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Ureato(1−) complexes of palladium(II) and platinum(II) Crystal structure of $[NBu_4][(C_6F_5)_2Pt\{PhNC(O)NPr^i_2\}]$

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Abstract

The reaction of $[NBu_4]_2[\{M(C_6F_5)_2(\mu\text{-}OH)\}_2]$ (M = Pd, Pt) with amines R_2NH in the presence of the heterocumulene phenylisocyanate, PhNCO, leads to the synthesis of the ureato(1−) complexes $[NBu_4][M(C_6F_5)_2\{PhNC(O)NR_2\}]$ (M = Pd, Pt). The single-crystal structure of $[NBu_4][(C_6F_5)_2Pt\{PhNC(O)NPr^i_2\}]$ revealed the presence of a *N*,*N*′-chelating ureato(1−) with a N(1)–Pt–N(2) bite angle of 64.0(3)°.

Keywords: Palladium complexes; Platinum complexes; Ureato complexes; Phenylisocyanate; Crystal structures

1. Introduction

Dimeric hydroxo complexes $M(\mu\text{-}OH)_2M$ of palladium(II) and platinum(II) [1] have shown to be excellent precursors in organic and organometallic synthesis of new compounds [2]. The acid–base reaction of these hydroxo complexes with a protic electrophile (HL) leads to the formation of mono- or binuclear complexes depending on the nature of the deprotonated $(L)^-$ ligand [3–7]. Furthermore, in the reaction of $[M_2(C_6F_5)_4(\mu\text{-}OH)_2]^{2-}$ with amines in the presence of CS_2, C–N bonds are formed to give the corresponding dithiocarbamate complexes $[M(C_6F_5)_2(S_2CNR_2)]^-$ (M = Pd, Pt) [8].

Following our systematic study of the reactivity of hydroxo complexes of Pd and Pt, we have now investigated the chemical behavior of $[M_2(C_6F_5)_4(\mu\text{-}OH)_2]^{2-}$ with amines R_2NH in the presence of the heterocumulene phenylisocyanate, PhCNO, to form the ureato(1−) complexes $[M(C_6F_5)_2\{PhNC(O)NR_2\}]^-$ (M = Pd, Pt), including the first crystal structure of a mononuclear chelate ureato(1−) platinum complex.

Urea is a final product of the nitrogen cycle in the human body and many bio-chemically important substances have an N(CO)N skeleton, such as uracil, cytosine and thymine. Urea and its alkyl or aryl derivatives act as weak N- or O-donor [9,10], and so far the ureato(1−) anion functions as an *N*-bonded monodentate terminal group [11–13], an *N*-bonded monodentate bridging group between two metal centres [14], an *N*,*O*-chelating ligand (generating highly strained, four-membered rings) [15], an *N*,*O*-bridging ligand between two metal centres [16] or an *N*,*N*′,*O*-bonded ureato(1−) anion bridging three metal ions [17]. To the best of our knowledge, no crystal structure of any *N*,*N*′-chelated ureato(1−) complex of any transition metal is available so far. Mononuclear ureato(2−) palladium [18] and platinum(II) complexes [19], and a number of tri- and tetranuclear palladium complexes containing urea dianions have also been reported [14].

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2. Experimental

2.1. Materials and physical measurements

C, H, and N analyses were performed with a Carlo Erba model EA 1108 microanalyzer. Decomposition temperatures were determined with a Mettler TG-50 thermobalance at a heating rate of 5 °C min^{-1} and the solid samples under nitrogen flow (100 mL min^{-1}). Molar conductivities were measured in acetone solution ($c \approx 5 \times 10^{-4}$ mol L^{-1}) with a Crison 525 conductimeter. The NMR spectra were recorded on a Bruker AC 200E or Varian Unity 300 spectrometer, using $SiMe_4$ and $CFCl_3$ as the standard, respectively. In the ^1H NMR spectra of the ionic compounds, the signal of the NBu_4^+ cation have been omitted. IR spectra were recorded on a Perkin–Elmer 1430 spectrophotometer using Nujol mulls between polyethylene sheets. Mass spectra were recorded on a Fisons V.G. Autospec spectrometer using the standard Cs^+ ion FAB (acceleration voltage 25 kV) and nitrobenzyl alcohol as matrix.

The starting complexes $[NBu_4]_2[\{M(C_6F_5)_2(\mu\text{-}OH)\}_2]$ (M = Pd [1a], Pt [1b]) were prepared by procedures described elsewhere. The commercially available chemicals were purchased from Aldrich Chemical Co. and were used without further purification. Solvents were dried by the usual methods.

2.2. Preparation of $[NBu_4][Pd(C_6F_5)_2\{PhNC(O)NR_2\}]$ [R = Me (1), Et (2), Pr^i (3)]

To a solution of $[NBu_4]_2[\{Pd(C_6F_5)_2(\mu\text{-}OH)\}_2]$ (100 mg, 0.071 mmol) in THF (6 mL) was added the corresponding amine (0.142 mmol) and PhNCO (15.4 μL, 0.142 mmol). The mixture was stirred at room temperature (r.t.) for 2 h. The solvent was completely evaporated under reduced pressure and the residue was treated with Et_2O/hexane, then the solid formed was collected by filtration and air-dried.

2.2.1. $[NBu_4][Pd(C_6F_5)_2\{PhNC(O)NMe_2\}]$ (1)

Yield: 60 mg, 74%. *Anal*. Found: C, 52.3; H, 5.4; N, 5.1. Calcd. for $C_{37}H_{47}F_{10}N_3OPd$: C, 52.5; H, 5.6; N, 5.0%. M.p.: 211 dec. $\Lambda_M = 112$ S cm^2 mol^{-1}. IR (Nujol, cm^{-1}): 1685 ν(C=O), 790, 780 (Pd–C_6F_5). ^1H NMR ($(CD_3)_2CO$): δ 7.18 (d, $2H_o$, $J_{om} = 7.4$ Hz), 6.92 (dd, $2H_m$, $J_{om} = J_{mp} = 7.4$ Hz), 6.69 (t, $1H_p$, $J_{mp} = 7.4$ Hz), 2.58 (s, 6H, $N(CH_3)_2$). ^{19}F NMR ($(CD_3)_2CO$): δ −113.0 (d, $2F_o$, $J_{om} = 27.7$ Hz), −114.9 (d, $2F_o$, $J_{om} = 28.8$ Hz), −164.6 (t, $1F_p$, $J_{pm} = 19.2$ Hz), −165.0 (t, $1F_p$, $J_{pm} = 19.5$ Hz), −166.1 (m, $4F_m$). Mass spectrum $(FAB)^-$: *m/z* 440 ($Pd(C_6F_5)_2$).

2.2.2. $[NBu_4][Pd(C_6F_5)_2\{PhNC(O)NEt_2\}]$ (2)

Yield: 65 mg, 76%. *Anal*. Found: C, 53.3; H, 5.7; N, 5.0. Calcd. for $C_{39}H_{51}F_{10}N_3OPd$: C, 53.6; H, 5.9; N, 4.8%. M.p.: 223 dec. $\Lambda_M = 125$ S cm^2 mol^{-1}. IR (Nujol, cm^{-1}): 1680 ν(C=O), 790, 780 (Pd–C_6F_5). ^1H NMR ($(CD_3)_2CO$): δ 7.18 (d, $2H_o$, $J_{om} = 7.4$ Hz), 6.91 (dd, $2H_m$, $J_{om} = J_{mp} = 7.4$ Hz), 6.68 (t, $1H_p$, $J_{mp} = 7.4$ Hz), 2.87 (m, 2H, NCH_2), 2.44 (m, 2H, NCH_2), 1.64 (t, 6H, $N(CH_2CH_3)_2$, $J_{HH} = 7.1$ Hz). ^{19}F NMR ($(CD_3)_2CO$): δ −113.6 (d, $2F_o$, $J_{om} = 29.3$ Hz), −113.9 (d, $2F_o$, $J_{om} = 28.8$ Hz), −164.9 (t, $1F_p$, $J_{pm} = 18.6$ Hz), −165.2 (t, $1F_p$, $J_{pm} = 19.8$ Hz), −166.2 (m, $4F_m$). Mass spectrum $(FAB)^-$: *m/z* 440 ($Pd(C_6F_5)_2$).

2.2.3. $[NBu_4][Pd(C_6F_5)_2\{PhNC(O)NPr^i_2\}]$ (3)

Yield: 67 mg, 75%. *Anal*. Found: C, 54.3; H, 6.2; N, 4.6. Calcd. for $C_{41}H_{55}F_{10}N_3OPd$: C, 54.6; H, 6.1; N, 4.7%. M.p.: 237 dec. $\Lambda_M = 112$ S cm^2 mol^{-1}. IR (Nujol, cm^{-1}): 1680 ν(C=O), 790, 780 (Pd–C_6F_5). ^1H NMR ($(CD_3)_2CO$): δ 7.10 (d, $2H_o$, $J_{om} = 7.4$ Hz), 6.88 (dd, $2H_m$, $J_{om} = J_{mp} = 7.4$ Hz), 6.65 (t, $1H_p$, $J_{mp} = 7.4$ Hz), 3.34 (m, 2H, NCH), 1.52 (d, 6H, $NCH(CH_3)_2$, $J_{HH} = 6.3$ Hz), 1.34 (d, 6H, $NCH(CH_3)_2$, $J_{HH} = 7.4$ Hz). ^{19}F NMR ($(CD_3)_2CO$): δ −113.7 (d, $2F_o$, $J_{om} = 31.3$ Hz), −114.2 (d, $2F_o$, $J_{om} = 30.5$ Hz), −165.4 (t, $1F_p$, $J_{pm} = 19.8$ Hz), −165.7 (t, $1F_p$, $J_{pm} = 19.8$ Hz), −166.7 (m, $4F_m$). Mass spectrum $(FAB)^-$: *m/z* 659 (M)

2.3. Preparation of $[NBu_4][Pt(C_6F_5)_2\{PhNC(O)NR_2\}]$ [R = Et (4), Pr^i (5)]

To a solution of $[NBu_4]_2[\{Pt(C_6F_5)_2(\mu\text{-}OH)\}_2]$ (100 mg, 0.063 mmol) in toluene (8 mL) was added the corresponding amine (0.252 mmol) and PhNCO (27.3 μL, 0.252 mmol). The solution was boiled under reflux for 7 h. The solvent was completely evaporated under reduced pressure and the residue was treated with Et_2O, then the solid formed was collected by filtration and air-dried.

2.3.1. $[NBu_4][Pt(C_6F_5)_2\{PhNC(O)NEt_2\}]$ (4)

Yield: 68 mg, 79%. *Anal*. Found: C, 48.6; H, 5.3; N, 4.3. Calcd. for $C_{39}H_{51}F_{10}N_3OPt$: C, 48.7; H, 5.3; N, 4.4%. M.p.: 217 dec. $\Lambda_M = 109$ S cm^2 mol^{-1}. IR (Nujol, cm^{-1}): 1690 ν(C=O), 805, 790 (Pt–C_6F_5). ^1H NMR ($(CD_3)_2CO$): δ 7.22 (d, $2H_o$, $J_{om} = 7.6$ Hz), 6.93 (dd, $2H_m$, $J_{om} = J_{mp} = 7.6$ Hz), 6.72 (t, $1H_p$, $J_{mp} = 7.2$ Hz), 3.10 (m, 2H, NCH_2), 2.62 (m, 2H, NCH_2), 1.58 (t, 6H, $N(CH_2CH_3)_2$, $J_{HH} = 7.1$ Hz). ^{19}F NMR ($(CD_3)_2CO$): δ −117.6 (d, $2F_o$, $J_{om} = 27.7$ Hz, J_{PtFo} 536.2 Hz), −118.1 (d, $2F_o$, $J_{om} = 27.7$ Hz, J_{PtFo} 468.5 Hz), −167.5 (t, $1F_p$, $J_{pm} = 19.8$ Hz), −166.2 (m, $1F_p + 4F_m$). Mass spectrum $(FAB)^-$: *m/z* 529 ($Pt(C_6F_5)_2$).

2.3.2. $[NBu_4][Pt(C_6F_5)_2\{PhNC(O)NPr^i_2\}]$ (5)

Yield: 79 mg, 86%. *Anal*. Found: C, 49.5; H, 5.5; N, 4.1. Calcd. for $C_{41}H_{55}F_{10}N_3OPt$: C, 49.7; H, 5.6; N, 4.2%. M.p.: 229 dec. $\Lambda_M = 121$ S cm^2 mol^{-1}. IR (Nujol, cm^{-1}): 1690 ν(C=O), 805, 795 (Pd–C_6F_5). ^{1}H NMR ($(CD_3)_2CO$): δ 7.16 (d, 2H$_o$, $J_{om} = 8.1$ Hz), 6.91 (dd, 2H$_m$, $J_{om} = J_{mp} = 8.1$ Hz), 6.69 (t, 1H$_p$, $J_{mp} = 8.1$ Hz), 3.54 (m, 2H, NCH), 1.52 (d, 6H, NCH(CH_3)$_2$, $J_{HH} = 6.4$ Hz), 1.39 (d, 6H, NCH(CH_3)$_2$, $J_{HH} = 6.3$ Hz). ^{19}F NMR ($(CD_3)_2CO$): δ −117.6 (d, 2F$_o$, $J_{om} = 29.1$ Hz, J_{PtFo} 554.3 Hz), −118.0 (d, 2F$_o$, $J_{om} = 29.1$ Hz, J_{PtFo} 516.4 Hz), −167.6 (t, 1F$_p$, $J_{pm} = 19.8$ Hz), −168.0 (m, 1F$_p$+4F$_m$). Mass spectrum (FAB)$^-$: *m*/*z* 748 (M).

2.4. Crystal structure determination of $[NBu_4][Pt(C_6F_5)_2\{PhNC(O)NPr^i_2\}]$ (5)

Suitable crystals for the X-ray diffraction study of compound **5** were grown from a dry CH_2Cl_2/hexane solution. X-ray diffraction experiment was carried out on an Enraf Nonius CAD4 diffractometer at r.t. The crystallographic data are shown in Table 1. Data were collected using a single crystal of dimensions 0.5 × 0.5 × 0.3 mm. Accurate cell parameters were determined by least-squares fitting of 25 reflections. The range of *hkl* was $0 \le h \le 11$, $0 \le k \le 25$, $-12 \le l \le 12$, corresponding to $2\theta_{max} = 50°$. The structure was solved by direct methods [20] and refined anisotropically on F^2 [21]. Hydrogen atoms were introduced in calculated positions. The final R factor was 0.031, $R_W = 0.069$ over 3472 observed reflections [$I > 2\sigma(I)$].

Table 1
Crystal data and summary of data collection and refinement for $[NBu_4][Pt(C_6F_5)_2\{PhNC(O)NPr^i_2\}]$ (**5**)

Empirical formula	$C_{41}H_{55}F_{10}N_3OPt$
Formula weight	990.97
Crystal system	monoclinic
Space group	$P2(1)$
Unit cell dimensions	
a (Å)	9.854(4)
b (Å)	21.094(6)
c (Å)	10.596(6)
β (°)	97.14(4)
V (Å^3)	2185(2)
Z	2
D_{calc} (Mg m^{-3})	1.51
F (mm^{-1})	3.29
λ (Å)	0.71073
Observed reflections	3472
Final R_1, wR_2 indices (all data)	0.041, 0.079
Goodness-of-fit	1.096

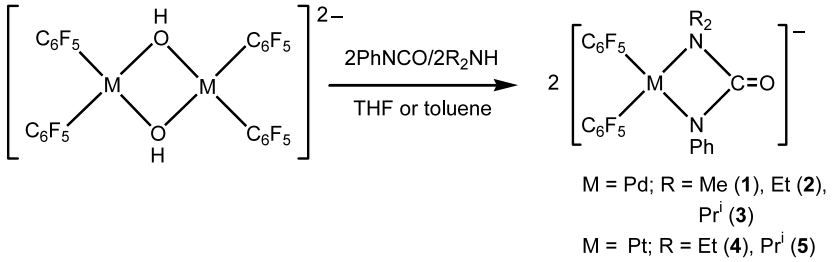
3. Results and discussion

The reaction of $[NBu_4]_2[\{M(C_6F_5)_2(\mu\text{-}OH)\}_2]$ (M = Pd, Pt) with amines R_2NH in the presence of PhNCO leads to the synthesis of the ureato(1−) complexes $[NBu_4][M(C_6F_5)_2\{PhNC(O)NR_2\}]$ **1**–**5** (M = Pd, Pt) shown in Scheme 1. The complexes are air-stable, both in the solid state and in solution. The new complexes have been characterized by partial elemental analyses and spectroscopic (IR and ^{1}H and ^{19}F NMR) methods. In acetone solution, these complexes behave as 1:1 electrolytes [22]. The presence of the ureato ligand is supported by the IR spectra, which show a band at approximately 1680 cm^{-1} assignable to ν(C=O) [11]. The IR spectra of complexes **1**–**5** show also the characteristic absorptions of the C_6F_5 group [23] at 1630, 1490, 1450, 1050, 950 cm^{-1} and a split band at approximately 800 cm^{-1}, derived from the so-called X-sensitive mode in C_6F_5 halogen molecules, which is characteristic of the *cis*-$M(C_6F_5)_2$ fragment [24,25] and behaves like a ν(M–C) band [26]. Mass spectra (FAB$^-$) of complexes **3** and **5** clearly support the proposed formulae showing the peaks corresponding to $[M(C_6F_5)_2\{PhNC(O)NR_2\}]^-$, with coincident experimental and calculated isotopic distributions.

The reaction of $[NBu_4]_2[\{M(C_6F_5)_2(\mu\text{-}OH)\}_2]$ (M = Pd, Pt) with amines R_2NH should give the amide complex $[NBu_4]_2[\{M(C_6F_5)_2(\mu\text{-}NR_2)\}_2]$ followed by insertion of PhNCO into the M–NR_2 bond. The synthesis of the related arylamide complexes $[NBu_4]_2[\{M(C_6F_5)_2(\mu\text{-}NHAr)\}_2]$ (M = Pd, Pt) and their reactions with CS_2 to yield the corresponding dithiocarbamate complexes $[M(C_6F_5)_2(S_2CNR_2)]^-$ have been previously reported [5]. External attack by NR_2^- of the coordinated heterocumulene is an alternative pathway to the formation of **1**–**5**.

It has also been reported the insertion of PhNCO into the Pt–NHPh bond of *trans*-$[PtH(NHPh)(PEt_3)_2]$ to give the monodentate *N*,*N*′-diphenylureato(1−)-*N* complex *trans*-$[PtH\{PhNC(O)NHPh\}(PEt_3)_2]$ [11] and the insertion of Bu^{t}NCO into Mo–N amido bonds to yield the molibdenum complex $[Mo\{Bu^tNC(O)NHBu^t\}_2(NBu^t)_2]$ [15], which has two chelating (*N*,*O*) ureato(1−) ligands. The X-ray diffraction study of complex **5** (vide infra) has revealed the presence of an *N*,*N*′-chelating ureato(1−) ligand.

The ^{19}F NMR spectra of complexes **1**–**5** are consistent with this structure, showing the presence of two nonequivalent freely rotating C_6F_5 groups, one trans to NR_2 and one trans to NPh, with two 2:2 resonances in the *o*- and *m*-fluorine region, and two 1:1 resonances in the *p*-fluorine region. As expected, the *ortho*-F signals of complexes **4** and **5** are flanked by the satellites due to the coupling to ^{195}Pt.



Scheme 1.

3.1. X-ray structure of $[NBu_4][Pt(C_6F_5)_2\{PhNC(O)NPr^i_2\}]$ (5)

Fig. 1 shows the X-ray structure of complex **5**, with selected bond lengths and angles listed in Table 2. The metal is coordinated in a distorted square-planar arrangement. The main distortion is the N(1)–Pt–N(2) bite angle, which is 64.0(3)°. This is due to the geometrical constraint set by the ligand. The angle between the two C_6F_5 rings is 87.8(4)°. The four-membered metallacycle N(2)–Pt–N(1)–C(19) is essentially planar (with no atom deviating from the least-square plane by more than 0.030(6) Å, for C(19)). The Pt–N(1) bond length (2.063(7) Å) is shorter than the Pt–N(2) bond length (2.158(8) Å). These values are similar to those reported in complexes containing the $\{Pt(C_6F_5)_2N_2\}$ moiety such as $[Pt(C_6F_5)_2(pz\cdots H\cdots pz)]$ (pz = pyrazolate) (2.091(5) Å) [24], $[Pt(C_6F_5)_2(cis\text{-chxn})]$ (chxn = cyclohexane-1,2-diamine) (2.125(5), 2.106(5) Å) [27], $[Pt(C_6F_5)_2(tmeda)]$ (tmeda = *N*,*N*,*N*′,*N*′-tetramethylethane-1,2-diamine) (2.141(5) Å) [28] and those observed in the ureato(2−) platinum complex [Pt{PhNC(O)NAd}(COD)] (Ad = 1-adamantyl, COD = 1,5-*cyclo*-octadiene) (2.048(8), 2.021(8) Å) [29].

The C(19)–O bond length (1.212(10) Å) is shorter than that found in the ureato(2−) palladium or platinum complexes [Pd{PhNC(O)NPh}(phen)] (phen = 1,10-phenantroline) (1.238(5) Å) [30], [Pt{PhNC(O)NAd}(COD)] (Ad = 1-adamantyl, COD = 1,5-*cyclo*-octadiene) (1.24(5) Å) [29] and $[Pd_2(a\text{-}cac)_2(\mu_4\text{-}NHCONH)]_2\cdot Et_2O$ (1.24(3) Å) [31].

The Pt–C_6F_5 distances (1.986(9) and 2.020(9) Å) are in the range found in the literature [1b,7].

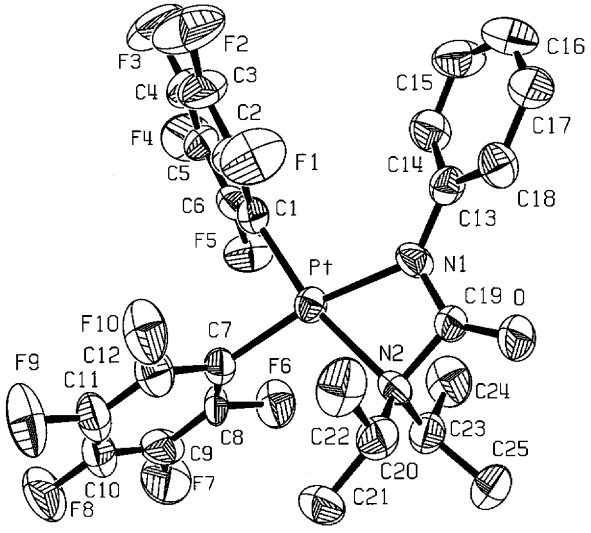


Fig. 1. An ORTEP drawing of the $[(C_6F_5)_2Pt\{PhNC(O)NPr^i_2\}]^-$ anion.

Table 2
Selected bond lengths [Å] and angles [°] for complex **5**

Bond lengths	
Pt–C(1)	1.986(9)
Pt–C(7)	2.020(9)
Pt–N(1)	2.063(7)
Pt–N(2)	2.158(8)
N(1)–C(19)	1.313(11)
N(2)–C(19)	1.505(11)
O–C(19)	1.212(10)
Bond angles	
C(1)–Pt–C(7)	87.8(4)
C(1)–Pt–N(1)	103.4(3)
C(7)–Pt–N(1)	168.6(3)
C(1)–Pt–N(2)	167.3(3)
C(7)–Pt–N(2)	104.9(3)
N(1)–Pt–N(2)	64.0(3)
O–C(19)–N(1)	134.1(9)
O–C(19)–N(2)	120.9(8)
N(1)–C(19)–N(2)	104.9(7)

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 195158 for compound **5**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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