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Syntheses of gold–manganese and gold–rhenium clusters, $[(Ph_3PAu)_3M(CO)_4]$ and $[(Ph_3PAu)_4M(CO)_4]^+$ (M = Mn, Re), and the crystal structure of $[(Ph_3PAu)_4Mn(CO)_4]BF_4$

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Abstract

The specific additions of one, three or four Ph_3PAu groups to $[M(CO)_5]^-$ (M = Mn, Re) are described. Thus $[M(CO)_5]^-$ in THF reacts with $[(Ph_3PAu)_3O]BF_4$ to give $[(Ph_3PAu)_4Mn(CO)_4]BF_4$. An X-ray crystal structure of the M = Mn example shows the cation to have a trigonal bipyramidal Au_4Mn core with the Mn in an equatorial site. The previously known neutral $(Ph_3PAu)_3M(CO)_4$ clusters are formed by addition of two Ph_3PAu groups, using the mixed reagent $[(Ph_3PAu)_3O]BF_4$ / $[ppn][Co(CO)_4]$, to $Ph_3PAuM(CO)_5$, which itself is readily prepared from $[M(CO)_5]^-$ and Ph_3PAuCl.

Introduction

The current interest in mixed-metal clusters incorporating gold in the core [1] stems partly from the development of synthetic routes involving (i) the reaction of cluster anions with Ph_3PAuX [2], or (ii) reaction of cluster hydrides with Ph_3PAuMe [3], or (iii) reaction of $[(Ph_3PAu)_3O]^+$ with cluster anions [4]. As this chemistry has developed, a parallel has been established between the addition of Ph_3PAu^+ and H^+ to cluster anions and this has been discussed in terms of an isolobal correspondence [5]. Recently this analogy has been extended by comparing the additions of H–H and Ph_3PAu–$AuPPh_3$ to neutral clusters [6].

In this paper, we report on the preparation of some neutral and cationic gold–manganese and gold–rhenium clusters. These are formed by the systematic addition of one, three, or four $AuPPh_3$ groups, respectively, to $[M(CO)_5]^-$ to give

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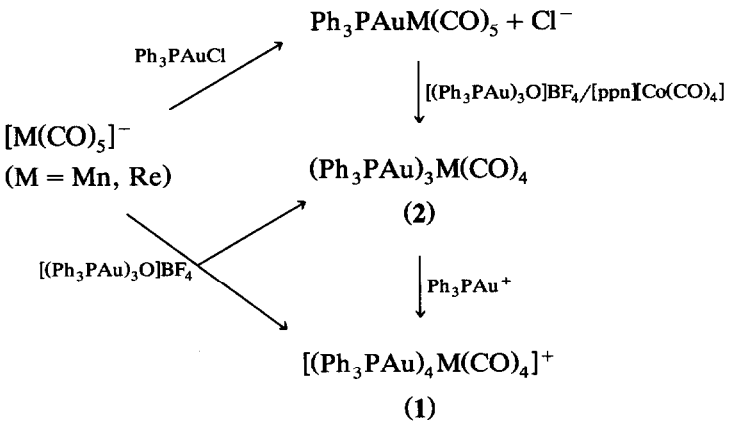


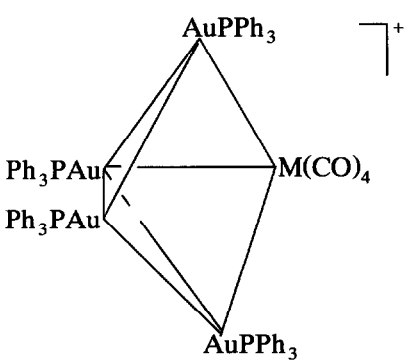
Scheme 1.

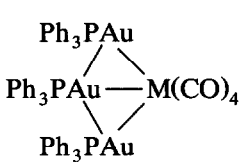
the neutral complexes $Ph_3PAuM(CO)_5$ and $(Ph_3PAu)_3M(CO)_4$ and the cationic clusters $[(Ph_3PAu)_4M(CO)_4]^+$ (M = Mn, Re).

Results and discussion

The chemistry described in this paper is summarized in Scheme 1. The addition of a THF solution of $[Mn(CO)_5]^-$ to a suspension of $[(Ph_3PAu)_3O]BF_4$ in the same solvent gives $Ph_3PAuMn(CO)_5$ and the light-orange cation $[(Ph_3PAu)_4Mn(CO)_4]^+$ (**1a**), which has been isolated as the BF_4^- salt and fully characterized by an X-ray crystal structure determination (see below).

With $[Re(CO)_5]^-$ and $[(Ph_3PAu)_3O]BF_4$, a similar reaction takes place to give $Ph_3PAuRe(CO)_5$ and a new brown-yellow cationic cluster $[(Ph_3PAu)_4Re(CO)_4]^+$ (**1b**) as the major product, together with a third component with ν(CO) bands at 1995 and 1884 cm^{-1} which could not be isolated pure from this reaction but was subsequently identified as the neutral species $(Ph_3PAu)_3Re(CO)_4$ (**2b**) via an alternative synthesis (see below). Thus, overall, $[Mn(CO)_5]^-$ and $[Re(CO)_5]^-$ react in a broadly similar manner with $[(Ph_3PAu)_3O]^+$, giving $[(Ph_3PAu)_4M(CO)_4]^+$ (M = Mn or Re) as the major products, but with differing amounts of by-products.





(**1a**) M = Mn
(**1b**) M = Re

(**2a**) M = Mn
(**2b**) M = Re

The correspondence of the species **1a** and **1b** was established by the closely similar infrared spectra; the profiles are essentially identical, with the band positions of the rhenium compound shifted by *ca.* 30 cm^{-1} to higher frequencies compared to the manganese example, as is usually observed for related complexes involving these metals. The formulation of the new clusters was indicated by FAB-MS, where the major peaks were assigned to $[(Ph_3PAu)_4Mn(CO)_4]^+$ and subsequent fragmentation ions, although this evidence has to be interpreted with caution since the observation of ions arising from combination of Ph_3PAu^+ with the parent ion is common in the FAB-MS of gold-containing clusters [7]. This is related in terms of isolobality to the frequently observed $[M + H]^+$ species found in the mass spectra of organic compounds. For example, the FAB-MS of $[(Ph_3PAu)_4M(CO)_4]BF_4$ and $(Ph_3PAu)_3M(CO)_4$ are indistinguishable, both containing a strong peak corresponding to $[(Ph_3PAu)_4M(CO)_4]^+$ (M = Mn or Re).

The unequivocal characterization of the clusters **1** was achieved by an X-ray crystal structure determination of $[(Ph_3PAu)_4Mn(CO)_4]BF_4$ (see below). Confirmation was also provided by comparison with a recent report of an independent synthesis of **1a** via photolysis of $Mn_2(CO)_{10}$ with Ph_3PAuN_3 [8].

It has been shown that the reagent combination $[(Ph_3PAu)_3O]BF_4$ + $[ppn][Co(CO)_4]$ ($[ppn]^+ = [N(PPh_3)_2]^+$) can act as a source of $Ph_3PAu–AuPPh_3$ which adds to neutral clusters, usually with concomitant loss of CO [6]. The mechanisms of these reactions are not known, but a reasonable sequence involves abstraction by $[Co(CO)_4]^-$ of one Ph_3PAu^+ unit from $[(Ph_3PAu)_3O]^+$ to give $Ph_3PAuCo(CO)_4$ (an observed by-product), the remaining $(Ph_3PAu)_2O$ species adding two Ph_3PAu units to the substrate as the O is eliminated, probably as CO_2. Although this is still speculative, reactions observed to date are consistent with this outline.

On the basis of these precedents, we envisaged an alternative route to the neutral clusters described above, involving addition of two gold units with the $[(Ph_3PAu)_3O]^+ + [Co(CO)_4]^-$ reagent to the mono-gold species $Ph_3PAuM(CO)_5$ (M = Mn, Re), which are readily available. The major products of these reactions were identified by their characteristic infrared spectra as the neutral clusters $(Ph_3PAu)_3M(CO)_4$ (**2**). The FAB-MS were also consistent with this formulation. These clusters have been previously synthesised by Ellis and Faltynek [9] by the procedure of eq. 1, and the example with M = Mn was structurally characterized [10], showing an unusual planar Au_3Mn core; *cf.* the more usual tetrahedral unit such as that in $(Ph_3PAu)_3V(CO)_5$ [11].

$$[M(CO)_4]^{3-} + 3Ph_3PAuCl \rightarrow (Ph_3PAu)_3M(CO)_4 + 3Cl^- \quad [M = Mn\ (\mathbf{2a}),\ Re\ (\mathbf{2b})] \qquad (1)$$

This new synthesis of the neutral species, **2**, is an improvement over the route devised by Ellis in that overall yields are better and the use of the highly reduced $[M(CO)_4]^{3-}$ anions is avoided.

Infrared monitoring of CH_2Cl_2 solutions of the neutral species **2** showed that slow rearrangement to the cationic **1** occurred (*ca.* 10% after 14 h), presumably by transfer of Ph_3PAu^+ entities during slow decomposition of part of the sample of **2**. In contrast, solutions of **1** showed no changes after 24 h, indicating greater stability.

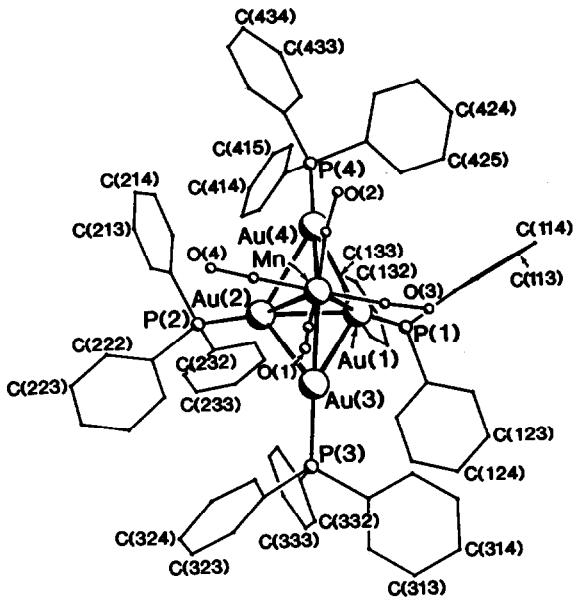


Fig. 1. The structure of the cation **1a**, $[(Ph_3P)_4Mn(CO)_4]^+$. Bond lengths (Å) include: Au(1)–Au(2) 2.789(2); Au(1)–Au(3) 2.925(2); Au(1)–Au(4) 2.800(2); Au(2)–Au(3) 2.822(2); Au(2)–Au(4) 2.949(2); Mn–Au(1) 2.766(5); Mn–Au(2) 2.756(5); Mn–Au(3) 2.658(4); Mn–Au(4) 2.671(4); Au(1)–P(1) 2.299(8); Au(2)–P(2) 2.28(1); Au(3)–P(3) 2.290(8); Au(4)–P(4) 2.274(9).

The ^{31}P NMR spectra of the cations $[(Ph_3PAu)_4M(CO)_4]^+$ gave single peaks at δ 45.7 (M = Mn) or 58.3 (M = Re) down to −40°C, indicating that fluxional processes exchange the Ph_3PAu groups between the axial and equatorial sites of the core; this is not unexpected for a trigonal bipyramidal molecule. Fluxionality leading to averaging over different Ph_3PAu sites has been previously described for a variety of other cluster geometries [1]. In contrast, the neutral species $(Ph_3PAu)_3Mn(CO)_4$ shows a single ^{31}P signal (δ 43.9) at room temperature but two peaks at −38°C which is consistent with the planar core found in the solid state being maintained in solution; a tetrahedral Au_3Mn molecule would have equivalent Ph_3P groups.

The crystal structure of **1a** · BF_4 was determined to establish its identity. The geometry of the cation is illustrated in Fig. 1. The structure of **1a** as the PF_6^- salt has been reported independently [8]; the two salts form isomorphous crystals, made possible by the disorder of the anions. The overall features of the cation are the same for the two determinations, although the Au–Au and Au–Mn bonds appear to be consistently 1–2% longer in **1a** · BF_4 than in **1a** · PF_6. The overall core geometry is that of an Au_4Mn trigonal bipyramid, with the Mn atom in an equatorial site. Each of the gold atoms carries a Ph_3P group, and the Mn atom is bonded to four CO ligands. These are arranged so that they nearly eclipse each of the Mn–Au bonds, and the short Au · · · C distances (2.59 Å av.) and non-linear Mn–C–O angles (171° av.) suggests that they can be regarded as weakly semi-bridging. Other aspects of the structure have been discussed in the report of the **1a** · PF_6 compound [8].

Experimental

Reactions were performed under dry nitrogen, but subsequent work-up involved no special precautions. $Ph_3PAuM(CO)_5$ (M = Mn, Re) [12], $[(Ph_3PAu)_3O][BF_4]$ [13], and $[ppn][Co(CO)_4]$ [14] were prepared by published methods.

Reaction of $[(Ph_3PAu)_3O][BF_4]$ with $[Re(CO)_5]^-$

A solution of $Re_2(CO)_{10}$ (130 mg, 0.2 mmol) in THF (30 ml) was reduced by stirring with 1% sodium amalgam for 90 min. Three millilitres of this solution (0.04 mmol $Re(CO)_5^-$) were added dropwise during 5 min to a suspension of $[(Ph_3PAu)_3O][BF_4]$ (60 mg, 0.04 mmol) in THF (5 ml). The solution turned dark yellow after 5 min, and a precipitate was present. After *ca.* 30 min, the reaction appeared to be complete, giving a brown, virtually homogeneous, solution with an unchanging infrared spectrum. The solvent was taken off and the residue extracted with Et_2O to remove $Ph_3PAuRe(CO)_5$, identified by IR (ν(CO) 2083m, 1969vs cm^{-1} [12]). The remaining solid was dissolved in the minimum of CH_2Cl_2, and the solution was filtered, layered with an equal volume of Et_2O, and left at −30°C. Golden-yellow crystals (which crumbled to a yellow powder on drying) of $[(Ph_3PAu)_4Re(CO)_4]BF_4$ (**1b**) were formed (32 mg, 47%), m.p. 140°C (dec). Anal. Found: C, 41.38; H, 2.53. $C_{76}H_{60}Au_4O_4P_4ReBF_4$ calc.: C, 41.08; H, 2.72%. IR (CH_2Cl_2): ν(CO) 2024s, 1953sh, 1938vs cm^{-1}. FAB-MS: $(m/e)^+$ at 2134s (M = $(Ph_3PAu)_4Re(CO)_4^+$); 1845s [$M - Ph_3P - CO$]; 1675w [$M - AuPPh_3$]; 1583m [$M - 2Ph_3P - CO$]; 1377w [$Au_3(PPh_3)_3$]; 1234w; 1115w [$Au_3(PPh_3)_2$]; 1101w. ^{31}P NMR ($CDCl_3$): δ 58.3.

Reaction of $[(Ph_3PAu)_3O][BF_4]$ with $[Mn(CO)_5]^-$

A solution of $Mn_2(CO)_{10}$ (130 mg, 0.33 mmol) in THF (25 ml) was reduced by stirring with 1% sodium amalgam for 90 min; 2.5 ml of this solution (0.066 mmol $Mn(CO)_5^-$) were added dropwise to a suspension of $[(Ph_3PAu)_3O][BF_4]$ (100 mg, 0.067 mmol) in THF (5 ml). The mixture was stirred for 90 min. The solvent was removed from the dark brown solution. Extraction of the residue with 8% CH_2Cl_2/92% Et_2O (3 ml) removed $Ph_3PAuMn(CO)_5$ (ν(CO) 2061s, 1953vs [12]) while CH_2Cl_2 dissolved a component, which was recrystallised twice from CH_2Cl_2/Et_2O to give light orange-brown crystals of $[(Ph_3PAu)_4Mn(CO)_4]BF_4$ (**1a**), ν(CO) (CH_2Cl_2) 1988s, 1928sh, 1911vs (*cf.* [8] 1985s, 1930m, 1905vs). FAB-MS: $(m/e)^+$ at 2003s [M = $[(Ph_3PAu)_4Mn(CO)_4]^+$]; 1975m [$M - CO$]; 1891m [$M - 4CO$]; 1629s [$M - 4CO - Ph_3P$]; 1544w [$M - AuPPh_3$]$^+$; 1377s [$Au_3(PPh_3)_3$]; 1114w; 1078w. ^{31}P NMR ($CDCl_3$): δ 45.7. The compound was further characterized by an X-ray crystal structure determination.

Reaction of $Ph_3PAuMn(CO)_5$ with $[(Ph_3PAu)_3O][BF_4]$ + $[ppn][Co(CO)_4]$

Solid $[ppn][Co(CO)_4]$ (36 mg, 0.051 mmol) was added in one portion to a mixture of $Ph_3PAuMn(CO)_5$ (33 mg, 0.051 mmol) and $[(Ph_3PAu)_3O][BF_4]$ (75 mg, 0.051 mmol) in THF (10 ml). Reaction seemed to be complete after 30 min, although some starting Au–Mn compound was still present. Distinct peaks from $Ph_3PAuCo(CO)_4$ (2054w, 1960s cm^{-1} [12]) were present in an infrared spectrum, with the main product peaks at 1979(sh)m, 1954s, 1884s, 1865(sh), corresponding to those of $(Ph_3PAu)_3Mn(CO)_4$ (**2a**) [9]. On leaving up to 80 min, the peaks due to

$[(Ph_3PAu)_4Mn(CO)_4]^+$ (**1a**) at 1987 and 1911 cm^{-1} grew slowly. After the THF was removed, a CH_2Cl_2/Et_2O (1:10) extract of the residue showed the products to be a mixture of **1a** and **2a**, with the latter predominating. Recrystallization from CH_2Cl_2/Et_2O gave $(Ph_3PAu)_3Mn(CO)_4$ as golden yellow microcrystals, ν(CO) (THF) 1983(sh)s, 1953vs, 1884vs, 1864(sh). ^{31}P NMR ($CDCl_3$): δ 43.87 (20°C); δ 43.84, 43.93 (−38°C).

Reaction of $Ph_3PAuRe(CO)_5$ with $[(Ph_3PAu)_3O][BF_4]$ + [ppn][$Co(CO)_4$]

Solid [ppn][$Co(CO)_4$] (36 mg, 0.051 mmol was added in one portion to a mixture of $Ph_3PAuRe(CO)_5$ (40 mg, 0.051 mmol) and $[(Ph_3PAu)_3O][BF_4]$ (75 mg, 0.051 mmol) in THF (10 ml). Reaction was complete after 10 min, leaving no starting Au–Re compound, and showing distinct infrared peaks from $Ph_3PAuCo(CO)_4$ (2054w, 1960s), and main product peaks at 1996m, 1916s, 1887s cm^{-1}. On leaving up to 30 min, the peaks at 2025 and 1940 cm^{-1} grew slowly. After THF was removed, an extraction with Et_2O (10 ml) removed the $Ph_3PAuCo(CO)_4$, and a CH_2Cl_2/Et_2O (1:4) extract of the residue showed the products to be a mixture of **1b** and **2b**, with the latter predominating. Recrystallization from CH_2Cl_2/Et_2O gave fairly pure $[(Ph_3PAu)_3Re(CO)_4]$ (**2b**), although it proved difficult to obtain a sample completely free from **1b**. The compound **2b** was identified by its infrared (ν(CO) 1999m, 1922(sh), 1912vs, 1892s cm^{-1} [9]) and FAB-MS characteristics. Crude yields were reasonable (*ca.* 60%), but losses occurred on recrystallization, and the compound can be chromatographed only with extensive loss.

X-Ray crystal structure of $[(Ph_3PAu)_4Mn(CO)_4]BF_4$

A small single crystal of $[(Ph_3PAu)_4Mn(CO)_4]BF_4$ was ultimately obtained from $CH_2Cl_2/^iPrOH$ and was embedded in epoxy resin to hinder loss of solvent from the lattice. Cell constants and intensity data were obtained on an Enraf–Nonius CAD4 diffractometer using monochromated Mo-K_α X-rays ($\lambda = 0.7107$ Å).

Crystal data. $C_{76}H_{60}Au_4MnO_4P_4^+BF_4^- \cdot 0.3CH_2Cl_2$, M_r 2119.1. Triclinic, space group $P\bar{1}$, $a = 17.147(1)$, $b = 20.361(2)$, $c = 11.842(1)$ Å, α 106.667(8), β 98.595(7), γ 103.052(8), $U = 3755.4$ Å^3, $D_c = 1.87$ g cm^{-3} for $Z = 2$. $F(000) = 2008$, μ(Mo-K_α) = 81 cm^{-1}, $T = 23$°C.

A total of 5782 unique reflections in the range $3° < 2\theta < 36°$ ($-16 < h < 0$; $-19 < k < 19$; $-11 < l < 11$) were collected using ω-scans. After corrections for Lorentz, polarization and absorption effects, 3892 data had $I > 3\sigma(I)$ and were used in all calculations. The structure was solved by direct methods for the metal atoms, with other atoms being located in subsequent difference maps. The BF_4^- anion was poorly defined because of disorder, while a penultimate difference map indicated residual electron density which was modelled as *ca.* $0.3CH_2Cl_2$. In the final cycles of blocked-matrix least-squares refinement, the Au, Mn, P and CO atoms were assigned anisotropic temperature factors, the phenyl rings were treated as rigid hexagons with isotropic temperature factors, the BF_4^- and solvent molecule atoms were treated isotropically, and H atoms were not included. Refinement converged with $R = 0.0681$, $R_w = 0.0689$ with $w = [\sigma^2(F) + 0.00227F^2]^{-1}$. Maximum shifts in the final cycle were 0.05σ, and the maximum feature in the final difference map was 2 e Å^{-3}, adjacent to Au(2). Final atomic parameters are listed in Table 1, while selected bond lengths are included in the caption to Fig. 1, which illustrates the structure of the cation.

Table 1

Final positional parameters for $[(Ph_3PAu)_4Mn(CO)_4]BF_4$

Atom	x	y	z	Atom	x	y	z
Au(1)	0.1434(1)	0.1754(1)	−0.2765(1)	Au(2)	0.1462(1)	0.2448(1)	−0.0352(1)
Au(3)	−0.0086(1)	0.1636(1)	−0.1901(1)	Au(4)	0.2591(1)	0.1581(1)	−0.1021(1)
Mn(1)	0.0993(3)	0.0976(3)	−0.1277(4)	P(1)	0.1662(5)	0.2092(5)	−0.4408(7)
P(2)	0.1833(6)	0.3567(6)	0.1044(8)	P(3)	−0.1217(5)	0.2006(6)	−0.2397(9)
P(4)	0.3986(6)	0.1857(6)	−0.0650(9)	C(1)	0.008(2)	0.057(2)	−0.110(3)
C(2)	0.155(2)	0.042(2)	−0.099(3)	C(3)	0.071(2)	0.050(2)	−0.289(4)
C(4)	0.123(3)	0.137(3)	0.033(5)	O(1)	−0.055(2)	0.020(2)	−0.101(3)
O(2)	0.187(2)	−0.004(2)	−0.091(3)	O(3)	0.049(2)	0.006(2)	−0.389(3)
O(4)	0.134(2)	0.158(2)	0.141(3)	C(111)	0.213(1)	0.153(1)	−0.542(2)
C(112)	0.266(1)	0.178(1)	−0.608(2)	C(114)	0.267(1)	0.057(1)	−0.712(2)
C(115)	0.213(1)	0.032(1)	−0.646(2)	C(116)	0.186(1)	0.079(1)	−0.561(2)
C(121)	0.073(1)	0.209(1)	−0.535(2)	C(122)	0.043(1)	0.163(1)	−0.654(2)
C(123)	−0.031(1)	0.164(1)	−0.722(2)	C(124)	−0.074(1)	0.211(1)	−0.671(2)
C(125)	−0.044(1)	0.257(1)	−0.552(2)	C(126)	0.030(1)	0.256(1)	−0.484(2)
C(131)	0.239(1)	0.297(1)	−0.402(2)	C(132)	0.310(1)	0.315(1)	−0.311(2)
C(133)	0.369(1)	0.381(1)	−0.282(2)	C(134)	0.356(1)	0.428(1)	−0.343(2)
C(135)	0.285(1)	0.410(1)	−0.434(2)	C(136)	0.227(1)	0.344(1)	−0.463(2)
C(211)	0.280(1)	0.379(1)	0.208(2)	C(212)	0.333(1)	0.449(1)	0.257(2)
C(213)	0.410(1)	0.463(1)	0.332(2)	C(214)	0.435(1)	0.407(1)	0.357(2)
C(215)	0.383(1)	0.337(1)	0.308(2)	C(216)	0.305(1)	0.323(1)	0.234(2)
C(221)	0.110(1)	0.373(1)	0.198(2)	C(222)	0.135(1)	0.422(1)	0.315(2)
C(223)	0.076(1)	0.435(1)	0.382(2)	C(224)	−0.007(1)	0.399(1)	0.332(2)
C(225)	−0.031(1)	0.349(1)	0.214(2)	C(226)	0.027(1)	0.336(1)	0.148(2)
C(231)	0.190(2)	0.425(1)	0.033(2)	C(232)	0.173(2)	0.489(1)	0.087(2)
C(233)	0.179(2)	0.541(1)	0.031(2)	C(234)	0.204(2)	0.530(1)	−0.078(2)
C(235)	0.222(2)	0.467(1)	−0.132(2)	C(236)	0.215(2)	0.414(1)	−0.077(2)
C(311)	−0.189(2)	0.144(1)	−0.387(2)	C(312)	−0.273(2)	0.138(1)	−0.410(2)
C(313)	−0.323(2)	0.098(1)	−0.525(2)	C(314)	−0.289(2)	0.065(1)	−0.618(2)
C(315)	−0.205(2)	0.072(1)	−0.595(2)	C(316)	−0.155(2)	0.111(1)	−0.480(2)
C(321)	−0.185(1)	0.200(1)	−0.130(2)	C(322)	−0.220(1)	0.255(1)	−0.089(2)
C(323)	−0.264(1)	0.255(1)	0.002(2)	C(324)	−0.271(1)	0.200(1)	0.051(2)
C(325)	−0.236(1)	0.145(1)	0.010(2)	C(326)	−0.193(1)	0.145(1)	−0.081(2)
C(331)	−0.097(2)	0.290(1)	−0.251(2)	C(332)	−0.149(2)	0.312(1)	−0.326(2)
C(333)	−0.125(2)	0.381(1)	−0.333(2)	C(334)	−0.050(2)	0.428(1)	−0.264(2)
C(335)	0.003(2)	0.406(1)	−0.190(2)	C(336)	−0.021(2)	0.338(1)	−0.183(2)
C(411)	0.451(2)	0.281(1)	−0.024(2)	C(412)	0.408(2)	0.332(1)	0.009(2)
C(413)	0.448(2)	0.404(1)	0.041(2)	C(414)	0.531(2)	0.427(1)	0.040(2)
C(415)	0.574(2)	0.376(1)	0.007(2)	C(416)	0.534(2)	0.303(1)	−0.025(2)
C(421)	0.433(2)	0.141(2)	−0.195(2)	C(422)	0.504(2)	0.119(2)	−0.185(2)
C(423)	0.531(2)	0.089(2)	−0.289(2)	C(424)	0.486(2)	0.082(2)	−0.403(2)
C(425)	0.415(2)	0.104(2)	−0.413(2)	C(426)	0.388(2)	0.133(2)	−0.309(2)
C(431)	0.440(2)	0.151(2)	0.047(2)	C(432)	0.407(2)	0.078(2)	0.031(2)
C(433)	0.439(2)	0.049(2)	0.113(2)	C(434)	0.504(2)	0.091(2)	0.212(2)
C(435)	0.537(2)	0.163(2)	0.228(2)	C(436)	0.505(2)	0.193(2)	0.146(2)
B(1)	0.733(5)	0.314(5)	0.380(7)	F(1)	0.726(4)	0.411(3)	0.447(5)
F(2)	0.726(7)	0.383(6)	0.281(9)	F(3)	0.799(6)	0.400(5)	0.364(9)
F(4)	0.681(6)	0.351(5)	0.340(9)	C(01) [a]	0.405(6)	0.708(6)	0.424(9)
Cl(1) [a]	0.496(3)	0.715(3)	0.462(4)	Cl(2) [a]	0.360(4)	0.668(4)	0.307(6)

[a] Site occupancy 0.3.

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