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Gold and silver complexes with the ferrocenyl-pyrazolate ligand FcCH₂pz

Eva M. Barranco, M. Concepción Gimeno, Antonio Laguna *, M. Dolores Villacampa

Departamento de Química Inorgánica, Instituto de Ciencia de Materiales de Aragón, Universidad de Zaragoza-CSIC, 50009 Zaragoza, Spain

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Dedicated to Professor Hubert Schmidbaur.

Abstract

We have synthesized a series of gold(I), gold(III) or silver(I) derivatives with ferrocenylmethylpyrazole (pzCH₂Fc) as ligand. X-ray structure determination for [Au(C₆F₅)(pzCH₂Fc)] reveals a linear geometry for the gold centre and association of the molecules in pairs through intermolecular gold(I)–gold(I) interactions of 3.1204(6) Å. These molecules are further assembled into chains and the chains into a 3D structure through several hydrogen bonds. [Ag(OTf)(pzCH₂Fc)] (OTf = triflate) is a dimer with a distorted tetrahedral geometry at the silver(I) centre and the triflate acting as bridging ligand. © 2005 Elsevier B.V. All rights reserved.

Keywords: Gold; Silver; Ferrocene; Pyrazole

1. Introduction

Ferrocene has been extensively used as starting product in the synthesis of new derivatives. It is a versatile molecule with important properties such as high electron density, aromaticity and reversible redox characteristics. These properties together with the ease of preparation of species in which one or two cyclopentadienyl rings are substituted with a great variety of organic fragments that may contain O, N, S, P, etc. as donor atoms make of ferrocene a very suitable building block in many fields of research [1–3]. Studies of these ligands are important because the complexes thus obtained may be interesting in many research areas looking for special properties, such as redox or non linear optical properties, charge transport, liquid crystals, electrochemical recognition or catalysis [1-12]. Ferrocenylpyrazole is one of the above systems and this ligand has recently been used

to produce polymetallic species owing to their versatile coordination possibilities [13–18].

In this paper, we report the synthesis of ferrocenylmethylpyrazole and its reactivity with gold or silver derivatives. The single crystal X-ray analysis of the new complexes shows the formation of dimers for the gold(I) or silver(I) derivatives, due to aurophilic interactions or the presence of triflate bridges, respectively. Also, the presence of hydrogen bonding leads to the formation of a supramolecular array for the gold compound.

2. Results and discussion

The ferrocenylmethylpyrazole (pzCH₂Fc) ligand (1) has been synthesized by reaction of *N*,*N*-dimethylaminomethylferrocene with pyrazole in acetic acid. The yellow solid is air- and moisture-stable and the LSIMS + mass spectrum shows the molecular peak at m/z = 266 (100%). The ¹H NMR spectrum shows the resonances of the ferrocenyl unit with a singlet for the

^{*} Corresponding author. Tel.: + 34 976 761185; fax: + 34 976 761187. *E-mail address:* alaguna@unizar.es (A. Laguna).

unsubstituted cyclopentadienyl ring and two multiplets for the α and β protons of the substituted cyclopentadienyl unit. The methylene protons appear as a singlet and for the pyrazole protons two doublets and one doublet of doublets are present. The ¹H NMR data are collected in Table 1. The reaction of 1 with [AuX(tht)] (tht = tetrahydrothiophene) gives the complexes [AuX(pzCH₂Fc)] (X = Cl (2) or C₆F₅ (3)) (Scheme 1), which are isolated as yellow air- and moisture-stable solids. IR spectra show the presence of Au–Cl [2, 325(m) cm⁻¹], or Au(I)–C₆F₅ bonds [3, 1505(vs), 956(s) and 808(m) cm⁻¹]. The ¹⁹F NMR spectra show the presence of one type of pentafluorophenylgold(I). The LSIMS + mass spectrum of 3 shows as the most intense peak the molecular peak at m/z = 630.

The crystal structure of complex **3** has been confirmed by an X-ray diffraction study; selected bond lengths and angles are collected in Table 2. The gold centre has a linear geometry with a N(2)–Au–C(21) angle of 178.9(2). In the lattice, the molecules are associated into pairs via an intermolecular Au···Au interaction of 3.1204(6) Å (Fig. 1) and the pairs form chains held together by H···Au and H···F interactions (Fig. 2). The shortest H1B···Au distance is 2.932 Å with a C1···Au distance of 3.568 Å and a C1–H1B···Au angle of 123°. The bond distance H4···F1 is 2.59 Å with a C4···F1 distance of 3.387 Å and a C4–H4···F1 angle of 141°. The chains are further linked to form a 3D structure through additional C–H···F hydrogen bonds (Table 3).

The Au–N and Au–C bond lengths, 2.072(5) and 2.023(7) Å, compare well with the values reported in complexes with the N–Au–C₆F₅ moiety as

Table 1 ¹H NMR data for complexes **1–6**

Complex	δ(C _c H _a)	δ(C _c H _c)	δ(CH ₂)	δ(nz)
1	4.20(m) 4.28(m)	4.19(s)	5.10(s)	6.24(dd), <i>J</i> (HH) 2.1, 1.4 Hz 7.36(d) 7.52(d)
2	4.13(m) 4.24(m)	4.13(m)	5.02(m)	6.21(m) 7.32(m) 7.46(m)
3	4.22(m) 4.45(m)	4.20(s)	5.45(s)	6.37(dd), <i>J</i> (HH) 2.6, 1.7 Hz 7.56(d) 7.64(d)
4	4.25(m)	4.08(m)	5.36(s)	6.44(m) 7.71(m) 8.02(m)
5	4.05(m) 4.22(m)	4.09(m)	5.10(m)	6.23(m) 7.54(m) 7.62(m)
6	4.10(m) 4.26(m)	4.26(m)	5.27(m)	6.33(m) 7.32(m) 7.55(m)

Table 2	
Selected bond	lengths (Å) and angles (°) for complex 3

	,	,	
Au-C(21)	2.023(7)	N(1)–C(1)	1.474(8)
Au-N(2)	2.072(5)	N(2)–C(2)	1.332(8)
Au–Au#1	3.1204(6)	C(2)–C(3)	1.390(9)
N(1)-C(4)	1.351(8)	C(3)–C(4)	1.355(10)
N(1)-N(2)	1.369(7)		
C(21)-Au-N(2)	178.9(2)	C(2)-N(2)-Au	129.3(4)
C(21)-Au-Au#1	87.89(17)	N(1)–N(2)–Au	123.9(4)
N(2)-Au-Au#1	91.28(14)	N(1)-C(1)-C(11)	112.1(5)
C(4)–N(1)–N(2)	108.6(5)	N(2)-C(2)-C(3)	110.1(6)
C(4)-N(1)-C(1)	128.6(5)	C(4)-C(3)-C(2)	105.5(6)
N(2)–N(1)–C(1)	122.5(5)	N(1)-C(4)-C(3)	109.1(6)
C(2)–N(2)–N(1)	106.8(5)		

Symmetry transformations used to generate equivalent atoms: #1 -x, y, -z + 1/2.

[Au(C₆F₅)(FcCH₂NHpyMe)] (2.073(4) and 1.988(4) Å) [19], [Au(C₆F₅)(3-Fcpy)] (2.124(15) and 2.00(2) Å) [20] or [Au(C₆F₅)(Ph₂C=N-N=CPh₂)] (2.069(5) and 1.992(6) Å) [21]. The cyclopentadienyl rings deviate slightly from an eclipsed geometry [torsion angle C(11)-Cp-Cp-C(18) 3.4°].

The reaction of 1 with $[M(OTf)PPh_3]$ (OTf = triflate, M = Au, Ag, 1:1 molar ratio, leads to the cationic complexes $[M(pzCH_2Fc)(PPh_3)]OTf [M = Au (4) or Ag (5)].$ These complexes are air- and moisture-stable yellow solids at room temperature and they behave as electrolyte 1:1 in acetone solutions [150 (4) and 107 (5)] $ohm^{-1} cm^2 mol^{-1}$]. They were readily characterized by elemental analysis, IR and NMR spectroscopy. A singlet is observed in the phosphorus spectra at room temperature [30.3 (4), 14.9 (5) ppm]. At -55 °C complex 5 shows two doublets because of the coupling with both silver nuclei, $[J(P^{109}Ag), 745.6; J(P^{107}), 657.7 Hz]$. IR spectra show the presence of the triflate group at 1265(vs, br), 1223(s), 1152(s) and 1029(s) cm⁻¹ or 1253(vs, br), 1223(s), 1152(s) and 1032(s) cm⁻¹, for 4 and 5, respectively. The LSIMS + mass spectrum shows the cationic molecular peak at m/z = 725 (30%, 4) or 636 (55%, 5).

The structure determination of complex 5 has been determined and reveals the presence of dimers in which triflate anions are acting as bridging ligands as depicted in Fig. 3. The complex crystallizes with one molecule of dichloroethane. Selected bonds and angles are collected in Table 4. The silver centre displays a highly distorted tetrahedral geometry; the angles around Ag range from 84.63(13)° [O(2)-Ag-N(2)] to 160.66(11)° [N(2)-Ag-P(1)]. The silver centre lies only 0.23 Å out of the plane formed by the atoms O(2), P(1) and N(2). The Ag-O distances, 2.544(4) and 2.612(4) A, longer than other values reported for tetracoordinate silver complexes [22–25], are consistent with weak bonds as has been found in other silver-triflate complexes. The longer 2.612(4) A Ag–O3#2 indicates a weaker bond to the second triflate unit.



Fig. 1. Molecular structure of the dimeric unit of 3 showing the atom numbering scheme. Radii are arbitrary.



Fig. 2. Association of dimers in complex 3 through C-H···Au and C-H···F interactions.

Table 3 Hydrogen bonds for complex **3** (Å and °)

D–H···A	<i>d</i> (D–H)	$d(H \cdot \cdot \cdot A)$	$d(D \cdots A)$	((DHA)
$C(4)-H(4)\cdots F(1)#2$	0.95	2.59	3.387(8)	141.5
$C(4)-H(4)\cdots F(2)#3$	0.95	2.56	3.199(8)	124.4
C(14)–H(14)···F(4)#4	0.95	2.65	3.334(8)	129.4
$C(1)-H(1B)\cdots Au#2$	0.99	2.93	3.568(6)	122.9

Symmetry transformations used to generate equivalent atoms: #2 -x, -y + 1, -z; #3 x, y - 1, z; #4 -x + 1/2, -y + 3/2, -z + 1.

The Ag–N distance, 2.196(4) Å, is shorter than those found in some four-coordinate pyrazole–silver complexes as $[Ag_2(pz)_2(PPh_3)_3]$ (2.295(2) and 2.323(2) Å for the tetracoordinated silver atom) [26], $[Ag{HB}(3,5-$

 $Me_2pz_{3}(PMePh_2)$] (from 2.316(6) to 2.336(5) Å) [27] or the complexes { $Fe[C_5H_4CH(pz)_2]_2AgX$ }_n (X = BF₄, PF₆, SO₃CF₃ and SbF₆) (from 2.230(3) to 2.420(4) Å) [28].

The Ag–P bond distance, 2.3670(14) Å, is similar to that reported for the complex [Ag{HB(3,5-Me₂pz)₃} (PMePh₂)] (2.336(2) Å) [27] but shorter than those found in [Ag₂(pz)₂(PPh₃)₃] (2.461(1) and 2.484(1) Å) [26], or other tetrahedral silver complexes such as [Ag(dppe)₂]NO₃ [29] (range 2.488(3)–2.527(3) Å), [Ag (phen){(PPh₂)₂C₂B₁₀H₁₀}]ClO₄ [30] (2.463(2), 2.479(2) Å) or [Ag{C₅(CO₂Me)₅}(PPh₃)₂] [22] (2.428(2), 2.414(2) Å). The cyclopentadienyl rings deviate slightly from an eclipsed geometry [torsion angle C(1)–Cp–Cp– C(6) 11.5°]. Additionally, complex **5** displays some



Fig. 3. Molecular structure of complex **5**. Radii are arbitrary. H atoms are omitted for clarity.

Table 4 Selected bond lengths (Å) and angles (°) for compound 5

Ag–N(2)	2.196(4)	N(1)-C(44)	1.345(6)
Ag-P(1)	2.3670(14)	N(1)–N(2)	1.358(5)
Ag–O(2)	2.544(4)	N(2)–C(42)	1.327(6)
Ag-O3#2	2.612(4)	C(42)–C(43)	1.393(7)
C(10)-C(41)	1.498(7)	C(43)-C(44)	1.374(7)
C(41)–N(1)	1.449(6)		
N(2)–Ag–P(1)	160.66(11)		
N(2)-Ag-O(2)	84.63(13)		
P(1)-Ag-O(2)	109.46(9)		
N(1)-C(41)-C(10)	113.3(4)		
C(44)-N(1)-N(2)	111.3(4)		
C(44)–N(1)–C(41)	128.3(4)		
N(2)–N(1)–C(41)	120.1(4)		
C(42)–N(2)–N(1)	105.3(4)		
C(42)–N(2)–Ag	127.9(3)		
N(1)–N(2)–Ag	126.8(3)		
N(2)-C(42)-C(43)	111.2(5)		
C(44)-C(43)-C(42)	105.0(5)		
N(1)-C(44)-C(43)	107.2(5)		

Symmetry transformations used to generate equivalent atoms: #2 1-x, 1-y, -z.

intra- or intermolecular C–H···O(triflate) hydrogen bonds that are collected in Table 5.

The ligand **1** also reacts with $[Au(C_6F_5)_3(OEt_2)]$ (1:1 molar ratio) to give the gold(III) complex $[Au(C_6F_5)_3(pzCH_2Fc)]$ (**6**) as a yellow air- and moisture-stable solid. The LSIMS + mass spectrum shows the molecular peak at m/z = 964 as the most intense peak. The signals due to three C_6F_5 groups are present in the IR spectrum. The ¹⁹F NMR spectrum shows two different types of pentafluorophenyl groups in a ratio 1:2, typical of the presence of a "Au(C_6F_5)₃" fragment (see Scheme 1).

Table 5	
Hydrogen bonds for complex 5 (Å and	°)

$D_H \dots A$ $d(D_H) d(H \dots A) d(D \dots A)$	(DHA)
$C(12)-H(12)\cdots O(1)$ 0.93 2.44 3.357(6) 1	70.3
C(41)-H(41A)···O(2)#1 0.97 2.54 3.322(6) 1	38.1
C(9)-H(9)···O(3)#2 0.93 2.57 3.413(6) 1	50.6
$C(36)-H(36)\cdots O(3)\#2$ 0.93 2.40 3.245(6) 1	51.1

Symmetry transformations used to generate equivalent atoms: #1 -x + 1, -y + 2, -z; #2 - x + 1, -y + 1, -z.



Scheme 1. (i) $[AuX(tht)] (X = Cl, C_6F_5)$; (ii) $[M(OTf)PPh_3] (M = Au, Ag)$; (iii) $[Au(C_6F_5)_3(OEt_2)]$.

3. Experimental

3.1. General procedures

All the reactions were carried out under an argon atmosphere at room temperature. IR spectra were recorded on a Perkin-Elmer 883 spectrophotometer, over the range 4000–200 cm^{-1} , by using Nujol mulls between polyethylene sheets. Conductivities were measured in about 5×10^{-4} M solutions with a Philips 9509 conductimeter. Mass spectra were recorded on a VG Autospec by the LSIMS technique with nitrobenzil alcohol as matrix. ¹H, ¹⁹F and ³¹P{¹H} NMR spectra were recorded on a Bruker ARX300 or GEMINI 2000 apparatus; chemical shifts are quoted relative to SiMe₄ (external, ¹H), CFCl₃ (external, ¹⁹F) and 85% H₃PO₄ (external, ³¹P). C, H and N analyses were performed with a Perkin-Elmer 2400 microanalyzer. The starting materials [AuX(tht)] (X = Cl, C₆F₅) [31], $[Ag(OTf)(PPh_3)]$ (M = Au, Ag) [32] and $[Au(C_6F_5)_3(OEt_2)_2]$ [33] were prepared by published procedures.

3.2. Preparation of $[pzCH_2Fc]$ (1)

To a 30 ml acetic acid solution of pyrazole (14 mmol) was added N,N-dimethylaminomethylferrocene (7 mmol). The orange solution was heated at 80 °C for 3 h and the solution was neutralized with a saturated aqueous solution of NaHCO₃. The product was extracted

with 3×10 ml of dichloromethane and dried with anhydrous magnesium sulfate. The dichloromethane solution was concentrated to ca. 3 ml and further addition of diethyl ether afforded complex **1** as a brown solid. It was purified by chromatography with alumina and a mixture of diethyl ether/hexane (80/20). Yield: 30%. C₁₄H₁₄FeN₂ requires: C, 63.2; H, 5.3; N, 10.5. Found: C, 62.95; H, 5.1; N, 10.35%.

3.3. Preparation of $[AuX(pzCH_2Fc)]$ (X = Cl (2) or C_6F_5 (3))

A dichloromethane solution (20 ml) of **1** (26.6 mg, 0.1 mmol) and [AuX(tht)] (0.1 mmol, X = Cl, 32 mg; C₆F₅, 45.2 mg) was stirred for about 30 min and then filtered through celite. Concentration to ca. 2 ml and addition of hexane (20 ml) afforded complexes **2** or **3** as yellow solids. Yield of **2**: 45%. C₁₄H₁₄AuClFeN₂ requires: C, 33.75; H, 2.85; N, 5.6. Found: C, 33.4; H, 2.9; N, 5.55%. Yield of **3**: 75%. C₂₀H₁₄AuF₅FeN₂ requires: C, 38.2; H, 2.25; N, 4.45. Found: C, 38.05; H, 2.0; N, 4.5%. ¹⁹F NMR: -112.1 (m, *o*-F), -155.2 (t, *p*-F, *J*(F-F) 20.1 Hz), -158.7 (m, *m*-F).

3.4. Preparation of $[M(pzCH_2Fc)(PPh_3)]OTf$ (M = Au (4) or Ag (5))

To a dichloromethane solution (20 ml) of 1 (26.6 mg, 0.1 mmol) was added a dichloromethane solution (10 ml) of [Au(OTf)PPh₃] (0.1 mmol, prepared in situ with [AuCl(PPh₃)] and AgOTf) or [Ag(OTf)PPh₃] (0.1 mmol, 51.9 mg). The mixture was stirred for about 1 h and then filtered through celite. Concentration to ca. 2 ml and addition of hexane (20 ml) afforded complexes 4 or 5 as yellow solids. Yield of 4: 65%. C₃₃H₂₉AuF₃Fe-N₂O₃PS requires: C, 45.35; H, 3.35; N, 3.2; S, 3.65. Found: C, 44.97; H, 3.2; N, 3.45; S, 3.55%. Yield of 5: 75%. C₃₃H₂₉AgF₃FeN₂O₃PS requires: C, 50.45; H, 3.7; N, 3.55; S, 4.05. Found: C, 50.4; H, 3.2; N, 3.5; S, 3.95%.

3.5. Preparation of $[Au(C_6F_5)_3(pzCH_2Fc)]$ (6)

A dichloromethane solution (20 ml) of **1** (26.6 mg, 0.1 mmol) and $[Au(C_6F_5)_3(OEt_2)]$ (77.2 mg, 0.1 mmol) was stirred at room temperature for about 1 h and then filtered through celite. Concentration to ca. 2 ml and addition of hexane (20 ml) afforded complex **6** as a yellow

Table 6

Details of crystal data and structure refinement for complexes 3 and 5

Compound	3	$5 \cdot C_2 H_4 Cl_2$
Empirical formula	C ₂₀ H ₁₄ AuF ₅ FeN ₂	C ₃₅ H ₃₃ AgCl ₂ F ₃ FeN ₂ O ₃ PS
Formula weight	630.15	884.28
Temperature (K)	173(2)	173(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	monoclinic	monoclinic
Space group	C2/c	P2(1)/n
Unit cell dimensions		
<i>a</i> (Å)	25.013(5)	14.994(5)
b (Å)	12.344(2)	9.961(5)
c (Å)	15.786(2)	24.530(7)
α (°)	90	90
β (°)	127.341(9)	106.22(2)
γ (°)	90	90
Volume (Å ³)	3875.1(11)	3518(2)
Z	8	4
Density (calculated) (Mg/m ³)	2.160	1.670
Absorption coefficient (mm^{-1})	8.366	1.281
<i>F</i> (000)	2384	1784
Crystal habit	orange prism	orange prism
Crystal size (mm ³)	$0.70 \times 0.45 \times 0.40 \text{ mm}^3$	$0.45 \times 0.23 \times 0.15$
Theta range for data collection (°)	2.58-25.00	2.22-25.09
Index ranges	$-1 \leq h \leq 29, -14 \leq k \leq 5, -18 \leq l \leq 15$	$0 \leqslant h \leqslant 17, -11 \leqslant k \leqslant 1, -29 \leqslant l \leqslant 28$
Reflections collected	5268	6760
Independent reflections	$3403 [R_{(int)} = 0.0395]$	$6201 [R_{(int)} = 0.0438]$
Completeness to theta = 25.09°	99.6%	99.1%
Absorption correction	Ψ-scans	Ψ-scans
Maximum and minimum transmission	0.1347 and 0.0674	0.894 and 0.807
Refinement method	full-matrix least-squares on F^2	full-matrix least-squares on F^2
Data/restraints/parameters	3403/0/262	6201/0/442
Goodness-of-fit on F^2	1.034	1.004
Final <i>R</i> indices $[I > 2\sigma(I)]$	$R_1 = 0.0400, wR_2 = 0.0952$	$R_1 = 0.0455, wR_2 = 0.0834$
R Indices (all data)	$R_1 = 0.0446, wR_2 = 0.0971$	$R_1 = 0.0872, wR_2 = 0.0954$
Largest difference between peak and hole (e Å $^{-3}$)	2.422 and -2.809	0.690 and -0.630

solid. Yield: 40%. $C_{32}H_{14}AuF_{15}FeN_2$ requires: C, 39.85; H, 1.45; N, 2.9. Found: C, 39.8; H, 1.6; N, 2.55%. ¹⁹F NMR: -122.7 (m, 2F, *o*-F), -124.0 (m, 4F, *o*-F), -156.9 (t, 2F, *p*-F, *J*(F–F) 20.0 Hz), -157.4 (t, 1F, *p*-F, *J*(F–F) 19.9 Hz), -160.6 (m, 4F, *m*-F) and -161.6 (m, 2F, *m*-F).

3.6. Crystal structure determinations

Data were recorded with a Siemens P4 (3) or a Stoe-AED (5) diffractometer. Data collection type: ω/θ -scans. Absorption correction was applied based on Ψ -scans. The structures were refined using the program SHELXL-97 [34]. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included using a riding model. Further details are given in Table 6. Refinement special details: the structure of complex 5 contains one well-resolved dichloroethane solvate molecule.

4. Supplementary material

Complete X-ray data (excluding structure factors) for complexes **3** and **5** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. 260995 and 260996. These data can be obtained free of charge at www.ccdc.cam.ac.uk/ conts/retrieving.htlm [or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (internat.) +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk].

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