perpendicular to this plane. One of the two planar nitrate groups is slightly tilted with respect to the equatorial plane (8°). The folding of the amide ligand, which allows the bidentate bonding, means that it spreads in a plane approximately perpendicular to the equatorial plane: the angle between the benzene ring and the equatorial hexagon is 76°.

Three of the butyl chains of the amide have the expected *trans* conformation, the fourth [C(35)···C(38)] a *cis* conformation. This seems to be due to the packing of the molecules: the distance O(2′)···C(38), which is 4·54 (2) Å, would be, in a *trans* conformation, too short (about 2·5 Å).

Complex 2. [UO$_2$(NO$_3$)$_2$(tbga)]. The structure is formed by the [UO$_2$(NO$_3$)$_2$(tbga$^{1/2}$)$_2$] units in which both nitrate and diamide ligands are in *trans* positions. The U atom lies on a center of symmetry. Two units are linked by a bridging diamide, each diamide being shared by two U atoms: the central C atom of the diamide C(2) lies on a diad axis. The bridging is repeated indefinitely to form a polymer chain as shown in Fig. 4. The six O atoms of the equatorial plane are not

rigorously coplanar, the maximum deviations of the atoms from this plane are ±0·17 (1) Å. The linear uranyl group is normal to this best equatorial plane. The nitrate group is perfectly planar and tilted 10° with respect to the equatorial plane.

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Structure of Chloro[1–3-η-(2-methylallyl)](perhydro-6-methyl-2-phenyl-1,3,6,2-dioxazaphosphocine)palladium

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Abstract. [PdCl(C$_4$H$_7$)(C$_{11}$H$_{16}$NO$_2$P)], $M_r = 422·2$, monoclinic, $P2_1$, $a = 8·631$ (1), $b = 11·756$ (2), $c = 8·824$ (1) Å, $\beta = 103·21$ (1)°, $V = 871·6$ (3) Å^3, $Z = 2$, $D_x = 1·61$ Mg m^{-3}, λ(Mo $K\overline{\alpha}$) = 0·7107 Å, $\mu = 1·29$ mm^{-1}, $F(000) = 428$, $T = 295$ K, $R = 0·027$ for 3437 unique observed reflections. The Pd^{II} atom is coordinated by the 2-methylallyl ligand [distance from Pd to middle of C–C bonds = 2·081 (4) and 2·010 (4) Å], the Cl atom [Pd–Cl = 2·3748 (9) Å] and the P atom of the aminophosphonite ligand [Pd–P = 2·2721 (6) Å]. Thus C$_6$H$_5$P(OC$_2$H$_4$)$_2$NCH$_3$ acts only as a monodentate ligand. The title compound is compared with other allylic complexes of palladium and some aminophosphonite complexes.

Introduction. The great synthetic and catalytic potential of allylic complexes of palladium (Trost, 1977, 1980; Tsuji, 1975, 1980; Maitlis, 1971) and their increasing use in homogeneous catalysis have attracted interest in the synthesis of new neutral and cationic species. We have been interested for many years in the use of particular ligands, namely cyclic aminophosphonites PNR (1). These compounds can act either as monodentate P or N ligands or as bidentate chelating or bridging ligands (Aresta, Ballivet-Tkatchenko & Bonnet, 1985). The structure of the title compound (2) is described and compared with other allylic complexes of palladium and some aminophosphonite (PNR) complexes of cobalt and rhodium. The synthesis and

$[PdCl(C_4H_7)(C_{11}H_{16}NO_2P)]$

catalytic properties of the complex have been previously reported (Agbossou, Bonnet & Tkatchenko, 1985).

(1a) R = H
(1b) R = Me

(1c)
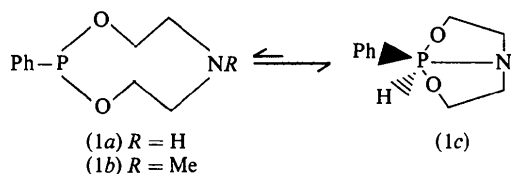
Experimental. Pale-yellow transparent crystal from dichloromethane, $0.2 \times 0.5 \times 0.5$ mm. Nonius CAD-4 diffractometer, graphite monochromator, Mo $K\alpha$ radiation, $\omega-\frac{4}{3}\theta$ scan, $2 \leq 2\theta \leq 70°$. Unit-cell parameters refined from setting angles of 25 selected reflections ($30.58 \leq 2\theta \leq 57.62°$). 3981 independent reflections, only 3437 observed $[I > 1.4\sigma(I)$ and $I > 1.3 \ I_{background}]$. $-13 \leq h \leq 13$, $0 \leq k \leq 18$, $0 \leq l \leq 14$. Standard reflection, 414, showed 4.9% intensity variation. Lp corrections, no absorption correction. Structure solved by Patterson and heavy-atom methods. Full-matrix refinements based on F with $w = (a + b|F_o|)^{-2}$ calculated from $|\Delta F|$ vs $|F_o|$ curves. H atoms located from ΔF syntheses, with isotropic B equal to the B_{eq} of bearing C atoms. Final refinement involving the x, y, z, β_{ij} parameters for the non-H atoms and x, y, z for the H atoms. $R = 0.027$, $wR = 0.033$, $S = 0.54$, $(\Delta/\sigma)_{max} = 0.30$ (non-H atoms), $|\Delta\rho| \leq 0.62$ e Å^{-3}. f from *International Tables for X-ray Crystallography* (1974). Computing by means of our library of classical crystallographic programs (Quaglieri, Loiseleur & Thomas, 1972), IBM 3090/200 computer, Centre Interégional de Calcul Electronique (CIRCE), Orsay.

Discussion. The refined atomic parameters are given in Table 1.* Main bond lengths and angles are in Table 2.

The drawing of the molecule in Fig. 1 was obtained by means of *ORTEP* (Johnson, 1965). The Pd atom is surrounded by a severely distorted square plane formed by the C(14) and C(15) atoms of the 2-methylallyl ligand, the Cl atom and the P atom of the cyclo-aminophosphonite ligand.

The Pd, P, Cl, C(14) and C(15) atoms are nearly coplanar, within ± 0.047 (6) Å, the Pd atom lying 0.033 (1) Å out of the mean plane. The dihedral angle between this coordination plane and the plane of the 2-methylallyl ligand defined by C(13), C(14) and C(15)

Table 1. *Relative atomic coordinates and thermal B_{eq} parameters*

$$B_{eq} = \tfrac{4}{3}\Sigma_i \Sigma_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	$B_{eq}(Å^2)$
Pd	0.01833 (2)	0	0.08285 (2)	2.5 (1)
Cl	−0.1316 (1)	−0.0956 (1)	−0.1399 (1)	4.6 (1)
P	0.27424 (8)	−0.04829 (7)	0.08732 (8)	2.3 (1)
O(1)	0.3196 (3)	−0.1298 (2)	−0.0431 (3)	3.3 (1)
O(2)	0.4097 (3)	0.0465 (2)	0.1385 (3)	3.0 (1)
N	0.2775 (4)	0.0906 (3)	−0.1710 (4)	3.4 (1)
C(1)	0.2592 (5)	−0.1115 (3)	−0.2085 (4)	3.6 (1)
C(2)	0.3291 (5)	−0.0022 (6)	−0.2541 (4)	4.2 (1)
C(3)	0.1317 (7)	0.1447 (5)	−0.2498 (7)	5.0 (2)
C(4)	0.3975 (5)	0.1674 (4)	−0.0845 (5)	4.1 (1)
C(5)	0.3877 (5)	0.1627 (3)	0.0836 (5)	3.6 (1)
C(6)	0.3462 (3)	−0.1456 (3)	0.2489 (3)	2.6 (1)
C(7)	0.2714 (5)	−0.2505 (3)	0.2476 (4)	3.5 (1)
C(8)	0.3231 (6)	−0.3282 (4)	0.3664 (5)	4.3 (2)
C(9)	0.4490 (7)	−0.3011 (4)	0.4897 (5)	4.5 (2)
C(10)	0.5228 (7)	−0.1976 (5)	0.4932 (5)	4.3 (2)
C(11)	0.4730 (5)	−0.1198 (3)	0.3730 (4)	3.4 (1)
C(12)	−0.0792 (7)	−0.0477 (5)	0.3985 (6)	4.8 (2)
C(13)	−0.0669 (4)	0.0465 (4)	0.2871 (4)	3.5 (1)
C(14)	−0.1909 (5)	0.0683 (5)	0.1599 (6)	4.6 (2)
C(15)	0.0824 (5)	0.0983 (4)	0.2862 (5)	3.9 (1)

Table 2. *Bond lengths (Å) and angles (°)*

$M(1)$ is the center of C(13)$\rightleftharpoons$C(14), $M(2)$ is the center of C(13)$\rightleftharpoons$C(15).

Pd–Cl	2.3748 (9)	Pd–P	2.2721 (6)
Pd–C(13)	2.167 (3)	Pd–C(14)	2.220 (4)
Pd–C(15)	2.099 (4)	Pd–$M(1)$	2.081 (4)
Pd–$M(2)$	2.010 (4)	P–C(6)	1.823 (3)
P–O(1)	1.613 (2)	P–O(2)	1.604 (2)
C(1)–O(1)	1.449 (4)	C(5)–O(2)	1.447 (4)
C(1)–C(2)	1.512 (5)	C(5)–C(4)	1.506 (5)
C(2)–N	1.441 (7)	C(4)–N	1.452 (5)
C(3)–N	1.439 (6)	C(7)–C(6)	1.390 (5)
C(8)–C(7)	1.385 (6)	C(9)–C(8)	1.387 (7)
C(10)–C(9)	1.370 (6)	C(11)–C(6)	1.393 (5)
C(11)–C(10)	1.393 (6)	C(13)–C(12)	1.500 (7)
C(14)–C(13)	1.386 (7)	C(15)–C(13)	1.427 (5)

Cl–Pd–P	103.89 (4)	Cl–Pd–C(14)	95.6 (2)
Cl–Pd–C(15)	162.8 (1)	P–Pd–C(14)	160.1 (1)
P–Pd–C(15)	93.3 (1)	C(14)–Pd–C(15)	67.2 (2)
Cl–Pd–C(13)	125.7 (1)	P–Pd–C(13)	124.2 (1)
C(14)–Pd–C(13)	36.8 (2)	C(15)–Pd–C(13)	39.0 (2)
$M(1)$–Pd–$M(2)$	33.9 (2)	$M(1)$–Pd–Cl	110.8 (1)
$M(1)$–Pd–P	142.5 (1)	$M(2)$–Pd–Cl	144.5 (1)
$M(2)$–Pd–P	109.4 (1)	Pd–P–O(1)	122.0 (1)
Pd–P–O(2)	118.3 (1)	Pd–P–C(6)	109.06 (9)
O(1)–P–O(2)	109.5 (1)	O(1)–P–C(6)	95.2 (1)
O(2)–P–C(6)	96.9 (1)	P–O(1)–C(1)	122.7 (2)
P–O(2)–C(5)	122.1 (2)	C(2)–N–C(3)	115.2 (4)
C(2)–N–C(4)	118.2 (3)	C(3)–N–C(4)	114.7 (4)
O(1)–C(1)–C(2)	108.7 (3)	N–C(2)–C(1)	108.6 (3)
N–C(4)–C(5)	107.5 (3)	O(2)–C(5)–C(4)	109.4 (3)
P–C(6)–C(7)	118.4 (2)	P–C(6)–C(11)	122.9 (3)
C(7)–C(6)–C(11)	118.7 (3)	C(6)–C(7)–C(8)	120.7 (4)
C(7)–C(8)–C(9)	120.1 (4)	C(8)–C(9)–C(10)	119.9 (4)
C(9)–C(10)–C(11)	120.3 (4)	C(6)–C(11)–C(10)	120.4 (4)
C(12)–C(13)–C(14)	120.7 (4)	C(12)–C(13)–C(15)	121.2 (4)
C(14)–C(13)–C(15)	116.6 (4)	C(13)–C(14)–Pd	69.5 (2)
C(13)–C(15)–Pd	73.0 (2)		

($108.8°$) is lower than the values found for other 2-methylallyl complexes of palladium, ranging from $111.6°$ for $[PdCl(\eta^3\text{-}C_4H_7)]_2$ (3) (Mason & Wheeler, 1968) to $116°$ for $[PdCl(\eta^3\text{-}C_4H_7)(PPh_3)]$ (4) (Mason & Russell, 1966). As generally observed, the methyl
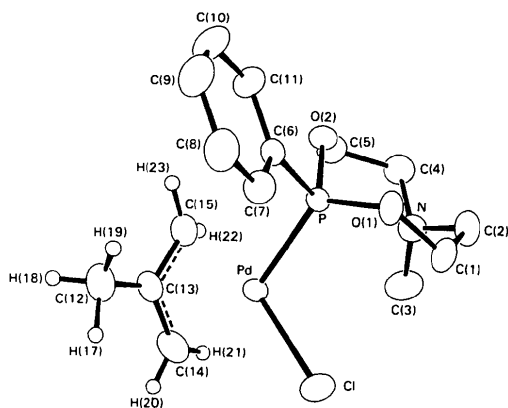
Fig. 1. *ORTEP* (Johnson, 1965) view of the complex with 40% probability thermal ellipsoids (only the H atoms of the methylallyl group are drawn).

C(12) atom is not in the plane of the allyl C(13), C(14) and C(15) atoms. It is displaced out of the plane by 0·315 (6) Å towards the Pd atom.

The Pd—Cl bond [2·3748 (9) Å] is similar to that observed in (4) [2·38 (3) Å] and in [PdCl(η^3-C$_3$H$_5$)-(2-amino-4-methylpyridine)] (5) (Reck, Heyn & Schröer, 1982) (2·396 Å) but slightly shorter than in the dimeric complexes (3) [2·413 (4)–2·395 (5)] and [PdCl(η^3-C$_3$H$_5$)]$_2$ (6) (Smith, 1965) [2·413 (2) Å] in which the Cl atoms act as bridging ligands.

The Pd—P bond length [2·2721 (6) Å] is shorter than that measured for the complex (4) [2·31 (1) Å] and for [Pd(η^3-C$_3$H$_5$)(SnCl$_3$)(PPh$_3$)] (7) (Mason & Whimp, 1969) [2·317 (3) Å]; this is in agreement with a smaller *trans* influence of PNMe ligand than for triphenyl-phosphine, the former being a better π-acceptor and a poorer σ-donor ligand. This *trans* influence is also reflected by the asymmetric bonding of the 2-methylallyl ligand to Pd: Pd—C(14) = 2·220 (4) Å (C *trans* to P) and Pd—C(15) = 2·099 (4) Å (C *trans* to Cl). These values are similar to those observed for the triphenylphosphine complex (4), 2·28 and 2·14 Å, respectively. The difference between the C(14)—C(13) [1·386 (7) Å] and C(15)—C(13) [1·427 (5) Å] bond lengths shows the same trend as for (4): 1·40 and 1·47 Å. The mean value of C⁼⁼C length within the allyl group (1·406 Å) compares well with those in [Pd(η^3-C$_4$H$_7$)(glycinato)] (8) (Benedetti, Maglio, Palumbo & Pedone, 1973) (1·42 Å) and [Pd(η^3-C$_4$H$_7$){2-(*R*,*S*)-α-phenylethylimino-3-penten-4-olato}] (9) (Claverini, Ganis & Pedone, 1973) (1·40 Å), but is greater than that in (3) [1·36 (3) Å] in accordance with the smaller *trans* influence of chlorine.

The angle C(14)—Pd—C(15) [67·2 (2)°] is very close to that determined for other 2-methylallyl complexes of palladium such as (8) (68°), (9) [67·7 (5)°] and (3) [66·0 (8)°]. Finally, the C(14)—C(13)—C(15) angle [116·6 (4)°] does not differ considerably from the usually observed values.

All the bond lengths of the (1*b*) ligand in the title complex (2) are very similar to those found in other cyclic aminophosphonite (1*a*) complexes of rhodium (Bondoux, Mentzen & Tkatchenko, 1981; Bonnet, Tkatchenko, Faure & Loiseleur, 1983), cobalt (Aresta, Ballivet-Tkatchenko, Bonnet, Faure & Loiseleur, 1985) or molybdenum (Wachter, Mitschler & Riess, 1981).

The P atom is coordinated to Pd as shown by the Pd—P bond length [2·2721 (6) Å]; in contrast, the Pd···N distance [3·667 (1) Å] precludes the possibility of bonding of the N atom to the metal, in accordance with the value [3·682 (6) Å] determined for the Co···N distance in the complex [CoCl(NO)$_2$(PNH)] (Aresta *et al.*, 1985) where the ligand (1*a*) is monodentate P-bound. The metal—N bond lengths found for the rhodium complexes (Bondoux *et al.*, 1981; Bonnet *et al.*, 1983) in which (1*a*) acts as bidentate P—N ligand are clearly shorter than in (2), ranging from 2·16 to 2·25 Å.

The P···N distance [2·809 (2) Å] lies within the range observed for the other aminophosphonite complexes (2·790–2·978 Å) and is shorter than the sum of the van der Waals radii of P and N (3·4 Å), but clearly larger than the value (1·986 Å) found in the compound [HP(OCH$_2$CH$_2$)$_3$N]BF$_4$ (Clardy, Milbrath, Springer & Verkade, 1976) which contains a P—N bond.

The O—P—C and O—P—Pd bond angles around the P atom show a strong distortion from the ideal tetrahedral configuration. This feature, previously observed in the rhodium complexes, thus appears to be characteristic of phosphonite complexes (Bondoux *et al.*, 1981).

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Bis(*O*-methyldithiocarbonato)mercury(II)

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Abstract. [Hg(S₂COCH₃)₂], $M_r = 414.9$, orthorhombic, *Pbca*, $a = 10.605$ (1), $b = 8.003$ (2), $c = 23.478$ (2) Å, $U = 1993$ (2) Å³, $D_x = 2.766$ Mg m⁻³, $Z = 8$, Mo $K\bar{\alpha}$ radiation, $\lambda = 0.7107$ Å, $\mu = 16.16$ mm⁻¹, $F(000) = 1520$, $T = 295$ (2) K, $R = 0.037$ for 1190 reflections with $I \geq 2.5\sigma(I)$. The immediate environment about the Hg atom is defined by two S atoms derived from two different xanthate ligands; Hg–S 2.365 (3) and 2.383 (3) Å, S–Hg–S 164.9 (1)°. One of the xanthate ligands also coordinates a neighbouring Hg atom, Hg–S 2.924 (3) Å, thereby generating a helical polymeric chain.

Introduction. The bis(xanthato) (⁻S₂CO*R*) complexes of the Zn triad elements are characterized by the formation of polymeric arrays and the presence of tetrahedrally coordinated metal centres. Thus far three structural prototypes have been discovered, all of which can be related to a 16-membered ring of four metal centres bridged by four xanthate ligands [see Watanabe (1981) for schematic representations of these structures]. An isolated tetrameric ring has been observed in Zn(S₂CO*ⁱ*C₃H₇)₂ (Ito, 1972) in which the two remaining tetrahedral sites on each Zn atom are occupied by a chelating xanthate ligand. In the structures of Zn(S₂COC₂H₅)₂ (Ikeda & Hagihara, 1966), Cd(S₂COC₂H₅)₂ (Iimura, Ito & Hagihara, 1972), Cd(S₂CO*ⁿ*C₄H₉)₂ (Rietveld & Maslen, 1965), and both polymorphs of Hg(S₂COC₂H₅)₂ (Watanabe, 1977; Chieh & Moynihan, 1980) the tetrameric rings are linked *via* bridging xanthate ligands which results in the formation of infinite two-dimensional arrays; as a consequence each central atom is coordinated by four bridging xanthate ligands. A third structural type was

found for Hg(S₂CO*ⁱ*C₃H₇)₂ (Watanabe, 1981) in which there are two crystallographically unique Hg atoms; the coordination geometry of two of the Hg atoms constituting the 16-membered ring is completed by a chelating xanthate ligand [as for Zn(S₂CO*ⁱ*C₃H₇)₂ above] while the remaining two Hg atoms link neighbouring tetrameric units *via* bridging xanthate ligands as for example in Zn(S₂COC₂H₅)₂ (Ikeda & Hagihara, 1966) so that the two-dimensional arrays of the tetramer are connected *via* helical chains. A recent X-ray study of the methylxanthates of the Group 15 elements (Snow & Tiekink, 1986) has shown that the polymeric structure found for Bi(S₂CO*ⁱ*C₃H₇)₃ (Hoskins, Tiekink & Winter, 1985) no longer persists in the Bi(S₂COCH₃)₃ analogue. These studies have now been extended to include Hg(S₂COCH₃)₂ to examine what effect the presence of the ⁻S₂COCH₃ anion has on the polymeric structures adopted by the closely related derivatives described above.

Experimental. Hg(S₂COCH₃)₂ prepared as in literature (Chieh & Moynihan, 1980). Colourless needle 0.09 × 0.88 × 0.03 mm grown from acetone. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω:2θ scan technique. Cell parameters from least-squares procedure (De Boer & Duisenberg, 1984) on 25 reflections (10 ≤ θ ≤ 15°). Analytical absorption correction: max./min. transmission factors 0.5385, 0.0902 (Sheldrick, 1976). Total of 3322 reflections (1 ≤ θ ≤ 25°) measured in the range −12 ≤ h ≤ 1, −9 ≤ k ≤ 1, −27 ≤ l ≤ 6. No significant variation in the intensities of three standards (33$\bar{6}$, 14$\bar{6}$, 2,3,$\overline{12}$) monitored every 3600 s. 1742 unique reflections (R_{int}