Supplementary Material (ESI) for Chemical Communications

Supporting Information

Isolable, Gold Carbonyl Complexes Supported by *N*-Heterocyclic Carbenes

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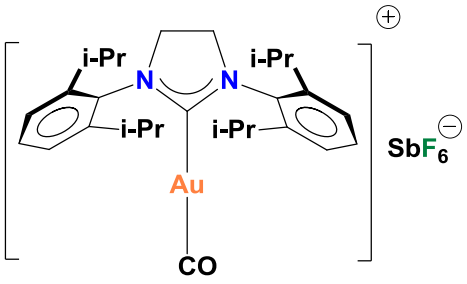
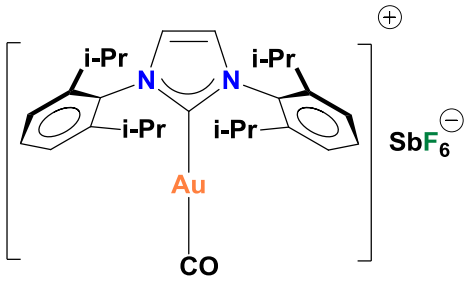


[(SIDipp)Au(CO)][SbF$_6$] (2)



[(IDipp)Au(CO)][SbF$_6$] (3)

Experimental Sections:

General Procedures. All manipulations were carried out under an atmosphere of purified nitrogen using standard Schlenk techniques or in a drybox. Solvents were purchased from commercial sources, purified using an Innovative Technology SPS-400 PureSolv solvent drying system degassed by the freeze-pump-thaw method twice prior to use. Glassware was oven-dried at 150 ^{o}C overnight. NMR spectra were recorded at 298 K on JEOL Eclipse 500 spectrometer (^{1}H: 500.16 MHz; ^{13}C: 125.77 MHz). Infrared spectra were recorded on a JASCO FT-IR 410 spectrometer operating at 2 cm^{-1} spectral resolution. IR spectroscopic data were collected using KBr pellets or mulls between NaCl plates prepared using solid material and Nujol or on a thin layer of solution (compound dissolved in CH_2Cl_2) placed between NaCl plates. Herein, we use abbreviations based on IUPAC guidelines, that is, υ for frequency and $\bar{\upsilon}$ for wavenumber. Elemental analyses were performed using a Perkin Elmer Series II CHNS/O analyzer. $AgSbF_6$ was purchased from Sigma-Aldrich and used as received. (SIDipp)AuCl and (IDipp)AuCl were synthesized using a reported procedure.[1]

[(SIDipp)Au(CO)][SbF$_6$], A mixture of (SIDipp)AuCl (0.175 g, 0.281 mmol) and $AgSbF_6$ (0.097 g, 0.281 mmol) in dichloromethane (*ca*. 10 mL) was stirred for 30 minutes to obtain an off-white precipitate. The resulting mixture was filtered through a pad of Celite *via* canula and the filtrate was concentrated to ~3 mL under reduced pressure. The mixture was cooled to -18 °C (using ice/acetone bath) and saturated with CO by bubbling CO through the solution for a few seconds. The solution was kept in refrigerator under CO atmosphere to obtain a colorless prism shaped crystals of [(SIDipp)Au(CO)][SbF_6] (0.124 g, 52 % yield): ^{1}H NMR (CD_2Cl_2, 500.16 MHz, 298 K): δ 7.51 (t, 2H, $^{3}J_{HH}$ = 8 Hz, $C_6\underline{H}_3$), 7.32 (d, 4H, $^{3}J_{HH}$ = 8 Hz, $C_6\underline{H}_3$), 4.28 (s, 4H, $C\underline{H}_2$), 2.99 (sept, 4H, $^{3}J_{HH}$ = 7 Hz, $C\underline{H}(CH_3)_2$), 1.36 (d, 12H, $^{3}J_{HH}$ = 7 Hz, $CH(C\underline{H}_3)_2$), 1.35 (d, 12H, $^{3}J_{HH}$ = 7 Hz, $CH(C\underline{H}_3)_2$). ^{13}C{^{1}H} NMR (CD_2Cl_2, 125.77 MHz, 298 K): δ 195.4 (Au–N$\underline{C}$N), 182.7 ($\underline{C}$O), 146.8, 132.0, 131.1, 125.2, 54.7 (N$\underline{C}H_2$), 29.1 ($\underline{C}H(CH_3)_2$), 25.5 ($CH(\underline{C}H_3)_2$), 23.8 ($CH(\underline{C}H_3)_2$). IR (KBr, selected bands) cm^{-1}: 2197 (CO), 2147 (^{13}CO isotopomer). IR (Nujol mull, selected bands) cm^{-1}: 2196 (CO), 2146 (^{13}CO isotopomer). Anal. Calcd. for $C_{28}H_{39}N_2OF_6AuSb\bullet 2CH_2Cl_2$: C, 35.25; H, 4.24; N, 2.74. Found: C, 34.68; H, 4.39; N, 2.72.

[(IDipp)Au(CO)][SbF$_6$], A mixture of (IDipp)AuCl (0.152 g, 0.245 mmol) and $AgSbF_6$ (0.084 g, 0.245 mmol) in dichloromethane (*ca*. 10 mL) was stirred for 30 minutes to obtain an off-white precipitate. The resulting mixture was filtered through a pad of Celite *via* canula and the filtrate was concentrated to ~3 mL under reduced pressure. The mixture was cooled to -18 °C (using ice/acetone bath) and saturated with CO by bubbling CO through the solution for a few seconds. The solution was kept in refrigerator under CO atmosphere to obtain [(IDipp)Au(CO)][SbF_6] as a colorless solid (0.121 g, 58 % yield): ^{1}H NMR (CD_2Cl_2, 500.16 MHz, 298 K): δ 7.62 (t, 2H, $^3J_{HH}$ = 8 Hz, $C_6\underline{H}_3$), 7.48 (s, 2H, C*H*N), 7.40 (d, 4H, $^3J_{HH}$ = 8 Hz, $C_6\underline{H}_3$), 2.41 (sept, 4H, $^3J_{HH}$ = 7 Hz, C*H*$(CH_3)_2$), 1.28 (d, 12H, $^3J_{HH}$ = 7 Hz, CH(C$\underline{H}_3)_2$), 1.23 (d, 12H, $^3J_{HH}$ = 7 Hz, CH(C$\underline{H}_3)_2$). ^{13}C{^{1}H} NMR (CD_2Cl_2, 125.77 MHz, 298 K): δ 182.9 (*C*O), 174.6 (Au–N*C*N), 145.7, 132.2, 131.8, 126.0, 124.9, 29.0 (*C*H$(CH_3)_2$), 24.8 (CH(*C*$H_3)_2$), 23.8 (CH(*C*$H_3)_2$). IR (KBr, selected bands) cm^{-1}: 2193 (CO), 2143 (^{13}CO isotopomer). IR (Nujol mull) cm^{-1}: 2192 (CO), 2142 (^{13}CO isotopomer).

X-ray crystallographic data:

A suitable crystal covered with a layer of paratone-N oil, was selected and mounted with in a cryo-loop and immediately placed in the low-temperature nitrogen stream. The X-ray intensity data were measured at 100(2) K on a Bruker Smart APEX CCD area detector system equipped with a Oxford Cryosystems 700 Series cooler, a graphite monochromator, and a Mo Kα fine-focus sealed tube (λ= 0.71073 Å).[2] The data frames were integrated with the Bruker SAINT-Plus software package.[3] Data were corrected for absorption effects using the multi-scan technique (SADABS). Structures were solved and refined using Bruker SHELXTL (Version 6.14) software package.[4] All the non hydrogen atoms were refined anisotropically. Hydrogen atoms were placed at calculated positions and refined riding on the corresponding carbons. The CCDC 807798 contains the supplementary crystallographic data. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge, CB2 1EZ, UK).

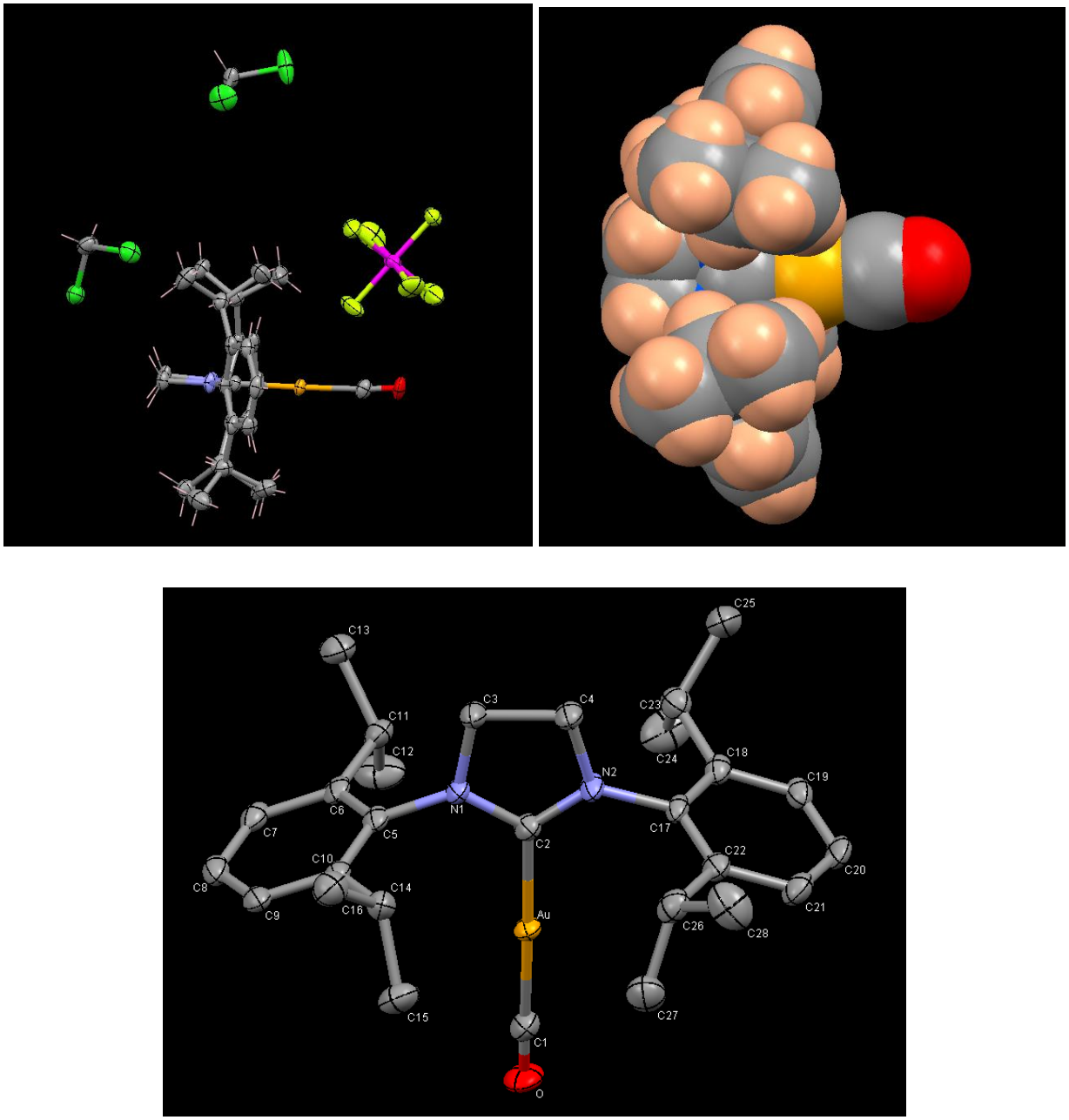


Figure S1. Views showing the (i) asymmetric unit of $[(SIDipp)Au(CO)][SbF_6]\bullet 2CH_2Cl_2$, space-filling diagram of $[(SIDipp)Au(CO)]^+$ moiety, and (iii) atom numbering scheme of $[(SIDipp)Au(CO)]^+$ moiety

Table S1. Crystal data and structure refinement for [(SIDipp)Au(CO)][SbF_6]•2CH_2Cl_2

Identification code	dias712_0m	
Empirical formula	C30 H42 Au Cl4 F6 N2 O Sb	
Formula weight	1021.17	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Cc	
Unit cell dimensions	a = 22.123(3) Å	α= 90°.
	b = 10.7279(14) Å	β= 122.014(1)°.
	c = 18.668(3) Å	γ = 90°.
Volume	3756.6(9) $Å^3$	
Z	4	
Density (calculated)	1.806 Mg/m^3	
Absorption coefficient	4.960 mm^{-1}	
F(000)	1984	
Crystal size	0.42 x 0.18 x 0.10 mm^3	
Theta range for data collection	2.17 to 27.00°.	
Index ranges	-28<=h<=28, -13<=k<=13, -23<=l<=23	
Reflections collected	14835	
Independent reflections	7294 [R(int) = 0.0202]	
Completeness to theta = 27.00°	99.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.6368 and 0.2298	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7294 / 2 / 414	
Goodness-of-fit on F^2	1.048	
Final R indices [I>2sigma(I)]	R1 = 0.0271, wR2 = 0.0677	
R indices (all data)	R1 = 0.0282, wR2 = 0.0687	
Absolute structure parameter	-0.004(3)	
Largest diff. peak and hole	2.364 and -1.511 e.$Å^{-3}$	

Table S2. Bond lengths [Å] and angles [°] for [(SIDipp)Au(CO)][SbF_6]•2CH_2Cl_2.

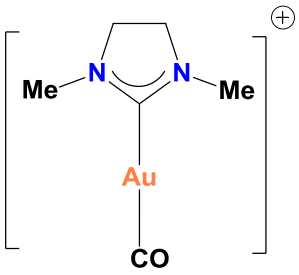
Au-C(1)	1.971(5)	C(6)-C(11)	1.513(7)
Au-C(2)	2.020(4)	C(7)-C(8)	1.389(7)
N(1)-C(2)	1.334(5)	C(7)-H(7)	0.9500
N(1)-C(5)	1.430(6)	C(8)-C(9)	1.374(7)
N(1)-C(3)	1.488(6)	C(8)-H(8)	0.9500
N(2)-C(2)	1.310(6)	C(9)-C(10)	1.397(6)
N(2)-C(17)	1.450(5)	C(9)-H(9)	0.9500
N(2)-C(4)	1.493(5)	C(10)-C(14)	1.529(7)
O-C(1)	1.110(6)	C(11)-C(13)	1.528(7)
C(3)-C(4)	1.548(6)	C(11)-C(12)	1.539(7)
C(3)-H(3A)	0.9900	C(11)-H(11)	1.0000
C(3)-H(3B)	0.9900	C(12)-H(12A)	0.9800
C(4)-H(4A)	0.9900	C(12)-H(12B)	0.9800
C(4)-H(4B)	0.9900	C(12)-H(12C)	0.9800
C(5)-C(6)	1.404(6)	C(13)-H(13A)	0.9800
C(5)-C(10)	1.411(7)	C(13)-H(13B)	0.9800
C(6)-C(7)	1.411(6)	C(13)-H(13C)	0.9800

C(14)-C(16)	1.527(7)	C(25)-H(25A)	0.9800
C(14)-C(15)	1.538(7)	C(25)-H(25B)	0.9800
C(14)-H(14)	1.0000	C(25)-H(25C)	0.9800
C(15)-H(15A)	0.9800	C(26)-C(27)	1.519(8)
C(15)-H(15B)	0.9800	C(26)-C(28)	1.538(9)
C(15)-H(15C)	0.9800	C(26)-H(26)	1.0000
C(16)-H(16A)	0.9800	C(27)-H(27A)	0.9800
C(16)-H(16B)	0.9800	C(27)-H(27B)	0.9800
C(16)-H(16C)	0.9800	C(27)-H(27C)	0.9800
C(17)-C(22)	1.397(7)	C(28)-H(28A)	0.9800
C(17)-C(18)	1.405(7)	C(28)-H(28B)	0.9800
C(18)-C(19)	1.393(7)	C(28)-H(28C)	0.9800
C(18)-C(23)	1.533(7)	Sb-F(2)	1.855(3)
C(19)-C(20)	1.378(8)	Sb-F(3)	1.862(4)
C(19)-H(19)	0.9500	Sb-F(1)	1.869(3)
C(20)-C(21)	1.389(9)	Sb-F(6)	1.875(4)
C(20)-H(20)	0.9500	Sb-F(4)	1.881(3)
C(21)-C(22)	1.405(7)	Sb-F(5)	1.889(4)
C(21)-H(21)	0.9500	C(29)-Cl(1)	1.739(5)
C(22)-C(26)	1.532(8)	C(29)-Cl(2)	1.749(6)
C(23)-C(24)	1.518(7)	C(29)-H(29A)	0.9900
C(23)-C(25)	1.535(7)	C(29)-H(29B)	0.9900
C(23)-H(23)	1.0000	C(30)-Cl(3)	1.755(6)
C(24)-H(24A)	0.9800	C(30)-Cl(4)	1.781(6)
C(24)-H(24B)	0.9800	C(30)-H(30A)	0.9900
C(24)-H(24C)	0.9800	C(30)-H(30B)	0.9900

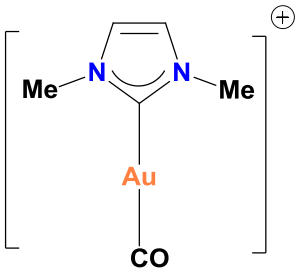
C(1)-Au-C(2)	177.7(2)	C(8)-C(7)-C(6)	120.7(4)
C(2)-N(1)-C(5)	126.0(4)	C(8)-C(7)-H(7)	119.6
C(2)-N(1)-C(3)	112.0(4)	C(6)-C(7)-H(7)	119.6
C(5)-N(1)-C(3)	121.9(3)	C(9)-C(8)-C(7)	120.9(4)
C(2)-N(2)-C(17)	125.2(4)	C(9)-C(8)-H(8)	119.6
C(2)-N(2)-C(4)	112.7(3)	C(7)-C(8)-H(8)	119.6
C(17)-N(2)-C(4)	122.1(3)	C(8)-C(9)-C(10)	121.2(5)
O-C(1)-Au	177.0(5)	C(8)-C(9)-H(9)	119.4
N(2)-C(2)-N(1)	110.8(4)	C(10)-C(9)-H(9)	119.4
N(2)-C(2)-Au	127.3(3)	C(9)-C(10)-C(5)	117.4(4)
N(1)-C(2)-Au	121.9(3)	C(9)-C(10)-C(14)	120.5(4)
N(1)-C(3)-C(4)	102.2(3)	C(5)-C(10)-C(14)	122.1(4)
N(1)-C(3)-H(3A)	111.3	C(6)-C(11)-C(13)	110.9(4)
C(4)-C(3)-H(3A)	111.3	C(6)-C(11)-C(12)	111.2(4)
N(1)-C(3)-H(3B)	111.3	C(13)-C(11)-C(12)	111.0(4)
C(4)-C(3)-H(3B)	111.3	C(6)-C(11)-H(11)	107.9
H(3A)-C(3)-H(3B)	109.2	C(13)-C(11)-H(11)	107.9
N(2)-C(4)-C(3)	101.9(3)	C(12)-C(11)-H(11)	107.9
N(2)-C(4)-H(4A)	111.4	C(11)-C(12)-H(12A)	109.5
C(3)-C(4)-H(4A)	111.4	C(11)-C(12)-H(12B)	109.5
N(2)-C(4)-H(4B)	111.4	H(12A)-C(12)-H(12B)	109.5
C(3)-C(4)-H(4B)	111.4	C(11)-C(12)-H(12C)	109.5
H(4A)-C(4)-H(4B)	109.2	H(12A)-C(12)-H(12C)	109.5
C(6)-C(5)-C(10)	122.8(4)	H(12B)-C(12)-H(12C)	109.5
C(6)-C(5)-N(1)	118.9(4)	C(11)-C(13)-H(13A)	109.5
C(10)-C(5)-N(1)	118.3(4)	C(11)-C(13)-H(13B)	109.5
C(5)-C(6)-C(7)	117.0(4)	H(13A)-C(13)-H(13B)	109.5
C(5)-C(6)-C(11)	122.9(4)	C(11)-C(13)-H(13C)	109.5
C(7)-C(6)-C(11)	120.0(4)	H(13A)-C(13)-H(13C)	109.5

H(13B)-C(13)-H(13C)	109.5
C(16)-C(14)-C(10)	111.2(4)
C(16)-C(14)-C(15)	110.8(4)
C(10)-C(14)-C(15)	110.0(4)
C(16)-C(14)-H(14)	108.3
C(10)-C(14)-H(14)	108.3
C(15)-C(14)-H(14)	108.3
C(14)-C(15)-H(15A)	109.5
C(14)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(14)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(14)-C(16)-H(16A)	109.5
C(14)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(14)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(22)-C(17)-C(18)	123.2(4)
C(22)-C(17)-N(2)	118.6(4)
C(18)-C(17)-N(2)	118.2(4)
C(19)-C(18)-C(17)	117.1(5)
C(19)-C(18)-C(23)	120.6(5)
C(17)-C(18)-C(23)	122.3(4)
C(20)-C(19)-C(18)	121.3(6)
C(20)-C(19)-H(19)	119.3
C(18)-C(19)-H(19)	119.3
C(19)-C(20)-C(21)	120.6(5)
C(19)-C(20)-H(20)	119.7
C(21)-C(20)-H(20)	119.7
C(20)-C(21)-C(22)	120.7(5)
C(20)-C(21)-H(21)	119.7
C(22)-C(21)-H(21)	119.7
C(17)-C(22)-C(21)	117.1(5)
C(17)-C(22)-C(26)	123.1(4)
C(21)-C(22)-C(26)	119.9(5)
C(24)-C(23)-C(18)	111.4(4)
C(24)-C(23)-C(25)	111.5(5)
C(18)-C(23)-C(25)	110.1(4)
F(3)-Sb-F(4)	89.5(2)
F(1)-Sb-F(4)	88.89(14)
F(6)-Sb-F(4)	90.48(16)
F(2)-Sb-F(5)	90.3(2)
F(3)-Sb-F(5)	178.2(2)
F(1)-Sb-F(5)	89.82(16)
F(6)-Sb-F(5)	88.4(2)
F(4)-Sb-F(5)	88.85(18)
Cl(1)-C(29)-Cl(2)	113.1(3)
Cl(1)-C(29)-H(29A)	109.0
Cl(2)-C(29)-H(29A)	109.0
Cl(1)-C(29)-H(29B)	109.0
Cl(2)-C(29)-H(29B)	109.0
H(29A)-C(29)-H(29B)	107.8
Cl(3)-C(30)-Cl(4)	111.0(3)
Cl(3)-C(30)-H(30A)	109.4
C(24)-C(23)-H(23)	107.9
C(18)-C(23)-H(23)	107.9
C(25)-C(23)-H(23)	107.9
C(23)-C(24)-H(24A)	109.5
C(23)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5
C(23)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
C(23)-C(25)-H(25A)	109.5
C(23)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5
C(23)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5
C(27)-C(26)-C(22)	111.1(5)
C(27)-C(26)-C(28)	110.0(5)
C(22)-C(26)-C(28)	111.0(5)
C(27)-C(26)-H(26)	108.2
C(22)-C(26)-H(26)	108.2
C(28)-C(26)-H(26)	108.2
C(26)-C(27)-H(27A)	109.5
C(26)-C(27)-H(27B)	109.5
H(27A)-C(27)-H(27B)	109.5
C(26)-C(27)-H(27C)	109.5
H(27A)-C(27)-H(27C)	109.5
H(27B)-C(27)-H(27C)	109.5
C(26)-C(28)-H(28A)	109.5
C(26)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
C(26)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
F(2)-Sb-F(3)	91.3(2)
F(2)-Sb-F(1)	91.12(15)
F(3)-Sb-F(1)	90.94(17)
F(2)-Sb-F(6)	89.49(17)
F(3)-Sb-F(6)	90.8(2)
F(1)-Sb-F(6)	178.2(2)
F(2)-Sb-F(4)	179.2(2)
Cl(4)-C(30)-H(30A)	109.4
Cl(3)-C(30)-H(30B)	109.4
Cl(4)-C(30)-H(30B)	109.4
H(30A)-C(30)-H(30B)	108.0

Table S3. Results from Computational Study, Bond lengths [Å] and Frequencies (cm^{-1})



[(SIMe)Au(CO)]$^+$ (4)



[(IMe)Au(CO)]$^+$ (5)

molecule	**d(C-O)**	$\bar{\nu}_{CO}$	**metal-CO**	
CO	1.1345	2200.5313		
[Au-CO]$^+$	1.1233	2297.0035	1.9404	
4	1.1272	2260.8110	1.9964	
5	1.1278	2255.3439	1.9889	
[Na-CO]$^+$	1.1233	2329.039	2.0000	fixed to 2.0
[Li-CO]$^+$	1.1218	2319.564	2.0000	fixed to 2.0

FREQUENCIES - original compared to SCALED
scaling = 0.974 = 2143/2200.53, chosen to match CO

molecule	**original**	**scaled**
CO	2200.5313	2143
[Au-CO]$^+$	2297.0035	2237
4	2260.8110	2201
5	2255.3439	2196

Table S3. Comparison of vibrational ($\bar{\upsilon}_{CO}$, cm^{-1}), structural (bond distances, Å), and NMR spectroscopic data (chemical shifts, ppm) for several gold(I) carbonyls.

Compound	$\bar{\upsilon}_{CO}$, **cm^{-1}**	**Au−CO, Å**	**C−O, Å**	**^{13}CO**	**ref.**
CO	2143		1.12822(7)	184 (CD_2Cl_2)	[5,6,7]
$[Au(CO)]^+$	2237				[8]
$[HB(3,5\text{-}(CF_3)_2Pz)_3]Au(CO)$	2144	1.862(9)	1.113(11)	173 ($CDCl_3$)	[9]
Au(CO)Cl	2162[a]	1.93(2)	1.11(3)	172 (CD_2Cl_2)	[10,11]
Au(CO)Br	2153[b]			174 (CD_2Cl_2)	[10]
$Au_2(CO)Cl_4$	2180[c]			171 (CD_2Cl_2)	[10,12]
$Au(CO)SO_3F$	2196			162 (HSO_3F)	[13,15]
$[(SIDipp)Au(CO)][SbF_6]$	2197	1.971(5)	1.110 (6)	183 (CD_2Cl_2)	this work
$[(IDipp)Au(CO)][SbF_6]$	2193			183 (CD_2Cl_2)	this work
$[Au(CO)][OTeF_5]$	2179				[14]
$[Au(CO)_2][Sb_2F_{11}]$	2217			174 (HSO_3F)	[15]
$[Au(CO)_2][UF_6]$	2200				[16]

[a] In CH_2Cl_2 [b] In *sym*-dibromoethane [c] In $SOCl_2$.

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