Reaction of $[Rh_2\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]$ with $[M(PPh_3)]^+$ (M = Au, Ag, or Cu): Formation of Unusual 44-Electron Rh_2M Clusters. Crystal Structure of $[Rh_2\{\mu\text{-}Au(PPh_3)\}\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]ClO_4{\cdot}2CH_2Cl_2$ †

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The complex *trans*-$[Rh_2\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]$ (**1**) reacts with $[M(PPh_3)]^+$ (M = Au, Ag, or Cu) to give $[Rh_2\{\mu\text{-}M(PPh_3)\}\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]ClO_4$ (**2**)—(**4**). Compound (**2**) was analysed by single-crystal *X*-ray diffraction: triclinic, space group $P\bar{1}$; a = 14.358(4), b = 20.775(6), c = 13.184(3) Å, α = 105.65(3), β = 115.66(2), γ = 69.96(3)°, Z = 2, and R = 0.071. The basic skeleton consists of a Rh_2Au triangle, the two Rh–Au distances [2.797(2) and 2.690(2) Å] being consistent with Rh–Au bonds. The co-ordination geometry around the rhodium atoms is square pyramidal. The Rh–Rh separation [2.815(2) Å] is at the upper part of the range of distances for a single Rh–Rh bond. Phosphorus-31 n.m.r. and i.r. data for compounds (**2**)—(**4**) are described, and are consistent with a bridging disposition of the $M(PPh_3)$ group in all the complexes.

Recently we reported[1,2] the preparation of binuclear rhodium complexes containing deprotonated diaminonaphthalene $(NH)_2C_{10}H_6{}^{2-}$ as the bridging ligand. These compounds were remarkable in that amido-rhodium complexes are relatively rare[1–6] and they appear to be the first compounds in which the two amide centres are bound to the two metals in both a bridging and chelating manner, but also because of the structural features[1] of $[Rh_2\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}I_2(CO)_2(PPh_3)_2]$ which has a short distance [2.540(1) Å] between the two rhodium atoms compared with the usual range (2.62—2.84 Å) for a single rhodium–rhodium bond.[7,8]

We report here the ability of *trans*-$[Rh_2\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]$ to react with the electrophilic metal fragments $M(PPh_3)^+$ (M = Au, Ag, or Cu) to give the 44-electron species $[Rh_2\{\mu\text{-}M(PPh_3)\}\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]^+$. Until now, the chemistry of 44-electron Rh_2M clusters has been limited to $[\{Rh(\eta^5\text{-}C_5Me_5)(\mu\text{-}CO)\}_2(\mu\text{-}L)]$ [L = AgCl, $AgONO_2$, AgO_2CCF_3, AgO_2CMe, $AgCN_2P(O)Ph_2$, or AuCl].[9]

Results and Discussion

Reaction of *trans*-$[Rh_2\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]$ (**1**) with $[M(PPh_3)(solv)]ClO_4$ (M = Ag, Au, or Cu; solv = acetone) in dichloromethane–acetone, for 30 min at room temperature, leads to the trinuclear clusters $[Rh_2\{\mu\text{-}M(PPh_3)\}\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]ClO_4$ [M = Au (**2**), Ag (**3**), or Cu (**4**)]. The complexes were obtained, after concentration of the solution and addition of diethyl ether, as red [(**2**) and (**3**)] or orange [(**4**)] solids in *ca.* 60% yield. The formulation of the compounds was deduced by elemental analysis, i.r. and ^{31}P n.m.r. spectroscopy; the crystal structure of compound (**2**) has been determined by single-crystal *X*-ray diffraction (see below).

The phosphorus resonance of the PPh_3 co-ordinated to the M atom appears as a triplet for complex (**2**), and is split into two doublets of triplets for complex (**3**) due to coupling with ^{107}Ag and ^{109}Ag, which is consistent with the $M(PPh_3)$ unit bridging between the two equivalent rhodiums; for complex (**4**) this resonance is broad because of the quadrupolar moment of the copper atom.[10] The phosphorus resonance of the PPh_3 co-ordinated to the rhodium atoms is a doublet for all the complexes, indicating a symmetrical structure. The ^1J(P–Rh) values are *ca.* 20 Hz lower than for complex (**1**) ($^{31}P\text{-}\{^1H\}$ ($CDCl_3$): δ 50.77 [d, ^1J(P–Rh) = 159 Hz]); this could be interpreted in terms of a decrease in the electron density on the rhodium atoms due to the co-ordination of the electrophilic $M(PPh_3)^+$ ion.[11] This effect can also be observed in the i.r. spectra which show a broad signal corresponding to $\nu(C{\equiv}O)$ at *ca.* 40 cm^{-1} higher than for complex (**1**) [$\nu(C{\equiv}O)$ 1 950 cm^{-1} (CH_2Cl_2)]. The conductivities of complexes (**2**)—(**4**) in dichloromethane are in the range 49—57 $ohm^{-1}\ cm^2\ mol^{-1}$ and indicate the ionic nature of the compounds.

The crystal structure of (**2**)$\cdot 2CH_2Cl_2$ (Figure,[12] Tables 1 and 2) consists of complex cations $[Rh_2\{\mu\text{-}Au(PPh_3)\}\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]^+$ and $ClO_4{}^-$ anions. The basic skeleton consists of a Rh_2Au triangle. The co-ordination geometry around the rhodium atoms is square pyramidal. The deviations of Rh(1) and Rh(2) from their co-ordination planes (N, N, C, P) are 0.055(1) and 0.040(1) Å, and the angles between the Au–Rh lines and the normal to the co-ordination planes are 4.2(2) and 5.4(3)° respectively. The Rh–N bond lengths [2.074(16), 2.130(10), 2.102(11), and 2.083(14) Å] are similar to those found in $[Rh_2\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}I_2(CO)_2(PPh_3)_2]$[1] and in other amido complexes.[4,6] The small N–Rh–N angles [75.3(5) and 75.7(5)°] are most likely imposed by the chelating nature of the ligand. The two Rh–Au distances [2.797(2) and 2.690(2) Å] and Rh–Au–P angles [144.9(2) and 153.4(2)°] are different; however, the ^{31}P n.m.r. spectrum is consistent with a symmetric $Au(PPh_3)$ bridge and no chemical significance is attached to those differences which are probably due to a combination of non-bonded repulsion between the phenyl rings and crystal-packing forces. The average Rh–Au distance of 2.743(2) Å is comparable to the average value [2.739(2) Å]

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† μ-Naphthalene-1,8-diamido-*NN'*-μ-triphenylphosphineaurio-bis-[carbonyl(triphenylphosphine)rhodium] perchlorate–dichloromethane (1/2).

Supplementary data available: see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1989, Issue 1, pp. xvii—xx.

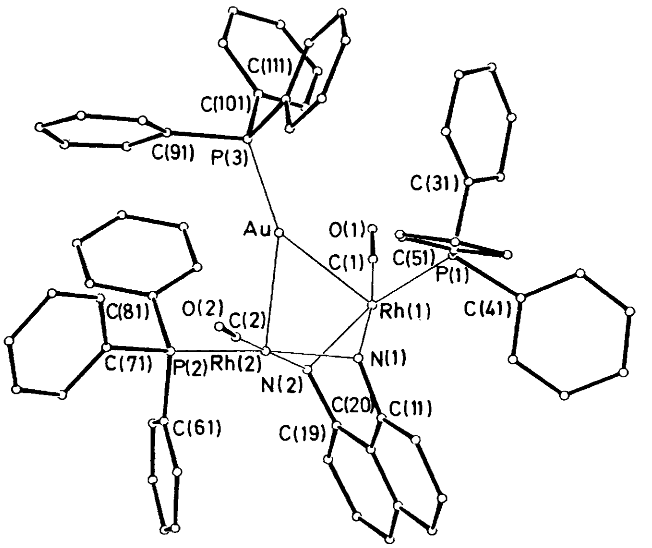

Figure. Crystal structure of complex (**2**) (cation only)

Table 1. Bond distances (Å) and angles (°) for complex (**2**)

Rh(1)–Rh(2)	2.815(2)	Au–P(3)	2.248(3)
Rh(1)–C(1)	1.804(20)	Rh(2)–C(2)	1.781(20)
Rh(1)–Au	2.797(2)	Rh(2)–Au	2.690(2)
Rh(1)–P(1)	2.268(4)	Rh(2)–P(2)	2.276(4)
Rh(1)–N(1)	2.074(16)	Rh(2)–N(1)	2.102(11)
Rh(1)–N(2)	2.130(10)	Rh(2)–N(2)	2.083(14)
P(1)–C(31)	1.835(16)	P(2)–C(61)	1.817(13)
P(1)–C(41)	1.823(14)	P(2)–C(71)	1.822(18)
P(1)–C(51)	1.809(20)	P(2)–C(81)	1.811(21)
P(3)–C(91)	1.787(15)	N(1)–N(2)	2.568(20)
P(3)–C(101)	1.787(23)		
P(3)–C(111)	1.833(16)		
C(1)–Rh(1)–P(1)	91.7(5)	C(2)–Rh(2)–P(2)	92.2(5)
C(1)–Rh(1)–Au	88.0(5)	C(2)–Rh(2)–Au	86.2(5)
C(1)–Rh(1)–N(2)	98.5(6)	C(2)–Rh(2)–N(1)	99.6(6)
Au–Rh(1)–N(2)	99.1(3)	Au–Rh(2)–N(2)	95.2(3)
Au–Rh(1)–N(1)	87.3(4)	Au–Rh(2)–N(1)	89.6(3)
Au–Rh(1)–P(1)	98.4(1)	Au–Rh(2)–P(2)	94.2(1)
N(1)–Rh(1)–N(2)	75.3(5)	N(1)–Rh(2)–N(2)	75.7(5)
N(1)–Rh(1)–P(1)	95.2(4)	N(2)–Rh(2)–P(2)	92.5(4)
Rh(2)–Rh(1)–Au	57.3(1)	Rh(1)–Rh(2)–Au	61.0(1)
Rh(1)–Au–P(3)	144.9(2)	Rh(1)–Au–Rh(2)	61.7(1)
Rh(2)–Au–P(3)	153.4(2)	Rh(1)–N(1)–Rh(2)	84.8(5)
Rh(1)–N(2)–Rh(2)	83.9(4)	Rh(1)–N(1)–C(11)	115.2(10)
Rh(1)–N(2)–C(19)	109.9(9)	Rh(2)–N(1)–C(11)	114.1(9)
Rh(2)–N(2)–C(19)	115.4(10)		

found[13] in $[Rh_2(\eta^5\text{-}C_5H_5)(\mu\text{-}CO)\{\mu\text{-}Au(PPh_3)\}(\mu\text{-dppm})]^+$ (dppm = $Ph_2PCH_2PPh_2$). The Rh–Rh separation [2.815(2) Å] is at the upper part of the range of distances (2.62—2.84 Å) observed for other rhodium dimers where a single bond is thought to exist.[7,8]

Heteronuclear gold–metal clusters containing a μ-bridging $Au(PR_3)$ fragment are generally said to involve a three-centre two-electron bond between the two metal atoms and the gold atom.[14] Thus, the 44-electron cluster (**2**) could be considered to contain a three-centre two-electron Rh_2Au group, in which the rhodium and gold atoms have 16- and 14-electron configurations; however, 18-electron configurations for the rhodium cannot be ruled out, since this assumption would lead to three electron pairs for the skeletal atoms[11] and imply a rhodium–rhodium bond. Clusters (**3**) and (**4**) have not been analysed by single-crystal *X*-ray diffraction, but the spectroscopic data (^{31}P n.m.r. and i.r.) suggest a similar mode of bonding of the $Ag(PPh_3)$ and $Cu(PPh_3)$ groups to the rhodium atoms.

Experimental

The reactions were carried out at room temperature under a nitrogen atmosphere using standard Schlenk techniques. The compounds $[Rh_2\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]$,[2] $[AuCl(PPh_3)]$,[15] $[\{AgCl(PPh_3)\}_4]$,[16] and $[\{CuCl(PPh_3)\}_4]$[17] were prepared as previously reported. Phosphorus-31 and ^1H n.m.r. spectra were recorded in $CDCl_3$ solution at room temperature on a Varian XL 200 spectrometer, i.r. spectra on a Perkin-Elmer 783 spectrophotometer. Elemental analyses were carried out with a Perkin-Elmer 240C elemental analyser. Conductivities were measured at room temperature with *ca.* 4×10^{-4} mol dm^{-3} dichloromethane solution using a Philips PW 9509 conductimeter.

Preparations.—$[Rh_2\{\mu\text{-}Au(PPh_3)\}\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]ClO_4$ (**2**). Silver perchlorate (55 mg, 0.26 mmol) was added to a dichloromethane (10 cm^3) solution of $[AuCl(PPh_3)]$ (132 mg, 0.26 mmol). The mixture was stirred for 15 min, then filtered through Kieselguhr and the filtrate added to a dichloromethane (10 cm^3) solution of $[Rh_2\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]$ (250 mg, 0.26 mmol). The resulting red solution was allowed to react for 20 min, after which partial concentration to *ca.* 2 cm^3 and addition of diethyl ether (15 cm^3) led to a red precipitate. The solid was filtered off, washed with diethyl ether, and dried under vacuum (327 mg, 82%) (Found: C, 52.9; H, 3.9; N, 1.8. Calc. for $C_{66}H_{53}AuClN_2O_6P_3Rh_2$: C, 52.8; H, 3.5; N, 1.8%). N.m.r. ($CDCl_3$): ^1H, δ 3.4 (br, 2 H, NH), 6.0 (d, 2 H, $C_{10}H_6$), and 6.8—7.8 (m, 49 H, C_6H_5, $C_{10}H_6$); $^{31}P\text{-}\{^1H\}$, δ 23.64 [t, P, ^2J(P–Rh) = 20] and 40.16 p.p.m. [d, 2 P, ^1J(P–Rh) = 139 Hz)]. I.r. (CH_2Cl_2): ν(C≡O) 1 990 cm^{-1}. $\Lambda_M(CH_2Cl_2)$: 57 Ω^{-1} cm^2 mol^{-1}.

$[Rh_2\{\mu\text{-}Ag(PPh_3)\}\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]ClO_4$ (**3**). The complex was prepared using the procedure described for (**2**), but with $[\{AgCl(PPh_3)\}_4]$ (32 mg, 0.02 mmol), $AgClO_4$ (17 mg, 0.08 mmol), and $[Rh_2\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]$ (75 mg, 0.08 mmol). The red complex was obtained in 68% yield (63 mg) (Found: C, 56.3; H, 4.2; N, 2.2. Calc. for $C_{66}H_{53}AgClN_2O_6P_3Rh_2$: C, 56.1; H, 3.8; N, 2.0%). N.m.r. ($CDCl_3$): ^1H, δ 3.5 (br, 2 H, NH), 6.0 (d, 2 H, $C_{10}H_6$), and 6.8—7.8 (m, 49 H, C_6H_5, $C_{10}H_6$); $^{31}P\text{-}\{^1H\}$, δ 9.30 [two d, t, P, $^1J(P\text{-}^{107}Ag)$ = 545, $^1J(P\text{-}^{109}Ag)$ = 630, ^2J(P–Rh) = 11] and 41.55 p.p.m. [d, 2 P, ^1J(P–Rh) = 140 Hz]. I.r. (CH_2Cl_2): ν(C≡O) 1 985 cm^{-1}. $\Lambda_M(CH_2Cl_2)$: 48 Ω^{-1} cm^2 mol^{-1}.

$[Rh_2\{\mu\text{-}Cu(PPh_3)\}\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]ClO_4$ (**4**). The complex was prepared using the procedure described for (**2**), but with $[\{CuCl(PPh_3)\}_4]$ (38 mg, 0.025 mmol), $AgClO_4$ (22 mg, 0.10 mmol), and $[Rh_2\{\mu\text{-}1,8\text{-}(NH)_2C_{10}H_6\}(CO)_2(PPh_3)_2]$ (100 mg, 0.10 mmol). The orange complex was obtained in 63% yield (92 mg) (Found: C, 58.0; H, 3.9; N, 2.0. Calc. for $C_{66}H_{53}ClCuN_2O_6P_3Rh_2$: C, 57.9; H, 3.9; N, 2.6%). N.m.r. ($CDCl_3$): ^1H, δ 3.4 (br, 2 H, NH), 6.0 (d, 2 H, $C_{10}H_6$), and 6.8—7.8 (m, 49 H, C_6H_5, $C_{10}H_6$); $^{31}P\text{-}\{^1H\}$, δ 31.25 (br, P) and 41.55 p.p.m. [d, 2 P, ^1J(P–Rh) = 140 Hz]. I.r. (CH_2Cl_2): ν(C≡O) 1 980 cm^{-1}. $\Lambda_M(CH_2Cl_2)$: 37 Ω^{-1} cm^2 mol^{-1}.

Crystal Structure Determination.—*Crystal data.* $C_{66}H_{53}AuClN_2O_6P_3Rh_2{\cdot}2CH_2Cl_2$ (**2**), triclinic, space group $P\bar{1}$, M = 1 671.2, a = 14.358(4), b = 20.775(6), c = 13.184(3) Å, α = 105.65(3), β = 115.66(2), γ = 69.96(3)°, U = 3 295.7(16) Å^3, D_c = 1.684 g cm^{-3}, Z = 2, F(000) = 1 652, $\lambda(Cu\text{-}K_\alpha)$ = 1.5418 Å, $\mu(Cu\text{-}K_\alpha)$ = 113.05 cm^{-1} (empirical absorption correction[18]

Table 2. Fractional atomic co-ordinates for complex **(2)**

Atom	X/a	Y/b	Z/c
Au	0.317 08(5)	0.265 78(4)	0.297 59(5)
Rh(1)	0.444 28(8)	0.333 75(5)	0.496 41(8)
Rh(2)	0.455 47(8)	0.192 40(5)	0.468 21(8)
C(1)	0.472 8(12)	0.371 2(7)	0.408 0(13)
O(1)	0.487 0(10)	0.395 5(7)	0.348 0(11)
C(2)	0.351 5(14)	0.153 1(6)	0.435 8(11)
O(2)	0.276 9(12)	0.130 6(8)	0.418 1(13)
N(1)	0.418 4(9)	0.277 7(6)	0.585 9(9)
N(2)	0.574 4(9)	0.243 1(6)	0.518 6(9)
C(11)	0.493 9(12)	0.273 5(7)	0.701 5(11)
C(12)	0.455 3(14)	0.287 6(8)	0.787 1(13)
C(13)	0.530 9(18)	0.287 1(8)	0.900 9(14)
C(14)	0.638 2(18)	0.273 5(10)	0.925 7(14)
C(15)	0.679 3(15)	0.260 0(8)	0.838 0(13)
C(16)	0.786 5(16)	0.246 4(12)	0.863 1(17)
C(17)	0.823 7(14)	0.235 0(11)	0.778 2(17)
C(18)	0.753 5(13)	0.233 1(8)	0.665 8(13)
C(19)	0.643 6(11)	0.244 8(7)	0.634 9(11)
C(20)	0.603 5(11)	0.258 4(7)	0.723 4(11)
P(1)	0.316 0(3)	0.430 9(2)	0.517 8(3)
C(31)	0.255 5(11)	0.490 1(7)	0.411 6(12)
C(32)	0.309 5(17)	0.534 4(10)	0.417 5(15)
C(33)	0.262 6(20)	0.580 3(11)	0.336 4(19)
C(34)	0.167 7(19)	0.577 3(12)	0.248 1(17)
C(35)	0.119 7(16)	0.530 3(11)	0.240 4(17)
C(36)	0.160 6(14)	0.484 9(9)	0.319 3(14)
C(41)	0.374 6(12)	0.482 9(7)	0.652 5(13)
C(42)	0.465 8(14)	0.448 4(9)	0.735 4(14)
C(43)	0.505 2(17)	0.481 9(11)	0.844 0(15)
C(44)	0.460 4(18)	0.551 4(11)	0.871 7(15)
C(45)	0.374 2(17)	0.584 5(9)	0.789 0(17)
C(46)	0.331 2(14)	0.553 2(8)	0.680 0(15)
C(51)	0.205 5(12)	0.417 6(8)	0.534 2(12)
C(52)	0.159 5(15)	0.460 6(9)	0.610 8(15)
C(53)	0.079 4(16)	0.447 8(10)	0.626 1(17)
C(54)	0.033 4(13)	0.394 8(10)	0.559 7(14)
C(55)	0.077 8(14)	0.350 3(10)	0.480 9(16)
C(56)	0.160 7(12)	0.364 4(9)	0.468 2(13)
P(2)	0.529 1(2)	0.105 9(2)	0.355 3(3)
C(61)	0.668 8(12)	0.074 3(7)	0.444 0(13)
C(62)	0.752 8(15)	0.075 0(11)	0.419 6(18)
C(63)	0.857 9(17)	0.053 2(14)	0.497 4(24)
C(64)	0.876 9(17)	0.034 5(14)	0.595 7(22)
C(65)	0.795 3(17)	0.032 1(13)	0.620 5(19)
C(66)	0.689 5(13)	0.55 7(10)	0.546 7(14)
C(71)	0.473 5(12)	0.031 1(8)	0.296 5(12)
C(72)	0.367 2(15)	0.041 0(9)	0.224 3(14)
C(73)	0.319 4(15)	−0.014 6(11)	0.177 5(15)
C(74)	0.381 0(22)	−0.079 7(10)	0.204 0(18)
C(75)	0.483 7(19)	−0.088 5(10)	0.277 1(19)
C(76)	0.533 7(15)	−0.035 9(8)	0.322 3(14)
C(81)	0.535 9(12)	0.126 4(7)	0.234 0(13)
C(82)	0.530 9(12)	0.191 8(9)	0.228 8(12)
C(83)	0.541 9(16)	0.208 2(9)	0.139 0(16)
C(84)	0.557 3(17)	0.154 6(10)	0.051 4(15)
C(85)	0.561 5(18)	0.090 9(10)	0.055 8(15)
C(86)	0.550 8(15)	0.074 0(8)	0.145 1(13)
P(3)	0.192 4(3)	0.277 4(2)	0.121 6(3)
C(91)	0.184 7(12)	0.196 5(8)	0.030 9(12)
C(92)	0.102 8(17)	0.166 2(11)	0.000 3(18)
C(93)	0.100 4(23)	0.102 5(16)	−0.071 3(24)
C(94)	0.184 0(24)	0.067 9(12)	−0.109 1(20)
C(95)	0.265 4(21)	0.096 0(12)	−0.071 4(21)
C(96)	0.266 7(16)	0.158 1(10)	−0.005 0(17)
C(101)	0.218 2(12)	0.325 0(9)	0.046 4(13)
C(102)	0.270 0(15)	0.374 4(11)	0.102 2(17)
C(103)	0.290 4(18)	0.413 7(11)	0.047 9(27)
C(104)	0.256 5(23)	0.402 8(14)	−0.065 7(28)
C(105)	0.203 3(22)	0.354 4(13)	−0.124 7(20)
C(106)	0.181 5(17)	0.316 2(10)	−0.072 4(17)
C(111)	0.057 7(11)	0.322 8(9)	0.120 2(12)
C(112)	0.019 9(13)	0.300 9(9)	0.182 8(14)
C(113)	−0.082 4(17)	0.336 2(12)	0.184 2(16)
C(114)	−0.145 4(15)	0.388 8(11)	0.120 6(15)
C(115)	−0.108 6(15)	0.409 8(12)	0.056 9(17)
C(116)	−0.008 0(14)	0.375 0(9)	0.055 8(14)
Cl(1)	0.804 2(5)	0.253 9(4)	0.378 3(8)
O(3)	0.710 4(15)	0.275 5(12)	0.402 8(18)
O(4)	0.854 1(22)	0.183 1(11)	0.391 5(27)
O(5)	0.770 8(21)	0.255 7(14)	0.249 0(23)
O(6)	0.870 5(22)	0.293 9(17)	0.434 7(44)
C(21)	0.110 4(37)	0.176 4(24)	0.583 2(39)
Cl(2)	0.230 1(8)	0.197 8(5)	0.663 4(9)
Cl(3)	0.055 6(20)	0.166 7(13)	0.667 4(22)
C(22)	0.094 9(37)	0.863 3(24)	0.813 6(40)
Cl(4)	0.117 2(14)	0.902 6(9)	0.964 6(15)
Cl(5)	−0.047 0(10)	0.873 0(7)	0.756 6(11)

applied). Red crystal samples obtained by vapour diffusion from dichloromethane–diethyl ether.

A crystal of 0.12 × 0.08 × 0.13 mm was used for the analysis on a Philips PW 1100 diffractometer, with Cu-K_α radiation, graphite monochromator, ω—2θ scans, bisecting geometry, and scan width 1.5°. The stability was checked every 90 min, with no observed decay. The cell parameters were obtained from a least-squares fit of the angular positions of 80 reflections with θ up to 45°.

The structure was solved from the Patterson function, completed through the DIRDIF system,[19] and refined by least-squares methods[20,21] on F_o. All hydrogen atoms were located in a Fourier difference synthesis and included isotropically in the last cycles of refinement where some hydrogen parameters had to be fixed. Of the 8 056 independent data, 6 421 were considered observed [$I > 3\sigma(I)$]. Refinement converged at $R = 0.071$ and $R' = 0.074$. Weights were chosen empirically so as to give no trends in $\langle w\Delta^2F\rangle$ *vs.* $<|F_o|g(\sin\theta/\lambda)$, K ensuring that $\langle w\Delta^2F\rangle \approx 1$. The final difference synthesis showed no peaks greater than 3.48 e Å^{-3}, the largest being near the Au atom. Final atomic co-ordinates are given in Table 2 for the non-hydrogen atoms, according to the numbering scheme in the Figure.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom co-ordinates and thermal parameters.

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