

Tabelle 3. Abstände (Å) und Winkel (°) in den Wasserstoffbrückenbindungen (Standardabweichungen in Klammern)

	O...O	H...O	Symmetrieeoperation
O(12)–H(122)···O(26)	2,73 (3)	2,2 (3)	x, y, z
O(12)–H(121)···O(215)	2,77 (4)	2,3 (3)	x, y, z
O(16)–H(162)···O(23)	2,75 (2)	1,8 (2)	x, y, z
O(16)–H(161)···O(212)	2,75 (2)	1,9 (2)	x, y, z
	Winkel		
O(26)···O(12)···O(215)	96 (2)		x, y, z
O(23)···O(16)···O(212)	110 (1)		x, y, z

Aufgrund der fehlenden H-Brückenbindung vom koordinierten Wasser zum Atom O(29) zeigt dieser Teil des Kronenethers eine größere thermische Beweglichkeit mit entsprechend höheren Temperaturfaktoren, es treten dadurch erhöhte Standardabweichungen bei den Bindungslängen und Winkeln an den Atomen O(29), C(210) und C(211) auf.

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### Structure of *cis*-Dichlorobis(perhydro-6-methylallyl-2-phenyl-1,3,6,2-dioxazaphosphocine)palladium(II)

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**Abstract.** [PdCl<sub>2</sub>(C<sub>14</sub>H<sub>20</sub>NO<sub>2</sub>P)<sub>2</sub>],  $M_r = 707.9$ , monoclinic,  $C2/c$ ,  $a = 25.516(3)$ ,  $b = 8.260(1)$ ,  $c = 17.151(2)$  Å,  $\beta = 113.70(1)^\circ$ ,  $V = 3309.9(9)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.420$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.84$  mm<sup>-1</sup>,  $F(000) = 1456$ ,  $T = 293$  K. Full-matrix least-squares refinement based on 2994 observed reflections led to  $R$  and  $wR$  values of 0.018 and 0.022 respectively. The molecular structure of the title

compound proves, in accordance with the analytical and spectroscopic measurements, the *cis* arrangement of the two P,N ligands.

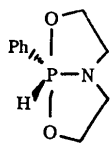
**Introduction.** The first synthesis of a transition-metal complex with the cyclic aminophosphonite (*2a*) derived from the bicyclic phosphorane (*1a*) by opening of the P–N bond was described ten years ago (scheme) and the structure of its carbonylchlororhodium complex determined by an X-ray diffraction study (Bondoux,

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Tkatchenko, Houalla, Wolf, Pradat, Riess & Mentzen, 1978).



(1a)



(2a) R = H

(2b) R = CH<sub>3</sub>(2c) R = CH<sub>2</sub>-C(CH<sub>3</sub>)=CH<sub>2</sub>

Nowadays, the coordination of such P,N hybrid ligands ( $R = \text{H, Me}$ ) has been extended to various transition metals with a large variety of coordination modes (monodentate, chelating, bridging, phosphoranide, . . .) and reactions, as proved by the number of references cited in recent papers (Aresta, Ballivet-Tkatchenko, Bonnet, Faure & Loiseleur, 1985; Riess, 1986).

This study is devoted to the structure of the title compound (3) with a new cyclic aminophosphonite ligand (2c). Interestingly, this compound offers, besides the P and N atoms, a novel potential coordination site, namely the double bond of the methylallyl substituent of the amino group.

**Experimental.** Yellow parallelepiped crystals obtained from a dichloromethane solution of (3) by slow diffusion of hexane. Crystal  $0.60 \times 0.40 \times 0.30$  mm, sealed on a glass fiber. CAD-4 diffractometer, graphite-monochromated Mo  $K\alpha$ , cell parameters from a least-squares fitting of 25 reflections with  $2\theta$  between 23 and  $32^\circ$ , 3710 reflections measured, using  $\theta-2\theta$  scans for  $2\theta$  from 3 to  $54^\circ$  ( $h-32$  to 32,  $k0$  to 10,  $l0$  to 21), scan range  $(0.85 + 0.35 \tan \theta)^\circ$ . Intensities of three reflections ( $2,0,10$ ,  $12,2,1$ ,  $538$ ) measured every 2 h during data collection varied less than 2% and thus indicated crystal stability. Corrections for Lp. Empirical absorption corrections (North, Phillips & Mathews, 1968):  $T_{\min} = 0.91$ ,  $T_{\max} = 1.0$  and merging equivalent reflections  $hk0$  and  $\bar{h}k0$ :  $R_{\text{av}} = 0.014$ . Heavy-atom method followed by Fourier and least-squares techniques using 2994 reflections having  $F_o^2 > 3\sigma(F_o^2)$ ,  $\sigma(F_o^2)$  based on counting statistics. Full-matrix least-squares refinement minimizing  $\sum w(|F_o| - |F_c|)^2$ , with anisotropic thermal parameters for all non-H atoms and constrained geometry for H atoms ( $\text{C}-\text{H} = 0.97 \text{ \AA}$ ), isotropic  $U_{\text{H}} = 0.05 \text{ \AA}^2$  kept fixed ( $0.06 \text{ \AA}^2$  for methyl H atoms).  $R = 0.018$ ,  $wR = 0.022$ , 177 variables, unit weights. Mean and max. parameter shifts  $0.07 \sigma$  and  $0.30 \sigma$  respectively. Max. and min. height in final  $\Delta F$  map  $0.34$  and  $-0.19 \text{ e \AA}^{-3}$ . Scattering factors including real and imaginary parts of anomalous dispersion from *International Tables for X-ray Crystallography* (1974) and

from Stewart, Davidson & Simpson (1965) for H atoms. VAX 730 computer. SDP (Frenz, 1982), SHELX76 (Sheldrick, 1976), ORFFE (Busing, Martin & Levy, 1964) and NRC (Ahmed, Hall, Pippy & Huber, 1966) programs.

**Discussion.** The final positional and equivalent isotropic thermal parameters are listed in Table 1.\* The thermal-ellipsoid plot of the molecule (Johnson, 1965) is shown in Fig. 1 with the atomic numbering. The packing of the molecules in the unit cell is shown in Fig. 2. Bond lengths and angles are listed in Table 2.

The coordination polyhedron of the Pd atom is roughly square-planar with two Cl and two P atoms in a *cis* arrangement, the Pd atom being in a special position on the mirror plane. The Cl atoms are  $\pm 0.2157(6) \text{ \AA}$  and the P atoms  $\pm 0.2072(6) \text{ \AA}$  from the least-squares plane defined by the Pd and its four nearest neighbors, larger deviations than those observed ( $\text{Cl} = \pm 0.13$ ,  $\text{P} = \pm 0.12 \text{ \AA}$ ) for the complex *cis*-dichlorobis(dimethylphenylphosphine)palladium(II) (Martin & Jacobson, 1971). The dihedral angle between the PdCl<sub>2</sub> and PdP<sub>2</sub> planes is  $14.8^\circ$ , a value very close to that of  $14.6^\circ$  found for the complex *cis*-[PdCl<sub>2</sub>{P(2-C<sub>5</sub>H<sub>4</sub>N)<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)}] (Newcome, Evans & Fronczek, 1987).

The Pd-Cl, 2.3563(5) Å, and Pd-P, 2.2256(5) Å, bond lengths are quite similar to those observed for other *cis*-[PdCl<sub>2</sub>(phosphine)<sub>2</sub>] complexes which are in the ranges 2.33–2.42 Å and 2.22–2.27 Å respectively (Martin & Jacobson, 1971; Alcock, Kemp & Wimmer, 1981; Alcock & Nelson, 1985; Newcome *et al.*, 1987). Owing to the *trans* effect of the phosphine ligands, the Pd-Cl bond in the title compound is longer and the Pd-P shorter than in the *trans* complexes, for example, in the *trans*-[PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>] species (Pd-Cl = 2.290, Pd-P = 2.337 Å) (Ferguson, McCrindle, McAlees & Parvez, 1982). The P-Pd-P' angle is slightly larger than  $90^\circ$  indicating less repulsion between the two P atoms than for other *cis*-[PdCl<sub>2</sub>(phosphine)<sub>2</sub>] complexes. Similarly the Cl-Pd-Cl' angle is close to  $90^\circ$ .

The values of the distances between the metallic center and the N atom, 4.164(2) Å, and the metallic center and the center of the double bond C(6)-C(7) of the methylallyl substituent of the amino group of the P,N ligand (2c), 5.356(3) Å, exclude any possibility of coordination of these two sites to the Pd, thus showing the monodentate behavior of (2c). This fact confirms the tendency of the cyclic aminophosphonite compounds (2b and 2c) bearing a tertiary amino group to act as monodentate ligands (Bonnet, Agbossou,

\* Lists of structure factors, anisotropic thermal parameters, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51823 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 100$ ) with e.s.d.'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
Pd	$\frac{1}{2}$	0.14645 (3)	$\frac{3}{4}$	3.07 (3)
Cl	0.46422 (2)	-0.05140 (7)	0.64247 (3)	4.36 (9)
P	0.48271 (2)	0.33498 (6)	0.64999 (3)	3.24 (9)
O(1)	0.52241 (6)	0.4883 (2)	0.68821 (9)	3.7 (2)
O(2)	0.49032 (6)	0.2835 (2)	0.56601 (9)	3.6 (2)
C(1)	0.5498 (1)	0.5802 (3)	0.6429 (2)	4.9 (4)
C(2)	0.6105 (1)	0.5246 (3)	0.6723 (2)	4.8 (4)
C(3)	0.5373 (1)	0.1885 (3)	0.5616 (2)	4.9 (4)
C(4)	0.5885 (1)	0.2922 (3)	0.5738 (2)	4.7 (4)
N	0.61565 (7)	0.3496 (2)	0.66247 (8)	4.4 (2)
C(5)	0.6750 (1)	0.2921 (3)	0.7063 (2)	5.2 (4)
C(6)	0.6780 (1)	0.1088 (3)	0.7169 (2)	5.3 (4)
C(7)	0.7021 (1)	0.0254 (3)	0.6696 (2)	5.6 (5)
C(8)	0.6544 (1)	0.0344 (3)	0.7772 (2)	5.9 (5)
C(9)	0.41012 (9)	0.4097 (3)	0.5996 (1)	4.2 (3)
C(10)	0.3956 (1)	0.5648 (3)	0.6165 (1)	4.5 (4)
C(11)	0.33966 (9)	0.6201 (3)	0.5759 (1)	4.6 (4)
C(12)	0.2979 (1)	0.5209 (3)	0.5184 (1)	4.8 (4)
C(13)	0.3123 (1)	0.3656 (3)	0.5013 (2)	4.9 (4)
C(14)	0.3683 (1)	0.3106 (3)	0.5419 (1)	4.7 (4)

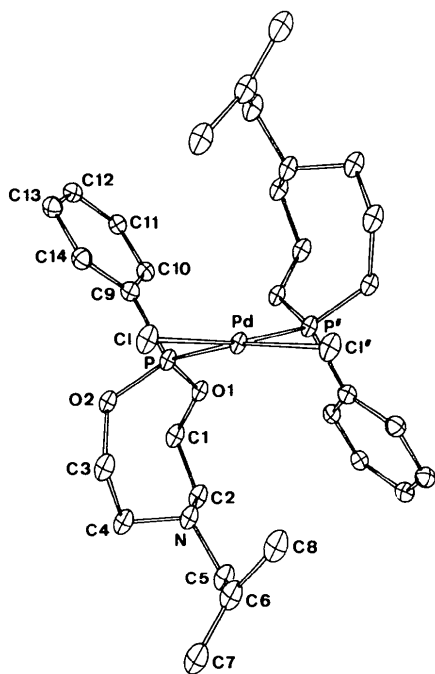


Fig. 1. ORTEP plot showing 25% probability thermal ellipsoids.

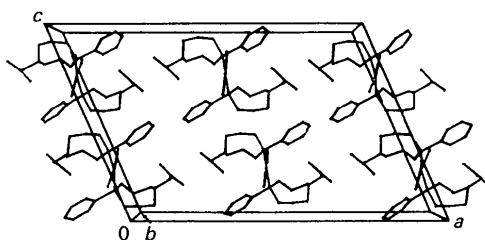


Fig. 2. An ORTEP view of the unit-cell contents.

Table 2. Interatomic bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

Pd-P, Pd-P'	2.2256 (5)	Pd-Cl, Pd-Cl'	2.3563 (5)
P-Pd-P'	91.20 (3)	Cl-Pd-Cl'	92.17 (3)
P-Pd-Cl, P'-Pd-Cl'	82.29 (2)	P'-Pd-Cl	169.39 (2)
P-O(1)	1.589 (1)	P-O(2)	1.587 (2)
	P-C(9)	1.809 (2)	
Pd-P-O(1)	110.10 (5)	O(1)-P-O(2)	107.32 (9)
Pd-P-O(2)	116.96 (6)	O(1)-P-C(9)	106.60 (9)
Pd-P-C(9)	117.07 (8)	O(2)-P-C(9)	97.63 (9)
O(1)-C(1)	1.451 (3)	O(2)-C(3)	1.460 (3)
C(1)-C(2)	1.494 (4)	C(3)-C(4)	1.505 (4)
C(2)-N	1.467 (3)	C(4)-N	1.474 (3)
N-C(5)	1.473 (3)	C(5)-C(6)	1.523 (4)
C(6)-C(7)	1.382 (5)	C(6)-C(8)	1.522 (5)
C(9)-C(10)	1.395 (3)	C(10)-C(11)	1.390 (3)
C(11)-C(12)	1.391 (3)	C(12)-C(13)	1.397 (4)
C(13)-C(14)	1.389 (3)	C(14)-C(9)	1.392 (3)
P-O(1)-C(1)	124.1 (1)	P-O(2)-C(3)	126.4 (1)
O(1)-C(1)-C(2)	108.3 (2)	O(2)-C(3)-C(4)	111.9 (2)
C(1)-C(2)-N	113.2 (2)	C(3)-C(4)-N	110.4 (2)
C(2)-N-C(4)	114.2 (2)	C(2)-N-C(5)	112.4 (2)
C(4)-N-C(5)	112.5 (2)	N-C(5)-C(6)	111.9 (2)
C(5)-C(6)-C(7)	116.0 (3)	C(5)-C(6)-C(8)	117.9 (3)
C(7)-C(6)-C(8)	126.1 (2)		
P-C(9)-C(10)	121.3 (2)	P-C(9)-C(14)	119.0 (2)
C(14)-C(9)-C(10)	119.7 (2)	C(9)-C(10)-C(11)	120.2 (2)
C(10)-C(11)-C(12)	120.0 (2)	C(11)-C(12)-C(13)	119.9 (2)
C(12)-C(13)-C(14)	119.9 (2)	C(13)-C(14)-C(9)	120.3 (2)

Tkatchenko, Faure & Loiseleur, 1987), in contrast to (2a), which shows a mainly bidentate coordination mode. The conformation of the eight-membered ring of (2c) is close to the chair-boat limiting form of the cyclooctane ring, as for other cyclic aminophosphonite complexes of rhodium (Bondoux, Mentzen & Tkatchenko, 1981; Bonnet, Tkatchenko, Faure & Loiseleur, 1983) and molybdenum (Wachter, Riess & Mitschler, 1984). The bond lengths and angles in the ligand are practically the same as those observed for the monodentate P bound to the metal ligands (2a) (Aresta *et al.*, 1985; Wachter *et al.*, 1984) and (2c) (Agbossou, Bonnet & Tkatchenko, 1985). The bond angles around the P atom exhibit a severe distortion from the ideal tetrahedron, as seems to be characteristic of cyclic aminophosphonites bound to a metallic center (Bondoux *et al.*, 1981). Finally, the geometric environment of the N atom is only slightly distorted from  $C_{3v}$  symmetry, probably because of the steric hindrance of the methylallyl group.

There is no evidence for any strong interactions between two neighbors, since all intermolecular contacts are greater than the van der Waals distances.

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## Structure of Bis(1,10-phenanthroline)trifluoromethanesulfonatolithium

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**Abstract.** [Li(CF<sub>3</sub>O<sub>3</sub>S)(C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>],  $M_r = 516.43$ , monoclinic,  $P2_1/n$ ,  $a = 10.540$  (4),  $b = 15.403$  (6),  $c = 14.624$  (6) Å,  $\beta = 90.55$  (3)°,  $V = 2374.1$  (17) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.44$  g cm<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu = 1.80$  cm<sup>-1</sup>,  $T = 298$  K,  $F(000) = 964$ ,  $R = 0.053$  for 2221 reflections with  $I > 3.0\sigma(I)$  (4197 unique observations). The Li is trigonal-bipyramidally coordinated to two phenanthroline molecules and one trifluoromethanesulfonate, with one equatorial Li–O bond [2.072 (9) Å] and four Li–N [2.115 (9)–2.166 (9) Å] bonds. The dihedral angle between the two planar 1,10-phenanthroline rings is 59.5°.

**Introduction.** Alkali-metal cations usually form 1:1 complexes with the 1,10-phenanthroline (phen) ligand, except for the *o*-nitrophenolates of Na<sup>+</sup>, K<sup>+</sup>, Rb<sup>+</sup> and Cs<sup>+</sup>, to which two molecules of phenanthroline are added (Layton, Nyholm, Banerjee, Fenton, Lestas & Truter, 1970; Poonia & Bajaj, 1979; Fenton, 1985). The relatively small Na<sup>+</sup> and K<sup>+</sup> cations form 1:2 complexes only when the reaction mixture contains foreign proton-donor species which stabilize the counter anion (Poonia, 1977). In this paper, we report that the small Li<sup>+</sup> cation forms a stable complex with

two equivalents of 1,10-phenanthroline in a non-protonic solvent, CH<sub>2</sub>Cl<sub>2</sub>, by use of the weakly basic trifluoromethanesulfonate counter ion (Nitschke, Schmidt & Trogler, 1985).

**Experimental.** Addition of 0.60 g (3.32 mmol) of 1,10-phenanthroline dissolved in 30 mL of CH<sub>2</sub>Cl<sub>2</sub>, to a stirred white slurry of 0.25 g (1.60 mmol) of Li(O<sub>3</sub>SCF<sub>3</sub>) in 30 mL of CH<sub>2</sub>Cl<sub>2</sub>, gave a yellow solution. After 18 h the solution was filtered, concentrated under vacuum, and layered with pentane to yield (0.51 g, 62%) white crystals of Li(O<sub>3</sub>SCF<sub>3</sub>)(phen)<sub>2</sub>. Analysis. Calculated for C<sub>25</sub>H<sub>16</sub>N<sub>4</sub>O<sub>3</sub>F<sub>3</sub>SLi: C 58.15; H 3.12; N 10.85%. Found: C 58.37; H 3.13; N 10.95%. m.p. 512–514 K.

X-ray data were collected with the use of a Nicolet R3m/V automated diffractometer equipped with a Mo X-ray tube and a graphite-crystal monochromator. Details on crystal data and intensity data are given in Table 1. The orientation matrix and unit-cell parameters were determined from 25 machine-centered reflections with  $15 < 2\theta < 30^\circ$ . The intensities of three check reflections (222, 105, 043) were monitored after every 100 reflections and showed statistical variations within 2%. Data were corrected for Lorentz and polarization effects. No absorption correction was

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