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Study on carbon–hydrogen activation of ketones by Gold(III) complexes and the synthesis and characterization of two ketonylgold(III) complexes

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Abstract

The acetonylgold(III) compound [Au(ppy)(CH_2COCH_3)Cl] (**1**) (ppy = 2-phenylpyridine) was unexpectedly obtained during the crystallization process of Au(III) lactate complex [Au(ppy)($CH_3CHOHCOO^-$)Cl]. This new structure prompted us to further study the role of Au(III) complexes on the carbon–hydrogen activation of ketones. Complex [Au(ppy)(CH_2COCH_3)NO_3] (**2**) was synthesized by reacting [Au(ppy)$(NO_3)_2$] with acetone while the ketonyl Au(III) complex [Au(apd)Cl_2] (**3**) (Hapd = 2-acetylpyridine) was obtained through carbon–hydrogen bond activation of the acetyl group. The crystal structures of **1** and **2** have common features: a square–planar Au(III) centre coordinated by one five-membered chelate ring, one acetonyl ligand and one anion (chloride or nitrate). Both structures show that carbon–hydrogen activation of acetone by 2-phenylpyridine–Au(III) complexes leads to the formation of acetonyl–Au(III) complexes. The Au–CH_2 bond lengths (2.067(7) Å, **1** and 2.059(5) Å, **2**) are similar to each other but longer than the Au–C (phenyl) bond lengths. The two softest ligands (carbanion) are also *cis* to each other in the thermodynamically most stable isomer. In complex **3**, the σ-bonded acetyl group is confirmed by ^{13}C DEPT NMR spectroscopy.

Keywords: Gold(III) complex; C–H activation; X-ray crystallography

1. Introduction

Metallated ketones play an important role in organic synthesis. There are several syntheses in which these compounds are involved, e.g., direct metallation of ketone by deprotonating agents, oxidative-addition reactions (with α-halogenocarbonyl compounds or epoxides) or transmetallation reactions [1–9]. In the work reported by Aoyama in 1987, acetone and methyl ethyl ketone underwent facile and direct metallation at the methyl groups by a cationic (octaethylporphyrinato) rhodium(III) complex with a non-coordinating perchlorate counteranion, (OEP)Rh^{III}(ClO_4) [9,10]. They presented an efficient catalytic cycle in the enolization of ketone under neutral or mild conditions by the Rh^{III} complex and in the enolated complex undergoing metallation to give organometalic derivatives or aldol reaction with free ketone.

In 1990, José Vicente and coworkers [11–14] reported that an unusual carbon–hydrogen activation of acetone occurred by intramolecular cooperation between the metal center Au(III) and a ligand attached to it. They investigated the reaction of [Au(pap)Cl_2] (pap = 2-phenylazophenyl) in acetone with various reagents such as

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Tl(acac) (acac = acetylacetonate), KCN, $AgClO_4$, HgR_2 (R = C_6F_5 or pap) or PdR_2 (R = $C_6H_4NO_2$-2) yielding an acetonyl product [Au(pap)(CH_2COMe)Cl]. They also proposed two plausible reaction pathways for the carbon–hydrogen activation of ketones [14]. In Scheme 1 [14], the substitution of the chloro ligands *trans* to the nitrogen atom is essential in the proposed pathway. First, it explains the labilization of the Au–N bond, thus permitting co-ordination of the acetone molecule, and then allows intramolecular transfer of the proton to form the deprotonated acetonyl Au(III) complex. In Scheme 1(**3**) for perchlorato-complex [14], the weakly bonding perchlorate ligand is directly replaced by acetone and the carbon–hydrogen activation is facilitated through a three-co-ordination of Au(III) intermediate. The reaction pathways in both schemes have in common the co-ordination of the ketone and the Au(III) ligand co-operation in carbon–hydrogen activation. The first process should be favoured by substitutes on the ketone with +I or +M effects (electron-releasing substituents, R = Et, Pr or Bu). It was also suggested that at least one of the substituents should be a methyl group (+I effect, low steric requirement).

Ketonylgold(III) complexes may play an important role in organic synthesis. As observed in other organo-transition–metal complexes [15–18], Au(III) complexes could serve as efficient deprotonating agents to generate enolization reagents or as metallo-enzyme catalyst in the biological enolization. The carbon–hydrogen activation of ketones by Au(III) complexes provides more options in the substitution at the α-position of the carbonyl group, such as in the halogenation and aldol reactions with C–C bond formation. There are a few syntheses and structures of ketonyl Au(III) complexes reported to date but their potential applications in organic reaction has not been explored extensively [11–14,19].

As part of our studies into the chemical and biological properties of Au(III) complexes, we recently reported synthesis of two series of Au(III) complexes with σ-bond C,N 2-phenylpyridine and *N*,*N* pyrazole or picolinamide ligands through direct deprotonation [20–23]. The strong polarizing property of Au(III) leads to direct metallation of Au(III) with 2-phenylpyridine under reflux and with pyrazole or picolinamide under mild conditions. During our study on 2-phenylpyridine Au(III) complexes, the crystallization of a Au(III) lactate complex [Au(ppy)($CH_3CHOHCOO^-$)Cl] in acetone gave an acetonyl Au(III) compound [Au(ppy)(CH_2COCH_3)Cl], instead of the expected product. This indicates that Au(III) complexes are important agents for the carbon–hydrogen activation of ketones. The unexpected carbon–hydrogen activation of acetone by Au(III) complexes motivated us to extend our research into the synthesis of new Au(III) complexes containing a Au–C σ-bonded ketonyl group. Thus, the intermediate [Au(ppy)$(NO_3)_2$] was reacted with solvent acetone for 3 days giving the acetonylgold(III) complex **2**. 2-Acetylpyridine (Hapd) reacted with $Na[AuCl_4]\cdot 2H_2O$ to give the neutral molecular ketonylgold(III) complex [Au(apd)Cl_2] (**3**). Herein, we report the structural features of two new acetonylgold(III) and one ketonylgold(III) complexes.

2. Results and discussion

In an attempt to synthesize a 2-phenylpyridine Au(III) lactate complex, [Au(ppy)Cl_2] was reacted with silver lactate for 10 h forming apparently a labile intermediate species, "[Au(ppy)($CH_3CHOHCOO^-$)Cl]", which in presence of acetone undergoes C–H activation yielding the [Au(ppy)(CH_2COCH_3)Cl] (**1**),

$$\begin{aligned}&[\mathrm{Au(ppy)Cl_2}] + \mathrm{Ag(C_3H_5O_3)}\\ &\quad \rightarrow \text{“}[\mathrm{Au(ppy)(CH_3CHOHCOO^-)Cl}]\text{”} + \text{acetone}\\ &\quad \rightarrow \mathbf{1}\end{aligned} \tag{1}$$

In fact, the crystals of **1** were grown during the process of crystallization of a 2-phenylpyridine Au(III) lactate complex in acetone. After two days, it was the compound [Au(ppy)(CH_2COCH_3)Cl] formed instead of the expected product. Complex **2** was prepared by treating intermediate [Au(ppy)$(NO_3)_2$] with acetone for 72 h at room temperature to complete the activation process.

In an attempt to extend the applicability of Au(III) species in the carbon–hydrogen activation of ketone, we explored the 2-acetylpyridine as a suitable ligand in the preparation of σ-bonded C–Au(III) complex through direct deprotonation of the methyl group. The reaction between 2-acetylpyridine and $Na[AuCl_4]\cdot 2H_2O$ in water was completed under mild conditions for 3 days to afford the ketonyl complex [Au(apd)Cl_2] (**3**), in a good yield,

$$\mathrm{Na[AuCl_4]\cdot 2H_2O} + \text{2-acetylpyridine} \rightarrow [\mathrm{Au(apd)Cl_2}] \tag{2}$$

3. Crystal structures

The structures of **1** and **2** were confirmed by X-ray crystallography. Figs. 1 and 2 depict the molecular structures **1** and **2**, respectively. Selected bond distances and angles are given in Table 2.

The two ketonyl Au(III) structures reveal neutral molecules with a square–planar geometry around the gold centre with both structures having common features. The chelate ring of the ppy ligand is associated with some distortion at the narrow C–Au–N bond angle (81.1(2)° in **1** and 81.8(2)° in **2**), and a concomitant

Table 1
Crystallographic data and structure refinement details for **1** and **2**

Complex	**1**	**2**
Formula	$C_{14}H_{13}AuClNO$	$C_{14}H_{13}AuN_2O_4$
Formula weight	443.67	470.23
Temperature (K)	293(2)	293(2)
Wavelength, λ (Å)	0.71073	0.71073
Crystal system	Triclinic	Monoclinic
Space group	$P2_1/c$	$P\bar{1}$
a (Å)	7.7676(3)	8.9969(3)
b (Å)	9.3952(3)	20.2059(6)
c (Å)	9.5779(3)	8.1420(3)
α (°)	90.193(1)	65.668(1)
β (°)	101.101(1)	106.632(1)
γ (°)	100.850(1)	64.721(1)
V (Å^3)	673.07(4)	1418.21(8)
Z	2	4
D_{calc} (g cm^{-3})	2.189	2.202
μ (mm^{-1})	11.113	10.390
Reflections collected	4368	8763
Independent reflections	3115	3507
R_{int}	0.0203	0.0376
Goodness-of-fit	1.053	0.999
Final $R[I > 2\sigma]$		
R_1[a]	0.0365	0.0304
wR_2[b]	0.0954	0.0611

[a] $R_1 = \sum \| F_o| - |F_c\| / \sum |F_o|$.

[b] $wR_2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{1/2}$.

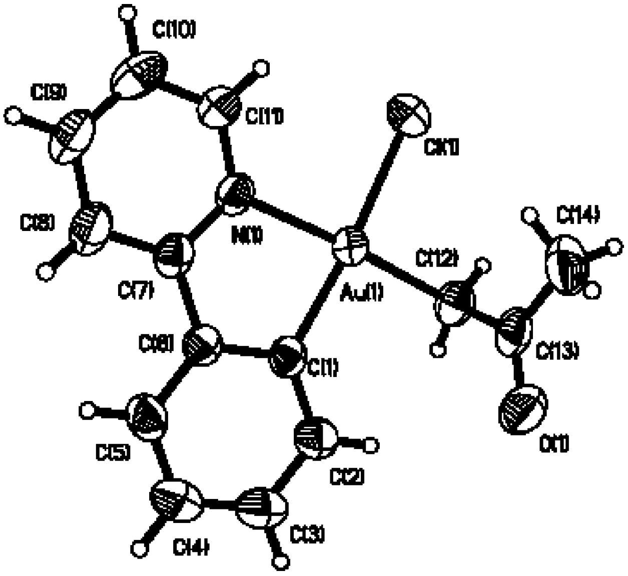


Fig. 1. An ORTEP view of **1** showing the numbering scheme.

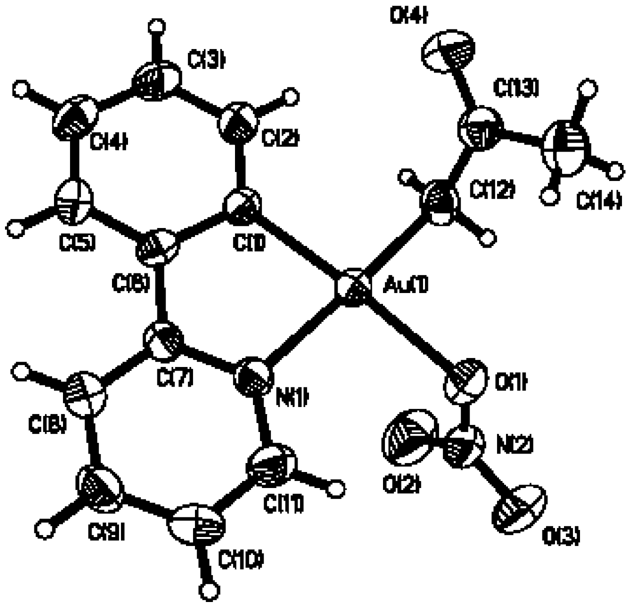


Fig. 2. An ORTEP view of **2** showing the numbering scheme.

Table 2
Selected bond lengths (Å) and angles (°) for **1** and **2**

1		**2**	
Au(1)–C(1)	2.018(7)	Au(1)–C(1)	2.003(4)
Au(1)–N(1)	2.110(5)	Au(1)–N(1)	2.114(4)
Au(1)–Cl(1)	2.370(2)	Au(1)–O(1)	2.128(3)
Au(1)–C(12)	2.059(5)	Au(1)–C(12)	2.059(5)
C(12)–C(13)	1.492(11)	C(12)–C(13)	1.472(7)
O(1)–C(13)	1.227(9)	O(4)–C(13)	1.209(6)
C(13)–C(14)	1.496(13)	O(1)–N(2)	1.323(6)
N(1)–C(7)	1.355(9)	O(2)–N(2)	1.221(6)
N(1)–C(11)	1.346(9)	O(3)–N(2)	1.219(5)
C(1)–Au(1)–N(1)	81.1(2)	C(1)–Au(1)–N(1)	81.8(2)
C(1)–Au(1)–C(12)	94.6(3)	C(1)–Au(1)–C(12)	93.1(3)
N(1)–Au(1)–C(12)	175.4(2)	N(1)–Au(1)–C(12)	177.7(2)
C(1)–Au(1)–Cl(1)	174.59(18)	C(1)–Au(1)–O(1)	174.6(2)
N(1)–Au(1)–Cl(1)	94.72(17)	N(1)–Au(1)–O(1)	92.96(14)
C(12)–Au(1)–Cl(1)	89.7(2)	C(12)–Au(1)–O(1)	89.2(2)
C(13)–C(12)–Au(1)	108.3(5)	C(13)–C(12)–Au(1)	112.1(3)
C(2)–C(1)–Au(1)	127.9(5)	N(2)–O(1)–Au(1)	112.7(3)
C(6)–C(1)–Au(1)	112.6(5)	O(2)–N(2)–O(1)	118.6(5)
C(7)–N(1)–Au(1)	114.0(4)	O(2)–N(2)–O(3)	125.1(5)

opening of N(1)–Au–Cl(1) (94.72(17)°) and C(1)–Au–C(12) (96.6(3)°) for **1** and of C(1)–Au–C(12) (96.0(2)°) and N(1)–Au–O(1) (92.96(14)°) in **2**. This structural feature is also observed in other 2-phenylpyridine Au(III) complexes, such as [Au(ppy)Cl_2] and [Au(ppy)(ace)$_2$] (ace = acetate) [21]. The two Au–C (phenyl) bond distances are similar (2.018(7) Å **1** and 2.003(4) Å **2**) to each other in spite of the different *trans* ligands. Similar Au–C (aryl) bond lengths are observed *trans* to an oxygen donor, as in [Au(ppy)(ace)$_2$] (2.005(8) Å), [Au(ppy)(bz)$_2$] (bz = $C_6H_5COO^-$, 2.001(9) Å), and [Au(η^2-$C_6H_4CH_2$-NMe_2-2)(η^2-quin)] (quin = quinolin-8-olate, 2.021(7) Å) [19,21]. The two gold-ketonyl bonds in both complexes (2.067(7) Å, **1** and 2.059(5) Å, **2**) are also similar to each other. However, the Au–CH_2 (sp^3 hybridization at carbon) bond lengths are longer than the Au–C (phenyl, sp^2 at carbon)) bonds in these two complexes, probably because of the different hybridization of the carbon atoms. In **2**, the nitrato N(2)–O(1) bond distance (1.323(6) Å) is longer than the other two N(2)–O(2) (1.221(6) Å) and N(2)–O(3) (1.219(5) Å) bonds as the O(1) atom is directly bound to gold.

Compared with other ketonyl Au(III) crystal structures of [Au{$C_6H_3(N{=}NC_6H_4Me\text{-}4')\text{-}2\text{-}Me\text{-}5$}(acac-*C*)Cl] (acac = acetylacetonate, Au–C(1) 2.083(4) Å) [13] and [Au(mpap)(CH_2COMe)(py)]ClO_4 (mpap = $C_6H_3(N{=}NC_6H_4Me\text{-}4)\text{-}2\text{-}Me\text{-}5$, py = pyridine, Au–C(1) 2.061(6) Å) [14], the Au–CH_2 bond length of 2.059(5) Å for **2** and 2.067(7) Å for **1** are similar. The angle of Au–CH_2–CO in **2** is 108.3(5)°, which is close to 109.1(2)° of [Au{$C_6H_3(N{=}NC_6H_4Me\text{-}4')\text{-}2\text{-}Me\text{-}5$}-(acac-*C*)Cl] but smaller than 112.1(3)° for **1** and 114.5(4)° of [Au(mpap)(CH_2COMe)(py)]ClO_4. Like other Au(III) complexes, these ketonyl complexes have the expected structures, with square–planar coordination at the gold atom. As a result of the *trans* influence, it was also expected that the two softest ligands (carboanion) would be *cis* to each other in the thermodynamically most stable isomer, as we observed in our structures.

4. Spectroscopic studies

The infrared spectra of **2** and **3** are consistent with the presence of a coordinated ketonyl group. A strong absorption band at ca. 1685 cm^{-1} corresponding to *v*(CO) is shown in both complexes. The IR spectrum of **2** has strong ppy ligand modes at ca. 1492, 1450, 786, 768, and 732 cm^{-1}, which are observed in other 2-phenylpyridine complexes [21–26]. The strong bands around 1600–1575 cm^{-1} correspond to the pyridine ligand in **3**.

The ^1H and ^{13}C DEPT(135) NMR chemical shifts of **2** and **3** are listed in Tables 3 and 4, respectively. The ^1H spectrum of the 2-phenylpyridine in **2** is similar to that in other 2-phenylpyridine Au(III) complexes [21,22], while two singlet peaks at δ 3.66 and 2.42 correspond to –CH_2– and –CH_3 groups, respectively. The ^{13}C DEPT(135) spectrum of **2** gave a negative peak at δ 56.8 showing the presence of a –CH_2– group and 9 positive peaks corresponding to 8 –CH– groups on the 2-phenylpyridine and one methyl group. It shows that the formation of Au–CH_2 bond leads to the methylene resonance at low filed compared with the methyl group. The proton spectrum of **3** shows a single peak at δ 3.72 for a methylene group and four peaks at δ 9.62(d), 8.55(t), 8.17(t), 8.08(d) for the pyridine ring protons. The integration ratio of H on the pyridine ring to on the acetyl group is 4:2. Compared with the ^1H spectrum of the ligand 2-acetylpyridine in DMSO-d_6 (δ 8.69 (1H, d), 7.93(2H, m), 7.62(1H, t), 2.60(3H, d)), there is a significant downfield shift of proton resonance after forming the Au(III) complex. Other ketonyl Au(III) complexes also show the methylene ^1H resonance as a singlet in the range δ 3.3–3.5 [14]. The chemical shift change of acetyl group indicates the deprotonation of the methyl group in the reaction and consequently the formation of the Au–CH_2 bond. The formation of a Au–CH_2 bond was also confirmed by a ^{13}C DEPT(135) spectrum, in which a negative peak for CH_2 is present at δ 57.7, whereas four positive peaks at δ 147.8, 144.1, 131.3 and 126.0 belong to the four CH groups on the pyridine ring.

Table 4
^1H and ^{13}C DEPT(45) NMR Data for complex **3** (δ, DMSO-d^6 solutions)

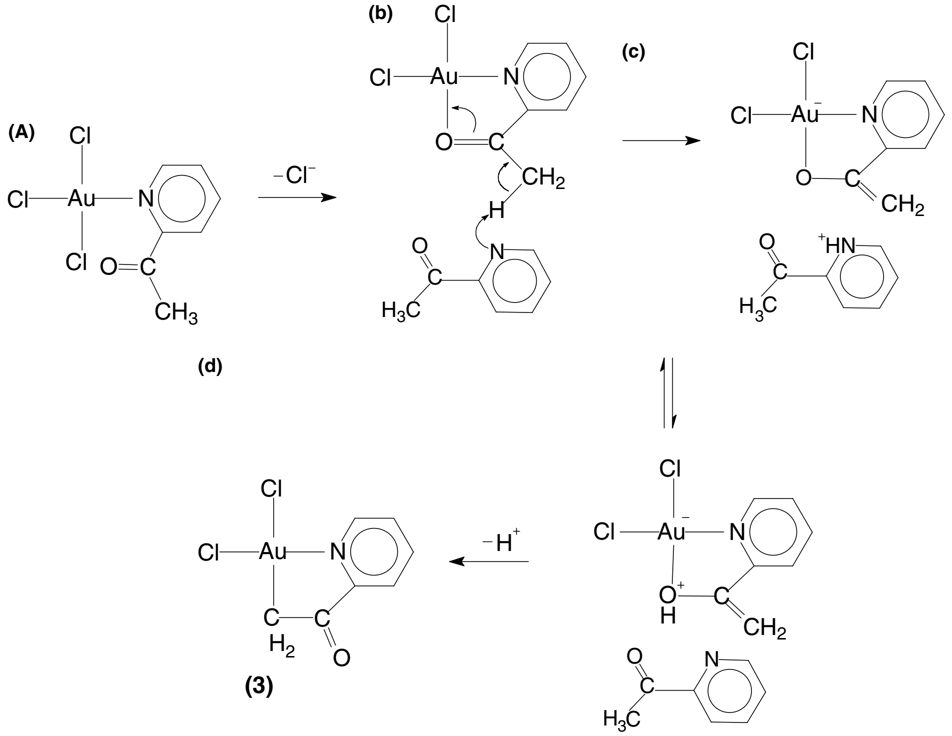
	Pyridine ring	–$COCH_2$
^1H	9.62(d), 8.55(t), 8.17(t), 8.08(d)	3.72(s)
^{13}C	147.8, 144.1, 131.3, 126.0	57.7

The detail mechanism of the carbon–hydrogen activation of ketones by Au(III) complexes is unclear to date. The proposed reaction pathway in Scheme 1(**3**) for the perchlorato-complex by Vicente [14] seems to be applicable to the formation of **2**, in which the enolization in the carbon–hydrogen activation process is facilitated by the three-co-ordination of Au(III) intermediates. Three-co-ordination of Au(III) intermediates have been established previously [27–29]. It is reasonable to postulate that the weakly bonding nitrate ligand is replaced by the acetone, instead of labilizing the ligand *trans* to it to allow the co-ordination of acetone *cis* to the nitrogen atom, as the proposed reaction pathway in Scheme 1 for other Au(III) complexes [14]. However, both schemes are unfit to the formation of **3** because there is neither the substitution of chloride ligand *trans* to the nitrogen atom as described in Scheme 1 nor the weakly bonding ligand replacement by acetone in Scheme 1(**3**). It is unlikely that methyl group could co-operate with the intramolecular base pyridine due to steric restraint. In this paper, we postulate a plausible

Table 3
^1H and ^{13}C DEPT(45) NMR Data for complex **2** (δ, DMSO-d^6 solutions)[a]

^1H	H^{11}	H^9	H^8	H^5	H^2	H^{10}	H^4	H^3	H^{12}	H^{14}
	8.75	8.44	7.94	7.78	7.52	7.40	7.33	7.26	3.66	2.42
^{13}C	2-Phenylpyridine								–CH_2COCH_3	
	121.8	125.2	126.3	128.3	129.1	131.5	146.5	152.8	33.5	56.8

[a] See Fig. 2 for ^1H numbering scheme.



Scheme 1. Proposed reaction pathway for the synthesis of complex **3**.

reaction pathway for the carbon–hydrogen activation of the acetyl group by Au(III) complex (See Scheme 1). First, it was the pyridine bound to Au forming **A** and secondly the oxygen atom on carbonyl group replaced the neighbouring chloride giving a five-membered chelating ring (**B**). With the aid of free trace 2-acetylpyridine in the solution, the highly electrophilic nature of the central ion Au(III) leads to the cooperation between the Au(III) center and the acetyl group to allow transfer of the proton shown in **C** or **D**, giving the deprotonated product **3**. The intramolecular co-operation between metal and internal ligands in C–H activation of ketones has been reported in a few cases [9,10,13,14]. However, the formation of **3** in this work is possibly through the intermolecular cooperation between 2-acetylpyridine and Au(III). Further studies on carbon–hydrogen activation of ketones will be devoted in order to fully understand the mechanism of formation of ketonyl Au(III) complexes as well as its potential application in the direct metallation of ketones and aldol reactions.

5. Experimental

$Na[AuCl_4] \cdot 2H_2O$ and 2-phenylpyridine were obtained from Aldrich Chemical Co. Milwankee, Wisconsin, USA. All other reagents were purchased from Fluka Chemie AG, Buchs, Switzerland.

NMR spectra were recorded on a Bruker ACF 300 (^1H, ^{13}C) spectrometer at 25 °C and were referenced to solvent. The Infrared spectra (KBr pellet) were recorded using an FTS165 Bio-Rad FTIR spectrophotometer in the range 4000–450 cm^{-1}. Elemental analyses were performed in the Microanalytical Laboratory, Department of Chemistry, National University of Singapore.

Complex $[Au(ppy)Cl_2]$ was synthesized according to literature methods [24].

5.1. $[Au(ppy)(CH_2COCH_3)Cl]$ (*1*)

Single crystals of **1** were obtained during the crystallization of a 2-phenylpyridine Au(III) lactate complex $[Au(ppy)(CH_3CHOHCOO^-)Cl]$. In an attempt to synthesize a 2-phenylpyridine Au(III) lactate complex, $[Au(ppy)Cl_2]$ was reacted with silver lactate in the dark for 10 h. The mixture was filtered and the filtrate was rotatoevaporated to a small volume to crystallize by vapour diffusion of diethyl ether into acetone in an ice bath. White crystals of **1** grew after 3 days.

5.2. $[Au(ppy)(CH_2COCH_3)NO_3]$ (*2*)

$[Au(ppy)Cl_2]$ (0.52 g, 1.2 mmol) was dissolved in acetone (30 ml) and silver nitrate (0.42 g, 2.4 mmol) added. The mixture was stirred in the dark for 2 h and the silver chloride filtered off. The colourless filtrate was stirred in the dark for 3 days and then it was rotatoevaporated to a small volume. The concentrated solution was crystallized by vapour diffusion of diethyl ether into it. White crystals of **2** were grown in a freezer. Yield: 0.12 g (21%). Anal. for $C_{14}H_{13}AuN_2O_4$ (Calc. in parenthesis): C, 35.86 (35.74); H, 2.73 (2.77); N, 5.88 (5.96); Au, 41.85 (41.91). IR (KBr, cm^{-1}): ν(CO) 1685, $\nu(C–N)_{ppy}$ 1492, 1450, 786, 768, 732.

*5.3. [Au(apd)Cl_2] (**3**)*

$Na[AuCl_4] \cdot 2H_2O$ (0.81 g, 2.0 mmol) was dissolved in water (20 ml) in a round bottom Schlenk flask. 2-Acetylpyridine (0.23 ml, 2.1 mmol) in water (10 ml) was then added dropwise with stirring and a yellow suspension formed. The mixture was stirred in the dark under nitrogen at 60 °C for 3 days. It was then filtered and washed with water; the residue was dried in vacuo, extracted with dichloromethane and filtered off. The solution was concentrated (3 ml), whereupon addition of diethyl ether (5 ml) and crystallization from dichloromethane/diethyl ether gave the corresponding complex **3**. Yield: 0.44 g (56%). Anal. for $C_7H_6AuNOCl_2$ (Calc. in parentheses): C, 21.45 (21.65); H, 1.60 (1.55); N, 3.71 (3.61); Au, 50.45 (50.77). IR (KBr, cm^{-1}): ν(CO) 1685, $\nu(C–N)_{py}$ 1600–1575.

6. X-ray crystallography

The diffraction experiments were carried out on a Bruker AXS SMART CCD diffractometer. The program SMART [30] was used for collecting the intensity data, for reflections indexing and for the determination of lattice parameters, SAINT [30] was used for integration of the intensity of reflections and scaling, SADABS [31] was used for absorption correction and SHELXTL [32] for space group and structure determination, least-squares refinements on F^2. All non-H atoms were refined with anisotropic thermal parameters. Selected crystallographic data and refinement details are displayed in Table 1.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2004.06.025.

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