TABLE 1—^{13}C NMR OF COMPOUND (I)

Carbon no.	ppm	Carbon no.	ppm
2	159.7 s	2′	77.2 s
3	112.9 s	3′	120.8 d
4	145.4 d	4′	124.5 d
4a	118.2 s	1″	40.2 s
5	130.8 d	2″	137.4 d
6	131.5 s	3″	111.9 t
7	154.4 s		
8	103.4 d	1″ < Me / Me	25.9 q*
8a	155.8 s	2′ < Me / Me	28.0 q*

*Values may interchange.

Acknowledgement

The authors express their thanks to Dr. S. C. Bhattacharyya, Director and Prof. A. K. Barua, Chairman, Department of Chemistry for their interest in the work.

References

1. D. P. CHAKRABORTY, *Fortschr. Chem. Org. Naturst.*, 1977, **34**, 299.
2. M. N. S. NAYER, M. K. BHAN and V. GEORGE, *Phytochemistry*, 1973, **12**, 2073.
3. R. LEMIEUX and E. RUDLOFF, *Can. J. Chem.*, 1955, **33**, 1701.

2-Hydroxy-5-carboxy-acetophenone Oxime as an Analytical Reagent for Gravimetric Determination of Palladium

B. L. SHAH and C. M. DESAI*

Department of Chemistry, P. J. Science College, Bardoli-394 601

Manuscript received 8 September 1981, accepted 26 May 1984

2-Hydroxy-5-carboxy-acetophenone oxime (HCAO) has been used as a reagent for gravimetric determination of copper[1] and nickel[2]. The present work describes the use of this ligand for the gravimetric determination of palladium(II). The reagent precipitates palladium ion at 0.8 and is quantitative between *p*H 1.5 to 3.0 and after drying at 110 to 120° the composition of the chelate corresponds to the formula $Pd(C_9H_8O_4N)_2$.

Experimental

Palladium chloride (1.0 g) was dissolved in minimum quantity of hydrochloric acid and the solution was made to 500 ml. with distilled water.

Preparation of reagent: *p*-Hydroxy-benzoic acid was acetylated[3] and the resulting compound *p*-acetoxy-benzoic acid was converted into ketone by the following method[4]. *p*-Acetoxy-benzoic acid (3.0 g, 1 mol) and anhydrous aluminium chloride (7.0 g, 3.3 mol) were intimately mixed and heated on an oil bath at 150 to 155° protected by $CaCl_2$ guard tube for 1 h. The reaction mass was cooled and treated with ice and conc. HCl. The mass was washed and crystallised from ethanol to yield the resulting ketone, 2-hydroxy-5-carboxy-acetophenone[4], m.p. 241°. The oxime was prepared by the reaction of the above ketone with hydroxylamine hydrochloride and sodium acetate in aqueous ethanol Crystallised from ethanol yielded the product, m p. 258°d.

Determination of palladium(II) ion: A known volume (10 ml) of palladium ion solution was diluted to 70-80 ml and *p*H was adjusted to 1.5-2.0 using acetic acid-sodium acetate buffer. The solution was warmed to 60-70° and ethanolic solution of the reagent (1.0%) was added with constant stirring followed by about 10% excess to ensure complete precipitation. The yellow precipitate obtained was digested on water bath (60-80°) for 30 min. It was filtered and washed with hot water and finally with ethanol to remove any contamination of ligand from the complex.

The precipitate was dried at 110-120° and weighed as $Pd(C_9H_8O_4N)_2$. It is insoluble in common organic solvents but soluble in pyridine, NaOH or NH_4OH solutions. Some of the results are given in Table 1. Conversion factor for Pd^{II} is 0.2152.

TABLE 1—DETERMINATION OF PALLADIUM(II)

Pd(II) taken mg	Weight of complex mg	Pd(II) found mg	Error mg
12.28	57.30	12.33	+05
6.14	28.60	6.15	+01
9.82	45.50	9.85	+03
18.42	85 8	18.46	+ 04

References

1. B. L. SHAH and C. M. DESAI, *South Gujarat Univ. J.*, 1981.
2. B. L. SHAH and C. M. DESAI, *Curr. Sci.*, submitted for publication.
3. A. I. VOGEL, "A Text Book of Practical Organic Chemistry", 3rd. ed., Longmans, London, 1955, p. 996.
4. D. N. SHAH and N. M. SHAH, *J. Indian Chem. Soc.*, 1949, **26**, 235.

* South Gujrat University, Surat.