Crystal and Molecular Structure of a Chelating Ylide Complex of Palladium : [(Benzoylmethylene)diphenyl-2-(diphenylphosphino)-ethylphosphorane]dichloropalladium(II) Toluene Solvate

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The crystal structure of the title compound was solved by single-crystal X-ray diffraction techniques. Crystals are monoclinic, space group $P2_1/c$, with cell dimensions $a=13.39(5)$, $b=16.88(1)$, $c=19.85(6)$ Å, $\beta=123.54(1)°$, $Z=4$. The final R was 0.065. The ylide ligand coordinates to palladium through the ylide carbon Pd–C, 2.115(15) Å and phosphine phosphorus Pd–P, 2.230(8)Å forming a six-membered chelate ring. The chelating ligand together with two chlorine atoms forms a square-planar geometry around the palladium. The Pd–Cl distances are discussed in terms of *trans*-influence of the ylide carbon.

We have reported syntheses of chelating ylide ligands derived from 1,2-bis(diphenylphosphino)ethane and bis-(diphenylphosphino)methane and their palladium(II) and platinum(II) complexes.[1-4] The coordination of the ylide carbon to the metal has been inferred from infrared, ^1H and ^{31}P NMR spectra.[4] The present X-ray structure determination of one of the ylide complexes, [(benzoylmethylene)diphenyl-2-(diphenylphosphino)ethylphosphorane]dichloropalladium(II) (formulated as $[PdCl_2(bdep)]$† in this paper), has confirmed that the ylide carbon and phosphine phosphorus coordinate to palladium. This paper describes the details of the X-ray study, the preliminary results of which have been published.[5]

Experimental

The chelating ylide ligand and its complex, $[PdCl_2(bdep)]$, were prepared as described in an earlier report.[4]

Preparation of the Crystal. $[PdCl_2(bdep)]$ (0.1 g) was dissolved in nitromethane (7 ml), and toluene (15 ml) was added to the solution. The solution was kept at 20 °C for three days. Yellow crystals were filtered, washed with diethyl ether, and dried *in vacuo*. A ^1H NMR spectrum and elemental analyses indicated that the crystals contained a toluene molecule per palladium atom as a crystallization solvent. A single crystal with dimensions *ca.* $0.25\times0.25\times0.10$ mm was used for measurement of diffraction intensities.

Crystal Data. $C_{34}H_{30}Cl_2P_2Pd\cdot C_7H_8$, $M=786.0$, monoclinic, $a=13.39(5)$, $b=16.88(1)$, $c=19.85(6)$ Å, $\beta=123.54(1)°$, $V=3740$ Å^3, $Z=4$, $D_m=1.42$ and $D_x=1.40$ g/cm^3, $\mu=7.47$ cm^{-1} (Mo-$K\alpha$). Space group $P2_1/c$ from systematic absences,, $h0l$, $l=2n+1$ and $0k0$, $k=2n+1$.

Intensity data were recorded on a Rigaku automated four-circle diffractometer, by use of graphite-monochromatized Mo-$K\alpha$ radiation. By the $\omega-2\theta$ scan technique, diffraction intensities were measured up to $2\theta=55°$ and 2652 independent reflections ($|F_o|\geq3\sigma$) were used for the structure analysis. Intensities were corrected for Lorentz and polarization effects but not for absorption and extinction.

Determination of the Structure. The position of the palladium atom was deduced from a three-dimensional Patterson map, and the positions of non-hydrogen atoms including carbon atoms of toluene were determined by the usual heavy-atom method. The structure was refined by block-diagonal least-squares, first with isotropic and then with anisotropic temperature factors. Of the 38 hydrogen atoms, 23 atoms were located from a difference Fourier map, and were refined with isotropic temperature factors. After inclusion of the rest of the hydrogen atoms whose positions were obtained by calculation, the final R value was reduced to 0.065.

Atomic scattering factors were taken from ref. 6. A weighting scheme adopted was $w=1.0$ for $|F_o|\geq36.19$ on an absolute scale and $w=0.5$ otherwise was employed. The calculation was performed on a HITAC 8800/8700 computer at the Computer Center of the University of Tokyo using a local version of UNICS programs.[7]**

Final positional and thermal parameters are given in Table 1, with their estimated standard deviations.

Results and Discussion

Molecular Structure. Final positional and thermal parameters are given in Table 1, with their estimated standard deviations. The molecular structure with the numbering scheme of atoms and a stereoview are

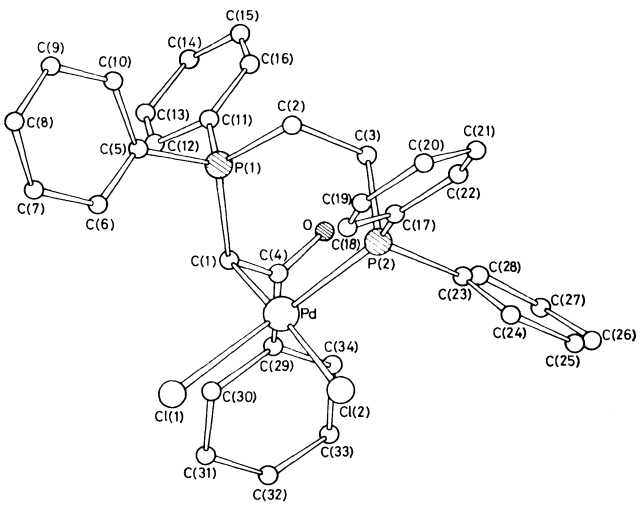


Fig. 1. Molecular structure of $[PdCl_2(bdep)]$ and numbering scheme of non-hydrogen atoms.

† bdep = C_6H_5-C(=O)-C$^-$H-P$^+$-$(CH_2)_2$-P-$(C_6H_5)_2$, with a second $(C_6H_5)_2$ on P.

** Observed and calculated structure factors are held by the office of the Chemical Society of Japan as Document No. 7710.

Table 1a. Final atomic coordinates ($\times 10^4$) and anisotropic thermal parameters ($\times 10^3$) of the non-hydrogen atoms with their estimated standard deviations in parentheses

The U_{ij}'s are defined by

$$\exp\left[-2\pi^2\left(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2hka^*b^*U_{12}+2hla^*c^*U_{13}+2klb^*c^*U_{23}\right)\right].$$

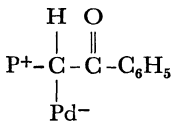
	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd	839(1)	1247(1)	3111(1)	51(1)	32(0)	43(1)	0(1)	33(0)	3(1)
Cl(1)	−922(4)	493(2)	2698(2)	74(3)	40(2)	66(3)	−11(2)	51(2)	−3(2)
Cl(2)	1898(4)	582(2)	4352(2)	71(3)	46(2)	55(2)	16(2)	41(2)	41(2)
P(1)	−183(3)	2784(2)	1875(2)	47(2)	33(2)	43(2)	2(2)	30(2)	5(2)
P(2)	2473(3)	1994(2)	3603(2)	51(2)	36(2)	42(2)	2(2)	30(2)	1(2)
O	1332(8)	1798(5)	1568(6)	56(7)	43(6)	60(6)	3(5)	36(6)	1(5)
C(1)	−99(12)	1723(7)	1929(7)	65(10)	23(7)	35(8)	12(7)	30(8)	4(6)
C(2)	1129(12)	3288(7)	2694(8)	58(10)	29(7)	52(9)	−7(7)	28(8)	−12(7)
C(3)	2305(12)	2865(8)	2995(8)	44(9)	44(8)	41(8)	6(7)	24(7)	17(7)
C(4)	583(12)	1411(7)	1597(8)	60(9)	35(8)	49(8)	−3(7)	35(8)	0(7)
C(5)	−1415(12)	3104(7)	1919(8)	51(9)	28(7)	49(8)	−4(7)	35(8)	−3(6)
C(6)	−2006(14)	2589(9)	2135(10)	63(11)	66(11)	78(12)	0(9)	52(10)	−1(9)
C(7)	−2982(16)	2882(11)	2183(11)	83(13)	82(13)	101(14)	2(11)	61(12)	−9(12)
C(8)	−3256(14)	3670(11)	2045(10)	73(11)	78(12)	89(12)	6(11)	56(10)	−7(11)
C(9)	−2690(15)	4177(9)	1814(9)	83(13)	59(10)	67(11)	24(9)	48(10)	2(9)
C(10)	−1732(13)	3895(8)	1787(8)	67(10)	34(9)	62(9)	−9(7)	43(8)	−3(7)
C(11)	−450(12)	3121(8)	924(8)	49(9)	52(9)	54(9)	19(8)	33(8)	17(8)
C(12)	−1422(18)	2816(13)	243(10)	114(17)	146(20)	47(11)	−38(15)	35(12)	17(12)
C(13)	−1641(23)	3012(15)	−538(12)	170(24)	167(24)	68(14)	−48(20)	52(16)	6(16)
C(14)	−946(16)	3553(11)	−575(11)	98(14)	84(14)	88(13)	29(11)	59(12)	28(11)
C(15)	−15(18)	3879(12)	101(12)	124(16)	106(16)	131(16)	−2(14)	103(14)	30(14)
C(16)	231(18)	3669(11)	877(10)	143(17)	67(12)	90(13)	−3(13)	84(13)	0(12)
C(17)	2952(13)	2467(8)	4581(8)	65(11)	49(9)	32(8)	3(8)	23(8)	−7(7)
C(18)	2230(15)	2452(9)	4874(8)	91(12)	55(9)	46(9)	3(9)	46(9)	1(8)
C(19)	2619(16)	2855(11)	5596(10)	100(14)	98(14)	62(11)	28(12)	56(11)	10(11)
C(20)	3657(17)	3255(10)	5975(9)	125(16)	79(12)	29(9)	11(11)	31(10)	2(9)
C(21)	4423(17)	3271(11)	5711(10)	101(15)	86(13)	51(11)	−14(11)	26(11)	−26(10)
C(22)	4001(14)	2876(10)	4978(9)	71(12)	70(11)	54(10)	−15(10)	25(10)	−20(9)
C(23)	3774(11)	1491(7)	3767(8)	37(8)	36(8)	51(9)	2(6)	23(7)	8(6)
C(24)	4590(13)	1133(10)	4499(8)	64(10)	68(11)	56(9)	3(10)	33(9)	−3(9)
C(25)	5581(14)	730(10)	4644(9)	63(12)	77(12)	68(11)	17(10)	40(10)	10(10)
C(26)	5735(15)	620(11)	4007(10)	79(14)	106(15)	84(13)	36(12)	57(12)	9(11)
C(27)	4868(17)	932(12)	3259(10)	101(14)	138(18)	73(12)	72(13)	65(12)	31(12)
C(28)	3932(15)	1365(11)	3123(9)	92(13)	89(13)	69(11)	44(12)	59(10)	11(11)
C(29)	404(14)	555(8)	1312(7)	79(12)	47(9)	24(7)	2(8)	25(8)	3(7)
C(30)	−618(16)	129(9)	1108(9)	105(14)	49(10)	91(10)	−13(10)	40(10)	−7(8)
C(31)	−807(17)	−641(9)	776(10)	130(17)	55(11)	66(11)	−20(11)	59(12)	−8(9)
C(32)	111(17)	−978(9)	729(10)	122(16)	53(10)	69(11)	11(10)	59(12)	−6(9)
C(33)	1118(18)	−584(10)	947(10)	120(17)	63(12)	74(12)	12(11)	55(12)	2(10)
C(34)	1277(15)	210(9)	1231(9)	73(12)	60(10)	68(11)	12(9)	35(10)	−11(9)
C(35)	5084(22)	4176(18)	3657(17)	116(21)	228(33)	188(26)	−97(21)	83(20)	−2(23)
C(36)	4171(23)	4700(15)	3228(17)	158(24)	109(19)	232(29)	−69(18)	127(24)	−38(20)
C(37)	3455(22)	4571(15)	2401(17)	145(23)	131(21)	243(30)	−40(18)	144(24)	9(21)
C(38)	3628(22)	3924(15)	2001(17)	141(21)	122(21)	236(28)	−14(17)	141(22)	10(20)
C(39)	4574(22)	3450(16)	2576(17)	135(20)	196(29)	212(26)	−33(19)	144(21)	−11(22)
C(40)	5395(25)	3454(17)	3445(20)	186(27)	173(28)	295(37)	−35(22)	193(29)	−65(26)
C(41)	5948(32)	4254(29)	4519(21)	192(35)	467(73)	194(33)	−159(41)	66(28)	31(39)

Table 1b. Final atomic coordinates ($\times 10^3$) and isotropic thermal parameters of the hydrogen atoms with their estimated standard deviations in parentheses

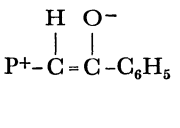
	x	y	z	B (Å^2)
H(2)	109(9)	378(7)	269(6)	2.8(27)
H(3)	115(9)	323(6)	321(6)	1.8(26)
H(4)	306(8)	314(6)	330(6)	0.4(22)
H(5)	223(10)	285(7)	253(7)	3.4(31)
H(6)	−176(10)	204(7)	227(6)	2.4(28)
H(8)	−402(11)	401(8)	205(7)	5.0(37)
H(9)	−279(9)	483(6)	185(6)	1.9(27)
H(10)	−121(10)	424(6)	159(6)	2.4(28)
H(12)	−195(14)	230(10)	15(9)	8.2(49)
H(13)	−217(15)	262(11)	−94(10)	10.0(56)
H(14)	−131(13)	365(10)	−121(9)	8.0(44)
H(15)	59(10)	429(7)	14(7)	2.6(30)
H(16)	81(11)	400(8)	134(7)	4.9(37)
H(18)	137(10)	210(7)	455(7)	2.9(30)
H(19)	207(12)	281(8)	580(8)	5.4(38)
H(20)	396(11)	345(7)	656(7)	3.8(34)
H(22)	460(10)	274(7)	484(6)	2.4(28)
H(24)	466(9)	130(7)	494(6)	2.5(26)
H(26)	639(9)	18(6)	410(6)	1.8(27)
H(27)	509(14)	62(9)	293(9)	8.3(49)
H(30)	−134(11)	38(8)	99(7)	4.3(34)
H(32)	−5(13)	−162(6)	78(9)	6.9(43)
H(34)	219(11)	57(7)	150(7)	3.7(32)

illustrated in Figs. 1 and 2, respectively. Bond lengths and bond angles are listed in Tables 2 and 3. The results show that [$PdCl_2$(bdep)] is an ylide complex in which the ylide carbon coordinates to palladium with Pd–C bond length of 2.115(15) Å. This value is slightly larger than the metal-ylide carbon bond lengths (1.91—2.10 Å) determined by X-ray diffraction for some other ylide complexes.[8–10] Although the ylide methine hydrogen atom could not be located, the bond angles around the ylide carbon ∠Pd–C(1)–P(1) (115.1(6)°), ∠Pd–C(1)–C(4) (102.9(9)°), and ∠P(1)–C(1)–C(4) (111.2(12)°), suggest that the C(1) carbon orbital is nearly sp^3 hybrid. This provides another example of the conversion of sp^2 hybridized ylide carbon to sp^3 state on coordination to the metal.[8,11] The phosphine P also is coordinated to palladium with the Pd–P(2) bond length of 2.230(8) Å, and a six-membered chelate ring is formed. The five atoms, Pd, C(1), P(2), Cl(1), and Cl(2), are approximately coplanar and the coordination around the palladium has square-planar geometry with the angles, ∠C(1)–Pd–P(2) (86.2(1)°), ∠C(1)–Pd–Cl(1) (87.3(4)°), ∠Cl(1)–Pd–Cl(2) (90.8(1)°), and ∠Cl(2)–Pd–P(2) (96.2(4)°).

The bond lengths of the ylide system in the present complexes, P(1)–C(1) (1.795(12) Å), C(4)–O (1.225(21) Å, and C(4)–C(1) (1.486(27) Å), indicate that the complex presumably takes the betaine structure (I) in contrast with the significant contribution of the betaine structure (II) in the free ligand which can be inferred from the lower ν(CO) frequency in an infrared spectrum.[4] The X-ray structure determination of a benzoyl stabilized ylide, benzoylchlorotriphenylphosphorane, has shown that the main contribution in the bonding is the betaine structure (II).[12]



(I)



(II)

It seems significant to compare the properties of ylides as ligands with those of phosphines, because both ylides (C ligands) and phosphines (P ligands) coordinate to the metal in similar fashion formally. Bdep which has both ylide and phosphine parts in a molecule appears to be a suitable ligand for the comparison of such properties as trans-influence (or -effect) simulta-

Table 2. Bond lengths (l/Å) with their estimated standard deviations in parentheses

Pd-C(1)	2.115(15)	C(9)-C(10)	1.397(29)	C(28)-C(23)	1.424(29)	H(6)-C(6)	0.97(11)
Pd-P(2)	2.230(8)	C(10)-C(5)	1.382(18)	C(29)-C(30)	1.393(26)	H(8)-C(8)	1.18(17)
Pd-Cl(1)	2.390(8)	C(11)-C(12)	1.358(20)	C(30)-C(31)	1.415(22)	H(9)-C(9)	1.12(11)
Pd-Cl(2)	2.340(9)	C(12)-C(13)	1.447(35)	C(31)-C(32)	1.403(34)	H(10)-C(10)	1.13(15)
P(1)-C(1)	1.795(12)	C(13)-C(14)	1.337(38)	C(32)-C(33)	1.343(30)	H(12)-C(12)	1.08(17)
P(1)-C(2)	1.816(14)	C(14)-C(15)	1.345(23)	C(33)-C(34)	1.423(23)	H(13)-C(13)	0.97(16)
P(1)-C(5)	1.783(19)	C(15)-C(16)	1.430(34)	C(34)-C(29)	1.391(29)	H(14)-C(14)	1.09(16)
P(1)-C(11)	1.808(19)	C(16)-C(11)	1.337(29)	C(35)-C(36)	1.361(35)	H(15)-C(15)	1.04(13)
P(2)-C(3)	1.836(15)	C(17)-C(18)	1.377(31)	C(36)-C(37)	1.387(40)	H(16)-C(16)	0.98(11)
P(2)-C(17)	1.856(17)	C(18)-C(19)	1.401(25)	C(37)-C(38)	1.442(45)	H(18)-C(18)	1.14(11)
P(2)-C(23)	1.797(17)	C(19)-C(20)	1.341(27)	C(38)-C(39)	1.396(32)	H(19)-C(19)	1.02(19)
C(1)-C(4)	1.486(27)	C(20)-C(21)	1.386(37)	C(39)-C(40)	1.445(41)	H(20(-C(20)	1.05(13)
C(2)-C(3)	1.519(22)	C(21)-C(22)	1.406(25)	C(40)-C(35)	1.425(47)	H(22)-C(22)	1.01(16)
C(4)-C(29)	1.522(19)	C(22)-C(17)	1.359(22)	C(41)-C(35)	1.447(40)	H(24)-C(24)	0.87(14)
C(4)-O	1.225(21)	C(23)-C(24)	1.384(18)			H(26)-C(26)	1.08(12)
C(5)-C(6)	1.394(26)	C(24)-C(25)	1.373(27)	H(2)-C(2)	0.84(12)	H(27)-C(27)	1.00(21)
C(6)-C(7)	1.448(32)	C(25)-C(26)	1.402(33)	H(3)-C(2)	1.02(14)	H(30)-C(30)	0.97(15)
C(7)-C(8)	1.367(25)	C(26)-C(27)	1.388(22)	H(4)-C(3)	0.97(9)	H(32)-C(32)	1.11(15)
C(8)-C(9)	1.380(30)	C(27)-C(28)	1.343(30)	H(5)-C(3)	0.87(15)	H(34)-C(34)	1.21(13)

Table 3. Bond angles (ϕ/°) with their estimated standard deviations in parentheses

Cl(1)-Pd-Cl(2)	90.8(1)	P(1)-C(5)-C(6)	121.6(12)	P(2)-C(23)-C(24)	121.4(13)
Cl(1)-Pd-C(1)	87.3(4)	P(1)-C(5)-C(10)	118.6(12)	P(2)-C(23)-C(28)	121.1(11)
Cl(2)-Pd-P(2)	96.2(4)	C(6)-C(5)-C(11)	139.4(14)	C(24)-C(23)-C(28)	117.1(15)
P(2)-Pd-C(1)	86.2(1)	C(5)-C(6)-C(7)	119.5(16)	C(23)-C(24)-C(25)	123.2(18)
Cl(1)-Pd-P(2)	174.1(2)	C(6)-C(7)-C(8)	118.2(20)	C(24)-C(25)-C(26)	118.9(16)
Cl(2)-Pd-C(1)	173.2(4)	C(7)-C(8)-C(9)	122.3(20)	C(25)-C(26)-C(27)	117.5(19)
Pd-C(1)-P(1)	115.1(6)	C(8)-C(9)-C(10)	119.1(16)	C(26)-C(27)-C(28)	124.0(21)
Pd-C(1)-C(4)	102.9(9)	C(5)-C(10)-C(9)	121.0(16)	C(23)-C(28)-C(27)	119.1(16)
P(1)-C(1)-C(4)	111.2(12)	P(1)-C(11)-C(12)	116.6(13)	C(4)-C(29)-C(30)	121.7(16)
C(1)-P(1)-C(2)	114.6(5)	P(1)-C(11)-C(16)	122.8(13)	C(4)-C(29)-C(34)	118.1(15)
C(1)-P(1)-C(5)	109.2(7)	C(12)-C(11)-C(16)	120.6(17)	C(30)-C(29)-C(34)	120.2(16)
C(1)-P(1)-C(11)	110.3(7)	C(11)-C(12)-C(13)	119.7(21)	C(29)-C(30)-C(31)	120.2(19)
C(2)-P(1)-C(5)	106.1(7)	C(12)-C(13)-C(14)	118.8(19)	C(30)-C(31)-C(32)	117.7(18)
C(2)-P(1)-C(11)	108.9(7)	C(13)-C(14)-C(15)	120.9(22)	C(31)-C(32)-C(33)	122.6(18)
C(5)-P(1)-C(11)	107.5(8)	C(14)-C(15)-C(16)	120.7(22)	C(32)-C(33)-C(34)	119.8(21)
Pd-P(2)-C(3)	115.6(5)	C(11)-C(16)-C(15)	119.1(17)	C(29)-C(34)-C(33)	119.2(17)
Pd-P(2)-C(17)	112.9(5)	P(2)-C(17)-C(18)	121.1(12)	C(36)-C(35)-C(40)	132.8(26)
Pd-P(2)-C(23)	115.5(5)	P(2)-C(17)-C(22)	118.3(14)	C(36)-C(35)-C(41)	122.4(29)
C(3)-P(2)-C(17)	100.9(7)	C(18)-C(17)-C(22)	120.5(16)	C(40)-C(35)-C(41)	104.7(24)
C(3)-P(2)-C(23)	104.7(7)	C(17)-C(18)-C(19)	118.2(16)	C(35)-C(36)-C(37)	115.7(28)
C(17)-P(2)-C(23)	105.8(7)	C(18)-C(19)-C(20)	120.2(21)	C(36)-C(37)-C(38)	123.7(24)
P(1)-C(2)-C(3)	114.3(10)	C(19)-C(20)-C(21)	123.4(19)	C(37)-C(38)-C(39)	109.5(24)
P(2)-C(3)-C(2)	111.1(12)	C(20)-C(21)-C(22)	115.2(18)	C(38)-C(39)-C(40)	136.2(29)
C(1)-C(4)-C(29)	119.5(14)	C(17)-C(22)-C(21)	122.4(19)	C(35)-C(40)-C(39)	101.7(23)
C(1)-C(4)-O	123.4(14)				
C(29)-C(4)-O	117.0(15)				

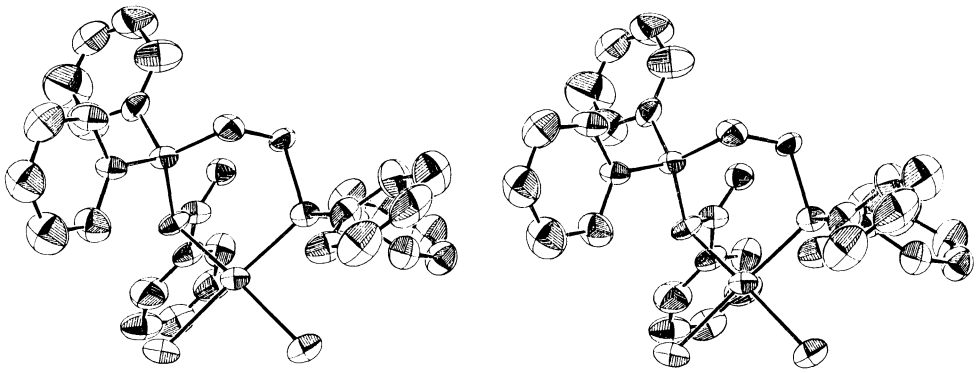


Fig. 2. A stereoscopic view of the molecule; the thermal ellipsoids are drawn at the 50% probability level (ORTEP).

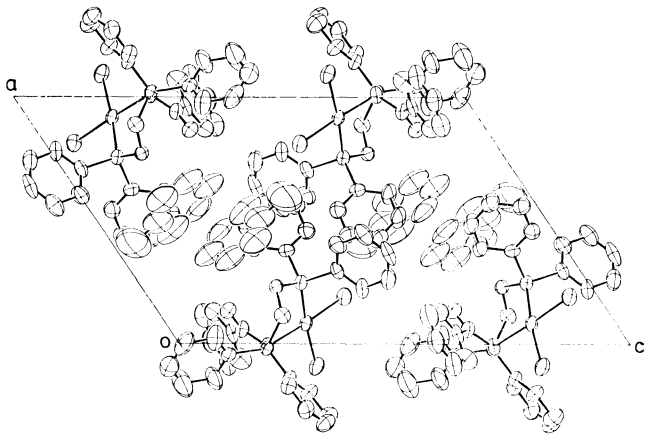


Fig. 3. Crystal structure projected along the b axis.

neously. Chlorine atoms in $[PdCl_2(bdep)]$ are bonded to palladium with bond lengths of Pd–Cl(1) (2.390(8) Å) and Pd–Cl(2) (2.340(8) Å). The Pd–Cl(2) which is trans to the ylide C is somewhat shorter than the other Pd–Cl(1) which is *trans* to the phosphine P. Since longer bond length of the bond *trans* to a ligand can be regarded as an indication of larger *trans*-influence of the ligand,[13] it is likely that the *trans*-influecne of the ylide C is comparable with, but slightly smaller than, that of the phosphine P in bdep.

Crystal Structure. The crystal structure projected along b axis is shown in Fig. 3. Toluene molecules are packed in voids formed by $[PdCl_2(bdep)]$ molecules, but the molecular interaction seems to be rather loose judging from the intermolecular contacts which are larger than 4 Å.

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