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### POLAROGRAPHIC DETERMINATION OF Pd(II) USING CATALYTIC H-WAVES

G. MOHAMED RAFI AND AGNES PAUL  
Department of Analytical Chemistry,  
A. C. College of Technology, Madras University,  
Madras 600 025, India

LITERATURE records many attempts to determine Pd(II) by D. C. Polarography<sup>1-5</sup>. With one exception<sup>6</sup>, the lower limits of estimation had hardly been 10 ppm and this exception has a prior concentration step of an extraction procedure before final estimation. It is well known that metal complexes of thiosemicarbazide (TS) give catalytic waves some of which have been analytically useful<sup>7-10</sup> Pd(II) at pH 1.5, in presence of TS, produces a catalytic hydrogen-wave with a large sharp maximum at potentials 170 mV more positive than the normal hydrogen-wave. The  $I_{max}$  at the peak potential is found to be proportional to the concentration of Pd(II) in the range, 0.5 – 11.0 ppm.

Polaritor P03 pen recording type polarograph, with a built-in saturated calomel anode is used. The characteristic of the capillary is  $2.069 \text{ mg}^{2/3} \text{ sec}^{-1/2}$  at  $h_{Hg} = 30 \text{ cm}$  at zero applied potential. Elico pH meter model LI 10 is used for adjusting the pH values correct to  $\pm 0.1$  pH.

Unless otherwise stated, all the chemicals are of reagent grade. PdCl<sub>2</sub> (John-Matthey) is dissolved in the minimum quantity of 4NHCl, made up to 100 ml with deionised water and standardised gravimetrically<sup>11</sup>. This solution (0.0948 M) is diluted appropriately for different studies. TS (Loba-Chemie) is prepared in deionised water to make a stock solution of 0.1 M. Sodium perchlorate is used as the supporting electrolyte to maintain a constant ionic strength of 0.6 M. Before subjecting to polarography the final solution is deoxygenated for 10 min in a stream of oxygen-free nitrogen.

Pd(II) in NaClO<sub>4</sub> medium, at pH 1.5, produces wave  $E_{1/2} = +0.06 \text{ V}$  vs SCE due to mercurous

ions<sup>12</sup> liberated chemically by Pd<sup>2+</sup> ions and an additional wave due to H<sup>+</sup> reduction with a small maximum at  $-1.67 \text{ V}$  vs SCE. In the presence of TS, the peak potential at  $-1.67 \text{ V}$  shifts to more positive values. With large excess of TS (0.02 M), the maximum at  $-1.50 \text{ V}$  ceases its further shift. Under these conditions addition of even a small amount of Pd(II) increases the  $I_{max}$  to about 3-fold compared to the increase of the limiting current. The maximum is however completely suppressed by 0.001% gelatin.

The fact that the pre-wave is catalytic in nature is clear from its independent behaviour with  $h_{Hg}$  at high TS concentration and its low temperature coefficient (0.4 %°K). Further the positive shift in potential of the hydrogen reduction wave by the complex also confirms the catalytic nature of the process. That the protons are involved in the reduction process is confirmed by the hydrogen evolution during the macroelectrolysis controlled at  $-1.60 \text{ V}$  vs SCE with a Hg pool cathode under polarographically identical conditions. Also any alteration in H<sup>+</sup> has a marked influence on the  $I_{max}$  at the peak potential. The relationship between  $I_{max}$  and Pd(II) concentration, is found to be proportional in the range 0.5 – 11.0 ppm with an accuracy of  $\pm 2\%$ .

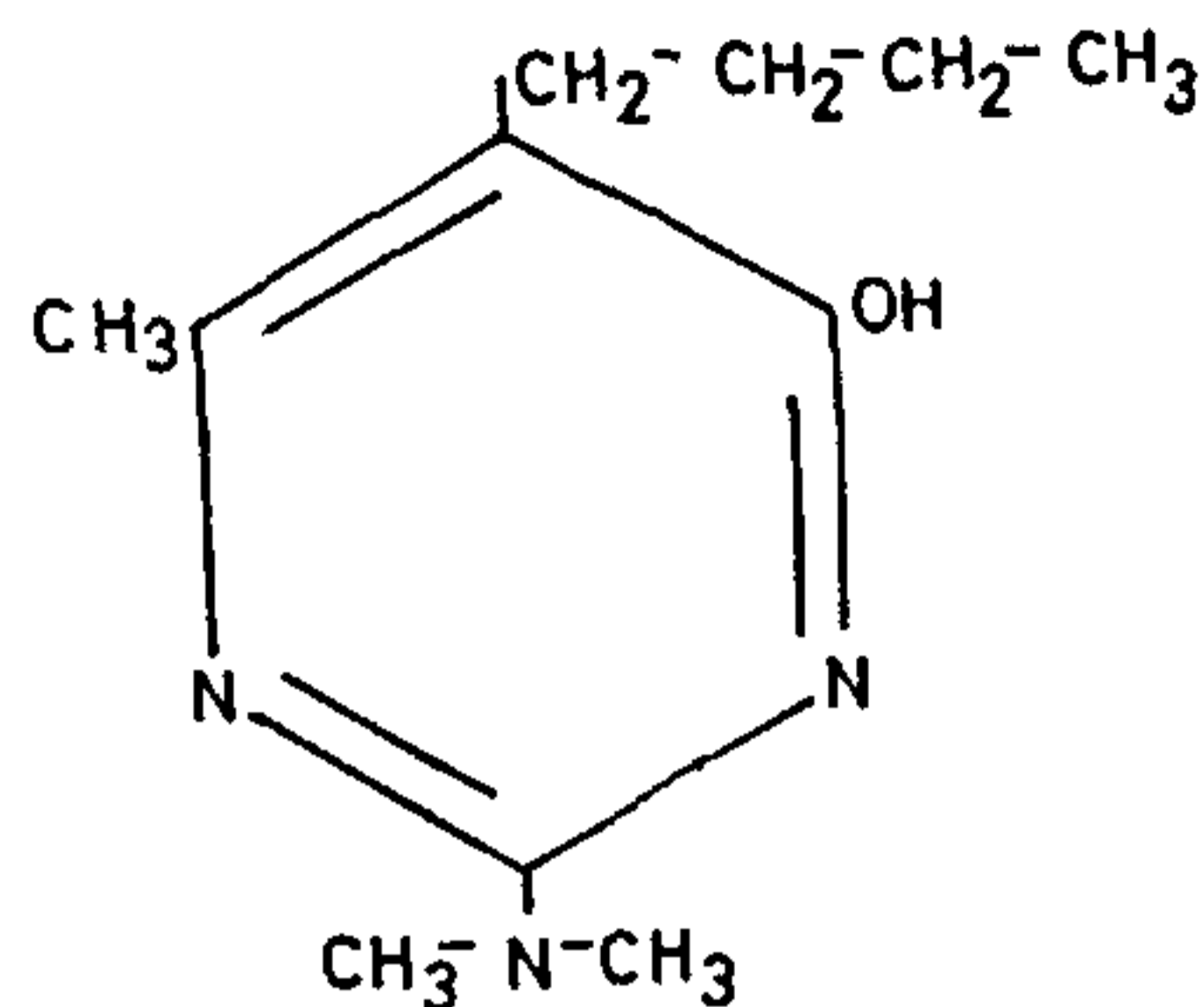
In order to assess the possible analytical applicability of the above method, the effect of other noble metals in the estimation of 2.2 ppm of Pd(II) has been studied, an error of  $\pm 2\%$  in the analysis being considered tolerable. The limits of interference are 1 ppm of Ir, 4 ppm of Rh, 10 ppm of Pt and 20 ppm of both Ru and Os. This method has been found to be quite satisfactory for the estimation of Pd(II) in synthetic mixtures simulating the compositions of jewellery metal<sup>13</sup>, electrical standard resistors<sup>14</sup> and catalyst<sup>15</sup> containing Pd(II) and other noble metals.

One of the authors (GM) thank UGC, New Delhi for a fellowship.

31 October 1981

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STRUCTURE OF DIMETHIRIMOL

## DIMETHIRIMOL: QUANTITATIVE ESTIMATION

R. C. DAS AND B. K. SAIKIA  
Regional Research Laboratory,  
Jorhat 785006, India

LITERATURE survey on dimethirimol shows the lack of analytical procedure for the quantitative estimation of this fungicide. As proton resonance spectroscopy is one of the standard methods for the quantitative estimations<sup>1,2</sup>, we have employed this technique and the results are discussed. Organic nitrogen is also estimated but it gives a lower percentage.

Dimethirimol, a pyrimidine<sup>2</sup> compound, is 5-*n*-butyl-2-dimethylamino-4-hydroxy-6-methyl pyrimidine. Its dimethylamino group gives a sharp singlet at 3.2 ppm without any overlapping of the other proton signals. Therefore, this peak is utilized for quantitative estimation. As the signal of chloroform (7.2 ppm) does not interfere with the analytical signal of dimethirimol, it is found suitable for use as internal standard.

Quantitative estimation is carried out by integrating two peaks, one at 3.2 ppm (dimethirimol) and the other at 7.2 ppm (chloroform) and the amount of substance found is calculated<sup>2-4</sup>.

Twenty estimations were carried out, starting from 0.10 g to 0.02 g and the percentage was found between 98.5 and 99.8.

Dimethirimol of 99.6% purity was obtained from National Physical Laboratory, Teddington, England and the spectra were recorded on Varian T-60 NMR spectrometer, using spectroscopic grade carbontetrachloride as solvent to which a known amount of chloroform is added as internal standard.

The integration values are the average of five integrations for each set. Organic nitrogen is also determined by standard methods<sup>5</sup> but it taken about 20 hr and gives 97-98% purity.

From experimental data, the per cent variation is found to be between -0.88 and +0.16 which is within the limits of experimental error. Therefore, this method can be used for quantitative estimation with a smaller quantity of compound.

24 November 1981

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## A NEW FLAVONOID GLYCOSIDE FROM THE LEAVES OF *IPOMOEA FISTULOSA*

P. DUBEY, N. KHARE AND P. C. GUPTA  
Department of Chemistry,  
University of Allahabad,  
Allahabad 211002, India

*IPOMOEA fistulosa* (I. carnea N. O. Convolvulaceae) is known as Behaya<sup>1</sup> in Hindi and is a wildly growing poisonous straggling shrub. The plant is not only used to protect the crops from cattle<sup>2</sup>, but as a hedge, purgative and laxative. An ester and two other compounds<sup>3</sup> have already been reported but the presence of the flavone glycoside and the phytosteroline has not been reported.

From the ethylacetate extract of leaves a new flavone glycoside (A) and a phytosteroline (B) have