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Note

Platina- and palladalactam complexes derived from 2-benzoylacetanilide; syntheses and X-ray structure of [Pd{NPhC(O)CHC(O)Ph}(bipy)]·CH₂Cl₂

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Abstract

Reactions of the complexes $[PtCl_2(cod)]$ (cod = cyclo-octa-1,5-diene), *cis*- $[PtCl_2(PPh_3)_2]$, and $[PdCl_2(bipy)]$ (bipy = 2,2'bipyridine) with 2-benzoylacetanilide and excess silver(I) oxide gives metallalactam complexes $[M{PhNC(O)CH(COPh)}L_2]$ in good yields. The complexes have been characterised by NMR and IR spectroscopies, elemental analysis, and electrospray mass spectrometry (ESMS). A single-crystal X-ray structure of the bipy palladium complex reveals the expected four-membered ring system, which is almost planar. No evidence was observed for platinum–oxygen bonded products, which were a possibility based on the result of the related system involving PhC(O)CH₂C(O)CH₂C(O)Ph reported in the literature, which yielded a six-membered dienediolate platinacycle. © 1999 Elsevier Science S.A. All rights reserved.

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1. Introduction

We recently reported on the synthesis of a range of metallalactam complexes of platinum(II) and palladium(II) formed by the reaction of the appropriate metal dihalide complex with either *N*-cyanoacetylurethane [1,2] or acetoacetanilide [3] mediated by silver(I) oxide, giving complexes 1 and 2, respectively. Other metallacyclic systems including platinalactone 3 [4], platinaureylene 4 [5], and platinacyclobutan-3-one



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 $(\eta^3$ -oxodimethylenemethane) **5** [6] complexes have also been prepared using the same methodology. These syntheses are successful providing that the organic precur-

0020-1693/99/\$ - see front matter © 1999 Elsevier Science S.A. All rights reserved. PII: S0020-1693(99)00190-5 sor contains sufficiently acidic NH and/or CH groups, which can be deprotonated by silver(I) oxide, which acts as a halide abstracting reagent and a strong base.

Following on from our studies on acetoacetanilide [3], in this paper we report on our investigations into the reactions of 2-benzoylacetanilide, $PhC(O)CH_2C$ -(O)NHPh, with various platinum(II) and palladium(II) dihalide complexes. It has previously been observed that the reactions of MeO₂CCH₂C(O)CH₂CO₂Me (dimethyl acetone-1,3-dicarboxylate) [6] or MeC(O)CH₂C(O)CH₂-C(O)Me (heptane-2,4,6-trione) [7] with a number of platinum(II) and platinum(0) complexes lead to platinum(II) oxodimethylenemethane (platinacyclobutan-3one) complexes of the type **5**.



In marked contrast, reaction with the phenyl analogue of the above triones, PhC(O)CH₂C(O)CH₂C(O)Ph, does not lead to oxodimethylenemethane complexes, but instead to the six-membered ring, platinum–oxygen bonded dienediolate complex 6 [7]. The phenyl groups presumably stabilise the extensively π -delocalised system. It was therefore of interest to examine whether metallalactam or six-membered ring analogues would be formed with the phenyl-rich 2-benzoylacetanilide system; possible complexes derived from 2-benzoylacetanilide containing Pt–O bonds are 7 and 8.



2. Results and discussion

2.1. Syntheses and structural characterisation

The reactions of the complexes $[PtCl_2(cod)]$ (cod = cyclo-octa-1,5-diene), *cis*- $[PtCl_2(PPh_3)_2]$, and $[PdCl_2(bipy)]$ (bipy = 2,2'-bipyridine) with one mole equivalent of 2-benzoylacetanilide and excess silver(I) oxide in refluxing dichloromethane give, after workup, the metallalactam complexes **9a**-**c**.



The complexes are isolated as white to pale yellow (platinum) or orange (palladium) air-stable solids which are soluble in dichloromethane and chloroform. Ligand displacement of the cod ligand of **9a** with phosphines also provides a useful route for the syntheses of **9b** and **9d**, and potentially a range of other derivatives. In all cases, the isolated products were of high purity, and there was no indication of any isomeric products containing platinum-oxygen bonds.

An X-ray diffraction study of the bipy palladium complex **9c** was undertaken in order to compare the structural features with those of other metallalactam complexes, in particular the related acetoacetanilide palladium complex **2a**. Details of the X-ray structure determination are given in Table 1, and in Section 3. Selected bond lengths and angles are given in Table 2. The complex crystallises with one molecule of dichloromethane per molecule of complex. However, elemental microanalysis on a vacuum-dried sample yielded results which better fit $0.5CH_2Cl_2$ molecules of crystallisation per molecule of complex (see Section 3).

The structure confirms the expected approximately square-planar palladium(II) centre which is part of a slightly puckered four-membered ring, and the structural features are similar overall to those of **1a** and the related acetoacetanilide complex **2a** reported previously. The fold angle between the N(1)–Pd–C(2) and N(1)–C(1)–C(2) planes is 5.2°. The metallacycle is therefore comparable, but somewhat more planar than that of complex **2a**, which has a fold angle of 17.5°. The plane of the bipy ligand is twisted slightly, by 2.60(5)°, relative to the plane of the palladacycle.

As expected from *trans*-influence considerations, the Pd–N(3) bond length (2.104(7) Å) is noticeably longer

Table 1

X-ray crystallographic data for $[Pd\{NPhC(O)CHC(O)Ph\}(bipy)]-(9c\cdot CH_2Cl_2)$

Crystal data	
Empirical formula	C ₂₅ H ₁₉ N ₃ O ₂ Pd·CH ₂ Cl ₂
Formula weight	584.76
Crystal system	monoclinic
Space group	$P2_{1}/c$
a (Å)	10.688(3)
b (Å)	12.074(7)
c (Å)	18.480(4)
β (°)	101.81(2)
$V(Å^3)$	2334(2)
Ζ	4
$D_{\rm c} ~({\rm g}~{\rm cm}^{-3})$	1.644
Data collection	
Diffractometer	Nonius CAD4
Radiation	Μο Κα
Wavelength (Å)	0.71073
Temperature (K)	193(2)
Crystal size (mm)	$0.25 \times 0.33 \times 0.33$
Data collection mode	ω -scans
θ Ranges for data collection (°)	1.95–24.95
Index ranges	$-12 \le h \le 0$
	$0 \le k \le 13$
	$-21 \le l \le 21$
Reflections collected	4291
Independent reflections	4062 [$R(int) = 0.0299$]
Absorption coefficient (mm ⁻¹)	1.054
<i>F</i> (000)	1176
Structural analysis and refinement	
Solution by	direct methods
Method of refinement	full-matrix least-squares
	based on F^2
Data/restraints/parameters	4062/0/307
Goodness-of-fit on F^2	1.041
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0562, \ wR_2 = 0.1420$
R indices (all data)	$R_1 = 0.0850, \ wR_2 = 0.1626$
	$w = [\sigma^2 (F_o^2) + (0.1041P)^2]$
	+3.1952P] ⁻¹ where
	$P = (F_{\rm o}^2 + 2F_{\rm c}^2)/3$
Largest difference peak, hole	1.945, -1.620
$(e \dot{A}^{-3})$	
Programs used	SHELXS-86 [15] and
	SHELXL-93 [16]

Ta	ble	2

Selected bond lengths (Å) and angles (°) for [Pd{NPhC(O)CHC(O)-
Ph}(bipy)](9c·CH ₂ Cl ₂) with e.s.d.s in parentheses

Bond lengths			
Pd-N(1)	2.021(5)	Pd-N(2)	2.042(5)
Pd-C(2)	2.054(6)	Pd-N(3)	2.104(5)
N(1)-C(1)	1.324(8)	C(1)–O(1)	1.239(8)
C(1)–C(2)	1.521(8)	C(3)–O(2)	1.249(8)
Bond angles			
N(1)-Pd-C(2)	67.1(2)	N(2)-Pd-C(2)	103.0(2)
N(1) - Pd - N(3)	110.7(2)	N(2)-Pd-N(3)	79.2(2)
C(1)–N(1)–Pd	98.0(4)	C(1)-N(1)-C(21)	122.9(5)
C(21)–N(1)–Pd	138.3(4)	N(1)-C(1)-C(2)	104.3(5)

than Pd–N(2) (2.042(5) Å), with N(2) being *trans* to a relatively low *trans*-influence nitrogen, and N(3) *trans* to carbon. Similar bond lengths were observed for the acetoacetanilide complex **2a**. The phenyl substituent on N(1) is bent away from the bipy ligand, as shown by bond angles Pd–N(1)–C(21) and C(1)–N(1)–C(21) (138.3(4) and 122.9(5)°, respectively). The benzoyl substituent on C(2) does not show this behaviour (bond angles Pd–C(2)–C(3) 112.7(4); C(1)–C(2)–C(3) 121.1(5)°), presumably because the steric bulk of the benzoyl substituent is further removed from the palladacycle, with the additional C–C bond. The benzoyl and phenyl substituents lie rotated out of the palladium coordination plane, presumably for steric reasons.

2.2. NMR spectroscopy

The NMR spectroscopic features are generally similar to those of other platina- and palladalactam complexes reported previously [1-3]. The cod complex 9a shows, as a result of the asymmetry of the metallacycle, four inequivalent cod CH groups, and four inequivalent cod CH₂ groups, in both the ¹H and $^{13}C{^{1}H}$ NMR spectra. In the $^{13}C{^{1}H}$ spectrum, two resonances at δ 106.6 and 105.8 both show ¹J(PtC) coupling constants of 62 Hz and are assigned to CH groups trans to the platinacyclic CH(COPh) group. Resonances at δ 87.9 and 86.1 show ¹J(PtC) values of 167 and 157 Hz, respectively, and are assigned to the cod CH groups trans to NPh, based on trans-influence considerations. Similar results were observed for the cod platinum cyanoacetylurethane and acetoacetanilide complexes 1a [2] and 2b [3], respectively. No conclusions regarding the differing trans influences of the NR groups could be made based on the ${}^{1}J(PtC)$ values.

2.3. Electrospray mass spectrometry (ESMS)

ESMS is a relatively new, powerful technique for the characterisation of inorganic and organometallic complexes [8]. For complexes which contain basic oxygen or nitrogen atoms, strong $[M + H]^+$ (or $[M + NH_4]^+$) ions are observed when low cone voltages are employed. At the low cone voltage of 20 V all metallalactam complexes give strong pseudo-molecular ion peaks $[M + H]^+$. Excellent agreement between observed and calculated isotope distribution patterns was observed for all major ions. Aggregate ions of the type [2M +H]⁺, $[2M + NH_4]^+$, $[3M + NH_4]^+$ etc. were also typically observed, although to variable extents. The source of the ammonium cations is undoubtedly the acetonitrile mobile phase used. The aggregates for the platinum-cod 9a and palladium-bipy 9c complexes were more intense than those for the complexes containing phosphine ligands, presumably because the reduced steric bulk of the bipy and cod ligands allows a closer association of the metallacycles around an ammonium ion.

3. Experimental

3.1. General

Electrospray mass spectra were recorded in positiveion mode on a VG Platform II instrument using CH_3CN/H_2O (1:1 v/v) as the mobile phase. Sample delivery was by means of a SpectraSystem P1000 HPLC pump, operating at a flow rate of 0.01 ml min⁻¹. The sample was introduced by means of a Rheodyne injector valve fitted with a 10 µl sample loop. Spectra were typically an average of ten scans. Spectra were recorded using cone voltages of 20 V while the capillary voltage was kept constant at 3.5 kV. Identification of all major ions was assisted by comparison of experimental and calculated isotope distribution patterns, the latter being obtained by use of the isotope simulation program [9]. ¹H and ¹³C{¹H} NMR data were recorded in CDCl₃ solution on a Bruker AC300P NMR spectrometer at 300.13 and 75.47 MHz, respectively. ³¹P{¹H} NMR spectra were recorded on a JEOL FX90Q NMR spectrometer at 36.23 MHz; samples were referenced to external 85% H₃PO₄ and were recorded in CH₂Cl₂ with a D₂O insert for lock. IR spectra were recorded as KBr discs on a BIO-RAD FTS-40 spectrometer. All melting points were recorded on a Reichert Thermopan apparatus in air, and are uncorrected. Elemental microanalyses were carried out at the University of Otago Microanalytical Unit.

The compounds [PtCl₂(cod)] [10], [PdCl₂(cod)] [11], [PdCl₂(bipy)] [12], 1,2-bis(diphenylphosphino)ethane [13] and silver(I) oxide [14] were prepared by literature methods. 2-Benzoylacetanilide (Aldrich) and triphenylphosphine (Pressure Chemical Co.) were used as received.

3.2. Synthesis of [Pt{PhNC(O)CHC(O)Ph}(cod)] (9a)

A mixture of $[PtCl_2(cod)]$ (0.140 g, 0.375 mmol), 2-benzoylacetanilide (0.092 g, 0.385 mmol) and Ag₂O (0.266 g, excess) was refluxed in dichloromethane (20 cm³) for 18 h. The silver salts were removed by filtration, giving a light yellow solution, which was concentrated under reduced pressure. The pale yellow product was precipitated by the addition of light petroleum, filtered off and dried (yield 0.139 g, 69%). M.p. 163– 165°C (decomp.). IR: v(CO) 1663 cm⁻¹ (vs). Found: C, 51.2; H, 4.2; N, 2.6. C₂₃H₂₄NO₂Pt requires: C, 51.1; H, 4.5; N, 2.6%. ESMS: $[M + H]^+$ (m/z 541, 100%), $[2M + H]^+$ (m/z 1081, 16%). ¹H NMR: δ 8.1–6.9 [m, 10H, Ph], 5.15–5.35 [m, 2H, cod CH, J(PtH) not discernible], 4.90–4.84 [m, 1H, cod CH, ${}^{2}J(PtH)$ 62.5], 4.16–4.09 [m, 1H, cod CH, ${}^{2}J(PtH)$ 59.5], 3.87 [s, 1H, ring CH, ${}^{2}J(PtH)$ 100], 2.70–1.90 [m, 8H, cod CH₂]. ${}^{13}C{}^{1H}$ NMR: δ 197.3 [s, COPh, ${}^{2}J(PtC)$ 51], 173.9 [s, ring CO, ${}^{2}J(PtC)$ 162], 144.5–124.1 [m, Ph], 106.6 [s, cod CH, ${}^{1}J(PtC)$ 62], 105.8 [s, cod CH, ${}^{1}J(PtC)$ 62], 87.9 [s, cod CH, ${}^{1}J(PtC)$ 167], 86.1 [s, cod CH, ${}^{1}J(PtC)$ 157], 40.5 [s, ring CH, ${}^{1}J(PtC)$ 435], 32.3 [s, cod CH₂], 30.5 [s, cod CH₂], 29.3 [s, cod CH₂], 28.1 [s, cod CH₂].

3.3. Synthesis of [Pt{PhNC(O)CHC(O)Ph}(PPh₃)] (9b) from 9a by ligand displacement

The complex [Pt{PhNC(O)CHC(O)Ph}(cod)] (9a) (0.038 g, 0.071 mmol) and triphenylphosphine (0.039 g, 0.148 mmol) were dissolved in dichloromethane (0.5 cm³). The complex was precipitated by the addition of light petroleum (5 cm^3) and filtered yielding the product as a light yellow solid after drying under vacuum (yield 0.066 g, 97%). M.p. 173-174°C (decomp.). IR: v(CO) 1636 cm⁻¹ (br, s). Found: C, 64.0; H, 4.2; N, 1.5. C₅₁H₄₁NO₂P₂Pt requires: C, 64.1; H, 4.3; N, 1.5%. ESMS: $[M + H]^+$ $(m/z 957, 100\%), [2M + H]^+$ (m/z1914, 5%), $[2M + NH_4]^+$ (*m*/*z* 1931, 3%). ¹H NMR: δ 7.80-6.50 (m, 40H, Ph), 3.53-3.36 [dd, ring CH, ${}^{2}J(\text{PtH})$ 41.2, ${}^{3}J(\text{PH})$ 4.9, ${}^{3}J(\text{PH})$ 4.0]. ${}^{13}C{}^{1}H{}$ NMR: δ 201.2 [s, COPh, ²J(PtC) 32, ³J(PC) 4], 174.1 [s, ring CO, ²J(PtC) 75, ³J(PC) 7], 144.0–123.1 (m, Ph), 45.9 [d, ring CH, ${}^{2}J(PC)$ 64 ${}^{1}J(PtC)$ 390]. ${}^{31}P{}^{1}H{}$ NMR: AB spin system δ 16.33 [P trans C, ¹J(PtP) 2351, ²J(PP) 17], 10.00 [P trans N, ¹J(PtP) 3733, ²J(PP) 17].

3.4. Preparation of [Pt{PhNC(O)CHC(O)Ph}(PPh_3)_2] (9b) from cis-[PtCl_2(PPh_3)_2]

The complex cis-[PtCl₂(PPh₃)₂] was prepared in situ from [PtCl₂(cod)] (0.065 g, 0.171 mmol) and triphenylphosphine (0.097 0.370 mmol) g, in dichloromethane (10 cm³). 2-Benzoylacetanilide (0.044 g, 0.186 mmol) and silver(I) oxide (0.289 g, excess) were added to this stirred solution with additional dichloromethane (10 cm³) and the mixture refluxed for 24 h. The mixture was filtered, the volume of the filtrate reduced and addition of light petroleum gave a light tan powder. This was filtered off and dried under vacuum to give the product **9b** (yield 0.118 g, 72%). The product was identified by ESMS and ${}^{31}P{}^{1}H{}$ NMR.

3.5. Preparation of [Pd{NPhC(O)CHC(O)Ph}(bipy)] (9c)

The complex [PdCl₂(bipy)] (0.177 g, 0.531 mmol), 2-benzoylacetanilide (0.128 g, 0.536 mmol) and silver(I) oxide (0.412 g, excess) in dichloromethane (20 cm³) were refluxed for 24 h. Filtration of the silver salts



Fig. 1. ZORTEP diagram of the complex $[Pd{NPhC(O)CHC(O)Ph}-(bipy)]$ (9c·CH₂Cl₂), showing the atom numbering scheme. Ellipsoids are shown at the 50% probability level, the dichloromethane of crystallisation, and all hydrogen atoms have been omitted. The inset shows a side view of the molecule showing the slight puckering of the palladalactam ring.

afforded a bright orange solution, which yielded a pale orange microcrystalline powder upon removal of the solvent. The product was recrystallised by liquid-liquid diffusion of a dichloromethane/methanol (0.5:0.1 cm³) solution layered with light petroleum (5 cm³) to give 0.210 g (78%) of 9c·CH₂Cl₂. Orange crystals of X-ray crystallographic quality were thus obtained. M.p. 167-168°C (decomp.). IR: v(CO) 1603 cm⁻¹ (br, s). A sample for elemental analysis was recrystallised from dichloromethane-light petroleum and dried under vacuum. Found: С, 56.1; H, 3.7; N, 7.8. C₂₅H₁₀N₃O₂Pd·0.5CH₂Cl₂ requires: C, 56.5; H, 3.7; N, 7.8%. ESMS: $[M + H]^+$ $(m/z 500, 100\%), [2M + H]^+$ $(m/z \ 1001, 46\%), [3M + H]^+ (m/z \ 1500, 3\%).$ ¹H NMR: δ 9.1–7.0 (m, 18H, Ar), 5.29 (s, CH₂Cl₂ of crystallisation), 3.58 (s, 1H, ring CH). ${}^{13}C{}^{1}H{}$ NMR: δ 201.2 (s, COPh), 171.7 (s, ring CO), 154.8-121.9 (m, Ar), 35.9 (s, ring CH).

3.6. Preparation of [Pt{PhNC(O)CHC(O)Ph}(dppe)] (9d) by ligand displacement from 9a

The complex $[Pt{PhNC(O)CHC(O)Ph}(cod)]$ (9a) (0.034 g, 0.064 mmol) and dppe (0.026 g, 0.066 mmol) were dissolved in dichloromethane (0.5 cm³). Addition

of light petroleum (5 cm³) precipitated the product as a tan solid, which was filtered off and dried under vacuum, (0.042 g, 89%). M.p. 131–133°C (decomp.). IR: v(CO) 1632 cm⁻¹ (br, s). Found: C, 59.4; H, 4.4; N, 1.7. C₄₁H₄₀NO₂P₂Pt requires: C, 59.4; H, 4.3; N, 1.7%. ESMS: $[M + H]^+$ (m/z 831, 100%), $[2M + H]^+$ (m/z 1662, 24%), $[2M + NH_4]^+$ (m/z 1679, 3%). ¹H NMR: δ 8.34 (m, 30H, Ph), 3.81–3.77 [dd, 1H, ring CH, ²J(PtH) 72.0, ³J(PH)_{trans} 9.1, ³J(PH)_{cis} 1.9], and 2.4–1.8 (m, br, 4H, CH₂, dppe). ¹³C{¹H} NMR: δ 199.3 [d, COPh, ³J(PC) 3], 174.5 [d, ring CO, ²J(PtC) 142, ³J(PC) 8], 146.4–123.0 (m, Ph), 44.3 [d, ring CH, ¹J(PtC) 323, ²J(PC) 61], 31.7–28.2 (m, dppe CH₂). ³¹P{¹H} NMR: AB spin system δ 44.3 [P trans C, ¹J(PtP) 2607], 35.9 [P trans N, ¹J(PtP) 3234].

3.7. X-ray diffraction study of [Pd{PhNC(O)CHC(O)Ph}(bipy)](9c·CH₂Cl₂)

X-ray crystallographic data for complex 9c and the details of the solution and refinement are summarised in Table 1, and in the supplementary data. Preliminary studies were performed by X-ray precession photography, giving approximate cell dimensions and the space group.

The complex crystallised with one molecule of dichloromethane in the lattice, and there appears to be a hydrogen-bonded network of symmetry-related dichloromethane molecules [Cl(2)–H(10B') 2.829 Å]. The closest intermolecular contacts of the dichloromethane to the palladalactam complex are C(101)…C(12) 3.856, Cl(1)…O(2) 3.114 and Cl(2)…N(2) 3.175 Å, none of which represent a hydrogen bond (Fig. 1).

4. Supplementary material

Full tables of atomic coordinates, bond lengths and angles, and thermal parameters have been deposited with the Cambridge Crystallographic Data Centre, and can be obtained from author W.H. on request.

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