Syntheses of Some Organogoldtriosmium Clusters; *X*-Ray Crystal Structure of $[Os_3(CO)_8(PPh_3)\{Au(PPh_3)\}(2\text{-}NHC_5H_4N)]$ †

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The clusters $[Os_3(CO)_9\{Au(PPh_3)\}(C_2Ph)]$ (1), $[Os_3(CO)_{10}\{Au(PPh_3)\}(OH)]$ (2), $[Os_3(CO)_{10}\{Au(PPh_3)\}(NHSO_2C_6H_4Me\text{-}p)]$ (3), and $[Os_3(CO)_8(Y)\{Au(PPh_3)\}(2\text{-}NHC_5H_4N)]$ [Y = PPh_3 (4) or CO (5)] were prepared from the appropriate neutral osmium cluster and chloro(triphenylphosphine)gold(I). $[Os_3(CO)_8(PPh_3)\{Au(PPh_3)\}(2\text{-}NHC_5H_4N)]$ (4) was shown by *X*-ray analysis to consist of an isosceles triangle of osmium atoms substituted by an equatorial phosphine ligand and eight carbonyls. A 2-pyridylamido ligand caps one side of the Os_3 triangle and a (triphenylphosphine)aurio group bridges the longest Os–Os bond. The complex crystallises in space group $P2_1/n$ with a = 14.567(5), b = 18.010(24), c = 18.456(7) Å, β = 92.22(3)°, and Z = 4. The structure was solved by a combination of direct methods and Fourier-difference techniques, and refined by blocked-cascade least-squares methods to R = 0.065 to 3 541 observed diffractometer data.

Hydride ligands and the *sp*-hybridized gold cation of a gold(I) phosphine fragment, $Au(PR_3)$, each can donate one electron to a metal cluster and there is increasing evidence that [gold(I) phosphine]triosmium clusters often bear a close structural analogy to the corresponding hydridotriosmium clusters.[1] Osmium cluster anions react readily with chlorogold(I) phosphines, $[Au(PR_3)Cl]$ (R = Et or Ph), affording goldpolyosmium clusters.[2-4] This paper describes some reactions in which hydridotriosmium clusters have been deprotonated and reacted *in situ* with a gold electrophile. This approach has proved to be especially useful in the synthesis of goldtriosmium clusters containing organic functionality.

Results and Discussion

Nucleophilic anions, such as Me^- and NR_2^- (R = alkyl), add to $[Os_3(CO)_{12}]$ by attack at a carbonyl ligand to give an intermediate which may then rearrange with loss of CO.[5,6] Attempts to react such anionic intermediates, formed from a variety of nucleophiles such as ethoxide or butyl-lithium, with chloro(triphenylphosphine)gold(I) gave complex mixtures of products, but in one case using phenylacetylide a product $[Os_3(CO)_9\{Au(PPh_3)\}(C_2Ph)]$ (1) was isolated in low and variable yields.

The complex (1) was characterised by mass spectroscopic, ^1H n.m.r., i.r. (Table 1), and analytical (Table 2) data. If, as in other cases,[2,4] the $Au(PPh_3)$ group adopts the same bonding mode as a bridging hydride the geometry of this complex is closely related to that of $[Ru_3(CO)_9H(C_2Ph)]$,[7] which is illustrated in Figure 1. Cluster (1) may also be prepared by the reaction of phenylethynyl(triphenylphosphine)gold(I), $[Au(PPh_3)(C_2Ph)]$, with $[Os_3(CO)_{10}(NCMe)_2]$ in 28% yield based on the acetonitrile cluster.

A more general method of forming organogoldtriosmium clusters is to form the intermediate anions by deprotonation. Lithium di-isopropylamide was chosen as a base so that addition to carbonyl ligands is sterically disfavoured. As a model reaction, $[Os_3(CO)_{10}H(OH)]$ in diethyl ether was treated with $Li[NPr^i_2]$ at −78 °C, warmed to 20 °C, and $[Au(PPh_3)Cl]$ with $Tl[PF_6]$ (to abstract the Cl^-) were added. The cluster $[Os_3(CO)_{10}\{Au(PPh_3)\}(OH)]$ (2) was isolated in 7% yield from the reaction mixture. Similar reactions using organotriosmium clusters are summarized in the Scheme. The chloro(triphenylphosphine)gold(I) compound as prepared here (see Experimental section) clearly contains some triphenylphosphine impurity and the substitution of a carbonyl ligand in complex (4) is a consequence of this. The method was most effective for clusters bearing ligands which could easily be deprotonated whereas other clusters, for example $[Os_3(\mu\text{-}H)(\mu\text{-}Cl)(CO)_{10}]$, gave complex mixtures and no products were isolated.

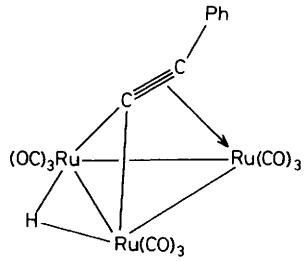


Figure 1. Structure of $[Ru_3(CO)_9H(C_2Ph)]$

The molecular geometry of $[Os_3(CO)_8(PPh_3)\{Au(PPh_3)\}(2\text{-}NHC_5H_4N)]$ (4) was established by a single-crystal *X*-ray analysis; the structure is shown in Figure 2 and the cluster core is illustrated in Figure 3. Selected bond lengths and angles are presented in Table 3. There are no intermolecular short contacts within the crystal structure.

The three Os atoms lie at the vertices of an isosceles triangle, the longer edge of which is asymmetrically bridged by the Au atom of the $Au(PPh_3)$ group. A 2-pyridylamido ligand caps the Os_3 triangle on the side away from the $Au(PPh_3)$ ligand; the N(1) atom symmetrically bridges the longer Os(1)–Os(2) bond, the same bond as the $Au(PPh_3)$ group, while the N(2) atom is co-ordinated to the third Os atom, Os(3), occupying an axial site. A PPh_3 ligand is terminally co-ordinated to Os(1) in an equatorial position. The eight carbonyl groups are terminally co-ordinated to the three Os atoms, two to Os(1), and three each to Os(2) and Os(3), in positions similar to those observed in a number of substituted $[Os_3(CO)_9H(Y)]$ cluster complexes.[8]

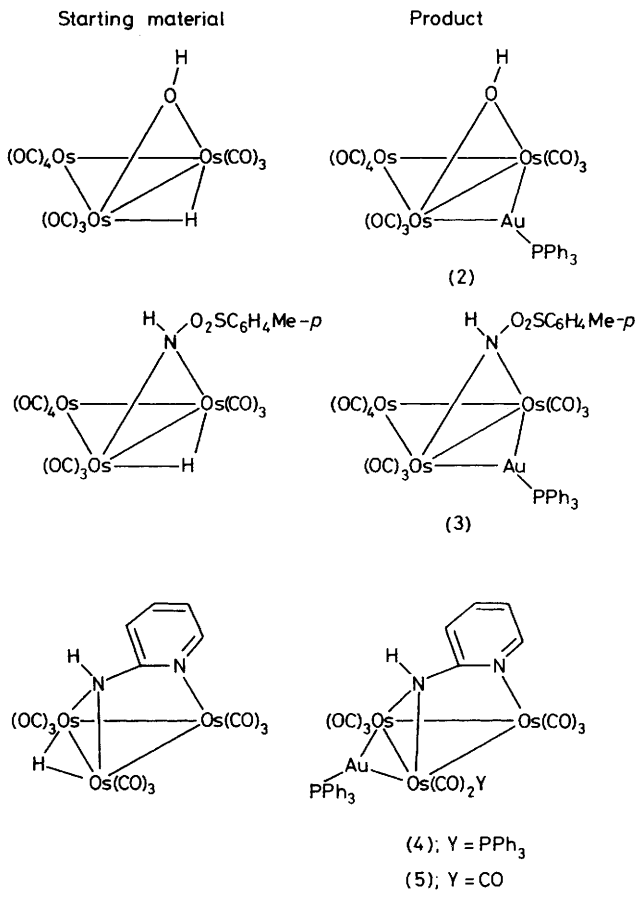
† 1,1,2,2,2,3,3,3-Octacarbonyl-μ_3-[2-pyridylamido-*N*(Os^{1,2})-*N'*(Os^3)]-1-(triphenylphosphine)-1,2-μ-(triphenylphosphineaurio)-*triangulo*-triosmium.

Supplementary data available (No. SUP 23628, 29 pp.): full bond length and bond angle data, hydrogen atom co-ordinates, least-squares planes, anisotropic thermal parameters, observed and calculated structure factors. See Notices to Authors No. 7, *J. Chem. Soc., Dalton Trans.*, 1981, Index issue.

Table 1. Spectral data for the complexes

Complex	*m/e* [M^+(^{192}Os,^{107}Au)]	^{1}H n.m.r. spectrum [a] δ/p.p.m.	I.r. spectrum [a] ν(CO)/cm^{-1}
(1)	1 388	7.69—7.45 (m)	2 094m, 2 070w, 2 041s, 2 013s, 1 993w, 1 973w, 1 964w
(2)	1 332	7.63—7.46 (m, 15 H), 3.44 (s br, 1 H)	2 092w, 2 040s, 2 004s, 1 968m, 1 956m [b]
(3)	1 485	7.86—7.35 (m, 19 H), 5.67 (s br, 1 H), 2.46 (s, 3 H)	2 090m, 2 047s, 2 038m, 2 006m, 1 989m, 1 976 (sh) [b]
(4)	*c*	8.55 (m, 1 H), 7.80—7.39 (m, 15 H), 6.71—6.41 (m, 3 H), 4.19 (s br, 1 H)	2 081w, 2 058w, 2 023s, 2 008m, 1 974m, 1 940m
(5)	*c*	8.53 (m, 1 H), 7.77—7.06 (m, 31 H), 6.61—6.42 (m, 2 H), 4.20 (s br, 1 H)	2 040m, 2 020m, 2 004s, 1 959s, 1 937m

[a] CD_2Cl_2 solution, unless otherwise stated. [b] Hexane solution. [c] Sample did not leave the probe at the ionising voltage.



Scheme. Yields of products: (2) 7, (3) 33, (4) 9, and (5) 8%

The overall structure of (4) closely resembles that of $[Os_3(CO)_9H(2\text{-}NHC_5H_4N)]$ [9] except that the bridging hydride in the latter has been replaced by the $Au(PPh_3)$ group. In both these clusters the di-bridged Os–Os bond is longer than other metal–metal bonds. However, the Os(1)–Os(2) bridged bond in (4) is *ca.* 0.09 Å longer than the equivalent bond [2.796(4) Å] in $[Os_3(CO)_9H(2\text{-}NHC_5H_4N)]$. Since the capping 2-pyridylamido ligand in the two complexes is the same the difference in this Os–Os bond length is either caused by the replacement of the bridging hydride by the $Au(PPh_3)$ group or by the replacement of an equatorial carbonyl group by the tri-

Table 2. Analytical data

Complex [a]	Formula	Found (%) [b] C	H	N
(1)	$C_{35}H_{20}AuO_9Os_3P$	30.6 (30.4)	1.5 (1.45)	—
(2)	$C_{28}H_{16}AuO_{11}Os_3P$	25.6 (25.35)	1.4 (1.2)	—
(3)	$C_{25}H_{23}AuNO_{12}Os_3PS$	29.05 (29.3)	1.8 (1.6)	1.2 (1.0)
(5)	$C_{32}H_{20}AuN_2O_9Os_3P$	28.35 (27.95)	1.9 (1.45)	—

[a] No analysis was obtained for complex (4) because of the low yield in which it was prepared. [b] Required values are in parentheses.

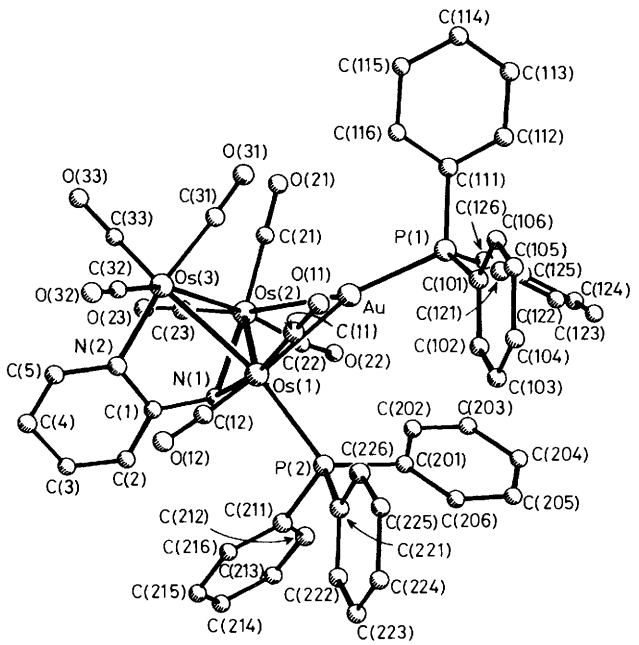


Figure 2. Structure of $[Os_3(CO)_8(PPh_3)\{Au(PPh_3)\}(2\text{-}NHC_5H_4N)]$ (4)

phenylphosphine group. The presence of a phosphorus donor in place of a carbonyl group in Os_3 systems causes an increase in metal–metal bond length of *ca.* 0.02 Å,[10] so that it seems that the main difference in the Os–Os bond length is due to the difference in the bridging functionality. A metal–metal bond

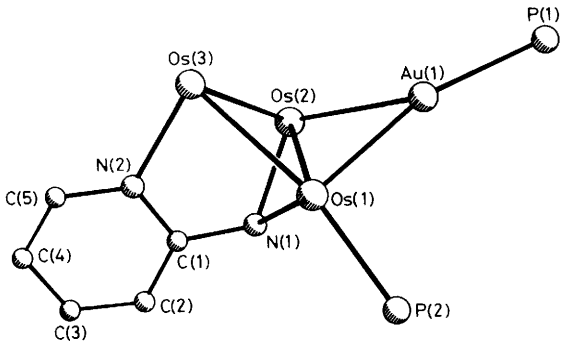


Figure 3. Geometry around the Os_3 cluster core of (4)

spanned only by a hydride is generally longer than a similar unbridged bond,[11] but a group with a ' bridgehead ' nitrogen atom often shortens a metal–metal bond. In this case it is probable that the Au atom has a slightly greater bond-lengthening influence than the hydride, and both these more than counterbalance the bond-shortening influence of the ' bridgehead ' N atom of the 2-pyridylamido ligand. The average Os–Os distance of 2.779(6) Å for the unbridged metal–metal bonds in $[Os_3(CO)_9H(2\text{-}NHC_5H_4N)]$[9] is *ca.* 0.014 Å shorter than the unbridged distances in (4), and both are shorter than the average Os–Os bond length of 2.877(3) Å in $[Os_3(CO)_{12}]$.[12] Capping groups similar to 2-pyridylamido appear to have a bond-shortening influence on all the metal–metal bonds that they cap.[8]

The N(1) atom of the 2-pyridylamido ligand symmetrically bridges the Os(1)–Os(2) bond with an average distance of 2.11(2) Å which is marginally shorter than the corresponding value of 2.18(2) Å in $[Os_3(CO)_9H(2\text{-}NHC_5H_4N)]$.[9] However, the σ-Os–N(pyridine) bond length of 2.164(13) Å in the latter complex is equivalent to the Os(3)–N(2) distance in (4). The differences in the bridged Os–N bond lengths between the two complexes may reflect differences in the electron distributions in the Os_2AuN and Os_2HN units. The other bond parameters within the 2-pyridylamido ligands in the two complexes are not significantly different, and do not deviate significantly from idealised values.

The Au atom of $Au(PPh_3)$ asymmetrically bridges the Os(1)–Os(2) bond, with the shorter distance to Os(2) which does not have the triphenylphosphine group co-ordinated to it. The average Os–Au bond length of 2.763(10) Å is similar to the average distance of 2.761(6) Å in $[Os_3Au(CO)_{10}(PPh_3)(SCN)]$, where the $Au(PPh_3)$ group also bridges a saturated Os–Os bond.[13] The Os–Au–Os angle in (4) is similar to the value of 63.1(1)° in the decacarbonyl cluster.[13]

All eight carbonyl groups are essentially linear with an average Os–C–O angle of 175(3)°. The average Os–C and C–O distances of 1.85(6) and 1.18(5) Å, respectively, are not significantly different from the lengths in the related cluster $[Os_3(CO)_9H(2\text{-}NHC_5H_4N)]$.[9] The bond parameters within the triphenylphosphine ligands associated with Os(1) and Au are in good agreement with the expected values.

Experimental

Proton n.m.r., i.r., and mass spectral data were recorded on Varian CFT20 at 80 MHz, Perkin-Elmer 257, and A.E.I. MS12 instruments respectively. Reactions were performed under nitrogen but t.l.c. separations were carried out in the air using commercial Merck silica plates.

Chloro(triphenylphosphine)gold(I) was prepared by adding a solution of triphenylphosphine (1.5 g) in acetone (5 cm^3) to a solution of ' chlorogold(III) acid ' ($HAuCl_4$) (1 g, 0.5 mol equiv.) in acetone at 20 °C. The volume of the solution was reduced to *ca.* 5 cm^3 and chloro(triphenylphosphine)gold(I) (1.2 g, 83%) precipitated.

$[Os_3(CO)_9\{Au(PPh_3)\}(C_2Ph)]$ (1).—(*A*) From $[Os_3(CO)_{12}]$. Dodecacarbonyltriosmium (0.111 g) and phenylethynyllithium (1 mol equiv.) were stirred in dry tetrahydrofurane (thf) (25 cm^3) for 10 min at 0 °C. $[Au(PPh_3)Cl]$ (1.1 mol equiv.) and $Tl[PF_6]$ (1.5 mol equiv.) were added and this mixture was then stirred at 20 °C for 24 h. Filtration (Celite), removal of the solvent, and t.l.c. [CH_2Cl_2–n-C_6H_{14} (40 : 60) eluant] gave the complex as an orange band (25 mg). A sample was recrystallized (Et_2O–n-C_6H_{14}) by slow evaporation at 0 °C.

(*B*) *From* $[Os_3(CO)_{10}(NCMe)_2]$. Bis(acetonitrile)decacarbonyltriosmium (90 mg) and $[Au(PPh_3)(C_2Ph)]$ (60 mg) were stirred in benzene (10 cm^3) for 2 h. Removal of the solvent and t.l.c. [as in (*A*)] gave $[Os_3(CO)_9\{Au(PPh_3)\}(C_2Ph)]$ (38 mg).

Formation of Clusters (2)—(5).—The appropriate triosmium cluster (Table 1) (*ca.* 100 mg) and $Li[NPr^i_2]$ (1.5 mol equiv.) were mixed in dry Et_2O (20 cm^3) at −78 °C. $[Au(PPh_3)Cl]$ (1 mol equiv.) and $Tl[PF_6]$ (1 mol equiv.) were added and the mixture was then stirred at 20 °C for 2 h. The clusters were separated in the same way as cluster (1) and recrystallized (CH_2Cl_2–n-C_6H_{14}) by slow evaporation at 0 °C.

Crystal Structure Determination.—Red ' block-shaped ' crystals of $[Os_3(CO)_8(PPh_3)\{Au(PPh_3)\}(2\text{-}NHC_5H_4N)]$ (4) were deposited from CH_2Cl_2–n-C_6H_{14}. 6 884 Intensities were recorded on a Stoe four-circle diffractometer using graphite-monochromated Mo-K_α radiation, and a crystal of dimensions *ca.* 0.51 × 0.27 × 0.12 mm. Unit-cell dimensions were determined from the angular measurements of 48 strong reflections with $15 < 2\theta < 25°$. Data were collected in the range $3.0 < 2\theta < 45.0°$ using 24-step ω—θ scan techniques. Three reflections were monitored every hour throughout the data collection and showed no significant variation.

A semi-empirical absorption correction, based on a pseudo-ellipsoid model, and Lorentz polarization corrections were applied to the ' on line ' profile-fitted data. Equivalent reflections were averaged to give 3 541 unique observed reflections [$F > 4\sigma(F)$].

Crystal data. $C_{49}H_{35}AuN_2O_8Os_3P_2$, $M = 1\,609.3$, Monoclinic, $a = 14.567(5)$, $b = 18.010(24)$, $c = 18.456(7)$ Å, $\beta = 92.22(3)°$, $U = 4\,838.3$ Å^3, $F(000) = 2\,976$, D_m not measured, $Z = 4$, $D_c = 2.21$ g cm^{-3}, Mo-K_α radiation, $\lambda = 0.710\,69$ Å, $\mu(\text{Mo-}K_\alpha) = 109.62$ cm^{-1}, space group $P2_1/n$ (non-standard setting of $P2_1/c$) from systematic absences.

The Au and Os atoms were located by multisolution Σ_2 sign expansion, and all the remaining non-hydrogen atoms from subsequent difference electron-density syntheses. The structure was refined by blocked-cascade least-squares methods with anisotropic thermal parameters assigned to Au, Os, P, N, O, and the C atoms of the 2-pyridylamido group, and isotropic temperature factors to the phenyl and carbonyl C atoms; the phenyl rings were refined as rigid groups with C–C set at 1.395 Å and C–C–C at 120.0°. The phenyl H atoms, and the H atoms co-ordinated to the 2-pyridylamido group were placed in idealised positions, and constrained to ride 1.08 Å from the atoms to which they are bonded; the H atoms were assigned a common isotropic thermal parameter. An unresolved solvent molecule was observed in the difference map, the electron density was refined as a lone carbon atom, C(100). In the final cycles of refinement the weighting scheme $w = [\sigma^2(F) + 0.001|F_o|^2]^{-1}$ was introduced. The converged residuals were $R = 0.065$ and

Table 3. Selected bond parameters

(*a*) Bond lengths (Å)

Os(1)–Os(2)	2.875(2)	N(1)–C(1)	1.395(33)	Os(2)–Os(3)	2.794(2)	Os(2)–N(1)	2.103(22)
Os(1)–Os(3)	2.793(2)	C(1)–C(2)	1.403(43)	Os(2)–Au	2.738(2)	C(2)–C(3)	1.342(53)
Os(1)–Au	2.788(2)	C(1)–N(2)	1.336(33)	Au–P(1)	2.304(9)	C(3)–C(4)	1.438(56)
Os(1)–P(2)	2.383(8)	C(5)–N(2)	1.350(40)	Os(1)–N(1)	2.108(22)	C(4)–C(5)	1.350(56)
Os(3)–N(2)	2.162(21)						

(*b*) Bond angles (°)

Os(2)–Os(1)–Os(3)	59.0(1)	Os(1)–N(1)–C(1)	116.6(15)	Os(3)–Os(2)–N(1)	74.1(6)
Os(2)–Os(1)–Au	57.8(1)	C(2)–C(1)–N(2)	119.6(24)	Au–Os(2)–N(1)	100.4(6)
Os(2)–Os(1)–P(2)	114.8(2)	C(1)–C(2)–C(3)	121.2(30)	Os(2)–Os(3)–N(2)	81.5(6)
Os(2)–Os(1)–N(1)	46.9(6)	C(3)–C(4)–C(5)	117.7(33)	Os(1)–Au–Os(2)	62.7(1)
P(2)–Os(1)–N(1)	91.9(6)	Os(3)–N(2)–C(1)	117.6(17)	Os(2)–Au–P(1)	155.6(2)
Os(1)–Os(2)–Os(3)	59.0(1)	Os(3)–N(2)–C(5)	122.4(20)	Os(2)–N(1)–C(1)	119.1(16)
Os(1)–Os(2)–Au	59.5(1)	Os(3)–Os(1)–Au	92.7(1)	N(1)–C(1)–C(2)	125.0(24)
Os(1)–Os(2)–N(1)	47.0(6)	Os(3)–Os(1)–P(2)	165.0(2)	N(1)–C(1)–N(2)	115.0(23)
Os(1)–Os(3)–Os(2)	61.9(1)	Os(3)–Os(1)–N(1)	74.1(6)	C(2)–C(3)–C(4)	118.2(33)
Os(1)–Os(2)–N(2)	82.1(6)	Au–Os(1)–P(2)	94.6(2)	C(4)–C(5)–N(2)	123.0(31)
Os(1)–Au–P(1)	141.5(2)	Au–Os(1)–N(1)	98.7(6)	C(1)–N(2)–C(5)	119.6(24)
Os(1)–N(1)–Os(2)	86.1(9)	Os(3)–Os(2)–Au	93.8(1)		

Table 4. Fractional atomic co-ordinates (× 10^4)

Atom	*X/a*	*Y/b*	*Z/c*	Atom	*X/a*	*Y/b*	*Z/c*
Os(1)	9 179(1)	3 778(1)	2 687(1)	C(214)	10 145(13)	6 913(12)	2 832(10)
Os(2)	8 898(1)	3 735(1)	4 220(1)	C(215)	9 625(13)	6 567(12)	2 278(10)
Os(3)	7 470(1)	3 404(1)	3 217(1)	C(216)	9 755(13)	5 815(12)	2 134(10)
Au	10 104(1)	2 777(1)	3 607(1)	C(221)	10 741(14)	4 377(10)	1 374(10)
P(1)	11 115(5)	1 794(6)	3 591(4)	C(222)	11 037(14)	5 010(10)	1 015(10)
C(101)	11 478(14)	1 663(10)	2 688(11)	C(223)	11 274(14)	4 965(10)	291(10)
C(102)	11 807(14)	2 298(10)	2 352(11)	C(224)	11 216(14)	4 287(10)	−73(10)
C(103)	11 979(14)	2 286(10)	1 614(11)	C(225)	10 921(14)	3 654(10)	286(90)
C(104)	11 822(14)	1 639(10)	1 212(11)	C(226)	10 683(14)	3 699(10)	1 010(10)
C(105)	11 493(14)	1 004(10)	1 549(11)	N(1)	8 883(15)	4 601(14)	3 455(10)
C(106)	11 321(14)	1 016(10)	2 287(11)	C(1)	8 076(16)	5 006(15)	3 328(13)
C(111)	10 677(10)	901(12)	3 845(10)	C(2)	8 006(23)	5 781(19)	3 375(14)
C(112)	11 296(10)	369(12)	4 116(10)	C(3)	7 193(30)	6 125(20)	3 278(17)
C(113)	10 993(10)	−348(12)	4 264(10)	C(4)	6 396(25)	5 680(24)	3 100(19)
C(114)	10 070(10)	−533(12)	4 141(10)	C(5)	6 502(23)	4 935(20)	3 091(18)
C(115)	9 451(10)	−1(12)	3 870(10)	N(2)	7 315(16)	4 597(12)	3 239(12)
C(116)	9 755(10)	716(12)	3 722(10)	C(11)	9 308(18)	2 917(17)	2 204(14)
C(121)	12 148(13)	1 952(12)	4 138(8)	O(11)	9 394(13)	2 364(12)	1 870(11)
C(122)	13 021(13)	1 919(12)	3 854(8)	C(12)	8 567(23)	4 150(20)	1 906(18)
C(123)	13 797(13)	2 058(12)	4 299(8)	O(12)	8 128(15)	4 399(16)	1 389(12)
C(124)	13 701(13)	2 231(12)	5 029(8)	C(21)	8 772(21)	2 868(20)	4 746(17)
C(125)	12 828(13)	2 264(12)	5 314(8)	O(21)	8 683(19)	2 287(14)	5 027(13)
C(126)	12 052(13)	2 125(12)	4 868(8)	C(22)	9 991(20)	4 058(18)	4 766(16)
P(2)	10 537(5)	4 408(5)	2 351(4)	O(22)	10 613(17)	4 178(15)	5 149(11)
C(201)	11 708(11)	4 189(12)	2 729(9)	C(23)	8 063(22)	4 234(21)	4 762(17)
C(202)	11 796(11)	4 031(12)	3 468(9)	O(23)	7 531(17)	4 549(17)	5 089(13)
C(203)	12 656(11)	3 856(12)	3 783(9)	C(31)	7 789(19)	2 390(19)	3 300(15)
C(204)	13 427(11)	3 839(12)	3 357(9)	O(31)	7 927(16)	1 762(13)	3 307(14)
C(205)	13 338(11)	3 997(12)	2 618(9)	C(32)	6 819(22)	3 282(20)	2 300(18)
C(206)	12 479(11)	4 172(12)	2 304(9)	O(32)	6 462(16)	3 192(16)	1 752(13)
C(211)	10 403(13)	5 408(12)	2 544(10)	C(33)	6 603(26)	3 294(22)	3 837(19)
C(212)	10 923(13)	5 754(12)	3 098(10)	O(33)	5 908(17)	3 141(15)	4 218(15)
C(213)	10 794(13)	6 506(12)	3 242(10)	C(100)	9 591(22)	663(21)	137(17)

$R' = \Sigma w^{\frac{1}{2}}\Delta/\Sigma w^{\frac{1}{2}}F_o = 0.064$. Complex neutral-atom scattering factors were employed [14] throughout the refinement. Table 4 lists the final atomic co-ordinates for the non-hydrogen atoms.

Initial data reduction was performed on the dedicated Nova 3 computer at Cambridge using programs written by Dr. W. Clegg, and crystal structure determination and refinement carried out on the University of Cambridge IBM 370/165 computer using programs written by Professor G. M. Sheldrick.

Acknowledgements

We thank the S.E.R.C. for financial support.

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Received 1*st December* 1982; *Paper* 2/2012