Kurzmitteilung / Short Communication

Gold Complexes with Selenium Ligands, I

Preparation and Crystal Structures of Phenylselenolato-Gold(I) Complexes

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(Triphenylphosphine)phenylselenolatogold(I), $Ph_3PAuSePh$ (**1**), and the corresponding dppm derivative, $[(dppm)(AuSePh)_2]$ (**3**), are prepared from $Me_3SiSePh$ and the appropriate (phosphine)chlorogold(I) complex. The reaction between Ph_3PAuCl, Ph_2Se_2 and $AgSbF_6$ leads to the salt $[(Ph_3PAu)_2SePh]^+SbF_6^-$ (**2**), with a bridging PhSe ligand. X-ray structure analyses of **1** and **2** confirm the expected linear geometry at gold and reveal short Au – Au contacts; both compounds may thus be regarded as loose dimers.

Gold complexes with selenium ligands have not been extensively studied. Crystal-structure determinations have been carried out for only five such compounds*); $Ph_3PSeAuCl$ 1), $[(Ph_3PAu)_3Se]^+PF_6^-$ 2), $Ph_4As^\bullet[Au(SeCN)_4]^-$ 3), the gold(II) complex $[\{Au(CH_2)_2PPh_2\}_2Cl(SePh)]$ 4) and the selenide/polyselenide complex $[Au_2Se_2(Se_4)_2]^{2-}$ 5). We have now begun a systematic study of gold-selenium complexes and report here the preparation of the phenylselenolato ($PhSe^-$) complexes $Ph_3PAuSePh$ (**1**), $[(Ph_3PAu)_2SePh]^+SbF_6^-$ (**2**) (both with crystal structures) and $[(dppm)(AuSePh)_2]$ (**3**). Amongst selenium ligands, phenylselenolate has a high nucleophilicity 6), comparable with that of selenourea $SeC(NH_2)_2$, with which we have also prepared some gold(I) complexes 7). Many metal complexes of the $PhSe^-$ ligand, both terminal and bridging, are known, mostly involving organometallic moieties 8).

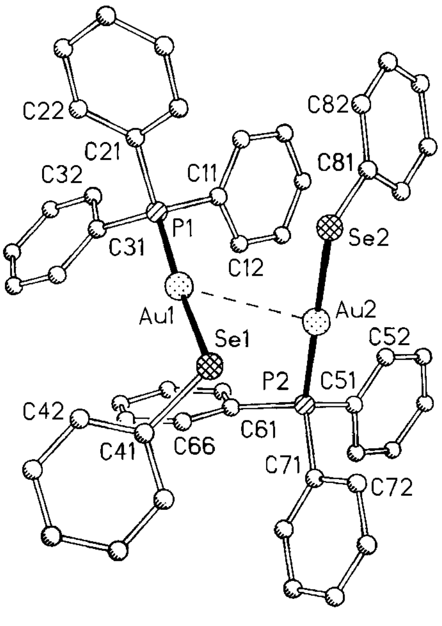

Figure 1. The loose dimer of compound **1** in the crystal (radii are arbitrary; H atoms omitted for clarity)

The reaction of (triphenylphosphine)chlorogold(I) Ph_3PAuCl with (trimethylsilyl)phenylselenide $PhSeSiMe_3$ 9) in THF leads smoothly to the formation of (triphenylphosphine)phenylselenolatogold(I) $Ph_3PAuSePh$ (**1**):

$$Ph_3PAuCl + PhSeSiMe_3 \longrightarrow Ph_3PAuSePh + Me_3SiCl$$

An exactly analogous reaction with $[(dppm)(AuCl)_2]$ leads to the corresponding binuclear complex $[(dppm)(AuSePh)_2]$ (**3**). Complexes **1** and **3** are air-stable crystalline solids; unusually for gold(I) complexes, which are generally colourless, **3** is yellow and **1** pale

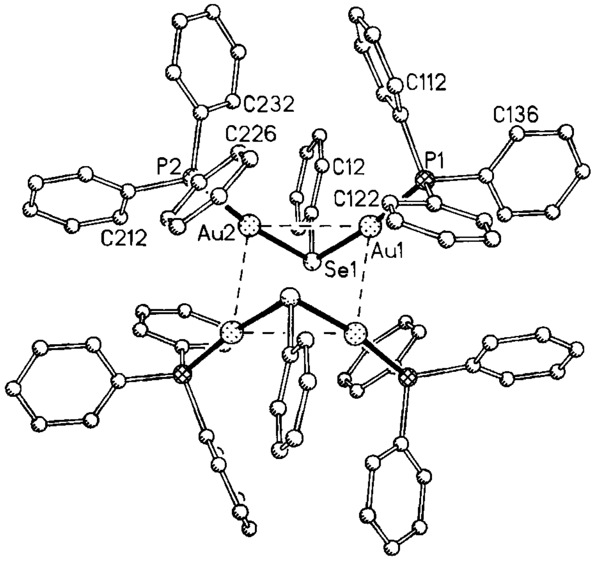

Figure 2. The loose dimer of the cation of compound **2** in the crystal (radii are arbitrary; H atoms omitted for clarity)

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*) Apart from simple binary and ternary gold selenides.

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green (we have observed a deeper green colour in the complex $Ph_2SeAuCl$ [7]). As far as we are aware, no other gold(I) complexes with RSe^- ligands have been reported at the time of writing, but a report of $Ph_3PAuSe(C_6H_2$-2,4,6-$tBu_3)$ will shortly appear [10].

The phenylselenolato ligand has further electron pairs that could be exploited to form bi- or trinuclear complexes. We are currently exploring such reactions of **1**. The reaction of Ph_3PAuCl with diphenyl diselenide Ph_2Se_2 in the presence of $AgSbF_6$ leads directly to $[(Ph_3PAu)_2SePh]^+SbF_6^-$ (**2**), a binuclear cationic complex with bridging SePh. The other products (apart from AgCl) have not been characterised. Complex **2** is a beige or pale orange microcrystalline solid, but its colour is probably due to small amounts of impurities; single crystals are colourless.

Compound **1** crystallises with two independent molecules in the asymmetric unit, linked by a gold–gold interaction of 311.8(1) pm into a loose dimer (Figure 1). Such interactions are well documented for Au(I) [11–13]. Each molecule displays the expected linear coordination at gold (175.6, 179.6°). The Au–Se bond lengths of 242.2, 241.5(1) pm are appreciably longer than the 237.1(2) pm observed in $Ph_3PSeAuCl$ [1], which, despite the differing nature of the Se ligands, may reflect the greater *trans* influence of phosphorus ligands.

The structure of **2** is less precisely determined because of the solvent of crystallisation. The geometry at gold shows appreciable deviations from linearity [171.7, 171.1(1)°]. The Au–Se bond lengths, 244.6 and 244.7(2) pm, are closely similar to those of the $[(Ph_3PAu)_3Se]^+$ cation (av. of six bonds 244.4 pm) [2]. There is a short intramolecular Au–Au contact of 324.0(2) pm with a correspondingly narrow Au–Se–Au angle of 83.0(1)°; the cations are linked in centrosymmetric pairs by an intermolecular Au–Au contact of 311.2(2) pm (Figure 2). The resulting Au_4 squares (Au–Au–Au 96.7, 83.3°) are similar to those observed in (piperidine)chlorogold(I) [14].

Table 1. Crystal data for **1** and **2**

Compound	**1**	**2**
Formula	$C_{24}H_{20}AuPSe$	$C_{42}H_{35}Au_2F_6P_2SbSe.CH_2Cl_2$
M_r	615.3	1395.0
Crystal habit	Pale green prism	Colourless prism
Crystal size (mm)	0.75 x 0.45 x 0.2	0.3 x 0.3 x 0.2
Space group	$P\bar{1}$	*Pbca*
Temperature (°C)	-95	20
Diffractometer	Siemens	Stoe
Cell constants :		
a (pm)	1106.1(4)	1697.3(2)
b (pm)	1149.3(4)	2019.9(3)
c (pm)	1673.8(7)	2664.9(7)
α (°)	97.74(3)	
β (°)	94.21(3)	
γ (°)	92.70(3)	
V (nm^3)	2.099	9.136
Z	4	8
D_x ($Mg\ m^{-3}$)	1.95	2.03
$F(000)$	1168	5248
μ (mm^{-1})	8.6	8.0
$2\theta_{max}$ (°)	50	50
Scan type	ω	ω/θ
No. of reflections :		
measured	7392	9879
independent	7235	7986
R_{int}	0.026	0.030
observed [$>4\ \sigma(F)$]	6107	4789
Absorption correction	DIFABS	ψ-scans
Transmissions	0.80-1.51	0.64-0.87
R	0.029	0.071
wR	0.036	0.068
g	0.0002	0.0004
No. of parameters	488	201
S	1.7	1.7
Max. Δ/σ	0.002	0.004
Max. $\Delta\rho$ ($10^{-6}\ e\ pm^{-3}$)	1.2	1.5

We thank the *Fonds der Chemischen Industrie* and the *Deutsche Forschungsgemeinschaft* for financial assistance and the firm *Degussa* for a gift of gold metal. The X-ray data for compound **2** were measured at the Institute for Inorganic Chemistry, University of Göttingen. The crystallographic program system used was Siemens SHELXTL PLUS. Prof. *W.-W. du Mont* kindly allowed us to see a preprint of his article [10].

Experimental

All syntheses were carried out in dried solvents under N_2. – NMR spectra: Bruker AC 200, $CDCl_3$, ^1H relative to TMS ext.,

Table 2. Atom coordinates ($\times 10^4$) and equivalent isotropic temperature factors (pm^2) for compound **1**

	x	y	z	U(eq)
Au(1)	755.2(2)	2701.0(2)	1763.9(1)	236(1)
Se(1)	-644.9(5)	3278.6(5)	2772.2(3)	286(2)
P(1)	2072(1)	2305(1)	800(1)	234(4)
C(11)	3628(5)	2827(5)	1129(3)	242(17)
C(12)	4150(6)	2400(6)	1811(3)	327(20)
C(13)	5332(6)	2731(6)	2095(4)	404(23)
C(14)	6021(6)	3475(6)	1691(4)	395(22)
C(15)	5518(6)	3895(6)	1024(4)	397(23)
C(16)	4324(5)	3582(5)	737(3)	295(19)
C(21)	1664(5)	2994(5)	-90(3)	246(17)
C(22)	1424(5)	2359(6)	-857(3)	309(19)
C(23)	1103(6)	2940(7)	-1516(4)	407(23)
C(24)	1046(6)	4138(7)	-1410(4)	447(26)
C(25)	1264(6)	4779(7)	-651(4)	443(25)
C(26)	1560(6)	4215(6)	9(4)	370(21)
C(31)	2214(5)	759(5)	425(3)	247(17)
C(32)	3202(6)	414(5)	-9(3)	302(19)
C(33)	3304(6)	-759(6)	-292(4)	376(22)
C(34)	2443(6)	-1582(6)	-156(4)	386(22)
C(35)	1459(6)	-1259(6)	283(4)	379(22)
C(36)	1356(5)	-89(5)	571(3)	301(19)
C(41)	-1553(5)	1865(5)	2902(3)	255(18)
C(42)	-1907(6)	994(6)	2278(4)	358(21)
C(43)	-2625(7)	15(6)	2389(4)	440(24)
C(44)	-3017(6)	-80(6)	3153(4)	457(25)
C(45)	-2653(7)	783(7)	3785(4)	495(27)
C(46)	-1936(6)	1747(6)	3663(4)	396(22)
Au(2)	2336.0(2)	4038.1(2)	3254.7(1)	234(1)
Se(2)	1948.5(5)	5538.8(5)	2403.3(3)	300(2)
P(2)	2691(1)	2635(1)	4059(1)	253(5)
C(51)	4199(6)	2771(6)	4568(3)	366(21)
C(52)	5042(6)	3564(7)	4365(5)	525(27)
C(53)	6207(7)	3715(8)	4747(7)	788(39)
C(54)	6523(9)	3056(11)	5333(6)	840(43)
C(55)	5710(11)	2238(13)	5545(5)	1004(56)
C(56)	4519(7)	2069(10)	5167(4)	740(40)
C(61)	2520(6)	1160(5)	3485(3)	273(18)
C(62)	3531(6)	513(6)	3275(3)	349(21)
C(63)	3340(7)	-550(6)	2780(4)	445(25)
C(64)	2183(7)	-986(6)	2503(4)	439(25)
C(65)	1186(6)	-347(5)	2705(3)	362(21)
C(66)	1365(6)	713(5)	3190(3)	318(20)
C(71)	1630(6)	2596(6)	4843(3)	314(20)
C(72)	1146(6)	3649(6)	5161(3)	362(21)
C(73)	358(6)	3633(6)	5769(4)	444(24)
C(74)	60(7)	2612(7)	6065(4)	452(25)
C(75)	539(7)	1575(7)	5756(4)	472(26)
C(76)	1326(7)	1564(6)	5148(4)	419(23)
C(81)	3514(5)	6095(5)	2153(3)	291(19)
C(82)	3557(7)	6646(6)	1460(4)	391(22)
C(83)	4631(9)	7105(7)	1260(5)	577(31)
C(84)	5673(8)	7038(7)	1724(5)	602(32)
C(85)	5662(8)	6498(8)	2430(6)	696(36)
C(86)	4561(6)	6018(6)	2641(5)	438(24)

Elemental analyses: **2**, Fa. Beller, Göttingen; Au analysis of **1**, Univ. of Murcia, Spain; otherwise locally.

Table 3. Selected bond lengths (pm) and angles (°) for compound **1**

Au(1)-Se(1)	242.2 (1)	Au(1)-P(1)	226.7 (1)
Au(1)...Au(2)	311.8 (1)	Se(1)-C(41)	191.5 (6)
P(1)-C(11)	181.7 (5)	P(1)-C(21)	181.8 (6)
P(1)-C(31)	182.1 (6)	Au(2)-Se(2)	241.5 (1)
Au(2)-P(2)	226.7 (2)	Se(2)-C(81)	191.3 (6)
P(2)-C(51)	180.6 (6)	P(2)-C(61)	182.7 (6)
P(2)-C(71)	182.7 (6)		

Se(1)-Au(1)-P(1)	175.6(1)	Se(1)-Au(1)...Au(2)	74.0(1)
P(1)-Au(1)...Au(2)	104.9(1)	Au(1)-Se(1)-C(41)	105.7(2)
Au(1)-P(1)-C(11)	113.6(2)	Au(1)-P(1)-C(21)	111.5(2)
C(11)-P(1)-C(21)	105.9(3)	Au(1)-P(1)-C(31)	116.5(2)
C(11)-P(1)-C(31)	103.0(3)	C(21)-P(1)-C(31)	105.4(3)
Au(1)...Au(2)-Se(2)	76.2(1)	Au(1)...Au(2)-P(2)	103.9(1)
Se(2)-Au(2)-P(2)	179.6(1)	Au(2)-Se(2)-C(81)	105.3(2)
Au(2)-P(2)-C(51)	113.6(2)	Au(2)-P(2)-C(61)	111.6(2)
C(51)-P(2)-C(61)	106.2(3)	Au(2)-P(2)-C(71)	113.6(2)
C(51)-P(2)-C(71)	106.7(3)	C(61)-P(2)-C(71)	104.4(3)

Table 4. Atom coordinates (× 10^4) and equivalent isotropic temperature factors (pm^2) for compound **2**

	x	y	z	U(eq)
Au(1)	4425.7(4)	4411.7(3)	4321.3(3)	60.5(2)
Au(2)	5661.2(4)	4167.7(3)	5229.6(3)	57.8(2)
Se(1)	5831(1)	4663(1)	4399(1)	59(1)
C(12)	5943(6)	3288(7)	4019(5)	118(7)
C(13)	6335	2768	3780	137(9)
C(14)	7093	2863	3592	102(6)
C(15)	7459	3478	3641	129(8)
C(16)	7068	3997	3880	115(7)
C(11)	6309	3902	4068	76(5)
P(1)	3177(3)	4023(2)	4222(2)	65(2)
C(112)	2582(7)	2926(6)	4775(5)	107(7)
C(113)	2624	2272	4943	127(8)
C(114)	3223	1857	4770	145(9)
C(115)	3780	2096	4431	159(10)
C(116)	3738	2749	4263	116(7)
C(111)	3139	3164	4435	71(5)
C(122)	2508(6)	4436(5)	5108(4)	82(5)
C(123)	1943	4750	5405	95(6)
C(124)	1318	5086	5179	87(5)
C(125)	1258	5107	4658	87(6)
C(126)	1822	4793	4361	79(5)
C(121)	2447	4457	4586	58(4)
C(132)	3109(7)	4500(5)	3248(5)	99(6)
C(133)	2786	4530	2767	123(8)
C(134)	2198	4085	2627	114(7)
C(135)	1932	3609	2966	128(8)
C(136)	2255	3579	3447	106(7)
C(131)	2843	4024	3588	77(5)
P(2)	5482(3)	3555(2)	5940(2)	61(2)
C(212)	7023(8)	3802(5)	6195(4)	95(6)
C(213)	7675	3800	6514	123(8)
C(214)	7597	3580	7008	109(7)
C(215)	6867	3363	7183	123(8)
C(216)	6215	3365	6864	104(7)
C(211)	6293	3584	6370	70(5)
C(222)	4630(5)	4231(5)	6669(4)	74(5)
C(223)	3934	4447	6894	82(5)
C(224)	3209	4211	6722	98(6)
C(225)	3182	3759	6327	83(5)
C(226)	3879	3543	6103	68(5)
C(221)	4603	3779	6274	59(4)
C(232)	5535(8)	2495(7)	5283(5)	120(7)
C(233)	5475	1828	5152	164(11)
C(234)	5265	1361	5514	131(8)
C(235)	5114	1560	6005	149(9)
C(236)	5174	2227	6136	130(8)
C(231)	5384	2695	5775	74(5)
Cl(1)	4002(8)	2361(6)	7302(5)	294(6)
Cl(2)	-84(10)	1495(8)	7204(6)	351(7)
Sb(1)	368(1)	4377(1)	6671(1)	85(1)
F(1)	591(10)	4653(9)	7320(5)	192(9)
F(2)	1287(9)	3925(9)	6612(7)	210(10)
F(3)	171(13)	4097(10)	6025(6)	223(11)
F(4)	-549(11)	4863(12)	6680(9)	275(14)
F(5)	849(12)	5110(7)	6420(6)	224(10)
F(6)	-105(14)	3677(9)	6958(8)	276(13)

$Ph_3PAuSePh$ (**1**): 0.46 g (2 mmol) $PhSeSiMe_3$[9] is slowly added to a cooled (0°C) solution of 1.00 g (2 mmol) Ph_3PAuCl in 50 ml THF. The solution is stirred for 1 h at 0°, during which time a yellow precipitate is formed. The solution is concentrated to 30 ml and 30 ml diethyl ether is added. After overnight cooling, the product is filtered off and recrystallised from dichloromethane/diethyl ether. Yield 0.90 g (73%), m. p. 180°C (dec.). The product contained single crystals suitable for X-ray analysis. — ^{31}P NMR: $\delta = 39.41$ (s). — MS (EI): $m/z = 616$ (weak) [M^+], 579 (base peak).

$C_{24}H_{20}AuPSe$ (615.3) Calcd. C 46.85 H 3.28 Au 32.01
Found C 47.35 H 3.54 Au 32.07

$[(Ph_3PAu)_2SePh]^+SbF_6^-$ (**2**): 1.0 g (2 mmol) Ph_3PAuCl is dissolved in 100 ml CH_2Cl_2/diethyl ether (3:2). To this solution are added 0.32 g (1 mmol) Ph_2Se_2 in 10 ml CH_2Cl_2 and 0.69 g (2 mmol) $AgSbF_6$. After 1 h stirring, the brown precipitate is filtered off (SiO_2/$MgSO_4$) and the solution concentrated to 30 ml. The product is precipitated with ether and recrystallised from CH_2Cl_2/ether. Yield 0.80 g (66%), m. p. 143°C (dec.). Single crystals for X-ray studies were obtained by liquid diffusion of ether into CH_2Cl_2 solutions. [The single crystals contained widely varying amounts of solvent, and thus displayed varying cell constants, although the space group was unchanged. The structure described above displayed the lowest solvent content (ca. 1 CH_2Cl_2 per monomer of **2**)]. — ^{31}P NMR: $\delta = 36.52$ (s).

$C_{42}H_{35}Au_2F_6P_2SbSe$ (1310.1) Calcd. C 38.51 H 2.69 Se 6.03
Found C 38.02 H 2.88 Se 5.84

$[(dppm)(AuSePh)_2]$ (**3**): 0.46 g (2 mmol) $PhSeSiMe_3$ is slowly added to a solution of 0.85 g (1 mmol) $[(dppm)(AuCl)_2]$ in 60 ml CH_3CN/DMF (5:1). The solution is refluxed for 3 h, after which the solvent is removed. The residue is recrystallised from dichloromethane/diethyl ether. Yield 0.95 g (87%), m.p. 145–148°C (dec.). — ^1H NMR: $\delta = 4.54$ (t, $J_{PH} = 12$ Hz, 2H, CH_2), 7.49 (m, 30H, Ph). — ^{31}P NMR: $\delta = 32.58$ (s). — Recrystallisation from DMF/CH_3OH gives an additional ^1H NMR, signal at $\delta = 3.4$ (s, 3H) from one mole of methanol of crystallisation.

$C_{37}H_{32}Au_2P_2Se_2$ (1090.5) Calcd. C 40.75 H 2.96 P 5.68
Found C 40.01 H 2.88 P 5.45

X-ray Structure Determinations[15]: See Table 1 for detailed crystal data. Intensity data were registered on four-circle diffractometers

Table 5. Selected bond lengths (pm) and angles (°) for compound **2** [symmetry operator (*i*): 1 − *x*, 1 − *y*, 1 − *z*]

Au(1)..Au(2)	324.0 (2)	Au(1)-Se(1)	244.7 (2)
Au(1)-P(1)	227.5 (4)	Au(1)..Au(2i)	311.2 (2)
Au(2)-Se(1)	244.6 (2)	Au(2)-P(2)	228.1 (4)
Se(1)-C(11)	194.8 (14)	P(1)-C(111)	182.7 (14)
P(1)-C(121)	180.2 (11)	P(1)-C(131)	178.1 (14)
P(2)-C(211)	179.3 (13)	P(2)-C(221)	179.6 (10)
P(2)-C(231)	180.1 (15)		

Se(1)-Au(1)-P(1)	171.5(1)	Se(1)-Au(2)-P(2)	171.1(1)
Au(1)-Se(1)-Au(2)	83.0(1)	Au(1)-Se(1)-C(11)	101.8(3)
Au(2)-Se(1)-C(11)	97.8(4)	Au(2)..Au(1)..Au(2i)	83.3(1)
Se(1)-Au(1)..Au(2i)	79.8(1)	P(1)-Au(1)..Au(2i)	108.6(1)
Au(1)..Au(2)-Se(1)	48.5(1)	Au(1)..Au(2)-P(2)	128.0(1)
Au(2)..Au(1)-Se(1)	48.5(1)	Au(2)..Au(1)-P(1)	129.6(1)
Au(1)..Au(2)..Au(1i)	96.7(1)	Se(1)-Au(2)..Au(1i)	88.7(1)
P(2)-Au(2)..Au(1i)	100.0(1)	Au(1)-P(1)-C(111)	108.9(4)
Au(1)-P(1)-C(121)	114.2(4)	C(111)-P(1)-C(121)	105.7(5)
Au(1)-P(1)-C(131)	114.0(4)	C(111)-P(1)-C(131)	106.6(6)
C(121)-P(1)-C(131)	106.9(5)	Au(2)-P(2)-C(211)	114.3(4)
Au(2)-P(2)-C(221)	112.7(4)	C(211)-P(2)-C(221)	108.2(5)
Au(2)-P(2)-C(231)	109.5(5)	C(211)-P(2)-C(231)	105.0(6)
C(221)-P(2)-C(231)	106.7(6)		

$^{31}P\{^1H\}$ relative to H_3PO_4 (ext.). — MS: Finnigan MAT 8430. —

using monochromated Mo-K_α radiation. Cell constants were refined from 2Θ values (**1**) or setting angles (**2**) of ca. 50 reflections in the range $2\Theta = 20-25°$. Structures were solved by the heavy-atom method and subjected to full-matrix least-squares refinement (for **1**, all non-H atoms anisotropic; for **2**, Au, Se, P, Sb and F anisotropic and phenyl groups idealised). H atoms were included using a riding model. Weighting schemes were of the form $w^{-1} = \sigma^2(F) + gF^2$. Final atomic coordinates and derived bond lengths and angles are given in Tables 2–5.

CAS Registry Numbers

1: 122711-32-4 / **2**: 129124-84-1 / **3**: 129124-85-2 / $\mathrm{Ph_3PAuCl}$: 14243-64-2 / $\mathrm{PhSeSiMe_3}$: 33861-17-5 / $\mathrm{Ph_2Se_2}$: 1666-13-3 / [(dppm)-$\mathrm{(AuCl)_2}$]: 37095-27-5

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[15] Further details of the structure determinations (complete bond lengths and angles, H atom coordinates, structure factors, temperature factors) have been deposited at the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-7514 Eggenstein-Leopoldshafen 2, Federal Republic of Germany. Any request for this material should quote a full literature citation and the reference number CSD-54769.

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