Synthesis and reactivity of [Au(2-CH$_2$-6-RC$_5$H$_3$N)(PPh$_3$)] (R = H, Me). X-Ray structure of [Ag{Au(2-CH$_2$-6-MeC$_5$H$_3$N)(PPh$_3$)}$_2$][ClO$_4$]

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The reaction of Li(2-CH$_2$-6-RC$_5$H$_3$N) with [AuCl(PPh$_3$)] leads to the mononuclear complexes [Au(2-CH$_2$-6-RC$_5$H$_3$N)(PPh$_3$)] (R = H, Me). These react further with other copper, silver or gold compounds to give heteronuclear derivatives, [M{Au(2-CH$_2$-6-RC$_5$H$_3$N)(PPh$_3$)}$_2$][X] (M = Cu, X = PF$_6$ or M = Ag, X = ClO$_4$), or the dinuclear complex [Au(2-CH$_2$-6-MeC$_5$H$_3$N)]$_2$. The crystal structure of [Ag{Au(2-CH$_2$-6-MeC$_5$H$_3$N)(PPh$_3$)}$_2$][ClO$_4$] has been determined by X-ray diffraction studies and shows short interactions between the gold and silver centres.

One of the more interesting developments in gold chemistry[1] over the last few years has been in the area of di- and polynuclear systems. Potentially bidentate ligands, with the correct geometry to hold two metal centres close to each other, have been widely investigated. However, anionic ligands obtained from 2-methylpyridine or 2,6-dimethylpyridine (2-CH$_2$-6-RC$_5$H$_3$N, R = H, Me) have been studied far less, and no gold derivative has been described. The only related derivative is the dinuclear [Au{2-C(SiMe$_3$)$_2$C$_5$H$_4$N}]$_2$.[2]

This paper reports the preparation of [Au(2-CH$_2$-6-RC$_5$H$_3$N)(PPh$_3$)] (R = H, Me) and the homo- and hetero-nuclear derivatives obtained from the reactions with other copper, silver or gold compounds. The molecular structure of [Ag{Au(2-CH$_2$-6-RC$_5$H$_3$N)(PPh$_3$)}$_2$][ClO$_4$] has been established by single-crystal X-ray studies, showing short interactions between the gold and silver centres.

Results and discussion

The reaction of 2-CH$_3$-6-RC$_5$H$_3$N (R = H, Me) in diethyl ether with LiBu^n leads to the lithium derivatives Li(2-CH$_2$-6-RC$_5$H$_3$N),[3–9] which react further with [AuCl(PPh$_3$)] to give the mononuclear complexes [Au(2-CH$_2$-6-RC$_5$H$_3$N)(PPh$_3$)] [R = H (1), Me (2)]. A similar reaction starting from tetramethylpyrazine (2,3,5,6-Me$_4$C$_4$N$_2$) gives the derivative [Au(2-CH$_2$-3,5,6-Me$_3$C$_4$N$_2$)(PPh$_3$)] 3. Complexes 1–3 are white air- and moisture-stable solids and are non-conducting in acetone solutions. Their IR spectra show the v(C=N) and v(C=C) vibrations of the heterocycle at 1587s and 1568s 1, 1589s and 1557s 2, and 1535s cm^{-1} 3. The positive liquid secondary ion mass spectra (LSIMS) show the cation molecular peaks at m/z (%): 551 (26) 1, 565 (22) 2, or 594 (22) 3. Other peaks appear at m/z 459 ([AuPPh$_3$]$^+$, 95 1, 70 2, or 100% 3) and 721 ([Au(PPh$_3$)$_2$]$^+$, 98 1, 70 2, or 90% 3). The most intense peaks correspond to the cation [M + AuPPh$_3$]$^+$ (m/z 1010 1, 1024 2, or 1053 3).

The ^{31}P-{^{1}H} NMR spectra at room temperature show a singlet at ca. δ 42.9. The ^{1}H NMR spectra of complexes 1 and 2 are as expected and are summarised in Table 1. The methylene protons appear as doublets because of coupling with the phosphorus atom, confirming the coordination of the gold centre to the CH$_2$ group. Some resonances (H^4 in 2, or H^5 in 1) are apparent triplets, rather than doublets of doublets, which is

probably attributable to similar J(HH) constants. The methylenic protons of 3 are not equivalent and appear as singlets at δ 2.39, 2.44, and 2.59.

As suggested by the formation of [M + AuPPh$_3$]$^+$ species in the mass spectra, complexes 1–3 can serve as building blocks for preparing heteronuclear complexes. We have studied their reactions with several silver or copper derivatives. Thus the treatment of complexes 1, 2 or 3 with AgClO$_4$ in molar ratio 2:1 gives the trinuclear derivatives [Ag{Au(2-CH$_2$-6-RC$_5$H$_3$N)(PPh$_3$)}$_2$][ClO$_4$] (R = H 4, Me 5) or [Ag{Au(2-CH$_2$-3,5,6-Me$_3$C$_4$N$_2$)(PPh$_3$)}$_2$][ClO$_4$] 6. A similar reaction of complexes 1 or 2 with [Cu(NCMe)$_4$][PF$_6$] leads to the complexes [Cu{Au(2-CH$_2$-6-RC$_5$H$_3$N)(PPh$_3$)}$_2$][PF$_6$] (R = H 7, Me 8) (Scheme 1). They are white or pale brown air- and moisture-stable solids. Complexes 4, 7 and 8 behave as 1:1 electrolytes in acetone solution; however the silver complexes 5 and 6 show conductivities (188 and 228 Ω^{-1} cm^2 mol^{-1}, respectively) higher than that of 1:1 electrolytes, probably because of interactions with the solvent forming new ions, such as [Ag(OCMe$_2$)$_4$]$^+$. Their IR spectra show, apart from the bands arising from the heterocycle or the phosphine, those of the perchlorate at 1100vs (br) and 625m cm^{-1} or hexafluorophosphate at 850s (br) and 558s cm^{-1}. The positive-ion LSIMS exhibit peaks corresponding to the cations [AuPPh$_3$]$^+$ (m/z = 459), [Au(PPh$_3$)$_2$]$^+$ (m/z = 721), [(2-CH$_2$-6-RC$_5$H$_3$N)(AuPPh$_3$)]$^+$ [m/z = 552 (4 or 7) or 566 (5 or 8)], [(2-CH$_2$-3,5,6-Me$_3$C$_4$N$_2$)(AuPPh$_3$)]$^+$ (m/z = 595, 6), [(2-CH$_2$-6-RC$_5$H$_3$N)(AuPPh$_3$)$_2$]$^+$ [m/z = 1010 (4 or 7) or 1024 (5 or 8)], or [(2-CH$_2$-3,5,6-Me$_3$C$_4$N$_2$)(AuPPh$_3$)$_2$]$^+$ (m/z = 1053, 6). The cation molecular peaks, [Ag{Au(2-CH$_2$C$_5$H$_4$N)(PPh$_3$)}$_2$]$^+$ (m/z = 1211, 30%) or [Ag{Au(2-CH$_2$-3,5,6-Me$_3$C$_4$N$_2$)(PPh$_3$)}$_2$]$^+$ (m/z = 1297, 30%) are only observed for complexes 4 and 6.

The ^{1}H NMR spectra show a doublet for the methylene protons, because of coupling with the phosphorus of the phosphine. Other resonances are collected in Table 1. Complex 6 presents a singlet for each of the three different methyl groups. The ^{31}P-{^{1}H} NMR spectra present only one singlet for the two equivalent phosphorus atoms.

The crystal structure of complex 5 has been confirmed by an X-ray diffraction study; its cation is shown in Fig. 1, with a selection of bond lengths and angles in Table 2. The silver centre lies on a two-fold axis and thus only half of the molecule corresponds to the asymmetric unit. The gold atoms have a

Table 1 ¹H NMR data for complexes.[a] Coupling constants in parentheses

Complex	CH₃	CH₂	H⁵	H³	H⁴	H⁶
2-MeC₅H₄N	2.50 (s)		7.01 (t)	7.07 (d)	7.33 (dt)	8.42 (d)
2,6-Me₂C₅H₃N	2.42 (s)		6.83 (d)	6.83 (d)	7.33 (t)	
1		2.88 (d) (10.8)	6.67 (t) (3.1)	7.02 (d) (7.8)	b	8.28 (d)
2	2.44 (s)	2.79 (d) (11.0)	6.58 (d) (7.3)	6.85 (d) (7.8)	7.25 (t)	
3	2.39 (s) 2.44 (s) 2.59 (s)	2.68 (d) (11.2)				
4		3.13 (d) (10.5)	6.54 (m)	7.02 (m)	b	8.05 (m)
5	2.20 (s)	3.24 (d) (10.5)	6.72 (d) (7.3)	6.98 (d) (7.8)	b	
6	2.41 (s) 2.56 (s) 2.59 (s)	2.88 (d) (9.5)				
7		3.10 (d) (9.5)	6.54 (m)	7.02 (m)	b	8.05 (m)
8	2.06 (s)	3.28 (d) (10.2)	6.60 (d) (7.3)	6.98 (d) (7.3)	b	
9		3.22 (d) (10.7)	b	b	b	8.34 (m)
10	2.80 (s)	3.28 (d) (10.6)	6.74 (d) (6.0)	6.99 (d) (8.1)	b	
11a	3.318 (s)	3.44 (s)	6.48 (d) (7.4)	7.0 (d) (7.4)	7.27 (t)	
11b	2.67 (s)	3.17 (s)	6.70 (d) (7.4)	7.03 (d) (7.4)	7.32 (t)	

[a] In CDCl₃ solutions at −60 °C, δ from external SiMe₄; s = singlet, d = doublet, t = triplet, dt = doublet of triplets, m = multiplet. [b] Overlapped with the resonances of the phenyl groups.
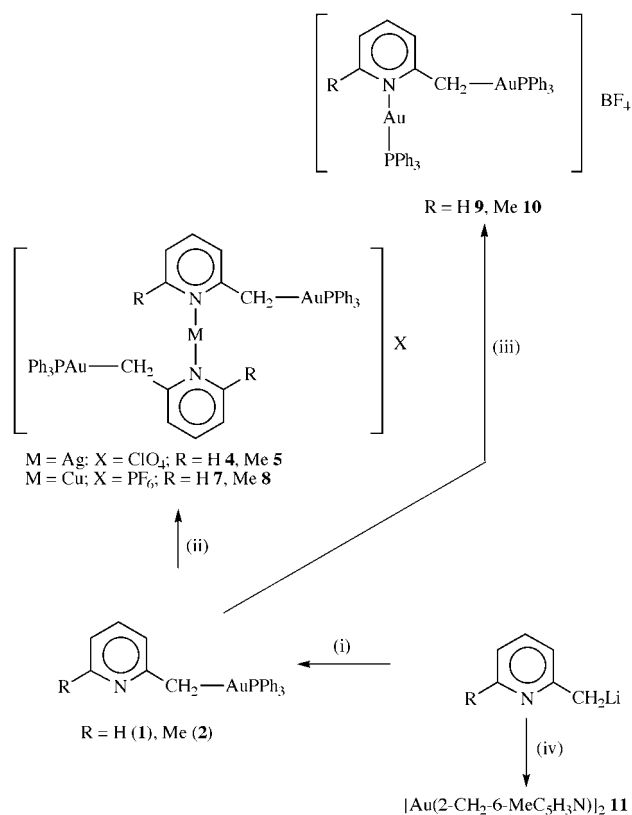

Scheme 1 (i) [AuCl(PPh₃)]; (ii) 1/2 AgClO₄ or 1/2 [Cu(NCMe)₄][PF₆]; (iii) HBF₄; (iv) [AuCl(AsPh₃)].

Table 2 Selected bond lengths (Å) and angles (°) for complex **5**

Au–C(1)	2.084(6)	P–C(31)	1.819(6)
Au–P	2.2813(16)	C(1)–C(11)	1.452(9)
Ag–N	2.162(5)	C(11)–N	1.347(7)
P–C(21)	1.815(6)	C(15)–N	1.346(8)
P–C(41)	1.818(6)		
C(1)–Au–P	175.32(17)	C(41)–P–Au	116.26(18)
N#1–Ag–N	159.6(2)	C(31)–P–Au	113.39(18)
C(21)–P–C(41)	105.7(2)	C(11)–C(1)–Au	112.3(4)
C(21)–P–C(31)	104.5(2)	C(15)–N–Ag	121.0(4)
C(41)–P–C(31)	104.6(2)	C(11)–N–Ag	116.2(4)
C(21)–P–Au	111.43(18)		

[a] Symmetry transformations used to generate equivalent atoms: #1 −x + 1, y, −z + 1/2.
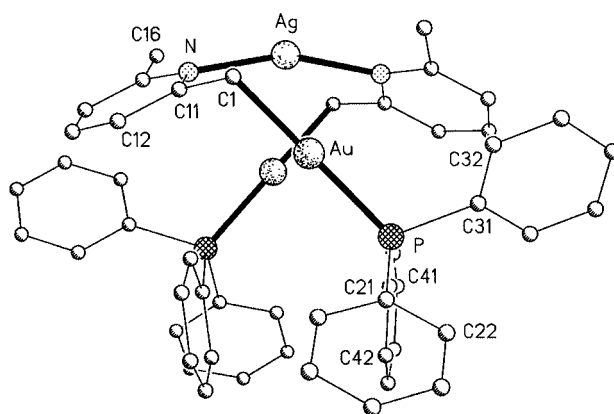
Fig. 1 Molecular structure of the cation of complex **5** showing the atom numbering scheme. Hydrogen atoms are omitted for clarity.

slightly distorted linear geometry with a P–Au–C(1) angle of 175.3(2)°, possibly associated with a weak interaction with the silver centre, Au···Ag 3.428(1) Å; in contrast, the gold atoms are located far from each other, Au···Au# 5.115(2) Å

(#: 1 − x, y, 0.5 − z). The silver centre has a very distorted linear geometry, N–Ag–N# 159.6(2)°, probably attributable to weak contacts to oxygen atoms of the (disordered) perchlorate
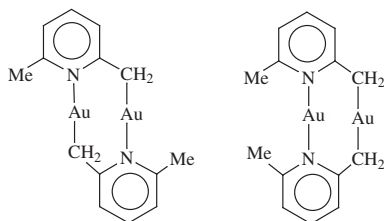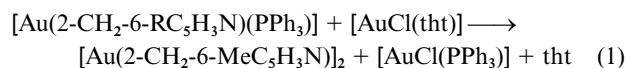
Fig. 2 The two isomers of complex **11**.

anion, Ag···O 3.10, 3.16 Å. The silver–nitrogen bond length of 2.162(5) Å is similar to values reported for two-coordinate and pseudo two-coordinate complexes of silver(I) with σ-bonding ligands such as [Ag(2,6-Me$_2$py)$_2$][ClO$_4$] (2.166(4) Å),[10] although shorter than in the complex [(Ph$_3$P)(O$_3$ClO)AgN-(Ph$_2$PAuPPh$_2$)$_2$NAg(OClO$_3$)(PPh$_3$)] (2.239(4) Å),[11] perhaps as a consequence of the *trans* influence of the phosphine ligand.

The reaction of complexes **1** or **2** with HBF$_4$ in a 2:1 molar ratio leads to protonation of half of the heterocyclic ligand and to the formation of the dinuclear complexes [(2-CH$_2$-6-RC$_5$H$_3$N)(AuPPh$_3$)$_2$][BF$_4$] (R = H **9**, Me **10**). They are white air- and moisture-stable solids and behave as 1:1 electrolytes in acetone solution. In the IR spectra the vibrations from the anion appear at 1100s and 1053s (br) cm^{-1}. The ^{31}P-{^{1}H} NMR spectra present two singlets at δ 42.7 and 30.3 **9** and δ 41.8 and 29.4 **10** from the two different phosphines and confirm the coordination of the second gold centre to the nitrogen atom. The ^{1}H NMR data are collected in Table 1. The mass spectra are similar to those of complexes **1** and **2** and the cations [M − BF$_4$]$^+$ appear at *m/z* (%) 1010 (55) **9** or 1024 (30) **10**.

The reaction of the lithium derivative Li(2-CH$_3$-6-MeC$_5$H$_3$N) with [AuCl(AsPh$_3$)], with the weakly coordinating ligand AsPh$_3$, leads to the dinuclear complex [Au(2-CH$_2$-6-MeC$_5$H$_3$N)]$_2$ **11**. It is a white air- and moisture-stable solid and a non-conductor in acetone solution. The ν(Au–Cl) vibrations and those of the arsine are not present in its IR spectrum and only those of the 2-CH$_2$-6-MeC$_5$H$_3$N ligand are observed. The ^{1}H NMR spectrum (Table 1) is in agreement with the presence of two isomers (Fig. 2), in a molar ratio **11a**:**11b** = 4:1.

The same complex is obtained from the reaction of complex **2** with [AuCl(tht)] (tht = tetrahydrothiophene) (eqn. (1)), but now with a different molar ratio for the two isomers (**11a**:**11b** = 1:4), as observed from the ^{1}H NMR spectrum. Complex **11** reacts with PPh$_3$ to give the mononuclear derivative **2** (84% yield).

$$[\text{Au}(2\text{-CH}_2\text{-6-RC}_5\text{H}_3\text{N})(\text{PPh}_3)] + [\text{AuCl(tht)}] \longrightarrow$$
$$[\text{Au}(2\text{-CH}_2\text{-6-MeC}_5\text{H}_3\text{N})]_2 + [\text{AuCl(PPh}_3)] + \text{tht} \quad (1)$$

Experimental

Infrared spectra were recorded on a Perkin-Elmer 883 spectrophotometer, over the range 4000–200 cm^{-1}, using Nujol mulls between polyethylene sheets. Conductivities were measured in *ca*. 5×10^{-4} mol dm^{-3} solutions with a Philips 9509 conductimeter. C, H, and N analyses were carried out with a Perkin-Elmer 2400 microanalyser. Mass spectra were recorded on a VG Autospec, with the liquid secondary-ion mass spectra (LSIMS) technique, using 3-nitrobenzyl alcohol as matrix. ^{1}H and ^{31}P-{^{1}H} NMR spectra were recorded on a Varian UNITY 300 or Bruker ARX 300 apparatus in CDCl$_3$ solutions (stored over Na$_2$CO$_3$); chemical shifts are quoted relative to SiMe$_4$ (external, ^{1}H) and 85% H$_3$PO$_4$ (external, ^{31}P). The starting materials [AuCl(tht)],[12] [AuCl(PPh$_3$)],[12] [AuCl(AsPh$_3$)],[13] [Au(C$_6$F$_5$)(tht)],[12] and [Cu(NCMe)$_4$][PF$_6$][14] were prepared by published procedures. All other chemicals used were commercially available and used without further purification. **CAUTION**: perchlorate salts with organic cations may be explosive.

Syntheses

[Au(2-CH$_2$-6-RC$_5$H$_3$N)(PPh$_3$)] (R = H, 1; Me, 2) and [Au(2-CH$_2$-3,5,6-Me$_3$C$_4$N$_2$)(PPh$_3$)] 3. To a solution of 2-CH$_3$-6-RC$_5$H$_3$N (R = H, 60 µl, 0.5 mmol; Me, 50 µl, 0.5 mmol) or 2,3,5,6-Me$_4$C$_4$N$_2$ (0.068 g, 0.5 mmol) in diethyl ether (40 cm^3) was added a *n*-hexane solution of LiBu^n (0.31 ml, 1.6 M, 0.5 mmol) and the mixture was stirred for 10 min. An orange, Li(2-CH$_2$-6-RC$_5$H$_3$N), or red Li(2-CH$_2$-3,5,6-Me$_3$C$_4$N$_2$), precipitate was formed. To this suspension was added [AuCl(PPh$_3$)] (0.247 g, 0.5 mmol). After stirring for 1 h the mixture was filtered through a layer of celite and the solvent was evaporated to *ca*. 5 cm^3. Addition of hexane (10 cm^3) gave complexes **1–3** as white solids. Complex **1**: yield 59%. Λ_M 1 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 51.85; H, 3.8; N, 2.3. Calc. for C$_{24}$H$_{21}$AuNP: C, 52.27; H, 3.8; N, 2.5. ^{31}P-{^{1}H} NMR, δ: 42.9 (s). Complex **2**: yield 67%. Λ_M 1 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 53.0; H, 4.15; N, 2.4. Calc. for C$_{25}$H$_{23}$AuNP: C, 53.1; H, 4.15; N, 2.45. ^{31}P-{^{1}H} NMR, δ: 43.0 (s). Complex **3**: yield 77%. Λ_M 8 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 52.15; H, 4.7; N, 4.25. Calc. for C$_{26}$H$_{26}$AuN$_2$P: C, 52.55; H, 4.4; N, 4.7. ^{31}P-{^{1}H} NMR, δ: 42.8 (s).

[M{Au(2-CH$_2$-6-RC$_5$H$_3$N)(PPh$_3$)}$_2$][X] (M = Ag, X = ClO$_4$, R = H, 4; Me, 5; M = Cu, X = PF$_6$, R = H, 7; Me, 8) or [Ag{Au(2-CH$_2$-3,5,6-Me$_3$C$_4$N$_2$)(PPh$_3$)}$_2$][ClO$_4$] 6. To a solution of AgClO$_4$ (0.031 g, 0.15 mmol) or [Cu(NCMe)$_4$][PF$_6$] (0.028 g, 0.15 mmol) in diethyl ether (**4**, **5**) or dichloromethane (**6–8**) (30 cm^3) was added complex **1** (0.165 g, 0.3 mmol), **2** (0.169 g, 0.3 mmol) or **3** (0.178 g, 0.3 mmol). The reaction mixture was stirred in the dark for 30 min. Evaporation of the diethyl ether to *ca*. 5 cm^3 or concentration of the solution (dichloromethane) to *ca*. 5 cm^3 and addition of diethyl ether (10 cm^3) gave complexes **4–8** as white (**4–6**) or pale brown (**7, 8**) solids. Complex **4**: yield 82%. Λ_M 152 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 45.15; H, 3.15; N, 2.0. Calc. for C$_{48}$H$_{42}$AgAu$_2$ClN$_2$O$_4$P$_2$: C, 44.9; H, 3.05; N, 2.15. ^{31}P-{^{1}H} NMR, δ: 42.9 (s). Complex **5**: yield 86%. Λ_M 188 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 45.2; H, 3.4; N, 1.95. Calc. for C$_{50}$H$_{46}$AgAu$_2$ClN$_2$O$_4$P$_2$: C, 44.75; H, 3.5; N, 2.1. ^{31}P-{^{1}H} NMR, δ: 43.8 (s). Complex **6**: yield 63%. Λ_M 227 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 52.15; H, 4.7; N, 4.25. Calc. for C$_{46}$H$_{40}$AgAu$_2$ClN$_4$O$_4$P$_2$: C, 52.55; H, 4.4; N, 4.7. ^{31}P-{^{1}H} NMR, δ: 43.5 (s). Complex **7**: yield 61%. Λ_M 147 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 43.55; H, 2.85; N, 1.85. Calc. for C$_{48}$H$_{42}$Au$_2$CuF$_6$N$_2$P$_3$: C, 43.95; H, 3.05; N, 2.15. ^{31}P-{^{1}H} NMR, δ: 42.7 (s). Complex **8**: yield 72%. Λ_M 164 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 44.3; H, 3.3; N, 3.25. Calc. for C$_{50}$H$_{46}$Au$_2$CuF$_6$N$_2$P$_3$: C, 44.8; H, 3.4; N, 2.1. ^{31}P-{^{1}H} NMR, δ: 42.4 (s).

[(2-CH$_2$-6-RC$_5$H$_3$N)(AuPPh$_3$)$_2$][BF$_4$] (R = H, 9; Me, 10). To a solution of complex **1** (0.110 g, 0.2 mmol) or **2** (0.112 g, 0.2 mmol) in 40 cm^3 of diethyl ether was added HBF$_4$ (54% diethyl ether solution, 13.6 µl, 0.1 mmol) and the mixture stirred for 15 min. A white precipitate of **9** or **10** was obtained. Complex **9**: yield 73%. Λ_M 103 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 45.6; H, 2.95; N, 1.35. Calc. for C$_{42}$H$_{36}$Au$_2$BF$_4$NP$_2$: C, 45.95; H, 3.3; N, 1.25. ^{31}P-{^{1}H} NMR, δ: 42.7 (s, C-Au-PPh$_3$), 30.3 (s, N-Au-PPh$_3$). Complex **10**: yield 78%. Λ_M 120 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 46.0; H, 3.3; N, 1.6. Calc. for C$_{43}$H$_{38}$Au$_2$BF$_4$NP$_2$: C, 46.45; H, 3.45; N, 1.25. ^{31}P-{^{1}H} NMR, δ: 41.8 (s, C-Au-PPh$_3$), 29.4 (s, N-Au-PPh$_3$).

[Au(2-CH$_2$-6-MeC$_5$H$_3$N)]$_2$ 11. To a suspension of Li(2-CH$_2$-6-MeC$_5$H$_3$N) (0.5 mmol), prepared as above (complex **2**), in 40 cm^3 of diethyl ether was added [AuCl(AsPh$_3$)] (0.269 g, 0.5 mmol). After stirring for 1 h the mixture was filtered through a layer of Celite and the solvent was evaporated to *ca*. 5

Table 3 Details of data collection and structure refinement for complex **5**

Formula	$C_{50}H_{46}AgAu_2ClN_2O_4P_2$
M	1388.08
Crystal system	Monoclinic
Space group	$C2/c$
a/Å	14.593(5)
b/Å	17.237(6)
c/Å	19.330(7)
β/°	108.11(3)
U/Å^3	4621(3)
Z	4
T/°C	−95
μ(Mo-Kα)/mm^{-1}	6.927
No. of reflections measured	4382
No. of unique reflections	4052
R_{int}	0.018
R^a (F, $F > 4\sigma(F)$)	0.031
wR^b (F^2, all reflections)	0.081

^{a} $R(F) = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. ^{b} $wR(F^2) = [\Sigma\{w(F_o^2 - F_c^2)^2\}/\Sigma\{w(F_o^2)^2\}]^{0.5}$; $w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$, where $P = [F_o^2 + 2F_c^2]/3$ and a and b are constants adjusted by the program.

cm^3. Addition of hexane (10 cm^3) gave complex **11** as a white solid. Yield 42%. Λ_M 12 Ω^{-1} cm^2 mol^{-1}. Elemental analysis (%), Found: C, 28.0; H, 2.85; N, 4.2. Calc. for $C_{14}H_{16}Au_2N_2$: C, 27.75; H, 2.65; N, 4.6.

Crystallography

The crystal was mounted in inert oil on a glass fibre and transferred to the cold gas stream of a Siemens R3 diffractometer equipped with an LT-2 low temperature attachment. Data were collected using monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Scan type ω. Cell constants were refined from setting angles of 50 reflections in the range 2θ 20–22°. An absorption correction was applied on the basis of ψ-scans. The structure was solved by the heavy-atom method and refined on F^2 using the program SHELXL-97.[15] The perchlorate anion was refined with a two-fold disorder model. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included using a riding model. A system of restraints to light-atom displacement factor components and to local ring symmetry was used. Further details are given in Table 3.

CCDC reference number 186/1548.

See http://www.rsc.org/suppdata/dt/1999/2819/ for crystallographic files in .cif format.

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