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Synthesis of some μ-hydroxo-, phenoxo- and O,O-acetylacetonato-arylgold(III) complexes. Crystal structure of $[Au(C_6H_4NO_2-2)_2(\mu\text{-}OH)]_2 \cdot 2Et_2O$

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Abstract

A new synthesis of *cis*-$Me_4N[Au(C_6H_4NO_2-2)_2Cl_2]$ (**1**) is reported, involving the reaction of $[Hg(C_6H_4NO_2-2)Cl]$ with $Me_4N[AuCl_4]$ in the presence of Me_4NCl. Reaction of **1** with NaOPh or with Tlacac (acac = acetylacetonate) in the presence of $NaClO_4$ yields $Na[Au(C_6H_4NO_2-2)_2(OPh)_2]$ (**2**) or *cis*-$[Au(C_6H_4NO_2-2)_2(O,O\text{-}acac)]$ (**3**), respectively. Complex **3** reacts with PPh_3 to give *trans*-$[Au(C_6H_4NO_2-2)_2(C\text{-}acac)(PPh_3)]$ (**4**). Treatment of **2** in acetone with NaOH gives $[Au(C_6H_4NO_2-2)_2(\mu\text{-}OH)]_2$ (**5**), which is also formed when a solution of **2** in $CHCl_3$/hexane is exposed to water. The crystal structure of $\mathbf{5} \cdot 2Et_2O$ shows that it is a centrosymmetric dimer with two hydroxo groups bridging two $Au(C_6H_4NO_2-2)$ moieties. The features of the structure include short Au–C bond distances (1.992(5) Å, 1.995(5) Å) and hydrogen bonding between the bridging OH groups and diethyl ether molecules.

Keywords: Gold; Crystal structure; Acetylacetonato μ-hydroxo complexes

1. Introduction

Few organogold(III) complexes with oxoanionic ligands are known and many of them are unstable or moisture sensitive [1]. Thus, $[AuMe_2(OH)]_4$ detonates when dry just by touching [2], and the complexes $[AuMe_2(X)L]$, where $X = ClO_4$, CF_3SO_3, *p*-$MeC_6H_4SO_3$ or NO_3 and $L = PPh_3$, decompose at room temperature to give ethane and [Au(X)L] [3]. $[AuMe_2(OSiMe_3)]_2$ is thermally stable but sensitive to moisture [4]. As far as we are aware, only three O,O-acetylacetonato- complexes are known [5], and no HO- or RO-arylgold(III) complexes. This paper reports the synthesis of new O,O-acetylacetonato- and the first HO- and RO-arylgold(III) complexes, advantage being taken of the high stability of the C–Au bonds in *ortho*-nitrophenyl gold complexes [6].

2. Results and discussion

The synthesis of *cis*-$Me_4N[Au(C_6H_4NO_2-2)_2Cl_2]$ (**1**) has been achieved by transmetallation (1:1, refluxing 18 h in acetone) [7]:

$$2[Hg(C_6H_4NO_2-2)_2] + [AuCl_4]^- \rightarrow [Au(C_6H_4NO_2-2)_2Cl_2]^- + 2[Hg(C_6H_4NO_2-2)Cl]$$

However, because Me_4NCl symmetrizes the by-product $[Hg(C_6H_4NO_2-2)Cl]$,

$$2[Hg(C_6H_4NO_2-2)Cl] + Cl^- \rightarrow [Hg(C_6H_4NO_2-2)_2] + [HgCl_3]^-$$

it is possible to improve the yield of **1**, with respect to the nitrophenyl group, by carrying out the reaction of $Me_4N[AuCl_4]$ with $[Hg(C_6H_4NO_2-2)Cl]$ in the presence of Me_4NCl (1:2:2, EtOH, refluxing 5 h, see Scheme

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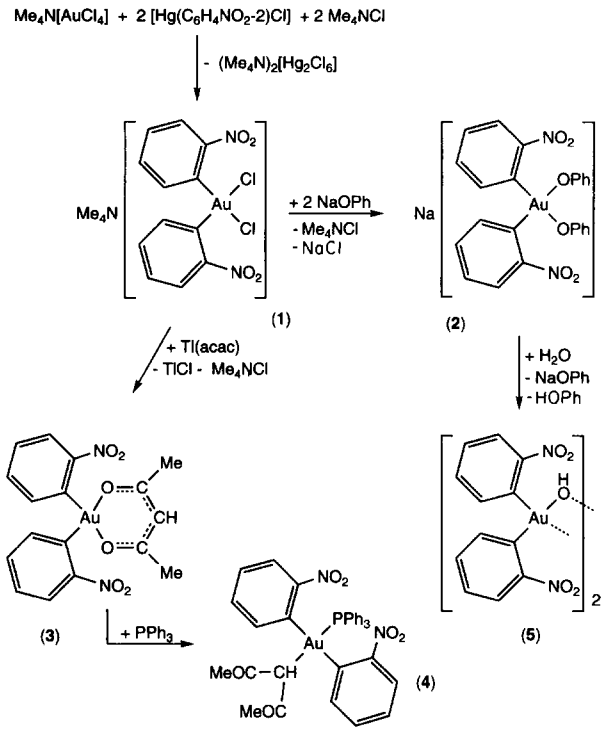

Scheme 1.

1). We have previously used a similar method to prepare $[Au(C_6H_4NO_2-2)Cl]^-$ [8].

Reactions of **1** with NaOR (R = Me, Et) gave metallic gold even at low temperature and under an inert atmosphere. However, **1** reacts with NaOPh (1:3, CH_2Cl_2, room temperature) to give $Na[Au(C_6H_4NO_2-2)_2(OPh)_2]$ (**2**). As far as we are aware, **2** is the first arylgold(III) species containing an RO^- ligand. Some aryl- or alkyl-oxodimethylgold(III) complexes ($AuMe_2(OR)PPh_3$) (R = Ph, tolyl, CH_2CF_3, $CH(CF_3)_2$) have recently been reported [9]. The complex $[AuMe_2(OH)]_4$ dissolves in aqueous NaOH to give a solution which is believed to contain $Na[AuMe_2(OH)_2]$ [10].

Tlacac (acac = acetylacetonate) reacts with **1**, in the presence of $NaClO_4$, (1:1, acetone) to give *cis*-$[Au(C_6H_4NO_2-2)_2(O,O\text{-acac})]$ (**3**). If the reaction is carried out in the absence of $NaClO_4$, a mixture of **1** and **3** is obtained, probably because formation of the partially soluble Me_4NCl leads to establishment of the equilibrium $\mathbf{3} + 2Cl^- \leftrightarrow \mathbf{1} + acac^-$. When $NaClO_4$ is present formation of the insoluble NaCl and Me_4NClO_4 displaces the equilibrium towards **3**. As far as we are aware, only two other (O,O-acac)diarylgold(III) complexes (*cis*-$[Au(R)_2(O,O\text{-acac})$) (R = 2,2'-biphenyl [5a], C_6F_5 [5b]) and one cationic (O,O-acac)monoarylgold(III) complex ($[Au(C,N'\text{-}C_6H_4N{=}NPh-2)(O,O\text{-acac})]ClO_4$ [5c]) have been described. The complex $[AuMe_2(O,O\text{-acac})]$ was reported long ago [11]. Addition of PPh_3 to **3** (1:1, CH_2Cl_2, room temperature) gives *trans*-$[Au(C_6H_4NO_2-2)_2(C\text{-acac})(PPh_3)]$ (**4**) in which the acac ligand is C-bonded rather than O,O-bonded and the cis geometry has changed to trans (as indicated by ^1H NMR spectroscopy, as described below). The reaction of $[AuMe_2(O,O\text{-acac})]$ with tertiary phosphines results in formation of an equilibrium with *cis*-$[AuMe_2(C\text{-acac})(PR_3)]$. Only in the case of PMe_2Ph could the C-acac complex be isolated [12]. Activation of C–H bonds in ketones (MeCOR) takes place with some (C-acac)(2-phenylazophenyl)gold(III) derivatives to give ketonyl complexes [5c]. The fact that **3** and **4** are stable in acetone is in accordance with the proposed pathway for this C–H activation process [5c].

Attempts were made to grow single crystals of **2** by the liquid diffusion method using CH_2Cl_2/n-hexane. The only suitable crystals obtained for X-ray diffraction turned out to be of $[Au(C_6H_4NO_2-2)_2(\mu\text{-}OH)]_2 \cdot 2Et_2O$ (**5**), presumably as a result of reaction with traces of water. This hydroxo-bridged complex was also made, although in low yield because of partial reduction to metallic gold, by reaction of **1** with NaOH. The homologous methylgold(III) derivative is a tetramer $[AuMe_2(\mu\text{-}OH)]_4$ [2] whereas 1-hydroxo-2,3,4,5-tetraphenylauracyclopentadiene is, like complex **5**, a dimer [13]. When dilute aqueous HCl was added to **5**, reaction took place with decomposition and no pure chloro complex was isolated.

Reactions between complex **2** and various protic acids did not lead to new complexes. Thus, **2** was stirred in acetone in an attempt to obtain new acetonylgold(III) complexes by C–H activation [5c], but no reaction was observed. Although some reaction took place with phenylacetylene in solution, as indicated by a change in colour from colourless to yellow, the starting complex was recovered unchanged after partial removal of the solvent and addition of Et_2O/n-hexane. Equilibrium reactions have previously been described between phenoxogold(III) complexes, *cis*-$[AuMe_2(OPh)(PPh_3)]$, and RH = methylcyanoacetate, malononitrile or phenylacetylene [9]. However, while the first gave a mixture of the starting complex and *cis*-$[AuMe_2(R)(PPh_3)]$, the latter two gave the corresponding R–Au(III) complexes. Complexes **3** and **4** were also recovered unchanged after treatment with phenylacetylene. This is in contrast with the observed behaviour of acetylacetonatogold(I) complexes towards alkynes [14].

2.1. Spectroscopic properties and structure of the complexes.

2.1.1. IR spectra

The phenoxo derivative **2** shows strong bands in the region 1250–1300 cm^{-1} assignable to ν(C–O) [9], and strong bands at 690 and 760 cm^{-1}, typical of monosub-

stituted phenyl groups (PhO). This last set of bands is not observed for the hydroxo species **5**. The acac complex **3** shows ν(CO) at 1550 and 1570 cm^{-1}, while the phosphine adduct **4** shows broad bands at 1650 and 1675 cm^{-1} as expected for chelating O,O-acac and terminal C-acac groups, respectively [5c]. All the complexes show bands at 1330–1340 cm^{-1} and ca. 1510 cm^{-1}, which have been shown to correspond to $\nu_{sym}(NO_2)$ and to $\nu_{asym}(NO_2)$, respectively, when the nitrophenyl group is not coordinated to the metal centre [15].

2.1.2. NMR spectra

The ^1H NMR spectrum of the acac complex **3** shows a singlet resonance at 2.0 ppm for methyl groups and at 5.5 ppm for the methynic proton. This resonance appears in **4** as a doublet at 4.2 ppm ($^3J_{HP}$ = 12 Hz). ^{13}C NMR of **4** shows only one resonance for the C attached to the nitro group (151.8 ppm) and 12 resonances in the range 122–135 ppm. The methyne carbon gives a doublet at 67.62 ppm with $^2J_{CP}$ = 86 Hz. These data indicate a trans geometry for **4**. The antisymbiotic effect has proved useful in understanding some related changes in geometry in arylpalladium(II) complexes [16]. In terms of this effect, an aryl and a phosphine ligand should have a destabilizing effect on each other when attached to class b metal ions such as palladium(II). No resonance assignable to μ-hydroxo protons was observed in the ^1H NMR spectrum of **5**. The same feature has been reported for other bridging hydroxo complexes [17].

2.2. Crystal structure of complex 5

The crystal structure of **5** is the first for a nitroarylgold(III) complex (see Fig. 1). The complex displays crystallographic inversion symmetry and the central Au_2O_2 ring is thus exactly planar; the transannular Au...Au distance is 3.150(1) Å. The coordination at gold is, as expected, planar, with a mean deviation of 0.06 Å. The Au–C bond distances (1.992(5) Å, 1.995(5) Å) are among the shortest Au(III)–C bond distances so far reported [18]. The only two other crystal structures reported of hydroxoorganogold(III) complexes ($[AuMe_2(OH)]_4$ [19] and 1-hydroxo-2,3,4,5-tetraphenylauracyclopentadiene [13b]) were not of sufficient accuracy to allow comparison with the data for complex **5**. The Au–O distances (2.073(4) Å, 2.075(4) Å) in **5** are longer than in $Sr[Au(OH)_4]_2$ (1.980(8) Å) [20]. This difference can partly be attributed to the change from a bridging to a terminal OH ligand. Thus, in $[Au_2O_6]^{6-}$ the Au–O distances are 2.17 Å and 2.12 Å, respectively [21]. Additionally, the greater trans influence of an aryl than of an OH ligand must be taken into account.

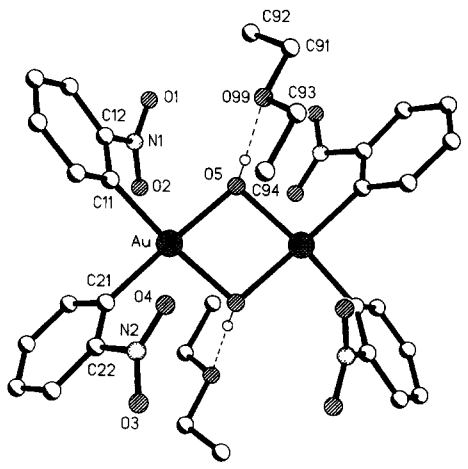

Fig. 1. The structure of compound **5** in the crystal. Radii are arbitrary.

Each nitro group is inclined to its own aryl ring (N1 by 35°, N2 by 20°) and in an anti disposition with respect to the hydrogen of the neighbouring *cis*-OH group. The OH hydrogen atoms are (necessarily) mutually trans and both are hydrogen bonded to a diethyl ether molecule (O...O 2.700(5) Å, O–H...O 178°).

The presence of the nitro group causes significant widening of the corresponding C–C(NO_2)–C angle (123.0(5)°, 123.9(5)°) with little change in the bond distances of the aryl ring (1.375(8)–1.404(8) Å). The opposite effect is observed for the C–C(Au)–C bond angle (117.0(5)°, 115.6(5)°). Both facts have been attributed to the operation of the -I rather than the -M effect of the nitro group [22]. The C–N (1.477(7) Å, 1.498(7) Å) and N–O (1.211(6)–1.230(7) Å) bond distances are similar to those previously reported [22]. The short intramolecular ONO...Au distances (Au...O2 2.866 Å, Au...O4 2.845 Å) correspond to weak axial interactions.

3. Experimental section

The compound [Hg($C_6H_4NO_2$ – 2)Cl] was prepared as previously described [23]. Reactions were carried out at room temperature and NMR spectra were recorded on a Bruker AC-200 or a Varian XL-300 spectrometer, and referenced to internal $SiMe_4$ (^1H, ^{13}C) or external H_3PO_4 (^{31}P) unless otherwise stated.

3.1. Cis-$Me_4N[Au(C_6H_4NO_2 - 2)_2Cl_2]$ (1)

To a yellow solution of $Me_4N[AuCl_4]$ (530 mg, 1.30 mmol) in EtOH (50 cm^3) were added solid [Hg($C_6H_4NO_2$ – 2)Cl] (919 mg, 2.60 mmol) and Me_4NCl (2.64 mg, 2.70 mmol). The mixture was stirred under reflux until complete (ca. 5 h). The solvent was removed decoloration and the residue extracted with CH_2Cl_2 (3 × 5 cm^3). The extract was filtered through anhydrous $MgSO_4$ and the filtrate evaporated to ca. 1 cm^3. Addition of Et_2O (15 cm^3) afforded a white

precipitate which was recrystallized from CH_2Cl_2/Et_2O (629 mg, 1.10 mmol) to give **1** [7]. Yield, 84%; ^1H NMR (DMSO): δ 3.10 (s, 12 H, Me_4N^+), 7.22 (m, 2H, C_6H_4), 7.33 (m, 2H, C_6H_4), 7.52 (m, 2H, C_6H_4), 7.87 (m, 2H, H3) ppm; ^{13}C NMR (DMSO): δ 150.97 (C2), 125.32, 126.27, 133.74, 135.12 (C1, C3–C6, two nuclei must be accidentally isochronous), 54.58 (Me_4N^+), ppm.

3.2. $Na[Au(C_6H_4NO_2-2)_2(OPh)_2]$ (2)

Solid **1** (189 mg, 0.33 mmol) was added to a suspension of NaOPh (170 mg, 0.99 mmol) in CH_2Cl_2 (30 cm^3), and the mixture stirred for 6 h. The solution was then filtered through anhydrous $MgSO_4$ and the filtrate evaporated to ca. 1 cm^3. Addition of Et_2O/n-hexane (1:1, 10 cm^3) produced a pale yellow precipitate which was filtered off, washed with Et_2O and dried under vacuum. Yield, 64%; M.p. 100°C (decomp.); $\Lambda_M = 87$ Ω^{-1} cm^2 mol^{-1} (5×10^{-4} mol dm^{-3}); ^1H NMR: δ 6.44 (m, 2H), 6.73 (m, 4H), 7.55 (m, 10H, C_6H_4 + PhO), 7.85 (m, 2H, H3) ppm. Anal. Calc. for $C_{24}H_{18}AuN_2NaO_6$: C, 44.3; H, 2.8; N, 4.3; Au, 30.3%. Found: C, 43.9; H, 3.0; N, 4.1; Au, 30.7%.

3.3. $[Au(C_6H_4NO_2-2)_2(O,O\text{-}acac)]$ (3)

To an acetone solution (50 cm^3) of **1** (56 mg, 0.10 mmol) were added solid Tl(acac) (acac = acetylacetonate) (29.5 mg, 0.10 mmol) and $NaClO_4$ (13.2 mg, 0.09 mmol). The mixture was stirred for 7 h and the solvent was then removed. The residue was extracted with CH_2Cl_2 (2×5 cm^3) and the solid filtered through anhydrous $MgSO_4$. Evaporation of the filtrate to a volume of ca. 1 cm^3 and addition of Et_2O (10 cm^3) gave a white precipitate (40 mg, 0.07 mmol) which was recrystallized from CH_2Cl_2/Et_2O. Yield, 75%; M.p., 205°C (decomp.); ^1H NMR: δ 2.00 (s, 6H, Me), 5.47 (s, 1H, CH), 7.30 (m, 4H, H4, H5), 7.52 (m, 2H, H6), 8.00 (m, 2H, H3) ppm. Anal. Calc. for $C_{17}H_{15}AuN_2O_6$: C, 37.8; H, 2.8; N, 5.2; Au, 36.5%. Found: C, 37.6; H, 2.4; N, 5.1; Au, 36.8 %.

Table 1
Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **5**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor

	x	y	z	U(eq)
Au	5185.7(1)	5783.4(1)	5619.9(1)	15.1(1)
C(11)	4935(4)	7109(4)	5626(3)	17.0(12)
C(12)	4071(4)	7437(4)	5503(4)	21.3(13)
C(13)	3882(5)	8349(4)	5413(3)	29(2)
C(14)	4583(5)	8959(4)	5455(4)	33(2)
C(15)	5461(4)	8654(4)	5572(4)	30.0(15)
C(16)	5637(4)	7746(4)	5651(4)	22.2(12)
N(1)	3293(3)	6812(4)	5499(3)	28.0(13)
O(1)	2650(3)	7001(4)	5071(3)	44.3(13)
O(2)	3321(3)	6156(3)	5945(3)	31.7(11)
C(21)	5695(4)	5873(4)	6726(3)	17.5(12)
C(22)	6590(4)	5774(4)	6930(3)	21.9(12)
C(23)	6905(4)	5780(5)	7710(4)	30.5(15)
C(24)	6313(4)	5928(5)	8327(4)	34(2)
C(25)	5398(4)	6031(4)	8156(4)	27(2)
C(26)	5101(4)	6010(4)	7370(3)	24.7(14)
N(2)	7281(3)	5622(4)	6286(3)	30.9(14)
O(3)	8010(3)	5295(4)	6492(3)	60(2)
O(4)	7107(3)	5846(3)	5603(3)	42.5(12)
O(5)	4558(3)	5587(3)	4519(2)	17.2(9)
C(91)	4349(5)	6695(5)	2671(4)	38(2)
C(92)	3519(5)	7018(5)	3095(5)	50(2)
C(93)	5912(5)	6417(5)	2885(4)	39(2)
C(94)	6598(5)	6298(6)	3544(5)	50(2)
O(99)	5054(3)	6613(3)	3250(3)	30.3(11)

Table 2
Selected bond lengths (Å) and angles (deg.) for **5**

Au–C(21)	1.992(5)	C(22)–N(2)	1.498(7)
Au–C(11)	1.995(5)	C(23)–C(24)	1.367(9)
Au–O(5)	2.073(4)	C(24)–C(25)	1.392(8)
Au–O(5)#1	2.075(4)	C(25)–C(26)	1.381(9)
C(12)–N(1)	1.477(7)	O(5)...O(99)	2.700(7)
N(1)–O(1)	1.221(6)	Au...Au#1	3.150(1)
N(1)–O(2)	1.222(6)		
C(21)–Au–C(11)	90.0(2)	O(1)–N(1)–O(2)	124.2(6)
C(21)–Au–O(5)	173.8(2)	O(1)–N(1)–C(12)	117.9(5)
C(11)–Au–O(5)	93.3(2)	O(2)–N(1)–C(12)	117.9(5)
C(21)–Au–O(5)#1	95.7(2)	O(4)–N(2)–O(3)	123.7(5)
C(11)–Au–O(5)#1	173.9(2)	O(4)–N(2)–C(22)	118.9(5)
O(5)–Au–O(5)#1	81.2(2)	O(3)–N(2)–C(22)	117.3(5)
C(12)–C(11)–C(16)	117.0(5)	Au–O(5)–Au#1	98.8(2)
C(11)–C(12)–C(13)	123.0(5)	C(14)–C(13)–C(12)	118.6(6)
C(13)–C(14)–C(15)	120.0(6)	C(16)–C(15)–C(14)	120.5(6)
C(15)–C(16)–C(11)	120.8(6)	C(22)–C(21)–C(26)	115.6(5)
C(21)–C(22)–C(23)	123.9(5)	C(24)–C(23)–C(22)	119.3(6)
C(23)–C(24)–C(25)	119.3(6)	C(26)–C(25)–C(24)	120.0(6)
C(25)–C(26)–C(21)	121.9(6)		

Symmetry transformation used to generate equivalent atoms: #1; $-x+1, -y+1, -z+1$.

3.4. $Trans\text{-}[Au(C_6H_4NO_2-2)_2(C\text{-}acac)(PPh_3)]$ (4)

Solid PPh_3 (26 mg, 0.10 mmol) was added to a CH_2Cl_2 solution (30 cm^3) of **3** (53 mg, 0.10 mmol) and the mixture stirred for 3 h. The solvent was evaporated to a volume of ca. 1 cm^3 and Et_2O/n-hexane (1:1, 10 cm^3) added to give a pale yellow precipitate (68 mg, 0.09 mmol). Yield, 89%; M.p., 165°C; ^1H NMR: δ 2.00 (s, 6H, Me), 4.23 [d, 1H, CH, $^3J_{HP} = 12$ Hz], 7.28–7.77 (m, 21H, $C_6H_4 + PPh_3$), 8.40 [d, 2H, H3, $^3J_{HH} = 8$ Hz] ppm; ^{31}P NMR, δ 24.93 ppm; ^{13}C NMR, δ 206.00 (CO), 151.87 (C2), 134.38, 134.12, 133.90, 132.59, 131.84, 131.79, 130.99, 129.25, 128.97, 128.75, 126.34, 122.97 (C1, C3–C6, Ph), 67.62 (CH), 30.62 (Me) ppm. Anal. Calc. for $C_{35}H_{30}AuN_2O_6P$: C, 52.4; H, 3.8; N, 3.5; Au, 24.5%. Found: C, 52.4; H, 3.9; N, 3.2; Au, 24.1 %.

3.5. $[Au(C_6H_4NO_2-2)_2(\mu\text{-}OH)_2]_2$ (5)

Solid NaOH (31.2 mg, 0.78 mmol) was added to an acetone solution (25 cm^3) of **1** (150 mg, 0.26 mmol)

and the suspension was stirred for 15 h, during which decomposition to metallic gold was observed. Evaporation of the solvent, extraction of the residue with CH_2Cl_2 and filtration of the extract through Celite gave a yellow solution which was concentrated to a volume of ca. 1 cm^3. Addition of n-hexane (20 cm^3) gave a pale yellow precipitate which was recrystallized from CH_2Cl_2/n-hexane. Yield, 35%; M.p., 210°C (decomp.). ^1H NMR: δ 7.9–6.4 (m, aryl) ppm. Anal. Calc. for $C_{24}H_{18}Au_2N_4O_{10}$: C, 31.5; H, 2.0; N, 6.1; Au, 43.0%. Found: C, 32.1; H, 2.3; N, 5.7; Au, 43.4%.

Crystal data. $C_{32}H_{38}Au_2N_4O_{12}$, $M_r = 1064.6$, orthorhombic, Pbca, $a = 14.801(2)$ Å, $b = 14.788(3)$ Å, $c = 16.627(2)$ Å, $V = 3639.4$ Å^3, $Z = 4$, $D_x = 1.943$ Mg m^{-3}, λ(Mo Kα) = 0.71073 Å, $\mu = 8.2$ mm^{-1}, $F(000) = 2048$, $T = -130$°C.

Data collection and reduction. A colourless flattened pyramid ca. $0.45 \times 0.4 \times 0.2$ mm^3 was mounted in inert oil and transferred to the cold gas stream of the diffractometer (Siemens P4 with LT-2 low temperature attachment). A total of 3671 unique intensities were measured to $2\theta = 50°$. Cell constants were refined from setting angles of 65 reflections to $2\theta = 25°$. An absorption correction based on ψ-scans gave transmission factors 0.55–0.97.

Structure solution and refinement. The structure was solved by the heavy-atom method and refined on F^2 using the program SHELXL-93. The hydroxyl H atom was refined with DFIX; others were included with a riding model. The weighting scheme was $w^{-1} = [\sigma^2(F_0^2) + (0.034P)^2]$, with $P = (F_0^2 + 2F_c^2)/3$. The final $wR(F^2)$ for all reflections was 0.059, with a conventional $R(F)$ of 0.026 for 2201 reflections with $I > 2\sigma I$, for 230 parameters; $S = 0.88$, max. $\Delta/\sigma = 0.001$, max. $\Delta\rho = 1.0$ e Å^{-3}. Final atomic coordinates are given in Table 1, with selected bond lengths and angles in Table 2.

Full details of the structure determination have been deposited at the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-76344 Eggenstein-Leopoldshafen, Germany, from where this material can be obtained on quoting the full literature citation and the reference number CSD 401615.

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