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Gold and silver derivatives with the carborane-selenolate ligand $[B_{10}H_{11}C_2Se]^-$

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Received 2nd September 2003, Accepted 11th September 2003
First published as an Advance Article on the web 25th September 2003

The carborane-selenol $[(B_{10}H_{11}C_2)SeH]$ has been isolated and its anion employed as a ligand. Reaction of the ligand with [AuClL] (L = PPh_3, $AsPh_3$) (1 : 1 molar ratio) or $PPN[AuCl_2]$ (PPN = $N(PPh_3)_2$) (2 : 1 molar ratio) affords the mononuclear compounds $[Au(SeC_2B_{10}H_{11})L]$ or $PPN[Au(SeC_2B_{10}H_{11})_2]$, respectively. Dinuclear derivatives $[Au_2(SeC_2B_{10}H_{11})_2(\mu\text{-PP})]$ (PP = 1,1′-bis(diphenylphosphino)ferrocene (dppf), 1,2-bis(diphenylphosphino)-ethane (dppe)) are synthesised by reaction of the ligand with $[Au_2Cl_2(\mu\text{-PP})]$ or by reaction of $[Au(SeC_2B_{10}H_{11})(AsPh_3)]$ with the bidentate ligand. The selenolate acts in a bridging mode in $[M_2(\mu\text{-SeC}_2B_{10}H_{11})(PPh_3)_2]X_2$ (M = Au, X = ClO_4; M = Ag, X = TfO), obtained by treatment of the ligand with $[O\{Au(PPh_3)\}_3]ClO_4$ or by reaction with $[Ag(TfO)(PPh_3)]$ in 1 : 2 molar ratio, respectively. The crystal structure of $[Au(SeC_2B_{10}H_{11})(PPh_3)]$ has been determined by X-ray diffraction studies.

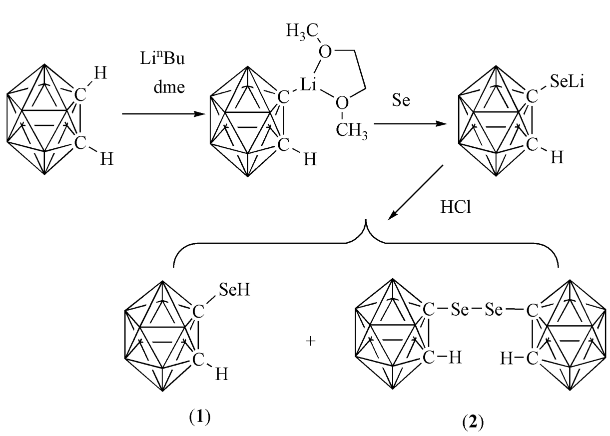
Introduction

There is a growing interest in the study and synthesis of gold selenolates because of their potential applications in the treatment of rheumatoid arthritis, in photography, or electronic microscope technology. The analyses of the gold complexes[1] described thus far, show mononuclear $[Au(SeR)(PPh_3)]$ (R = Ph, naphthyl),[2,3] dinuclear $[Au_2(SeR)(PR_3)_2]^+$ (R = Ph, C_6F_4Cl, CH_2Ph, naphthyl, 4-$NH_2C_6H_4$, 4-ClC_6H_4; PR_3 = PPh_3, PPh_2Me, $PPhMe_2$),[2–5] $[Au_2(SePh)_2(\mu\text{-PP})]$ (PP = dppm, dppe),[3] or heteronuclear $[\{Au(PPh_3)\}\{WCp(CO)_3\}\{\mu\text{-Se}(CH_2SiMe_3)\}]SbF_6$[6] complexes. The gold(III) compound with two [bispyrazine-2,3-diselenolato]$^{2-}$ ligands,[7] and the gold(II) derivative $[Au_2Cl(SePh)\{\mu\text{-}Ph_2P(CH_2)_2\}_2]$[8] have also been described. This brief overview reveals that the most represented oxidation state is +I, and also that this chemistry has been scarcely studied until now, in comparison with the related thiolate gold chemistry. Silver derivatives[2] include stoichiometries $[Ag\{SeC(SiMe_3)_3\}]_4$,[9] $(NMe_4)_2[Ag_4(SePh)_6]$,[10] $[Ag\{Se(2,4,6\text{-}^iPr_3C_6H_2)\}]_8$,[11] $[Ag_6\{\mu_3\text{-Se-}(4\text{-}ClC_6H_4)\}_4(\mu\text{-dppm})](PF_6)_2$,[12] $[Ag_4(Se^iPr)_4(dppm)_2]$,[13] and $[Ag_8(SeEt)_8(dppm)]_n$.[13] As commented above, the development of this area can provide compounds with interesting properties. These facts prompted us to introduce the properties of carborane clusters (derived both from the specific properties of the deltahedral borane clusters and on those of elemental boron itself), to gold and silver selenolate chemistry. Some publications have reported interesting Rh, Ir and Fe compounds[14–21] that incorporate the $[B_{10}H_{10}C_2Se_2]^{2-}$ unit, but the diselenolcarborane ligand has not been isolated. Herein, we present the synthesis and isolation of the moisture- and air-stable selenol $[(B_{10}H_{11}C_2)SeH]$. The reactivity of the corresponding selenolate ligand towards gold and silver derivatives is also explored. The crystal structure of $[Au(SeC_2B_{10}H_{11})(PPh_3)]$ has been determined by X-ray diffraction studies.

Discussion

Because of the disproportionation of monolithio-*o*-carborane, which leads to the undesired di-C-substituted product, the synthesis of mono-C-substituted *o*-carborane derivatives has been a difficult problem, as well as a topic of interest. In the literature three different methods have been developed with the aim of affording such species: (a) the use of a bulky ether as solvent (dimethoxyethane, dme)[22] which coordinates to the lithium atom and avoids the dilithiation of the carborane, (b) the protection of one position (much work has been done based on C-substituted silyl carboranes),[23] incorporation of the desired substituent and deprotection of the original C-position, (c) the reaction of $B_{10}H_{12}(CH_3CN)_2$ with the corresponding acetylene derivative R–C≡CH. In fact, this is the way in which *o*-carborane itself is prepared.[24]

The synthesis of $[(B_{10}H_{11}C_2)SeH]$ (**1**) has been carried out according to method (a) described above (Scheme 1), in a similar experiment to that from which $[(B_{10}H_{11}C_2)SH]$ is obtained.[22] The reaction of 1,2-dicarba-*closo*-dodecaborane in dme, with Li^nBu, followed by the addition of Se and HCl affords $[(B_{10}H_{11}C_2)SeH]$ (**1**) and $[(B_{10}H_{11}C_2)Se]_2$ (**2**). Compound **2** separates from the mixture upon addition of light petroleum, in which it is insoluble.



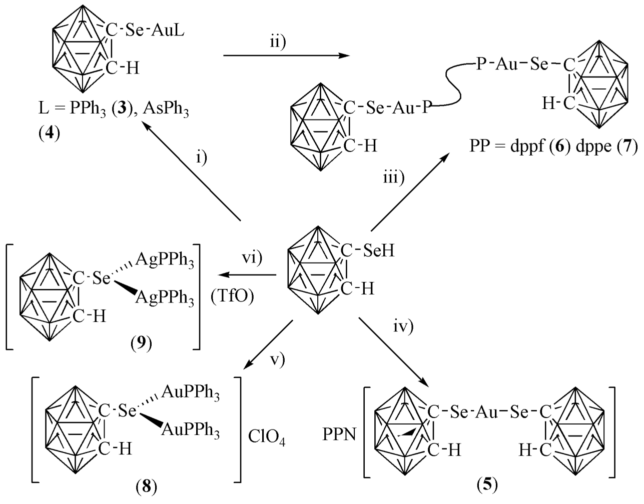
Scheme 1

Compound **1** is isolated as a yellow solid. In its IR spectra the ν(BH) vibrations appear as a broad band at about 2600 cm^{-1}. These bands are observed in the same region for all the

DOI: 10.1039/b310658f

compounds described below. A broad signal, corresponding to the hydrogen atoms of the carborane cage, between 1 and 3 ppm, is shown in its ^{1}H NMR spectrum, the resonance due to the Se–H proton appears at 2.54 ppm, and that due to the C–H hydrogen atom at 3.73 ppm. The ^{77}Se NMR spectrum shows one signal at 733 ppm. A phase-sensitive (^{1}H–^{77}Se) HMQC experiment was recorded and the transfer delay was set for a $^3J(^1\text{H}–^{77}\text{Se})$ of 50 Hz. The positive Liquid Secondary Ion Mass Spectrum (LSIMS(+)) shows the molecular peak at m/z = 233 (40%). No signal corresponding to the Se–H hydrogen is present in the ^{1}H NMR spectrum of **2**. The ^{77}Se NMR spectrum shows a singlet at 750 ppm. The peak in the LSIMS(+) spectrum at m/z = 445 (30%) shows an experimental isotopic distribution consistent with that calculated for the species $\{[(B_{10}H_{11}C)Se]_2\}^+$. A peak corresponding to the species $\{(B_{10}H_{11}C)Se_2\}^+$ at m/z = 302 (20%) is also present. These data and the behavior as a non-electrolyte in acetone solutions ($\Lambda_M = 1.6\ \Omega^{-1}$ cm^2 mol^{-1}) are consistent with the structure shown in Scheme 1 for compound **2**.

Reaction of **1** with chlorogold derivatives, in the presence of Na_2CO_3, affords mono- or di-nuclear complexes (Scheme 2). Thus, reaction with [AuClL] affords $[Au(SeC_2B_{10}H_{11})L]$ (L = PPh_3 (**3**), $AsPh_3$ (**4**)). The phosphorus atom of the PPh_3 ligand in **3** displays one signal at 36.8 ppm in the ^{31}P(^{1}H) NMR spectrum. Both ^{1}H NMR spectra are consistent with the deprotonation of the ligand. The selenide atoms display resonances in the ^{77}Se NMR spectrum shifted about 300 ppm (δ 453 ppm (**3**), 440 ppm (**4**)), to high field from that corresponding to the free ligand (**1**). The LSIMS(+) mass spectra show the molecular peak at m/z = 682 (10%, **3**), and 727 (5%, **4**), respectively.



Scheme 2 i) [AuClL}, Na_2CO_3; ii) L = $AsPh_3$, ½PP; iii) ½[Au_2Cl_2(μ-PP], Na_2CO_3; iv) ½PPN[$AuCl_2$], Na_2CO_3; v) [O{Au(PPh$_3$)}$_3$]ClO$_4$; vi) 2[Ag(TfO)(PPh$_3$)], Na_2CO_3.

The crystal structure of compound **3** has been determined by X-ray diffraction studies. The gold atom (Fig. 1) displays a slightly distorted linear geometry (P–Au–Se 175.08(3)°, Table 1). Close to ideal values are observed in $[Au(SC_2B_{10}H_{11})L]$[25] (L = $AsPh_3$, 178.34°, PPh_3, 179.70°). In **3** the molecules associate in pairs through gold ··· gold interactions of 3.3035(4) Å, but not in the $[Au(SC_2B_{10}H_{11})L]$ derivatives mentioned above. This could explain the distortion from linear geometry. Intermolecular Au ··· Au contacts of this type have been observed for some of the gold complexes with selenido containing ligands reported thus far, and the value found in **3** is among the shortest. The Au–P (2.2690(10) Å) and Au–Se (2.417(4) Å) bond distances are within the expected range, they resemble the shortest found in other selenolate gold(I) complexes.

Compound PPN$[Au(SeC_2B_{10}H_{11})_2]$ (**5**) is afforded from the reaction of **1** with PPN[$AuCl_2$] in 2 : 1 molar ratio. The spectroscopic data are consistent with the deprotonation of the ligands. The LSIMS(–) spectrum shows the anionic molecular peak at m/z = 641(60%).

Table 1 Bond lengths (Å) and angles (°) for compound **3**

Au–P	2.2690(10)	C(1)–C(2)	1.682(6)
Au–Se	2.4167(4)	P–C(21)	1.813(4)
Au–Au#1	3.3035(4)	P–C(11)	1.821(4)
Se–C(1)	1.940(4)	P–C(31)	1.821(4)
P–Au–Se	175.08(3)	C(21)–P–C(31)	105.65(17)
P–Au–Au#1	110.77(3)	C(11)–P–C(31)	107.04(18)
Se–Au–Au#1	73.740(10)	C(21)–P–Au	111.96(12)
C(1)–Se–Au	100.37(12)	C(11)–P–Au	112.89(14)
C(21)–P–C(11)	106.57(17)	C(31)–P–Au	112.25(12)

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

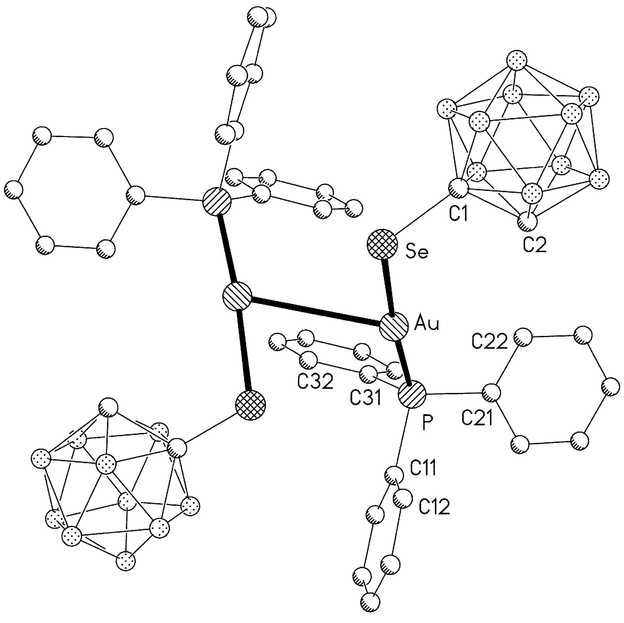


Fig. 1 Molecular structure of compound **3**. Radii are arbitrary. Hydrogen atoms have been omitted for clarity.

The use of the dinuclear derivatives [Au_2Cl_2(μ-PP)] affords the synthesis of $[Au_2(SeC_2B_{10}H_{11})_2(\mu\text{-PP})]$ (PP = dppf (**6**), dppe (**7**)). The compounds display only one signal in their ^{31}P(^{1}H) NMR spectra at 30.5 (**6**) and 33.3 (**7**) ppm. For both derivatives the LSIMS(+) mass spectra show the peak arising from the loss of one carborane ligand $\{M - SeC_2B_{10}H_{11}\}^+$ at m/z = 1171 (10%, **6**) and 1015 (100%, **7**). These compounds can be also synthesized by reaction of **4** with the diphosphines in 2 : 1 molar ratio.

The selenolate acts in a bridging mode in $[M_2(\mu\text{-SeC}_2B_{10}H_{11})(PPh_3)_2]X$ (M = Au, X = ClO_4 (**8**); M = Ag, X = TfO (**9**), prepared by addition of **1** to [O{Au(PPh$_3$)}$_3$]ClO$_4$ (molar ratio 1 : 1) or to [Ag(TfO)(PPh$_3$)] (molar ratio 1 : 2). Compound **8** shows the vibrations corresponding to the perchlorate anion (T_d) at about 1100 and 619 cm^{-1}, in **9** those corresponding to the trifluoromethanesulfonate anion arise at about 1270 ($\nu_{asym}(SO_3)$), 1225 ($\nu_{sym}(SO_3)$), 1151 ($\nu_{asym}(CF_3)$) and 1033 cm^{-1} ($\nu_{sym}(SO_3)$). The coordination of two "AuPPh$_3$" fragments in **8** is consistent with the ^{31}P{^{1}H} NMR data, the resonance due to the two triphenylphosphine ligands (δ 35.8 ppm) is shifted 1 ppm to higher field from that in **3**. The shift of these resonance to higher field in the ^{31}P{^{1}H} NMR spectrum as the nuclearity grows, has been observed in other gold selenide complexes.[26] The ^{31}P{^{1}H} NMR of **9** shows a broad resonance centred at 12.7 ppm when the experiment is carried out at 25°C. This signal is split into two doublets, centred at 11.4 ppm at −55°C, $J(^{109}\text{AgP})$ 558, $J(^{107}\text{AgP})$ 483 Hz. The cationic molecular peaks of compounds **8** and **9** are present in their LSIMS(+) mass spectra at m/z = 1141 (**8**, 5%), 962 (**9**, 1%).

Unfortunately no crystal suitable for X-ray studies has been obtained. It is possible that compound **9**, in the solid state, shows the structure of its analogous sulfur derivative $[Ag_2(SC_2B_{10}H_{11})(PPh_3)_2]TfO$[27] (Fig. 2), in which two "$Ag_2(SC_2B_{10}H_{11})(PPh_3)_2$" units are linked through two further Ag–S bonds. In addition, triflate oxygen atoms chelate the silver atoms. The centre of the molecule consists on a four-membered ring formed by alternating sulfur and silver atoms. The other two silver atoms are exocyclically bonded to the thiolate, which acts in a triply bridging mode.

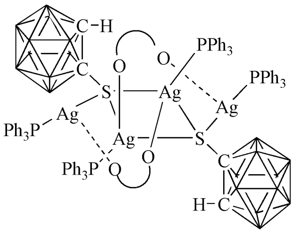


Fig. 2 Scheme of the molecular structure of $[Ag_2(SC_2B_{10}H_{11})(PPh_3)_2]$-TfO; TfO = trifluoromethanesulfonate.

Conclusions

The first carborane selenol $[(B_{10}H_{11}C_2)SeH]$ has been isolated as an stable solid by a conventional method, in which it is easily separated from undesired products. Its reactivity towards gold complexes has been studied and has led to the synthesis of the first gold carborane-selenolate complexes with different structural patterns.

Unlike the analogous $[Au(SC_2B_{10}H_{11})L]$ ($AsPh_3$, PPh_3), the structure of $[Au(SeC_2B_{10}H_{11})(PPh_3)]$ displays gold ··· gold interactions in the solid state. This interactions are probably responsible of a more distorted linear geometry towards the gold centre.

Experimental

Reagents

o-Carborane was purchased from Katchem Ltd., dppe and dppf from Aldrich. All were used as received. [AuClL] (L = PPh_3, $AsPh_3$),[28] $[Au_2Cl_2(\mu\text{-PP})]$ (PP = dppe,[29] dppf[30]), $[O\{Au(PPh_3)\}_3]ClO_4$,[28b] PPN[$AuCl_2$][31] and $[Ag(TfO)(PPh_3)]$[32] were synthesized according to literature methods.

General procedure

Infrared spectra were recorded on a Perkin-Elmer FT-IR Spectrum 1000 spectrophotometer, in the range 4000–400 cm^{-1}, using Nujol mulls between polyethylene sheets. Conductivities were measured in *ca.* 5×10^{-4} M acetone solutions with a Jenway 4010 conductimeter. C, H, N and S analysis were carried out with a Perkin-Elmer 240C microanalyser. Mass spectra were recorded on a VG Autospec using the LSIMS (+, −) techniques and nitrobenzyl alcohol as matrix and on a HP59987 A ELECTROSPRAY. ^1H, ^{19}F, ^{77}Se and ^{31}P NMR spectra were recorded on a Bruker ARX 300 or Varian Unity 300 in $CDCl_3$ solutions. Chemical shifts are quoted relative to $SiMe_4$ (^1H, external), $CFCl_3$ (^{19}F, external), H_3PO_4 (85%) (^{31}P, external) and Me_2Se_2 (^{77}Se, external).

Synthesis of the complexes

Synthesis of $[(B_{10}H_{11}C_2)SeH]$ (1). In a three-necked round-bottomed flask (100 mL) with a dinitrogen inlet/outlet and a dropping funnel was prepared a solution of 1,2-dicarba-*closo*-dodecaborane (1 mmol, 0.144 g) in 25 mL of 1,2-dimethoxyethane. The flask was cooled at 0 °C during the addition of 1 mmol of Li^{n}Bu (1.6 M, 0.625 mL). After 1 h of reaction Se powder (1 mmol, 0.079 g) was added. The resulting suspension was stirred for one day. The 1,2-dimethoxyethane was evaporated and diethyl ether (30 mL) and HCl (20 ml, 1 M) were then added. After 2 h of stirring the organic phase was dried by adding anhydrous $MgSO_4$, which was then filtered off. The solution was dried *in vacuo* and a mixture of **1** and **2** was obtained. Light petroleum (bp 40–60 °C) was added, and the suspension filtered. The solid was identified as **2**, compound **1** was isolated upon concentration of the filtrate. **1**: Yellow solid. Yield 40%. Λ_M = 0.8 Ω^{-1} cm^2 mol^{-1} (Found: C, 10.9; H, 5.25. $C_2H_{12}B_{10}Se$ requires C, 10.75; H, 5.4%). δ_H ($CDCl_3$) 3.48–1.19 (10H, m, $B_{10}H_{10}$), 3.73 (1H, s, CH), 2.54 (1H, s, SeH). **2**: Orange–brown solid. Yield 28%. Λ_M = 2 Ω^{-1} cm^2 mol^{-1} (Found: C, 10.70; H, 4.75. $C_4H_{22}B_{20}Se_2$ requires C, 10.8; H, 5.0%). δ_H ($CDCl_3$) 3.60–1.15 (20H, m, $B_{10}H_{10}$), 3.69 (2H, s, CH).

Synthesis of $[Au(SeC_2B_{10}H_{11})L]$ [L = PPh_3 (3), $AsPh_3$ (4)]. To a solution of **1** (0.1 mmol, 0.0223 g) in dichloromethane (20 mL), Na_2CO_3 and [AuClL] [0.1 mmol, L = PPh_3 0.0495 g; $AsPh_3$ 0.0538 g)] were added. The mixture was stirred for 30 min and the excess of Na_2CO_3 filtered off. Removal of solvent to *ca.* 5 mL and addition of *n*-hexane afforded a white solid. **3**: Yield 89%. Λ_M = 2.5 Ω^{-1} cm^2 mol^{-1} (Found: C, 35.50; H, 3.82. $C_{20}H_{26}AuB_{10}PSe$ requires C, 35.24; H, 3.82%). δ_H ($CDCl_3$) 3.60–1.20 (10H, m, $B_{10}H_{10}$), 3.83 (1H, s, CH), 7.56–7.47 (15H, m, Ph). **4**: Yield 82%. Λ_M = 99^{-1} cm^2 mol^{-1} (Found: C, 33.20; H, 3.68. $C_{20}H_{26}AuAsB_{10}Se$ requires C, 33.10; H, 3.58%). δ_H ($CDCl_3$) 3.40–1.40 (10H, m, $B_{10}H_{10}$), 3.83 (1H, s, CH), 7.54–7.44 (15H, m, Ph).

Synthesis of PPN$[Au(SeC_2B_{10}H_{11})_2]$ (5). To a solution of **1** (0.2 mmol, 0.0446 g) in dichloromethane (20 mL), PPN[$AuCl_2$] (0.1 mmol, 0.0805 g) and Na_2CO_3 were added. The mixture was stirred for 30 min and the excess of Na_2CO_3 filtered off. Removal of solvent to *ca.* 5 mL and addition of *n*-heptane afforded a white solid. Yield 78%. Λ_M = 99 Ω^{-1} cm^2 mol^{-1} (Found: C, 40.89; H, 4.36; N, 1.40. $C_{40}H_{52}AuB_{20}NP_2Se_2$ requires C, 40.71; H, 4.41; N, 1.19%). δ_H ($CDCl_3$) 3.30–1.35 (20H, m, $B_{10}H_{10}$), 3.85 (2H, s, CH), 7.50–7.20 (30H, m, Ph).

Synthesis of $[Au_2(SeC_2B_{10}H_{11})_2(\mu\text{-PP})]$ [PP = dppf (6), dppe (7)]. *Method a.* To a solution of **1** (0.2 mmol, 0.0446 g) in dichloromethane (20 mL), Na_2CO_3 and $[Au_2Cl_2(\mu\text{-PP})]$ (0.1 mmol, PP = dppf 0.1019 g; dppe 0.087 g) were added. The mixture was stirred for 30 min and the excess of Na_2CO_3 filtered off. Removing of solvent to *ca.* 5 mL and addition of diethyl ether afforded an orange solid.

Method b. To a solution of **4** (0.2 mmol, 0.1448 g) in dichloromethane (20 mL) PP (0.1 mmol, PP = dppf 0.0554 g; dppe 0.0398 g) was added. The mixture was stirred for 30 min. Removal of solvent to *ca.* 5 mL and addition of *n*-hexane afforded a white solid.

6: Yield 87%. Λ_M = 2.1 Ω^{-1} cm^2 mol^{-1} (Found: C, 32.94; H, 3.01. $C_{38}H_{50}Au_2B_{20}FeP_2Se_2$ requires C, 32.76; H, 3.59%). δ_H ($CDCl_3$) 3.40–1.40 (20H, m, $B_{10}H_{10}$), 4.51 (4H, m, C_5H_4), 4.41 (4H, m, C_5H_4), 3.73 (2H, s, CH), 7.59–7.46 (20H, m, Ph). **7**: Yield 69%. Λ_M = 2.3 Ω^{-1} cm^2 mol^{-1} (Found: C, 28.45; H, 3.36. $C_{26}H_{46}Au_2B_{20}P_2Se_2$ requires C, 28.15; H, 3.56%). δ_H ($CDCl_3$) 3.40–1.20 (20H, m, $B_{10}H_{10}$), 3.85 (2H, s, CH), 2.67 (4H, s, CH_2), 7.68–7.41 (20H, m, Ph).

Synthesis of $[Au_2(\mu\text{-}SeC_2B_{10}H_{11})(PPh_3)_2]ClO_4$ (8). To a solution of **1** (0.1 mmol, 0.0223 g) in dichloromethane (20 mL), $[O\{Au(PPh_3)\}_3]ClO_4$ (0.1 mmol, 0.1492 g) was added. The mixture was stirred for 1 h. Removing of solvent to *ca.* 5 mL and addition of diethyl ether afforded a white solid. Yield 75%. Λ_M = 100 Ω^{-1} cm^2 mol^{-1} (Found: C, 36.95; H, 3.35. $C_{38}H_{41}Au_2B_{10}ClO_4PSe$ requires C, 36.80; H, 3.31%). δ_H ($CDCl_3$) 3.40–1.20 (10H, m, $B_{10}H_{10}$), 3.92 (1H, s, CH), 7.60–7.38 (30H, m, Ph).

Synthesis of [$Ag_2(\mu\text{-}SeC_2B_{10}H_{11})(PPh_3)_2$]TfO (9). To a solution of **1** (0.1 mmol, 0.0223 g) in dichloromethane (20 mL), Na_2CO_3 and [AgTfO(PPh_3)] (0.2 mmol, 0.1038 g) were added. The mixture was stirred for 1 h and the excess of Na_2CO_3 filtered off. Removal of solvent to *ca.* 5 mL and addition of *n*-hexane afforded a brown solid. Yield 62%. $\Lambda_M = 127\ \Omega^{-1}\ cm^2\ mol^{-1}$ (Found: C, 42.32; H, 3.34; S, 2.45. $C_{39}H_{41}Ag_2B_{10}F_3O_3P_2SSe$ requires C, 42.12; H, 3.69; S, 2.88%). δ_H ($CDCl_3$) 3.0–1.20 (10H, m, $B_{10}H_{10}$), 3.71 (1H, s, CH), 7.66–7.33 (30H, m, Ph).

Crystal structure determination of compound 3

A single crystal of **3** was mounted in inert oil on a glass fibre and transferred to the cold gas stream of a Bruker SMART 1000 CCD system diffractometer. *Crystal data*: $C_{20}H_{26}AuB_{10}PSe$, M = 681.40, orthorhombic, space group *Fdd*2, a = 22.810(2), b = 41.698(4), c = 10.4693(11) Å, U = 9957.9(17) Å^3, T = 143 K, Z = 16, μ(Mo-Kα) = 7.445 mm^{-1}, 29202 reflections measured ($2\theta_{max}$ = 60°, ω-scans), 7236 unique (R_{int} = 0.0428), absorption correction based on multiple scans (program SADABS[33]). The structures were refined on F^2 using the program SHELXL-97.[34] All non-hydrogen atoms were refined anisotropically. The structure was refined as an enantiomeric twin. Final R = 0.0252, R_w = 0.0585, for 299 parameters, 211 restraints; max. $\Delta\rho$ = 1.99 e Å^{-3}.

CCDC reference number 218880.

See http://www.rsc.org/suppdata/dt/b3/b310658f/ for crystallographic data in CIF or other electronic format.

Acknowledgements

We thank the Dirección General de Investigación n° BQU2001–2409-C02–01 for financial support.

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