

**Complexes of Uranyl Nitrate and Dialkyldiamides: Structures of  
Dinitratodioxo[*N,N,N',N'*-tetrabutyl-1,2-phenylenebis(oxyacetamide)-*O,O'*]uranium(VI)  
and *catena*-Poly{[dinitratodioxouranium(VI)]- $\mu$ -(*N,N,N',N'*-tetrabutylglutaramide-*O:O'*)}**

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**Abstract.** (1)  $[\text{U}(\text{C}_{26}\text{H}_{44}\text{N}_2\text{O}_4)(\text{NO}_3)_2\text{O}_2]$ ,  $M_r = 843$ , monoclinic,  $P2_1/c$ ,  $a = 17.280$  (3),  $b = 10.024$  (4),  $c = 21.500$  (4) Å,  $\beta = 110.41$  (1)°,  $V = 3490$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.60$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 4.465$  mm<sup>-1</sup>,  $F(000) = 1664$ ,  $T = 295$  K,  $R = 0.037$  for 2396 unique reflections. (2)  $[\text{U}(\text{C}_{21}\text{H}_{42}\text{N}_2\text{O}_2)(\text{NO}_3)_2\text{O}_2]$ ,  $M_r = 748$ , monoclinic,  $C2/c$ ,  $a = 16.247$  (5),  $b = 12.759$  (2),  $c = 16.526$  (8) Å,  $\beta = 118.40$  (5)°,  $V = 3013$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.659$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 5.157$  mm<sup>-1</sup>,  $F(000) = 1472$ ,  $T = 295$  K,  $R = 0.032$  for 1602 unique reflections. In both complexes of formulae  $\text{U}(\text{O})_2(\text{NO}_3)_2L$  ( $L = \text{diamide}$ ), the linear uranyl group is equatorially surrounded by six O atoms from two bidentate nitrates and one bidentate diamide. However, the structure of (1) is of molecular type while the structure of (2) is of linear polymeric type, two  $\text{UO}_2(\text{NO}_3)_2(L^{1/2})_2$  units sharing one diamide. All U–O distances are very similar: the uranyl bond lengths are 1.72 (2) and 1.749 (7) Å, the O atoms of the nitrates are 2.52 (2) and 2.518 (8) Å from the U atom and those of the amides either bidentate at 2.52 (2) Å or bridging at 2.518 (8) Å, respectively, in (1) and (2).

**Introduction.** Substituted amides are effective in extracting metallic ions from aqueous solutions (Fritz & Orf, 1975; Pohlandt & Fritz, 1979), particularly actinides (Siddall, 1960; Hubert, 1985). In the extraction process of  $\text{U}^{\text{VI}}$ , the aqueous solvation sphere of  $\text{UO}_2^{2+}$  is replaced by amide ligands to form a complex. In the case of diamides, the coordination to the metallic ion is not fully understood, the diamide acting either as a monodentate or as a bidentate ligand. We have studied two complexes of formulae  $\text{UO}_2(\text{NO}_3)_2(\text{bboa})$  and  $\text{UO}_2(\text{NO}_3)_2(\text{tbga})$  in which  $\text{bboa} \equiv N,N,N',N'$ -tetrabutyl-1,2-phenylenebis(oxyacetamide)  $\{\text{C}_6\text{H}_4[\text{OCH}_2\text{CON}(\text{C}_4\text{H}_9)_2]_2\}$  and  $\text{tbga} \equiv N,N,N',N'$ -tetrabutylglutaramide  $\{(\text{CH}_2)_3[\text{CON}(\text{C}_4\text{H}_9)_2]_2\}$ .

**Experimental.** Compounds obtained from slow precipitation of a *bboa* (1) or *tbga* (2) toluene solution

when saturated by  $\text{UO}_2(\text{NO}_3)_2$ ; saturation achieved by extraction of  $\text{UO}_2(\text{NO}_3)_2$  from an aqueous solution of  $\text{LiNO}_3$ ; suitable crystals available from the precipitate, separated, then washed with toluene and vacuum dried; chemical analysis suggested the formula  $\text{UO}_2(\text{NO}_3)_2L$ ; IR spectroscopy showed that all the C=O amide groups were bonded since a single C=O stretching band was observed at 1560 cm<sup>-1</sup>.

(1) Crystal  $0.25 \times 0.1 \times 0.05$  mm; Enraf–Nonius CAD-4 diffractometer; unit-cell parameters refined from 25 reflections in range  $16 < 2\theta < 24^\circ$ ; three standard reflections monitored every 100 reflections, loss of 4% in 31 h; 2725 reflections with  $4 < 2\theta < 50^\circ$  ( $0 < h < 15$ ,  $0 < k < 8$ ,  $-18 < l < 18$ ); 2396 with  $I > 3\sigma(I)$ ; Lorentz and polarization corrections; empirical absorption correction based on  $\psi$  scans (North, Phillips & Mathews, 1968), average transmission coefficient 0.957; structure solved by Patterson function and difference Fourier syntheses, refined by full-matrix least squares ( $F$ ); anisotropic thermal parameters for U; H atoms introduced at calculated positions ( $\text{C}–\text{H} = 0.95$  Å,  $B = 5$  Å<sup>2</sup>), not refined but constrained to ride on their C atoms; scattering factors including anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974);  $R = 0.037$ ,  $wR = 0.043$  ( $w = 1$ ); max. (shift/e.s.d.) = 0.3,  $S = 4.96$ , highest peak in final difference map =  $0.6 \text{ e } \text{Å}^{-3}$ ; program: *Enraf–Nonius Structure Determination Package, SDP-Plus* (Frenz, 1983); PDP 11/23 Plus.

(2) Crystal  $0.35 \times 0.25 \times 0.075$  mm; Enraf–Nonius CAD-4 diffractometer; unit-cell parameters from 25 reflections in range  $16 < 2\theta < 24^\circ$ ; no significant variations for three standard reflections; 2881 reflections with  $4 < 2\theta < 50^\circ$  ( $0 < h < 20$ ,  $0 < k < 16$ ,  $-20 < l < 20$ ); 1604 reflections with  $I > 3\sigma(I)$ ; Lorentz and polarization corrections; empirical absorption correction using *DIFABS* (Walker & Stuart, 1983), transmission coefficients min. 0.668 and max. 1.333; structure solved by the heavy-atom method and refined by full-matrix least squares ( $F$ ); anisotropic thermal parameters for U and O (nitrate groups); H atoms

introduced at calculated positions ( $C-H = 0.95 \text{ \AA}$ ,  $B = 5 \text{ \AA}^2$ ) not refined but constrained to ride on their C atoms; scattering factors including anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974);  $R = 0.032$ ;  $wR = 0.042$  ( $w = 1$ ); max. (shift/e.s.d.) =  $0.13$ ;  $S = 1.76$ ; largest peak in final difference map:  $0.8 e \text{ \AA}^{-3}$ ; program *SDP-Plus* (Frenz, 1983), PDP 11/23 Plus.

**Discussion.** Atomic coordinates for (1) are given in Table 1 and those for (2) in Table 2,\* while associated bond lengths and angles for the two structures are presented in Tables 3 and 4 respectively. The numbering scheme of (1) is given in Fig. 1 and that of (2) in Fig. 2. The packings of (1) and (2) are shown in Figs. 3 and 4, respectively.

\* Lists of structure factors and anisotropic thermal parameters for (1) and (2) and H-atom parameters and least-squares-planes data for (1) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43452 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates with e.s.d.'s and equivalent isotropic thermal parameters (complex 1)

	x	y	z	$B(\text{\AA}^2)$
U	0.23252 (7)	0.2182 (1)	0.19627 (5)	4.26 (2)*
O(1)	0.161 (1)	0.015 (2)	0.1952 (7)	4.9 (4)
O(2)	0.218 (1)	0.188 (2)	0.1133 (8)	6.0 (5)
O(3)	0.2456 (8)	0.251 (2)	0.2765 (7)	4.7 (4)
O(4)	0.237 (1)	-0.028 (2)	0.3373 (8)	5.7 (5)
O(5)	0.0801 (9)	0.244 (2)	0.1760 (7)	6.3 (5)
O(6)	0.139 (1)	0.421 (2)	0.1607 (8)	6.4 (5)
O(7)	0.011 (1)	0.430 (2)	0.152 (1)	8.5 (6)
O(8)	0.294 (1)	0.440 (2)	0.1877 (8)	5.6 (5)
O(9)	0.380 (1)	0.278 (2)	0.2155 (8)	7.0 (5)
O(10)	0.427 (1)	0.472 (2)	0.2083 (9)	7.8 (6)
O(11)	0.403 (1)	-0.028 (2)	0.3997 (8)	5.2 (4)
O(14)	0.329 (1)	0.040 (2)	0.2326 (8)	5.1 (4)
N(1)	0.072 (1)	0.364 (2)	0.157 (1)	7.2 (7)
N(2)	0.367 (1)	0.403 (2)	0.202 (1)	7.2 (7)
N(3)	0.108 (1)	-0.184 (2)	0.2104 (9)	5.4 (5)
N(4)	0.383 (1)	-0.155 (2)	0.2684 (9)	4.6 (5)
C(2)	0.142 (2)	-0.065 (3)	0.236 (1)	5.2 (7)
C(3)	0.150 (2)	-0.019 (3)	0.303 (1)	6.3 (8)
C(5)	0.265 (1)	0.030 (3)	0.399 (1)	4.8 (7)
C(6)	0.217 (2)	0.080 (3)	0.433 (1)	7.6 (9)
C(7)	0.259 (2)	0.138 (3)	0.495 (2)	9 (1)
C(8)	0.338 (2)	0.146 (3)	0.520 (1)	8.2 (9)
C(9)	0.388 (2)	0.090 (3)	0.489 (1)	7.7 (9)
C(10)	0.350 (2)	0.036 (3)	0.427 (1)	6.3 (8)
C(12)	0.413 (2)	0.040 (3)	0.345 (1)	4.8 (7)
C(13)	0.371 (2)	-0.030 (3)	0.281 (1)	4.6 (6)
C(31)	0.081 (2)	-0.281 (3)	0.252 (1)	6.7 (7)
C(32)	0.148 (2)	-0.363 (3)	0.298 (1)	7.2 (8)
C(33)	0.118 (2)	-0.462 (4)	0.343 (2)	12 (1)
C(34)	0.181 (3)	-0.526 (5)	0.391 (2)	14 (1)
C(35)	0.101 (2)	-0.221 (3)	0.143 (1)	7.6 (8)
C(36)	0.020 (2)	-0.201 (4)	0.094 (2)	11 (1)
C(37)	0.010 (3)	-0.254 (6)	0.022 (2)	18 (2)
C(38)	0.058 (4)	-0.193 (7)	-0.001 (3)	24 (3)
C(41)	0.437 (1)	-0.241 (3)	0.318 (1)	5.3 (7)
C(42)	0.394 (2)	-0.342 (3)	0.347 (1)	6.4 (8)
C(43)	0.453 (2)	-0.442 (4)	0.393 (2)	9 (1)
C(44)	0.413 (2)	-0.560 (4)	0.415 (2)	11 (1)
C(45)	0.340 (1)	-0.207 (3)	0.2028 (9)	5.4 (5)
C(46)	0.387 (2)	-0.197 (3)	0.155 (1)	7.6 (8)
C(47)	0.332 (2)	-0.244 (4)	0.083 (2)	12 (1)
C(48)	0.366 (3)	-0.229 (6)	0.036 (3)	19 (2)

$$* B_{eq} = \frac{4}{3} \sum_i \sum_j a_i a_j \beta_{ij}$$

Table 2. Atomic coordinates with e.s.d.'s and equivalent isotropic thermal parameters (complex 2)

	x	y	z	$B(\text{\AA}^2)$
U	0.250	0.250	0.000	2.766 (8)*
O(1)	0.2187 (4)	0.2571 (7)	-0.1559 (3)	3.3 (1)
O(2)	0.2849 (6)	0.0814 (6)	-0.0567 (5)	5.6 (2)*
O(3)	0.3409 (5)	0.0919 (6)	0.0888 (4)	4.5 (2)*
O(4)	0.3582 (7)	-0.0523 (7)	0.0292 (6)	8.7 (3)*
O(1-1)	0.1412 (5)	0.1962 (6)	-0.0260 (4)	3.8 (1)
N(1)	0.1601 (5)	0.2760 (6)	-0.3082 (5)	3.3 (2)
N(2)	0.3280 (7)	0.0359 (8)	0.0188 (6)	5.0 (2)
C(1)	0.1570 (5)	0.2319 (7)	-0.2364 (5)	2.9 (2)
C(2)	0.0782 (6)	0.1586 (8)	-0.2495 (6)	2.9 (2)
C(3)	0.000	0.2259 (9)	-0.250	2.7 (3)
C(10)	0.2320 (8)	0.357 (1)	-0.2930 (7)	4.4 (2)
C(11)	0.2128 (8)	0.459 (1)	-0.2603 (8)	5.1 (3)
C(12)	0.1140 (9)	0.500 (1)	-0.3137 (9)	6.1 (3)
C(13)	0.100 (1)	0.609 (2)	-0.282 (1)	9.0 (5)
C(14)	0.0924 (6)	0.257 (1)	-0.4048 (6)	4.5 (2)
C(15)	0.131 (1)	0.174 (1)	-0.4445 (9)	6.3 (3)
C(16)	0.054 (1)	0.158 (2)	-0.551 (1)	10.9 (6)
C(17)	0.075 (2)	0.094 (2)	-0.600 (2)	16 (1)

$$* B_{eq} = \frac{4}{3} \sum_i \sum_j a_i a_j \beta_{ij}$$

Table 3. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with e.s.d.'s in parentheses (complex 1)

(a) Uranium environment			
U-O(2)	1.74 (2)	O(2)-U-O(3)	178.8 (7)
U-O(3)	1.69 (1)	O(1)-U-O(14)	70.3 (6)
U-O(1)	2.38 (2)	O(14)-U-O(9)	64.6 (6)
U-O(14)	2.39 (2)	O(9)-U-O(8)	50.4 (6)
U-O(5)	2.53 (2)	O(8)-U-O(5)	109.9 (6)
U-O(6)	2.55 (2)	O(5)-U-O(1)	65.2 (6)
U-O(8)	2.50 (2)		
U-O(9)	2.50 (2)		
(b) Nitrates			
N(1)-O(5)	1.26 (3)	O(5)-N(1)-O(6)	115 (2)
N(1)-O(6)	1.27 (3)	O(6)-N(1)-O(7)	120 (2)
N(1)-O(7)	1.21 (3)	O(7)-N(1)-O(5)	123 (3)
N(2)-O(8)	1.24 (3)	O(8)-N(2)-O(9)	115 (2)
N(2)-O(9)	1.28 (3)	O(9)-N(2)-O(10)	118 (2)
N(2)-O(10)	1.22 (3)	O(10)-N(2)-O(8)	127 (2)
(c) Amide			
<N-C butyl>	1.45 (4)	<C-N(4)-C>	120 (2)
<N-C amide>	1.33 (3)	<C-N(3)-C>	120 (2)
<C-O amide>	1.26 (4)		
<C-O chain>	1.43 (3)		
<C-C>benzene	1.36 (4)		
<C-C>chain	1.49 (6)		

Table 4. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with e.s.d.'s in parentheses (complex 2)

(a) Uranium environment			
U-O(1-1)	1.749 (7)	O(1)-O(2)	2.68 (1)
U-O(1)	2.378 (6)	O(2)-O(3)	2.13 (1)
U-O(2)	2.517 (9)	O(3)-O(1')	2.63 (1)
U-O(3)	2.519 (7)	O(1)-O(2)-O(3)	117.9 (4)
		O(2)-O(3)-O(1')	118.5 (4)
		O(3')-O(1)-O(2)	118.5 (3)
(b) Nitrate			
N(2)-O(2)	1.25 (1)	O(2)-N(2)-O(3)	114 (1)
N(2)-O(3)	1.29 (1)	O(2)-N(2)-O(4)	125 (1)
N(2)-O(4)	1.21 (1)	O(3)-N(2)-O(4)	120 (1)
O(2)-O(4)	2.18 (1)		
O(3)-O(4)	2.17 (1)		
(c) Amide			
O(1)-C(1)	1.269 (8)	C(10)-C(11)	1.50 (2)
C(1)-N(1)	1.33 (1)	C(11)-C(12)	1.51 (2)
N(1)-C(10)	1.49 (1)	C(12)-C(13)	1.54 (3)
N(1)-C(14)	1.46 (1)	C(14)-C(15)	1.53 (2)
C(1)-C(2)	1.52 (1)	C(15)-C(16)	1.61 (2)
C(2)-C(3)	1.53 (1)	C(16)-C(17)	1.60 (4)
O(1)-C(1)-N(1)	118.7 (8)	C(1)-N(1)-C(10)	120.0 (7)
O(1)-C(1)-C(2)	119.9 (8)	C(10)-N(1)-C(14)	125.0 (8)
C(2)-C(1)-N(1)	121.3 (6)	C(1)-N(1)-C(14)	114.8 (9)

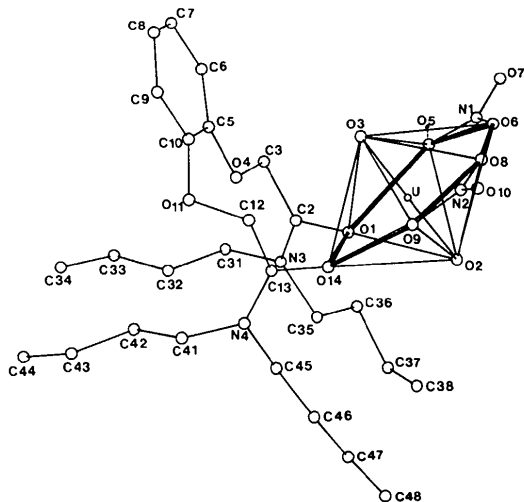


Fig. 1. View of the molecule [UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>(bboa)] showing the atomic numbering (ORTEP; Johnson, 1976).

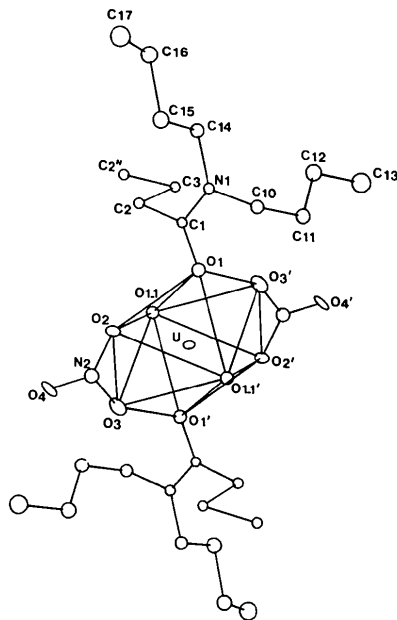


Fig. 2. View of the molecule [UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>(tbga)] showing the atomic numbering (ORTEP; Johnson, 1976). (Primed atoms are related by a center of symmetry and double-primed atoms by a twofold axis to unprimed atoms.)

In both complexes, uranium is eight-coordinated at the center of a distorted hexagonal bipyramid involving four O atoms of two bidentate nitrates and two atoms of the diamide around the uranyl group. The bond lengths U—O in UO<sub>2</sub><sup>2+</sup> are normal [U—O 1.72 (2) for (1) and 1.749 (7) Å for (2)] as are the equatorial U—O ligand distances [U—O<sub>NO<sub>3</sub></sub> 2.52 (2) for (1) and 2.518 (8) Å for (2); U—O diamide 2.38 (2) Å for (1)

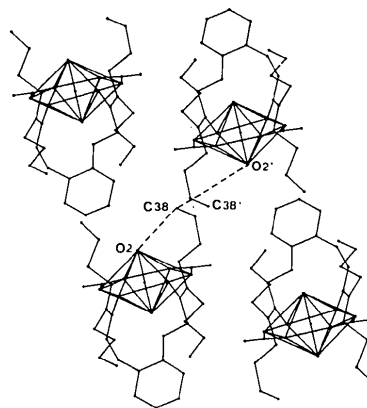


Fig. 3. Packing of [UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>(bboa)] (ORTEP; Johnson, 1976) viewed down the [010] axis. (Primed atoms are related to unprimed by a center of symmetry.)

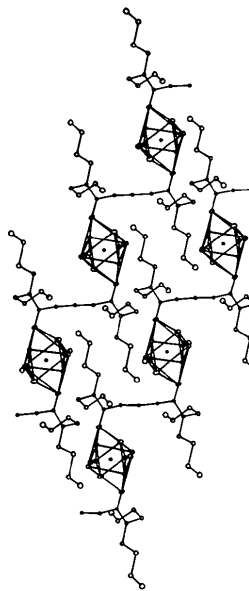


Fig. 4. Packing of [UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>(tbga)] (ORTEP; Johnson, 1976) viewed down the [010] axis.

and 2.378 (6) Å for (2)]. However, the distributions of the ligands in the equatorial plane are not the same in the two compounds, leading to very different structures.

**Complex (1).** [UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub>(bboa)]. The structure is of a molecular type. The diamide ligand is bidentate with its two O atoms adjacent in the equatorial plane; the nitrate groups which complete the plane are, as usual, bidentate and then adjacent. The six O atoms form a nearly planar slightly puckered hexagon around the U atom: the deviations of the atoms from this plane are between 0.14 (1) and -0.15 (1) Å. The linear UO<sub>2</sub><sup>2+</sup> is

perpendicular to this plane. One of the two planar nitrate groups is slightly tilted with respect to the equatorial plane ( $8^\circ$ ). 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  $76^\circ$ .

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.**  $[\text{UO}_2(\text{NO}_3)_2(\text{tbga})]$ . The structure is formed by the  $[\text{UO}_2(\text{NO}_3)_2(\text{tbga}^{1/2})_2]$  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  $\pm 0.17(1)$  Å. The linear uranyl group is normal to this best equatorial plane. The nitrate group is perfectly planar and tilted  $10^\circ$  with respect to the equatorial plane.

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## Structure of Chloro[1–3- $\eta$ -(2-methylallyl)](perhydro-6-methyl-2-phenyl-1,3,6,2-dioxaphosphocine)palladium

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**Abstract.**  $[\text{PdCl}(\text{C}_4\text{H}_7)(\text{C}_{11}\text{H}_{16}\text{NO}_2\text{P})]$ ,  $M_r = 422.2$ , monoclinic,  $P2_1$ ,  $a = 8.631(1)$ ,  $b = 11.756(2)$ ,  $c = 8.824(1)$  Å,  $\beta = 103.21(1)^\circ$ ,  $V = 871.6(3)$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.61$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.7107$  Å,  $\mu = 1.29$  mm<sup>-1</sup>,  $F(000) = 428$ ,  $T = 295$  K,  $R = 0.027$  for 3437 unique observed reflections. The Pd<sup>II</sup> 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  $\text{C}_6\text{H}_5\text{P}(\text{OC}_2\text{H}_4)_2\text{NCH}_3$  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