

The Micro Determination of Palladium with *p*-Ethoxy-chrysoidine

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The use of a number of chromogenic reagents in the determination of platinum metals has been reported. For instance, Grigore Popa and his co-workers¹⁾ described the spectrophotometric determination of palladium with chrysoidine within the range from 1 to 15 μg . palladium/ml. In the present investigations, the use of *p*-ethoxy-chrysoidine, which contains the chromogenic group ($-\text{OC}_2\text{H}_5$), in the spectrophotometric determination of palladium will be described; it will be shown to be more sensitive than chrysoidine.

The absorption spectra of the dye and palladium complex are shown in Fig. 1. The maximum difference in absorbance (Δ -absorbance) between

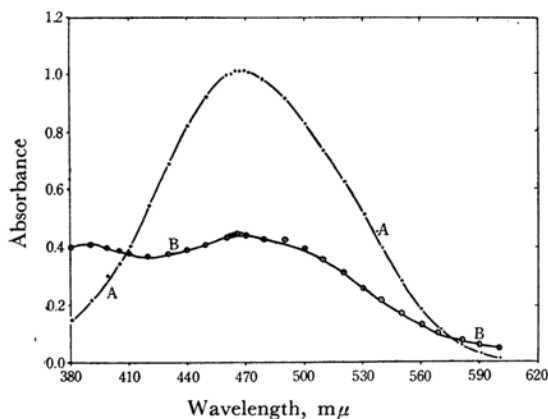


Fig. 1. Absorption spectra. pH 2.2

A: Reagent ($4 \times 10^{-5} \text{ M}$), against water
B: Reagent ($4 \times 10^{-5} \text{ M}$) + Pd ($2 \times 10^{-5} \text{ M}$), against water

the dye and the palladium complex is found at $465 \text{ m}\mu$ at pH 2.2. The complex is formed by adding 0.5 ml. of a 0.002 M palladium solution to a mixture of 10 ml. of a sodium acetate - hydrochloric acid buffer solution (pH 2.2) and 1.0 ml. of a

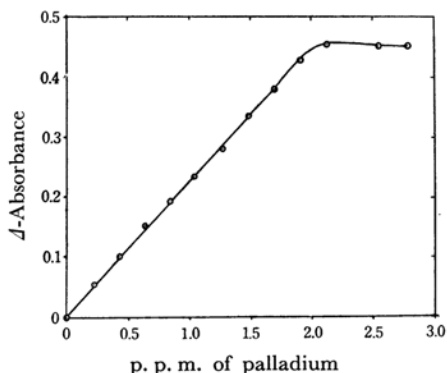


Fig. 2. Beer's law curve. pH 2.2

0.002 M dye solution in a 50-ml. volumetric flask. The solution is then diluted to the mark with double-distilled water and mixed thoroughly. A dye blank is also prepared under similar conditions. The Δ -absorbance between the dye blank and the palladium complex is at its maximum in the 2.0—2.4 pH range. The colour under these conditions develops instantaneously and is stable for at least one hour. There is no variation in the Δ -absorbance over the temperature range of 15—45°C. The presence of excess palladium, however, helps in the coagulation of the complex.

Beer's law holds over the 0—1.9 p. p. m. palladium/ml. range at $465 \text{ m}\mu$ (Fig. 2); the molar absorptivity is 2.5×10^4 , and the sensitivity is $0.004 \mu\text{g}$. palladium/ cm^2 , corresponding to $\log I_0/I = 0.001$.

Composition studies by Vosburgh and Cooper's method²⁾ and by the slope-ratio method³⁾ indicate the formation of only one compound, with a ratio of palladium to dye of 1 : 2.

Further, more detailed studies are now in progress.

2) W. C. Vosburgh and G. R. Cooper, *J. Am. Chem. Soc.*, **63**, 437 (1941).

3) A. E. Harvey and D. L. Manning, *ibid.*, **72**, 4448 (1950).

1) G. Popa, V. Croitoru and D. Costache, *Studia Univ. Babes-Bolyai, Ser. Chem.*, **8**(1), 195 (1963).