comparison of the dihedral angles between the Co, N(3), N(13), N(18) and Co, N(3), N(8), N(13) planes and the ring planes [176·8 (3), 92·9 (3)° and 110·1 (3), 164·6 (3)° for the N(11)–C(15) and N(1)–C(5) rings, respectively].

The structure contains a three-dimensional network of hydrogen bonds (Fig. 2) involving N^{τ}—H, amino, water and C(12)—H groups as donors, and water O atoms, carbonate and Cl^- anions as acceptors. The N(1)—H group forms a hydrogen bond, the N(1)···O(3) ($-x$, $-y$, $-1-z$) distance being 2·755 (4) Å. The N(8) amino group is a donor in two hydrogen bonds. With N(8)···Cl($1+x$, y, $1+z$) and N(8)···O*W*($1+x$, y, z) distances of 3·318 (4) and 2·922 (5) Å, respectively. The N(11)—H group of the second histamine ligand forms a hydrogen bond to the Cl^- ion [N(11)···Cl($1-x$, $1-y$, $-z$) = 3·175 (4) Å]. The C(12)—H group acts as a donor in a bifurcated hydrogen bond, the C(12)···O(1) and C(12)···Cl($1+x$, y, z) distances being 2·838 (5) and 3·448 (5) Å, respectively. The N(18) amino group is a donor in two hydrogen bonds, the N(18)···Cl and N(18)···O(3) (x, y, $1+z$) distances being 3·484 (5) and 2·930 (5) Å, respectively. Both water O—H groups form hydrogen bonds [O*W*···Cl = 3·240 (4) and O*W*···O(2) (x, y, $1+z$) = 2·772 (4) Å]. The Cl^- ion participates in five hydrogen bonds, and the carbonate ion is an acceptor in four such bonds, whereas the water O atom is a single-hydrogen-bond acceptor.

This research was partly supported by project RP.II.10.

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Structure of *trans*-Bromodicarbonyl[*o*-phenylenebis(diphenylphosphine)]-(*p*-tolylmethylidyne)tungsten(0)

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(*Received* 23 *January* 1989; *accepted* 6 *July* 1989)

Abstract. $[WBr(CO)_2(C_8H_7)(C_{30}H_{24}P_2)]$, $M_r = 869{\cdot}39$, monoclinic, $P2_1/n$, $a = 9{\cdot}505$ (5), $b = 18{\cdot}333$ (4), $c = 20{\cdot}775$ (8) Å, $\beta = 96{\cdot}68$ (4)°, $V = 3595$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}606$ Mg m^{-3}, Mo $K\alpha$ radiation (graphite monochromator, $\lambda = 0{\cdot}71073$ Å), $\mu = 4{\cdot}50$ mm^{-1}, $F(000) = 1704$, $T = 291$ K, final $R = 0{\cdot}025$ for 4337 observed reflections and 509 variables. The W atom is in a distorted octahedral environment formed by the two P atoms of the chelating diphosphine, two mutually *cis* carbonyl groups and a bromide ligand *trans* to a methylidyne group. The W≡C—*R* ($R = C_6H_4CH_3$-4) group deviates slightly from linearity and the short W—C distance suggests a formal metal—carbon triple bond.

Introduction. Very few X-ray structure determinations have been carried out on neutral six-coordinated methylidyne complexes of tungsten(0), and the majority of them are *trans*-tetracarbonyls, having also halogens (Huttner, Lorenz & Gartzke, 1974; Neugebauer, Fischer, Dao & Schubert, 1978; Fischer, Gammel & Neugebauer, 1980) or the

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$Co(CO)_4$ group (Fischer, Friedrich, Lindner, Neugebauer, Kreissl, Uedelhoven, Dao & Huttner, 1983). Here we report the first crystal structure of a stable methylidyne complex of the type *trans*-*X*(CO)$_2$(*L*–*L*)W(≡C*R*), where *X* = Br, *L*–*L* = *o*-phenylenebis(diphenylphosphine) and *R* = C_6H_4Me-4, prepared by Carriedo, Riera & Sánchez (1988) following a method similar to that published recently for other dicarbonyl methylidyne complexes (McDermott, Dorries & Mayr, 1987).

Experimental. A crystal was selected and mounted on an Enraf–Nonius CAD-4 four-circle diffractometer. The unit-cell parameters were determined from 23 reflections ($8 \le \theta \le 12°$) and refined by least-squares method. Intensities were collected, using the ω–2θ scan technique, up to $\theta = 30°$ in the *hkl* range from −13,0,0 to 13,25,19. Three reflections were measured each hour as orientation and intensity control; no significant intensity decay was observed. 9194 reflections were measured, corresponding to 8631 unique reflections ($R_{int} = 0{\cdot}024$), 4337 of which were assumed as observed applying the condition $I \ge 3\sigma(I)$. Lorentz and polarization corrections were applied.

The structure was solved by locating the W and Br atoms with *MULTAN*80 (Main *et al.*, 1980); the other atoms were found by Fourier synthesis with *SHELX*76 (Sheldrick, 1976). The structure was refined by block-matrix least squares, using *SHELX*76 (Sheldrick, 1976). The function minimized was $\sum[w(|F_o| - |F_c|)^2]$, where $w = [\sigma^2(F_o) + 0{\cdot}0002|F_o|^2]^{-1}$. After the isotropic refinement, an empirical absorption correction was applied (Walker & Stuart, 1983), correction factors were in the range 0·905 to 1·090. Anisotropic thermal parameters were comparable after refinement with and without absorption correction. Except for those of the methyl group [C(10)], which were located by difference Fourier synthesis, the H atoms were inserted geometrically. All the H atoms were refined freely with an overall isotropic temperature factor (final U = 0·107 Å^2). The remaining atoms were refined with individual anisotropic temperature factors. The final R was 0·025 and wR was 0·024. Max. shift/e.s.d. was 2·43 for the overall temperature factor of H atoms. For non-H atoms the max. shift/e.s.d. was 1·47 for U_{11} of C(33). Owing to restrictions in calculation time, in each run of the *SHELX* program each block was refined only once. The truncation of the results in each run of the program resulted in numerical noise. Finally, successive executions of the program led to no further progress in the refinement. The final difference Fourier map showed a residual electron density between −0·42 and 0·45 e Å^{-3}. The values of f, f' and f'' were taken from *International Tables for X-ray Crystallography* (1974). The geometry calculations were performed with *PARST* (Nardelli, 1983), and Fig. 1 was drawn with *PLUTO* (Motherwell, 1976).

Discussion. Final atomic parameters are listed in Table 1 and selected bond lengths and angles in Table 2.* The structure consists of discrete molecules (Fig. 1) linked by van der Waals forces. The shortest intermolecular separation is 2·480 (0) Å [H(24)···H(34^i); (i) = $x - \frac{1}{2}$, $-y + \frac{1}{2}$, $z - \frac{1}{2}$]. The W atom is in a slightly distorted octahedral environment, with two P atoms of the chelating ligand and two carbonyl ligands in the equatorial positions. The Br atom and the methylidyne group are in the apical positions.

The Br(1)—W(1) bond distance [2·675 (1) Å] is very similar to that observed in [Br(CO)$_4$W(≡CMe)] [2·648 (6) Å] (Neugebauer, Fischer, Dao & Schubert, 1978). The W(1)—C(3) distance [1·798 (5) Å)] may be compared with those found in the related complexes [(η-C$_5$H$_5$)(CO)$_2$W≡C(C$_6$H$_4$Me-4)] [1·82 (2) Å] (Fischer, Lindner, Huttner, Friedrich, Kreissl & Besenhard, 1977) and [I(CO)$_4$W≡CPh)] [1·90 (5) Å] (Huttner, Lorenz & Gartzke, 1974). In all of these complexes the W—C separation is considerably shorter than that observed in [W(=CPh$_2$)(CO)$_5$] [2·14 (2) Å] (Casey, Burkhardt, Bunnell & Calabrese, 1977) and this has been taken to imply a W=C bond because it is significantly less than the corresponding distance [2·34 (1) Å] in the anion {W[CH(OMe)Ph](CO)$_5$}$^-$ (Casey, Polichnowski, Tuinstra, Albin & Calabrese, 1978). The angles Br(1)—W(1)—C(3) [173·4 (2)°] and W(1)—C(3)—C(4) [171·5 (4)°] deviate slightly from linearity.

The W(1)—CO bond lengths [2·012 (5) Å] are shorter than those observed in [Br(CO)$_4$W(≡CMe)] [average value 2·12 (5) Å] (Neugebauer, Fischer, Dao & Schubert, 1978). This may be attributed to the presence of a P atom in a *trans* position to each CO group. The W(1)—C—O groups are almost linear [average value 176·8 (5)°]. Both W(1)—P distances [average value 2·513 (1) Å] are close to those found in the related complex [W(CO)$_3$(dppe)(C=CHCO$_2$-Me)] [average value 2·550 (3) Å] (Birdwhistell, Nieter Burgmayer & Templeton, 1983). The Br(1)—W(1)—P angles are smaller than the Br(1)—W(1)—CO angles, but all of them are about 90°. Similarly, the C(3)—W(1)—CO angles [average value 87·0 (2)°] are smaller than those corresponding to the C(3)—W(1)—P angles [average value 98·4 (1)°]. The angle between the two carbonyl groups is near 90°

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52399 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional positional and thermal parameters* ($Å^2 \times 10^2$) *with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
W(1)	0·49916 (2)	0·22404 (1)	0·08264 (1)	4·26 (1)
Br(1)	0·49150 (6)	0·30205 (3)	−0·02654 (3)	6·15 (2)
P(1)	0·3103 (1)	0·30583 (7)	0·11781 (6)	4·40 (4)
P(2)	0·2757 (1)	0·16591 (7)	0·02763 (6)	4·68 (4)
C(1)	0·6574 (5)	0·2873 (3)	0·1241 (3)	5·8 (2)
O(1)	0·7441 (4)	0·3217 (3)	0·1506 (2)	9·4 (2)
C(2)	0·6319 (5)	0·1559 (3)	0·0436 (3)	6·4 (2)
O(2)	0·7050 (5)	0·1163 (2)	0·0236 (3)	9·8 (2)
C(3)	0·5256 (4)	0·1702 (3)	0·1554 (2)	5·0 (2)
C(4)	0·5675 (5)	0·1317 (3)	0·2155 (3)	5·7 (2)
C(5)	0·7055 (6)	0·1340 (4)	0·2437 (3)	9·2 (3)
C(6)	0·7422 (8)	0·0985 (5)	0·3024 (4)	13·5 (4)
C(7)	0·6501 (9)	0·0614 (4)	0·3338 (3)	9·9 (3)
C(8)	0·5181 (8)	0·0576 (4)	0·3051 (4)	11·9 (4)
C(9)	0·4739 (7)	0·0925 (4)	0·2467 (3)	10·5 (3)
C(10)	0·698 (1)	0·0242 (5)	0·3978 (4)	18·8 (6)
C(21)	0·1490 (4)	0·2957 (3)	0·0608 (2)	4·7 (2)
C(22)	0·1306 (4)	0·2323 (3)	0·0249 (2)	4·8 (2)
C(23)	0·0026 (5)	0·2200 (3)	−0·0139 (3)	6·4 (2)
C(24)	−0·1046 (5)	0·2715 (4)	−0·0157 (3)	7·5 (2)
C(25)	−0·0834 (5)	0·3353 (4)	0·0175 (3)	7·8 (3)
C(26)	0·0428 (5)	0·3481 (3)	0·0563 (3)	6·4 (2)
C(31)	0·2524 (5)	0·2820 (3)	0·1959 (2)	4·9 (2)
C(32)	0·1122 (6)	0·2753 (4)	0·2056 (3)	8·3 (3)
C(33)	0·0777 (6)	0·2623 (4)	0·2670 (3)	10·0 (3)
C(34)	0·1746 (8)	0·2540 (4)	0·3176 (3)	8·6 (3)
C(35)	0·3132 (7)	0·2599 (4)	0·3095 (3)	9·2 (3)
C(36)	0·3535 (6)	0·2738 (3)	0·2481 (3)	7·0 (2)
C(41)	0·3364 (5)	0·4040 (3)	0·1245 (3)	5·1 (2)
C(42)	0·3554 (6)	0·4433 (3)	0·0700 (3)	7·3 (2)
C(43)	0·3740 (7)	0·5180 (3)	0·0720 (3)	8·2 (3)
C(44)	0·3765 (7)	0·5546 (3)	0·1285 (4)	8·8 (3)
C(45)	0·3592 (9)	0·5166 (4)	0·1832 (4)	11·1 (4)
C(46)	0·3381 (8)	0·4410 (3)	0·1812 (3)	9·1 (3)
C(51)	0·2712 (5)	0·1377 (3)	−0·0569 (3)	5·1 (2)
C(52)	0·2356 (5)	0·1862 (3)	−0·1075 (3)	6·4 (2)
C(53)	0·2382 (6)	0·1649 (4)	−0·1705 (3)	8·2 (3)
C(54)	0·2777 (7)	0·0949 (5)	−0·1843 (4)	9·5 (3)
C(55)	0·3183 (7)	0·0473 (4)	−0·1349 (4)	9·0 (3)
C(56)	0·3134 (6)	0·0677 (3)	−0·0717 (3)	7·0 (2)
C(61)	0·2156 (6)	0·0858 (3)	0·0694 (3)	6·0 (2)
C(62)	0·3131 (7)	0·0330 (3)	0·0893 (3)	8·2 (3)
C(63)	0·2717 (10)	−0·0296 (4)	0·1199 (4)	11·1 (4)
C(64)	0·138 (1)	−0·0395 (5)	0·1312 (4)	12·9 (5)
C(65)	0·0433 (9)	0·0133 (6)	0·1139 (5)	15·1 (5)
C(66)	0·0801 (7)	0·0766 (4)	0·0821 (4)	10·7 (3)

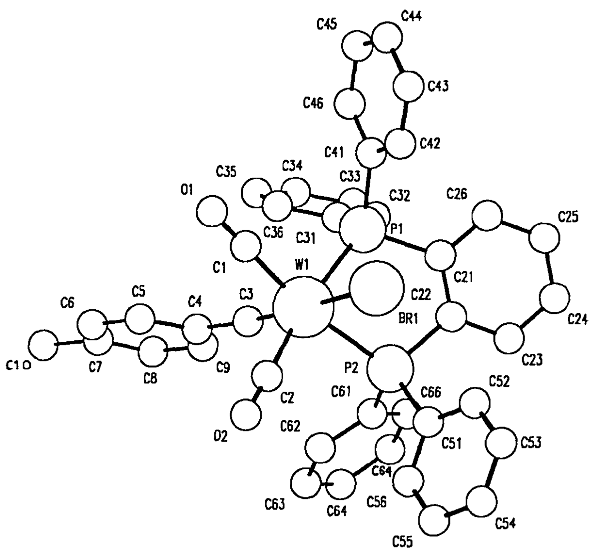

Fig. 1. Geometry and crystallographic numbering scheme for the complex {*trans*-Br(CO)$_2$[*o*-Ph(PPh$_2$)$_2$]W(C≡*R*)}. *R* = C_6H_4Me-4.

Table 2. *Selected bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

W(1)—Br(1)	2·675 (1)	W(1)—C(3)	1·798 (5)
W(1)—P(1)	2·512 (1)	C(1)—O(1)	1·128 (6)
W(1)—P(2)	2·528 (2)	C(3)—C(4)	1·449 (7)
W(1)—C(1)	2·012 (5)	P(1)—C(21)	1·834 (4)
		C(21)—C(22)	1·381 (7)
C(3)—W(1)—C(2)	88·0 (2)	P(1)—W(1)—P(2)	78·0 (1)
C(3)—W(1)—C(1)	86·1 (2)	Br(1)—W(1)—C(3)	173·4 (2)
C(1)—W(1)—C(2)	93·3 (2)	Br(1)—W(1)—C(2)	86·9 (1)
C(3)—W(1)—P(2)	100·2 (2)	Br(1)—W(1)—C(1)	89·9 (1)
C(2)—W(1)—P(2)	95·1 (2)	Br(1)—W(1)—P(2)	84·4 (1)
C(1)—W(1)—P(2)	169·6 (2)	Br(1)—W(1)—P(1)	88·7 (1)
C(3)—W(1)—P(1)	96·8 (2)	W(1)—C(1)—O(1)	176·2 (5)
P(1)—W(1)—C(2)	172·2 (2)	W(1)—C(2)—O(2)	177·5 (5)
P(1)—W(1)—C(1)	93·2 (2)	W(1)—C(3)—C(4)	171·5 (4)

[93·3 (2)°], and considerably greater than that found between the two P atoms of the chelating ligand [78·0 (1)°].

The W(1)—P(2) bond makes an angle of 21·26 (1)° with the plane formed by W(1), P(1) and C(21). The plane containing the C(24), C(25) and C(26) atoms is almost perpendicular to the *p*-tolyl ring [dihedral angle 81·44 (1)°] and forms a dihedral angle of 16·8 (1)° with the plane formed by the W(1), P(21) and C(21) atoms.

We thank the Spanish CAICYT for financial support and the Spanish Ministerio de Educacion y Ciencia for a fellowship (to MGS).

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Acta Cryst. (1990). C**46**, 584–587

Structure of 2-Adamantylammonium Trichloro(ethylamine)platinate(II)

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(*Received* 26 *April* 1989; *accepted* 19 *July* 1989)

Abstract. 2-Adamantylammonium trichloro(ethylamine)platinate(II), [C$_{10}$H$_{18}$N][PtCl$_3$(C$_2$H$_7$N)], M_r = 498·79, monoclinic, $P2_1/n$, with a = 12·401 (10), b = 6·859 (15), c = 20·199 (15) Å, β = 100·72 (6)°, V = 1688 (2) Å^3, Z = 4, D_x = 1·962 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 8·86 mm^{-1}, F(000) = 960, T = 295 K, R = 0·060 for 1801 unique observed reflections. Pt has square-planar coordination with bond distances Pt—Cl = 2·281 (4), 2·278 (4), 2·301 (4) Å and Pt—N = 2·03 (1) Å. The [Pt(C$_2$H$_5$NH$_2$)Cl$_3$]$^-$ anion is normal but the C atoms in the adamantane ring show very high thermal motion. The crystal structure is stabilized by hydrogen bonding between the amine groups and the chloro ligands. The N···Cl distances range from 3·12 (2) to 3·34 (1) Å.

Introduction. The cage molecule 1-adamantanamine (C$_{10}$H$_{15}$NH$_2$) has been the subject of some attention lately, chiefly because of its antiviral (Hay, Wolstenholme, Shehel & Smith, 1985; Widell, Hansson, Oeberg & Nordenfelt, 1986; Fletcher, Hirschfield & Forbes, 1965) and antitumor activity (Ho, Hakala & Zakrsewski, 1972). Moreover, platinum(II) complexes containing the related 1,2-adamantanediamine ligand (Shionogi & Co. Ltd, 1983) apparently possess antitumor activity comparable to that of the commercially licensed drug cisplatin, *cis*-Pt(NH$_3$)$_2$Cl$_2$ (Rosenberg, van Camp, Trosko & Mansour, 1969). A convenient synthetic route to mixed-amine platinum(II) complexes of the type *cis*-Pt(L)(L')Cl$_2$ (L,L' = methylamine, ethylamine, cyclobutylamine, cyclopentylamine *etc.*) has been developed recently and we felt that it would be worthwhile attempting to synthesize similar complexes containing various adamantanamine ligands. It is our ultimate hope that such synthetic combinations might result in compounds with enhanced antitumor activity and reduced toxicity.

Reaction of K[Pt(EtNH$_2$)Cl$_3$] with 2-adamantanamine (2-adam) does apparently yield the mixed-amine complex [Pt(EtNH$_2$)(2-adam)Cl$_2$] but, upon treatment with dilute HCl during the work-up of the product, yellow crystals of the [2-adamH]$^+$.-[Pt(EtNH$_2$)Cl$_3$]$^-$ salt are also produced. This complex is probably the result of displacement of the coordinated adamantanamine ligand by Cl$^-$ and subsequent quarternization of the amine group. We have now characterized the ionic compound by X-ray diffraction and report the results here.

The crystal structure was also of interest to us in view of our ongoing work on order–disorder phase transitions in organic molecular crystals, including substituted adamantane derivatives (Bélanger-Gariépy, Brisse, Harvey, Butler & Gilson, 1987, 1990; Harvey, Gilson & Butler, 1987). The room-temperature phases in such 'plastic crystalline' materials are usually disordered and the phase transitions take place at low temperatures or under high external pressures. The transitions are exothermic and consequently the materials are beginning to be exploited in various passive heat-storage systems. In the case of our complex, we anticipated that the adamantylammonium ion might be disordered at room temperature.

Experimental. 1 mmol of K[Pt(EtNH$_2$)Cl$_3$], synthesized by the method already reported (Rochon, Melanson & Doyon, 1987) and 1·1 mmol of 2-adamantanamine were stirred together in a minimum quantity of water for 90 min. Then 0·1M HCl (10 ml) was added to the solution and the mixture was stirred for a further 10 min. The resulting yellow

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