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Communications

Peri-Diaurated Naphthalene: Synthesis and Reactions of a New Class of Organogold(I) Complexes Containing Bridging, Dianionic Naphthalenediyl Ligands

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Summary: The dinuclear gold(I) compound $[Au_2(\mu\text{-}C_{10}H_6)\{\mu\text{-}Ph_2P(CH_2)_2PPh_2\}]$ *was prepared in high yield by the reaction of 1,8-bis(trimethylstannyl)naphthalene with* $[Au_2Cl_2(\mu\text{-}Ph_2P(CH_2)_2PPh_2)]$*. Oxidative addition with halogens results in the formation of the dihalodigold(II) complexes* $[Au_2(X)_2(\mu\text{-}C_{10}H_6)\{\mu\text{-}Ph_2P(CH_2)_2PPh_2\}]$ *(X = Cl, Br, I) containing a gold−gold bond.*

Dinuclear gold(I) complexes in which two gold atoms are bridged by a pair of suitable ligands have been known and studied for a number of years. These types of compounds can be broadly classified into three main categories: neutral or cationic complexes of the type $[Au_2(\mu\text{-E-E})_2]$ (E-E = neutral or monoanionic bidentate donor ligands. including $Ph_2P(CH_2)PPh_2$,[1−3] dithiocarbamates,[4−6] methylenethiophosphinate,[7] and (2-pyridyl)dimethylphosphine[8]), the bis(ylide) derivatives $[Au_2(\mu\text{-}\{(CH_2)_2PR_2\})_2]$,[9,10] and the cycloaurated complexes $[Au_2(\mu\text{-C-E})_2]$ (C-E = cyclometalated phosphine or arsine).[11] In addition, there are examples of heterobridged dinuclear gold(I) complexes in which there are two different bridging ligands (from the categories mentioned above) in the same molecule.[12−18] One class of

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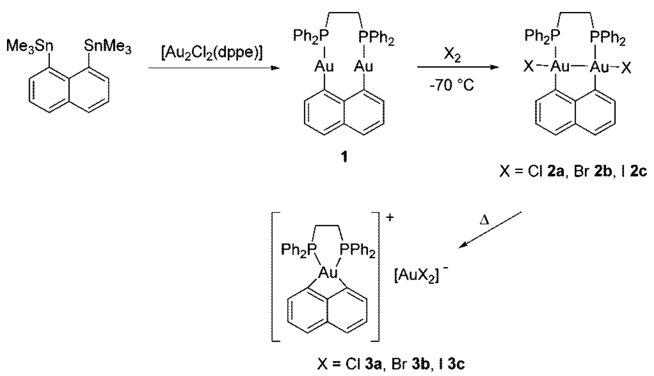
Scheme 1. Preparation and Reactions of Complex 1



dinuclear gold(I) complexes that has hitherto been unknown consists of derivatives of the type [Au_2(μ-C-C)(μ-E-E)], where C-C represents a dianionic carbon-only ligand and E-E a neutral, bidentate ligand. Given the required geometry and bite angle to bridge two metal atoms, the 1,8-naphthalenediyl dianion, $C_{10}H_6^{2-}$, seemed a suitable choice for our purpose. Indeed, there are prior examples of metal compounds in which 1,8-$C_{10}H_6^{2-}$ adopts a bridging coordination mode. These include derivatives of Li,[19] Mg,[20] Ge,[21] Sn,[22] Ga,[23] and Hg.[24,25] In the mercury derivative [Hg_2(μ-$C_{10}H_6$)$_2$],[24] the two Hg atoms are held 2.797(1) Å apart, a distance which is close to that typically also observed in binuclear gold complexes. We therefore attempted to prepare the binuclear gold(I) complex [Au_2(μ-$C_{10}H_6$){μ-$Ph_2P(CH_2)_2PPh_2$}] and to study its chemistry. Some preliminary results of our studies are communicated herein. The desired dinuclear gold(I) complex [Au_2(μ-$C_{10}H_6$){μ-$Ph_2P(CH_2)_2PPh_2$}] (**1**), containing both bridging 1,8-$C_{10}H_6^{2-}$ and $Ph_2P(CH_2)_2PPh_2$ (dppe) ligands, was obtained in 84% yield from the reaction of 1,8-bis(trimethylstannyl)naphthalene with [Au_2Cl_2(μ-$Ph_2P(CH_2)_2PPh_2$)] in dichloromethane (Scheme 1).[35] Alternatively, the complex can also be prepared from the reaction of 1,8-naphthalenediboronic acid with [Au_2Cl_2(μ-$Ph_2P(CH_2)_2$-PPh_2)] in the presence of base.[26]

Complex **1** was isolated as a pale yellow air- and moisture-stable solid, which is soluble in halogenated solvents and acetone but insoluble in diethyl ether and hexane. The ^{31}P NMR spectrum of **1** shows a singlet resonance, suggesting the presence of equivalent phosphorus atoms in the molecule. The MALDI-mass spectrum shows a peak at *m*/*z* 918, corresponding to the molecular ion peak of the compound, in addition to peaks with higher *m*/*z* values due to formation of various adducts, typically observed in mass spectra of gold(I) compounds. The proposed structure of complex **1** was confirmed by an X-ray diffraction study (Figure 1).[36]

The complex crystallizes in the space group $P2_1/n$ and contains three independent molecules of **1** in the unit cell. Each molecule of **1** consists of a pair of gold atoms bridged by a $C_{10}H_6^{2-}$ group and a molecule of $Ph_2P(CH_2)_2PPh_2$. The coordination geometry about gold is almost linear, with C−Au−P angles ranging from 169.60(16) to 175.22(19)°. The Au···Au distances range from 2.8868(3) to 2.9239(3) Å, which are greater than those found in the eight-membered gold(I) dimers [Au_2(μ-2-$C_6H_4PPh_2$)$_2$] (Au···Au = 2.8594(3) Å)[27] and [Au_2(μ-C_6H_3-2-PPh_2-6-Me)$_2$] (Au···Au = 2.861(2) Å)[27] but less than those of the ten-membered gold(I) dimer [Au_2(μ-2-$C_6H_4CH_2PPh_2$)$_2$] (Au···Au = 3.0035(9) Å).[28] The average Au−P and Au−C distances in **1** (2.293(5) and 2.060(5) Å) are similar to those observed in other organometallic gold(I) phosphine complexes. In general, there is good agreement of all equivalent bond lengths among the three molecules. Tables S1 and S2 (Supporting Information) show a comparison and statistical analysis of selected equivalent bonds. The highest degree of conservation is observed for the naphthyl rings. These exhibit only slight twisting (atoms deviate between 0.003 and 0.097 Å from the least-squares planes), and the sum of the bond angles surrounding the carbon atom to which the gold atoms are attached falls into the narrow range between 359.2(9) and 360.0(9)°. The largest variance is observed for the Au···Au distance, which correlates with the C′−C−Au angle, with C′ being the bridging carbon atom of the naphthyl ring. The dihedral angles of the ethyl bridge of the chelating phosphine ligands deviate substantially from a gauche conformation; however, the resulting P···P distance does not correlate with the corresponding dihedral angle. Instead, the C−C−P bond angles also deviate from the ideal 109.5°. This flexibility of some parts of the molecule (most likely crystal-packing effects will contribute significantly to the conformation of the dppe ligand) result in a twisting of the whole molecule expressed through the dihedral angle between the intramolecular C−Au−P vectors. This angle varies between 6.3° for the almost planar molecule (Au1 and Au2) and 28.6° (Au5 and Au6).

In dichloromethane, complex **1** absorbs strongly in the UV region, with the absorption bands (Figure 2) being attributed to an admixture of spin-allowed metal-perturbed intraligand ^{1}IL (naphthyl and phosphine) and metal-centered transitions.[29] The aerated solution is weakly emissive upon photoexcitation at room temperature (Figure 2). However, the intense green emission of the solution is "turned on" under deoxygenated conditions (Figure 2) with a lifetime in the microsecond range (21.8 μs). The emission is vibronically structured with vibra-

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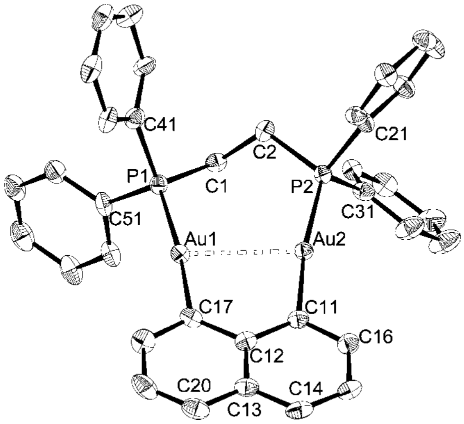

Figure 1. Molecular structure of one of the independent molecules of complex **1**. Ellipsoids are given at the 50% probability level, and H atoms as well as CH_2Cl_2 molecules of solvation have been omitted for clarity. Selected bond distances (Å) and angles (deg) of one of the molecules: Au1···Au2 = 2.9239(3), P1−Au1 = 2.2988(16), P2−Au2 = 2.2908(16), C17−Au1 = 2.062(6), C11−Au2 = 2.063(6), C1−P1 = 1.829(6), C2−P2 = 1.819(6), C1−C2 = 1.547(9), C11−C12 = 1.435(9), C12−C13 = 1.434(9), C13−C14 = 1.418(9), C14−C15 = 1.371(10), C15−C16 = 1.415(9), C16−C11 = 1.387(9); C11−Au2−P2 = 171.94(18), C17−Au1−P1 = 174.52(18), C2−P2−Au2 = 119.1(2), C1−P1−Au1 = 116.1(2), C2−C1−P1 = 114.3(4), C1−C2−P2 = 116.1(4), C12−C11−Au2 = 123.7(4), C16−C11−Au2 = 117.8(5), C16−C11−C12 = 118.5(6), C12−C17−Au1 = 124.9(4), C18−C17−Au1 = 117.4(5), C18−C17−C12 = 117.7(6).

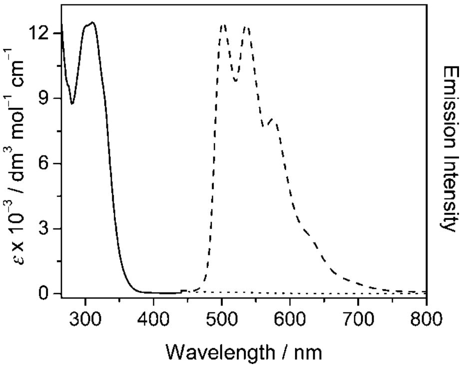

Figure 2. Electronic absorption spectrum (—) and emission spectra under aerated (···) and deoxygenated (- - -) conditions of complex **1** in dichloromethane at room temperature.

tional progressional spacings that are typically observed in naphthalene-based systems.[30] The complex in the solid state at room temperature and at 77 K also shows structured emission with long emission lifetimes, 7.6 and 50.9 μs, respectively, with a slight red shift of ca. 10 nm in wavelength. In view of the long emission lifetimes, large Stokes shifts, severe oxygen quenching, and vibronic structures, the intense emission is assigned to originate mainly from spin-forbidden ^{3}IL (naphthyl) states, stemming from the large spin−orbit coupling associated with the presence of the heavy gold(I) atoms. The assignment is supported by the fact that the ^{3}IL emission of naphthalene-based compounds occurs at similar energies.[30] Although the contribution from metal-centered d−p/d−s excited states modified by Au(I)···Au(I) interactions cannot be completely excluded, given the short intramolecular Au(I)···Au(I) distance in the current system as revealed by X-ray structure determination (vide supra), we do favor the assignment of a ^{3}IL (naphthyl) origin with its highly structured emission band.

Treatment of **1** with 1 equiv of $PhICl_2$, Br_2, or I_2 in dichloromethane at ca. −70 °C gives a red (**2a**), brown (**2b**), or rust red (**2c**) solution, out of which solid compounds can be isolated by precipitation with hexane at low temperature.[37] These colored solids are stable to air and moisture and show singlet $^{31}P\{^1H\}$ NMR resonances indicative of equivalent phosphorus atoms. The Raman spectra of the chloro- and bromo derivatives show Au−Au stretching vibrations at 174 and 135 cm^{-1}, similar to those observed for the bis(ylide) dihalodigold(II) complexes $[Au_2(X)_2(\mu\text{-}\{(CH_2)_2PPh_2\})_2]$ (ν(Au−Au) 162 and 132 cm^{-1} for X = Cl, Br, respectively).[31] On the basis of these data, we formulate these colored solids as the dihalodigold(II) complexes $[Au_2(X)_2(\mu\text{-}C_{10}H_6)\{\mu\text{-}Ph_2P(CH_2)_2PPh_2\}]$ (X = Cl (**2a**), Br (**2b**), I (**2c**)). Although the oxidation state +2 is very rare for mononuclear gold compounds, it is by now well established for dinuclear gold complexes.[11] Among the known cyclometalated aryldihalodigold(II) complexes, there are examples of various isomerization processes occurring in solution.[27,28,32,33] In the present case too, solutions of **2** undergo a change of color over a period of hours at room temperature; the half-life of **2a** in CH_2Cl_2 is about 1.5 h, as estimated by UV−vis spectroscopy.[38] Out of these solutions yellow (**3a**), pale yellow (**3b**), and gray (**3c**) solid products were isolated in good yields. These compounds show singlet resonances in their $^{31}P\{^1H\}$ NMR spectra (δ(P) 32.6 (**3a**), 35.2 (**3b**), 38.8 (**3c**)), indicative of equivalent phosphorus atoms. The electrospray mass spectra of these materials all show a strong peak at *m/z* 721.15 in positive ion mode, which corresponds to loss of AuX_2 from the parent dihalodigold(II) complex. In negative ion mode signals due to $[AuX_2]^-$ anions can be observed. These data suggest that the isolated complexes are mixed-valence Au(I)/Au(III) salts consisting of Au(III) cations containing chelating $C_6H_{10}{}^{2-}$ and $Ph_2P(CH_2)_2PPh_2$ ligands as well as $[AuX_2]^-$ anions. While we so far have not been able to unambiguously confirm this structure by X-ray diffraction, there is precedence for both the chelating mode of the naphthalenediyl dianion in transition-

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(35) Synthesis of **1**: to a solution of 1,8-bis(trimethylstannyl)naphthalene (277 mg, 0.610 mmol) in dichloromethane (5 mL) was added $[Au_2Cl_2(\mu\text{-}Ph_2P(CH_2)_2PPh_2)]$ (500 mg, 0.579 mmol). The mixture was stirred overnight at room temperature. Hexane (5 mL) was subsequently added to the solution, and the resulting solid was isolated by filtration and washed with boiling hexane to give 447 mg (84%) of complex **1** as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.63 (d, J = 10.7 Hz, 4 H, PCH_2CH_2P), 7.38 (dd, J = 8.1/6.6 Hz, 2 H, naph-H3), 7.42−7.48 (m, 12 H, *m*-PPh_2, *p*-PPh_2), 7.69 (dd, J = 8.1/1.0 Hz, 2 H, naph-H2), 7.79−7.85 (m, 8 H, *o*-PPh_2), 7.84 (d, J = 6.6 Hz, 2 H, naph-H4). $^{31}P\{^1H\}$NMR (161.98 MHz, $CDCl_3$): δ 38.3 (s). MALDI-MS (*m/z*): 595 $[Au(dppe)]^+$, 918 $[M]^+$, 1115 $[M + Au]^+$, 1513 $[M + Au(dppe)]^+$, 2033 $[2 M + Au]^+$. Anal. Calcd for $C_{36}H_{30}Au_2P_2$: C,47.07; H, 3.29. Found: C, 47.11; H, 3.3.

(36) X-ray crystal structure analysis of **1**: $C_{37}H_{30}Au_2Cl_2P_2$, $M_r = 1001.38$, colorless plate, crystal size 0.15 × 0.12 × 0.04 mm, monoclinic, space group $P2_1/n$, $a = 22.5380(3)$ Å, $b = 11.1475(2)$ Å, $c = 39.7366(6)$ Å, $\beta = 92.7680(10)°$, $V = 9971.9(3)$ Å^3, $T = 100$ K, $Z = 12$, $D_{calcd} = 2.001$ g cm^{-3}, $\lambda = 0.710\,73$ Å, μ(Mo Kα) = 9.100 mm^{-1}, empirical absorption correction ($T_{min} = 0.74$, $T_{max} = 0.99$), Nonius KappaCCD diffractometer, $2.92 < \theta < 33.17°$, 218 901 measured reflections, 37 781 independent reflections, 24 867 reflections with $I > 2\sigma(I)$, structure solved by direct methods and refined by full-matrix least squares against F^2 to R1 = 0.053 ($I > 2\sigma(I)$), wR2 = 0.143, 1162 parameters, H atoms riding, $S = 1.054$, residual electron density 2.8/−3.7 e Å^{-3}.

(37) Oxidative addition reactions: a stirred solution of **1** (60 mg, 0.065 mmol) in dichloromethane (5 mL) at −78 °C was treated with an equimolar amount of $PhICl_2$, bromine, or iodine. The mixture was stirred for 30 min at ca. −65 °C. An approximately equal amount of hexane was then added, and the solution was concentrated under reduced pressure at or below −20 °C until the product began to precipitate. The solid was isolated by filtration, washed with hexane, and dried under vacuum. The complexes were obtained in yields of 60−90%. Characterization data for these compounds is given in the Supporting Information.

metal complexes of Pt(II) (isoelectronic with Au(III)), Rh(III), and Ir(III)[34] as well as the isomerization of dihalodigold(II) compounds to Au(I)/Au(III) salts.[28] Thus, the chemistry of complex **1** known so far is that it has similarities to both the bis(ylide) and the cyclometalated dimers. Oxidative addition with halogens occurs to give gold−gold-bonded Au(II) complexes. However, oxidative addition reactions with MeI or dibenzoyl peroxide were unsuccessful with **1**. As is the case for the cyclometalated complexes, the dihalodigold(II) derivatives **2a**−**c** are unstable in solution and isomerize to ionic Au(I)/Au(III) complexes. In this respect, **2a**−**c** behave more like the 10-membered-ring cyclometalated dihalodigold(II) derivatives $[Au_2(\mu\text{-}2\text{-}C_6H_4CH_2PPh_2)_2]$ and $[Au_2(\mu\text{-}2\text{-}CH_2C_6H_4PPh_2)_2]$, which isomerize to the salts $[Au(\kappa^2 C,P\text{-}2\text{-}C_6H_4CH_2PPh_2)_2][AuX_2]$ and $[Au(\kappa^2 C,P\text{-}2\text{-}CH_2C_6H_4PPh_2)_2][AuX_2]$, respectively.[28] Further studies of the reactivity of complex **1** and its derivatives are currently ongoing.

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Supporting Information Available: Text and tables containing full experimental and characterization details of all new compounds as well as a CIF file giving crystallographic details of complex **1**. This material is available free of charge via the Internet at http://pubs.acs.org.

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[38] The half-life was estimated by observing the decrease in absorbance of the peak due to **2a** at 320 nm over time.