Journal of Organometallic Chemistry, 208 (1981) 253—260
Elsevier Sequoia S.A., Lausanne — Printed in The Netherlands

THE CRYSTAL STRUCTURE OF η^2-*C,C'*-[THIOCARBOXY-*C,C'*-SULPHIDO-*N*-METHYL-*N*-TOLYLAMINOCARBENE]HYDRIDOCARBONYLBIS-(TRIPHENYLPHOSPHINE)OSMIUM(II), $\overline{\mathrm{Os[CS_2C}}\mathrm{N}(p\text{-tolyl})\mathrm{Me]H(CO)(PPh_3)_2}$

SUZANNE M. BONIFACE and GEORGE R. CLARK *
Department of Chemistry, University of Auckland, Auckland (New Zealand)

(Received September 15th, 1980)

Summary

Crystal and molecular structures of the title compound have been determined from a three-dimensional X-ray analysis using diffractometer data. The crystals are orthorhombic, space group *Pbca*, with $Z = 8$ in a unit cell of dimensions a = 18.298(1), b = 18.474(7), c = 24.583(2) Å. The observed and calculated densities are 1.51 and 1.522 g cm^{-3}, respectively. The crystals decompose slowly under X-rays, and two crystals were required to complete data collection. The structure was solved by conventional heavy-atom methods, and refined by least-squares techniques to final residuals R and R_w of 0.070 and 0.071, respectively.

The crystals contain neutral monomers of the complex with no solvent molecules of crystallisation. The coordination about the osmium atom is that of a distorted octahedron, and consists of *trans* triphenylphosphine ligands, with the hydride, carbonyl, and bidentate carbene ligands occupying the equatorial sites. The hydride ligand is situated *trans* to the thiocarbonyl carbon atom of the carbene. The Os—P distances of 2.326(9) and 2.344(10) Å are short compared to those found in other osmium bis(triphenylphosphine) complexes.

Introduction

Reaction of [OsCl(CS)(CO)(CNR)(PPh$_3$)$_2$]$^+$, R = *p*-tolyl, with SH$^-$ yields Os(η^2-SCNR)(CS)(CO)(PPh$_3$)$_2$, which can be methylated with MeI to form the dihapto-thiocarboxamido cation [Os(η^2-SCNMeR)(CS)(CO)(PPh$_3$)$_2$]$^+$. Upon reaction with NaBH$_4$ the thiocarboxamido ligand presumably becomes monodentate, but the complex undergoes a further rearrangement to form a four-membered metallocycle by coalescence of the monohapto-thiocarboxamido and thiocarbonyl ligands. The above reaction sequence has recently been

0022—328X/81/0000—0000/$02.50, © 1981, Elsevier Sequoia S.A.

described, together with a preliminary account of the structure of the metallocycle complex product [1]. We now describe details of the refined crystal structure.

Experimental

The crystals were prepared by Dr. T.J. Collins and Dr. W.R. Roper of the University of Auckland. Preliminary data from oscillation and Weissenberg photography showed that they belonged to the orthorhombic crystal class, with systematic absences ($0kl$, $k = 2n + 1$; $h0l$, $l = 2n + 1$; $hk0$, $h = 2n + 1$) characteristic of space group *Pbca*. Accurate unit cell parameters were obtained from a least-squares refinement of the setting angles of twelve high-theta reflections using a Hilger and Watts four-circle automatic diffractometer [2]. Details of unit cell parameters and intensity data collection procedures are summarised in Table 1. It was found that the count rates of three standard reflections declined steadily with time of irradiation by X-rays, and it was necessary to use two separate crystals to complete data collection. The first crystal was replaced when the count rates of the standard reflections had declined by approximately 40% of their initial values. At the end of data collection the count rates for the second crystal had declined by approximately 32%. After scaling and averaging, the data were corrected for Lorentz and polarization factors, and for absorption [3]. The observed data criterion was $I > 3\sigma(I)$ $\{\sigma(I) = [T + t^2B + (pI)^2]^{1/2}$, T = integrated peak count, B = average background count, t = ratio of scan to background times, p was assigned as 0.04 [4]$\}$.

TABLE 1

SUMMARY OF CRYSTAL DATA AND INTENSITY DATA COLLECTION FOR $\overline{\text{Os}[\text{CS}_2\text{C}}\text{N}(p\text{-tolyl})\text{Me}]\text{H(CO)(PPh}_3)_2$

Compound: η^2-*C*,*C*′-[thiocarboxy-*C*,*C*′-sulphido-*N*-methyl-*N*-tolylaminocarbene]-hydridocarbonylbis(triphenylphosphine)osmium(II)	
Formula: $C_{47}H_{41}NOP_2S_2Os$	Molecular weight: 952.07
Crystal habit: Needles with diamond-shaped cross-section	
$a = 18.298(1)$ Å	$b = 18.474(7)$ Å
$c = 24.583(2)$ Å	$V = 8309.97$ Å^3
$Z = 8$	Temperature = 291 K
$p_c = 1.522$ g cm^{-3}	$p_o = 1.51$ g cm^{-3} (by flotation in aqueous KCl/KI)
Space group: *Pbca* D_{2h}^{15} (No. 61)	
Dimensions:	Mosaic spread:
Crystal 1 0.10 × 0.20 × 0.19 mm	0.13°
Crystal 2 0.12 × 0.20 × 0.24 mm	0.12°
Crystal faces developed: $\{011\}$, $\{100\}$	
X-radiation: Cu $K_{\bar{\alpha}}$, $\lambda = 1.5418$	$\mu = 77.66$ cm^{-1}
Collimators: Primary 0.7 mm	Secondary 5.0 mm
Take-off angle: 4°	Theta(max) 56.5°
Scan speed: 0.01° in θ per second using $2\theta/\omega$ scan	
Scan range: Symmetric scan of 0.80° in θ	
Background: 15 s stationary count at each end of scan range	
Standard reflections:	
3 standards remeasured after every 200 reflections	
Maximum decline:	Crystal 1 40.8%
	Crystal 2 32.3%
Observed data criterion: 1317 unique reflections with $I > 3\sigma(I)$	

Structure determination and refinement

The position of the osmium atom was found from the solution of a three-dimensional Patterson map, and a subsequent electron density synthesis revealed the positions of all other non-hydrogen atoms *. Atomic scattering factors and dispersion corrections were from standard listings [6]. Refinement employed the full least-squares matrix and minimised the function $\Sigma w(|F_0| - |F_c|)^2$, with weights $w = 4|F_0|^2/(\sigma^2(F_0)^2)$. Residuals quotes are $R = \Sigma(|F_0| - |F_c|)/\Sigma|F_0|$ and $R_w = [\Sigma w(|F_0| - |F_c|)^2/\Sigma w|F_0|^2]^{1/2}$. Initial refinement cycles used isotropic temperature factors for all atoms, but thereafter, the osmium, sulphur and phosphorus atoms were assigned anisotropic thermal parameters. Several attempts were made to assign anisotropic thermal parameters to all atoms except the carbon atoms of the triphenylphosphine groups, but these were not successful since several atoms returned non-positive definite temperature factors. Relocation of the affected atoms in difference maps gave little improvement in subsequent refinement. A critical examination of the intensity data revealed that the only obvious systematic irregularity was that the very low theta reflections ($\sin^2\theta/\lambda^2 < 0.02$) suffered from rather uneven background counts, and these were removed from the data set. Analysis of the weighting scheme showed that $\langle w(|F_0| - |F_c|)^2\rangle$ was very satisfactorily constant throughout the full range of the data set, which indicated that the form of the weighting expression, and the particular value assigned to the p factor, were well chosen. It was concluded that the least-squares refinement was being affected by the general paucity of reliable data (probably resulting from the effects of crystal decomposition during data collection) and that it would not be possible to obtain the high accuracy in atomic coordinates that one would normally be seeking. Two final cycles were computed before refinement was terminated with R and R_w equal to 0.070 and 0.071, respectively.

There was no evidence in any difference maps for any solvent molecules or misplaced atoms, nor was it possible to locate the hydrogen atom presumed to be bonded to osmium.

Final atomic positions are listed in Table 2. The atomic numbering scheme is outlined in Fig. 1. Tables of thermal parameters, bond distances and angles involving phenyl rings, and observed and calculated structure factors are available on request from the authors (G.R.C.).

Description of the crystal structure

The compound is neutral and monomeric. The overall geometry is shown in Fig. 1. The position of the hydrogen atom has not been located from an electron density map, but has been placed in a calculated position for the purpose of clarity in the diagram. The molecular packing can be seen in the stereoscopic diagrams of Fig. 2. The more important bond distances and angles are listed in Table 3.

Coordination about the osmium atom is that of a distorted octahedron. The

---

* All computing was carried out on the University of Auckland Burroughs B 6700 computer [5].

TABLE 2

ATOMIC POSITIONS FOR $\overline{\mathrm{Os}[\mathrm{CS}_2\mathrm{C}}\mathrm{N}(p\text{-tolyl})\mathrm{Me}]\mathrm{H}(\mathrm{CO})(\mathrm{PPh}_3)_2$

Atom	x/a	y/b	z/c
Os	0.1828(1)	0.2232(1)	0.0908(1)
S(1)	0.0311(5)	0.2599(5)	0.1247(4)
S(2)	0.0455(6)	0.1063(6)	0.1447(5)
P(1)	0.2252(5)	0.2451(5)	0.1792(4)
P(2)	0.1570(5)	0.2046(4)	−0.0008(4)
C(1)	0.2420(22)	0.1403(20)	0.0850(18)
C(2)	0.0757(25)	0.1713(22)	0.0973(20)
C(3)	0.1040(20)	0.3038(19)	0.1206(17)
C(4)	0.1590(26)	0.4281(19)	0.0663(15)
O	0.2846(15)	0.0985(15)	0.0819(13)
N	0.0990(16)	0.3737(16)	0.0924(17)
C(t1)	0.0312(23)	0.4247(20)	0.1070(20)
C(t2)	0.0400(27)	0.4711(23)	0.1422(18)
C(t3)	−0.0244(25)	0.5127(23)	0.1538(17)
C(t4)	−0.0884(25)	0.5007(23)	0.1291(18)
C(t5)	−0.0948(21)	0.4515(20)	0.0841(21)
C(t6)	−0.0356(23)	0.4048(20)	0.0749(18)
C(t7)	−0.1584(22)	0.5453(22)	0.1415(16)
Phenyl Groups			
Ph1			
C(11)	0.2745(22)	0.1713(21)	0.2065(15)
C(12)	0.2424(23)	0.1007(21)	0.2096(15)
C(13)	0.2827(22)	0.0434(21)	0.2332(17)
C(14)	0.3566(25)	0.0589(22)	0.2555(18)
C(15)	0.3877(22)	0.1228(20)	0.2492(16)
C(16)	0.3510(18)	0.1772(16)	0.2280(14)
Ph2			
C(21)	0.2890(19)	0.3245(19)	0.1970(16)
C(22)	0.3047(22)	0.3378(16)	0.2461(15)
C(23)	0.3592(27)	0.3981(23)	0.2558(20)
C(24)	0.3850(20)	0.4355(18)	0.2144(17)
C(25)	0.3657(24)	0.4298(23)	0.1606(20)
C(26)	0.3116(28)	0.3578(22)	0.1518(16)
Ph3			
C(31)	0.1499(18)	0.2594(17)	0.2323(15)
C(32)	0.1314(22)	0.3356(20)	0.2342(17)
C(33)	0.0637(23)	0.3511(19)	0.2692(15)
C(34)	0.0323(21)	0.2975(18)	0.2981(15)
C(35)	0.0580(18)	0.2323(21)	0.2970(13)
C(36)	0.1180(18)	0.2103(16)	0.2583(13)
Ph4			
C(41)	0.2215(17)	0.2392(16)	−0.0501(13)
C(42)	0.2971(20)	0.2309(23)	−0.0341(15)
C(43)	0.3497(21)	0.2586(19)	−0.0660(18)
C(44)	0.3375(21)	0.2863(22)	−0.1203(16)
C(45)	0.2584(21)	0.2867(21)	−0.1390(14)
C(46)	0.2063(18)	0.2624(19)	−0.0992(17)
Ph5			
C(51)	0.0683(27)	0.2452(29)	−0.0288(19)
C(52)	0.0675(32)	0.3147(33)	−0.0374(21)
C(53)	0.0017(28)	0.3524(25)	−0.0621(19)
C(54)	−0.0531(31)	0.3060(31)	−0.0682(20)
C(55)	−0.0553(25)	0.2317(33)	−0.0638(17)
C(56)	0.0098(38)	0.1909(32)	−0.0380(27)
Ph6			
C(61)	0.1449(20)	0.1068(20)	−0.0205(18)
C(62)	0.1536(19)	0.0918(20)	−0.0750(16)
C(63)	0.1454(28)	0.0227(30)	−0.0975(26)
C(64)	0.1172(30)	−0.0250(29)	−0.0545(29)
C(65)	0.1101(34)	−0.0145(36)	0.0030(31)
C(66)	0.1351(28)	0.0571(30)	0.0198(23)

TABLE 3

BOND DISTANCES (Å) AND ANGLES (°) FOR $\overline{\mathrm{Os[CS_2C}}\mathrm{N(}p\text{-}\mathrm{tolyl)Me]H(CO)(PPh_3)_2}$

Os—P(1)	2.344(10)	S(1)—C(2)	1.83(4)
Os—P(2)	2.326(9)	S(1)—C(3)	1.70(4)
Os—C(1)	1.88(3)	S(2)—C(2)	1.45(5)
Os—C(2)	2.30(4)	C(1)—O	1.10(4)
Os—C(3)	2.08(4)	C(3)—N	1.30(5)
N—C(4)	1.62(5)	N—C(t1)	1.60(6)
C(t1)—C(t2)	1.23(6)	C(t2)—C(t3)	1.44(6)
C(t3)—C(t4)	1.34(6)	C(t4)—C(t5)	1.44(6)
C(t5)—C(t6)	1.40(6)	C(t6)—C(t1)	1.50(6)
C(t4)—C(t7)	1.55(6)		
P(1)—C(11)	1.77(4)	P(2)—C(41)	1.81(3)
P(1)—C(21)	1.93(4)	P(2)—C(51)	1.92(5)
P(1)—C(31)	1.92(4)	P(2)—C(61)	1.88(4)
P(1)—Os—P(2)	172.1(6)	Os—P(2)—C(61)	114.5(12)
P(1)—Os—C(1)	91.1(10)	C(2)—S(1)—C(3)	93(2)
P(1)—Os—C(2)	93.3(12)	C(3)—N—C(4)	127(2)
P(1)—Os—C(3)	92.0(12)	C(3)—N—C(t1)	128(2)
P(2)—Os—C(1)	85.7(10)	C(4)—N—C(t1)	104(2)
P(2)—Os—C(2)	94.3(12)	Os—C(3)—N	138.9(15)
P(2)—Os—C(3)	92.3(12)	S(2)—C(2)—S(1)	123.2(8)
C(1)—Os—C(2)	100.1(15)	S(1)—C(3)—N	117(2)
C(1)—Os—C(3)	171.2(15)	N—C(t1)—C(t2)	118(3)
C(2)—Os—C(3)	71.6(16)	N—C(t1)—C(t6)	112(2)
Os—C(2)—S(2)	143.2(5)	C(t6)—C(t1)—C(t2)	130(3)
Os—C(3)—S(1)	103.5(4)	C(t1)—C(t2)—C(t3)	114(3)
Os—C(1)—O	170.0(15)	C(t2)—C(t3)—C(t4)	123(3)
Os—C(2)—S(1)	91.4(3)	C(t3)—C(t4)—C(t5)	122(3)
Os—P(1)—C(11)	112.8(12)	C(t3)—C(t4)—C(t7)	123(2)
Os—P(1)—C(21)	112.8(11)	C(t5)—C(t4)—C(t7)	115(2)
Os—P(1)—C(31)	114.7(10)	C(t4)—C(t5)—C(t6)	117(2)
Os—P(2)—C(41)	117.7(10)	C(t5)—C(t6)—C(t1)	113(2)
Os—P(2)—C(51)	117.4(15)		

triphenylphosphine ligands occupy the axial sites, while the hydride, carbonyl, and bidentate carbene groups lie in the equatorial plane.

The ligand of interest in this compound results from an inter-ligand reaction in the parent compound which leads to the formation of a four-membered metallocycle. It is unfortunate that the estimated standard deviations in the final atom parameters allow little significance to be attached to specific bond lengths and angles in such a novel ligand. However the basic geometry can be described and several observations made.

The ligand is bound to the osmium atom through two carbon atoms, each of which is bound to a sulphur atom to complete the metallocycle. The Os—C distances, 2.08 and 2.30(4), are both longer than the sum of the covalent radii of the atoms [7] and the significant differences in the bond distances reflects in part the differing *trans* directing capacities of the hydrido and carbonyl ligands. Although the Os—C(2) distance of 2.30 Å is apparently longer than any Os—C distance previously reported, it does not differ significantly from the distance of 2.137(5) Å found in the Os-monohaptoester complex $\mathrm{OsH(CO)_2(CS_2Me)(PPh_3)_2}$ [8]. The C(3)—S(1) distance, 1.70(4), is characteristic of C—S distances in carbenoid complexes where the C—S bond order is ca. 1.5 [9]. The

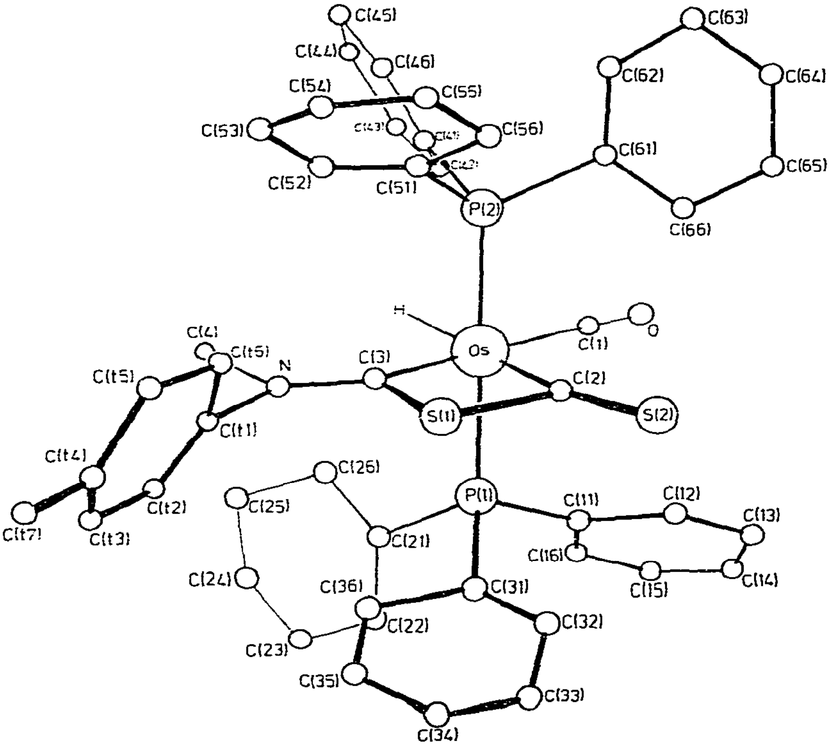

Fig. 1. The molecular geometry and atomic numbering for $\overline{\mathrm{Os[CS_2C}}\mathrm{N}(p\text{-tolyl})\mathrm{Me]H(CO)(PPh_3)_2}$.

C(2)—S(1) distance, 1.83(4), although characteristic of C—S single bonds [10], is not significantly different from C(3)—S(1) and no conclusions can be drawn as to the exact nature of the bonding in the metallocycle. For the C(3)—S(2) bond, the measured distance of 1.45(5) is within 3σ of the value for C=S double bonds (1.55—1.56) [11]. The angles within the metallocycle are subject to the constraints of a four-membered ring and consequently the Os—C—S angles (91.4(3) and 103.5(4) are considerably smaller than the ideal angle for sp^2 hybrid orbitals and the C—S—C angle (93(2)) is smaller than the ideal tetrahedral angle. The *p*-tolyl group is planar, but lies 70.6° out of the equatorial plane of the molecule as a result of rotation about the N—C(t1) bond. In this position the *p*-tolyl ring is sandwiched between two phenyl rings from adjacent phosphines, viz. Ph2 (C(21)—C(26)) from one molecule and Ph5 (C(51)—C(56)) from a second molecule. The closest approach made by the *p*-tolyl atoms to phenyl carbon atoms is 3.4 Å. If, however, the tolyl group were assumed to lie in the equatorial plane, the atoms C(t1)—C(t7) approach more closely to both these phenyl groups. When atomic sites are estimated for such a model the following intermolecular contacts are noted: C(t3)....C(23) 2.9, C(t3).... C(24) 3.4, C(t5)....C(52) 3.1, C(t5)....C(53) 2.4, C(t6)....C(53) 2.7 Å. The observed twist of the group out of the equatorial plane undoubtedly occurs to minimise these steric interactions.

The Os—CO bond distance, 1.88(3), is normal, and agrees with those found

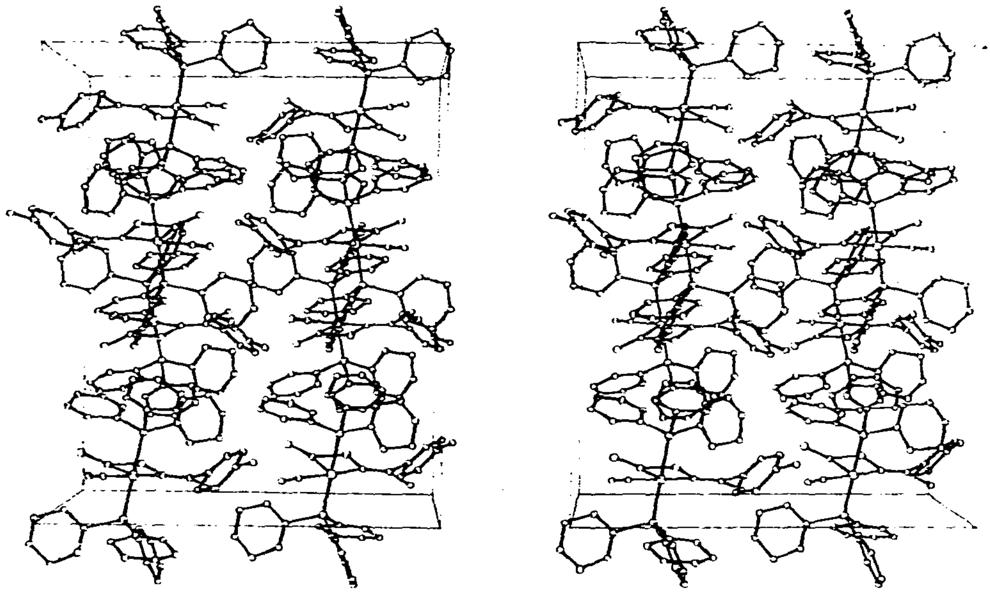

Fig. 2. A stereoscopic view of the molecular packing of $\overline{\mathrm{Os[CS_2C}}\mathrm{N(}p\mathrm{\text{-}tolyl)Me]H(CO)(PPh_3)_2}$.

in other osmium carbonyl complexes, e.g. $OsH(CO)_2(CS_2Me)(PPh_3)_2$ (1.941, 1.914(5)) [8], $Os(CO)_3(PPh_3)_2$ (1.90, 1.93(5)) [12] and $[Os(\eta^2\text{-}S_2Me)(CO)_2(PPh_3)_2]ClO_4$ (1.90, 1.88(2)) [13].

These values are consistent with the π-backbonding properties of the carbonyl ligand. The C—O distance, 1.10(4), agrees with those previously reported but the Os—C—O bond angle, 170(2), shows some deviation from linearity. This is possibly the result of the packing of the molecules into the unit cell, since the carbonyl ligand lies between a phenyl ring of an adjacent molecule and the *N*-methyl group of a second neighbouring molecule.

The Os—P distances of 2.326(9) and 2.344(10) Å are at the lower end of the range typically found in osmium bis(triphenylphosphine) complexes, e.g. $Os(CO)_3(PPh_3)_2$ (2.32, 2.35(3) Å) [12]; $OsH(CO)(N_2C_6H_5)(PPh_3)_2$ (2.333, 2.341 Å) [14]; $OsH(CO)_2(CS_2Me)(PPh_3)_2$ (2.371, 2.381(2) Å) [8]; $OsCl_2(HgCl)(NO)(PPh_3)_2$ (2.39, 2.40(2) Å) [15]; $[Os(CO)_2(NO)(PPh_3)_2]ClO_4$ (2.401, 2.405(3) Å) [16]; $[Os(CO)(CN\text{-}p\text{-}tolyl)(NO)(PPh_3)_2]ClO_4$ (2.400, 2.406(7) Å) [17]; $OsCl_3(NH_3)(PPh_3)_2$ (2.411(2) Å) [18]; $[Os(\eta^2\text{-}S_2Me)(CO)_2(PPh_3)_2]ClO_4$ (2.419, 2.420(3) Å) [13]; $OsCl_2(CO)(CS)(PPh_3)_2$ (2.431, 2.432(3) Å) [19]; $[Os(OH)(NO)_2(PPh_3)_2]PF_6$ (2.432(4), 2.436(3) Å) [20]. Clearly the Os—P distances in such complexes are subject to considerable variation. In view of the very short Os—P bond lengths in the present complex, it would appear that the phosphines (and carbonyl) are acting as receptors for any π-electron density available for back-donation from the metal, in preference to the bidentate carbene ligand. However, it must be remembered that the relative influences of electronic and steric factors in determining a specific coordination geometry are not well understood, and in this context, it is unfortunate that the quality of the data set does not allow a more conclusive study of such an unusual compound.

References

1 G.R. Clark, T.J. Collins, D. Hall, S.M. James and W.R. Roper, J. Organometal. Chem., 141 (1977) C5.
2 W.R. Busing and H.A. Levy, Acta Cryst., 22 (1967) 457.
3 The absorption programme used was DABS, a modification of DATAPH, described by P. Coppens in paper G1, International Summer School of Crystallographic Computing, Ottawa, 1969.
4 P.W. Corfield, R.J. Doedens and J.A. Ibers, Inorg. Chem., 6 (1967) 197.
5 Major programs used were HILGOUT, FOURIER and CUCLS, written by R.J. Delaca of the University of Canterbury, Christchurch, New Zealand.
6 International Tables for X-ray Crystallography, Vol. III, Kynoch Press, Birmingham, 1962, pp. 202—214.
7 L. Pauling, The Nature of the Chemical Bond, 3rd edition, Cornell University Press, Ithaca, New York, 1960, p. 249.
8 J.M. Waters and J.A. Ibers, Inorg. Chem., 16 (1977) 3273.
9 W.M. Butler and J.H. Enemark, Inorg. Chem., 12 (1973) 540; L. Ricard, J. Estienne and R. Weiss, Inorg. Chem., 12 (1973) 2182; R.J. Hoare and O.S. Mills, J. Chem. Soc. Dalton Trans., (1972) 653.
10 R.O. Harris, L.S. Sadavy, S.C. Nyburg and F.H. Pickard, J. Chem. Soc., Dalton Trans., (1973) 2646; J.M. Coleman, A. Wojcicki, P.J. Pollick and L.F. Dahl, Inorg. Chem., 6 (1967) 1236; W. Maier, Angew. Chem., 73 (1961) 120; R. Thomas, C.B. Shoemaker and K. Eriks, Acta Cryst., 21 (1966) 12.
11 M.J. Calloman and H.W. Thompson, Proc. Royal Soc. London Ser. A, 222 (1959) 431; W.A. Hardy and G. Silvey, Phys. Rev. 95 (1954) 385; G.C. Dousmanis, T.M. Sanders, C.H. Townes and H.J. Zeiger, J. Chem. Phys., 21 (1953) 1416.
12 J.K. Stalick and J.A. Ibers, Inorg. Chem., 8 (1969) 419.
13 G.R. Clark and D.R. Russell, J. Organometal. Chem., 173 (1979) 377.
14 M. Cowie, B.L. Haymore and J.A. Ibers, Inorg. Chem., 14 (1975) 2617.
15 G.A. Bentley, K.R. Laing, W.R. Roper and J.M. Waters, Chem. Commun., (1970) 998.
16 G.R. Clark, J.M. Waters and K.R. Whittle, J. Chem. Soc., Dalton Trans., (1975) 2233.
17 G.R. Clark, J.M. Waters and K.R. Whittle, J. Chem. Soc., Dalton Trans., (1976) 2029.
18 D. Bright and J.A. Ibers, Inorg. Chem., 8 (1969) 1078.
19 G.R. Clark and K. Marsden, to be published.
20 G.R. Clark, J.M. Waters and K.R. Whittle, J. Chem. Soc., Dalton Trans., (1975) 463.