composition) at 25± 1°C

Method	Log K_1	Log K_2	Log K_1K_2
Leden's[6]	4.62	4.15	8.77
Yatsimirskii's[7]	4.36	4.05	8.41
Harvey-manning	-	-	8.24

Result and Discussion:

The stepwise formation constants were calculated from the extrapolated values of the subsidiary functions. From the established composition 1:2 (metal-ligand) the free ligand concentration for each solution was calculated from Beer's Law data. It is assumed that the absorbance corresponds mainly due to mononuclear coloured complex species of palladium.

In order to evaluate the two formation constants by Leden's method from spectrophotometric data, a series of subsidiary functions were constructed and extrapolated to zero value of variable (L). The intercept values obtained leads to the calculation of K_1 and K_2. The extrapolation was necessary since the experimental points obtained from absorbance values corresponding to free ligand concentrations in the range of 0 to 4.0 x 10^{-5} mole/litre indicated some deviation from the usual plot. Inherent experimental error lead to deviations in the said range, as the absorbance values were sufficiently low in the region. The molar absorptivity e_1 and e_2 for the species ML_1 and ML_2 are 6.02 x 10^3 and 6.84 x 10^3 respectively.

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