

A coordination number greater than six is commonly observed for the K ion in crown-ether complexes. Thus both K ions in this molecule are seven coordinated. In addition to the six O atoms from the macrocycle, the seventh coordinating atom is the N atom of a cyanide group in one case and the O atom of a water molecule in the other. The K(1)···N(3) and K(2)···O(71) distances are 2.759 (8) and 2.672 (7) Å respectively.

In both the [K-DB18C6]⁺ ions the K is nearly coplanar with the six O atoms of the crown ether. All the O···K···O angles are close to 60°, ranging from 56.1 (2) to 62.3 (1)° in one case and from 56.4 (2) to 62.0 (2)° in the other. K(1) is 0.28 (1) Å from the O₆ plane while K(2) is -0.38 (1) Å. K···O distances vary from 2.709 (3) to 2.766 (7) Å for an average of 2.741 (6) Å for K(1) while the corresponding values are 2.691 (7), 2.835 (5) and 2.759 (6) Å respectively for K(2). Both the range and the average of the K···O distances are in agreement with values reported elsewhere (Hilgenfeld & Saenger, 1981; Poonia & Bajaj, 1979; Rogers & Atwood, 1984; Wink *et al.*, 1985).

Within the polyether macrocycle the aliphatic C—C distances average 1.484 (14) Å for the K(1) complex ion and 1.487 (11) Å for K(2) with a spread of less than 0.02 Å. These values are slightly shorter than that expected for single C—C bonds but are within the range usually observed in crown-ether complexes (Bush & Truter, 1971). Bond distances and angles within the benzene rings are normal. Average O—C(aliphatic) distances are 1.416 (9) and 1.423 (11) Å for the two macrocycles while average O—C(aromatic) distances are 1.386 (11) and 1.383 (10) Å respectively, again in good agreement with reported values.

The second water molecule was located from the difference Fourier map after all the other non-H atoms were refined anisotropically. It is not bonded to the K atoms.

References

- ALDOSHI, S. M., D'YACHENKO, O. A., TKACHEV, V. V. & ATOVMYEN, L. O. (1981). *Koord. Chim.* **7**, 287–296.
- ATWOOD, J. L., HUNTER, W. E., ROGERS, R. D. & WEEKS, J. A. (1985). *J. Inclusion Phenom.* **3**, 113–123.
- BRODERSEN, K., BECK, I., BECK, R., HUMMEL, H. U. & LIEHR, G. (1984). *Z. Anorg. Allg. Chem.* **516**, 30–34.
- BUSH, M. A. & TRUTER, M. R. (1971). *J. Chem. Soc. B*, pp. 1440–1446.
- FAN, Y. P., ZHANG, Z. Y., WANG, X. M., ZHANG, J. Z. & HAN, J. F. (1985). *Kexue Tongbao* (foreign language edition), **30**, 910–915.
- HILGENFELD, R. & SAENGER, W. (1981). *Angew. Chem. Int. Ed. Engl.* **20**, 1045–1046.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- IZATT, R. M., EATOUGH, D. J. & CHRISTENSEN, J. J. (1973). *Thermodynamics of Cation-Macrocyclic Compound Interaction. In Structure and Bonding*, No. 16, New York: Springer-Verlag.
- KLUEFERS, P., FUESS, H. & HAUSUEHL, S. (1981). *Z. Kristallogr.* **156**, 255–263.
- POONIA, N. S. & BAJAJ, A. V. (1979). *Chem. Rev.* **79**, 289–445.
- ROGERS, R. D. & ATWOOD, J. L. (1984). *J. Cryst. Spectrosc. Res.* **14**, 1–11.
- SHELDRICK, G. M. (1984). *SHELXTL*. Revision 4.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.
- SHELDRICK, G. M. (1985). *SHELXTL*. Revision 5.1 (Eclipse 32K Version). Nicolet XRD Corporation, Madison, Wisconsin, USA.
- WINK, D. J., FOX, J. R. & COOPER, N. J. (1985). *J. Am. Chem. Soc.* **107**, 5012–5014.
- ZAWOROTKO, M. J., REID, C. & ATWOOD, J. L. (1985). *Organometallics*, **4**, 238–241.

Acta Cryst. (1987). **C43**, 2096–2099

Two Crystalline Phases of Chlorotris(2-pyridyl)phosphinegold(I)

BY C. J. L. LOCK AND M. A. TURNER

Laboratories for Inorganic Medicine, Departments of Chemistry and Pathology, McMaster University, SSC-426, Hamilton, Ontario, Canada L8S 4M1

(Received 5 May 1987; accepted 30 June 1987)

Abstract. The title compound, [AuCl(C₅H₄N)₃P], *M_r* = 497.68, crystallizes in two different crystalline phases *A* and *B*. *A* is triclinic, *P* $\bar{1}$, *a* = 13.050 (1), *b* = 14.276 (2), *c* = 11.263 (2) Å, α = 115.86 (1), β = 92.10 (1), γ = 118.80 (1)°, *V* = 1571.5 Å³, *Z* = 4, *D_m* = 2.13 (2), *D_x* = 2.10 g cm⁻³, graphite-monochromated Mo *K* α radiation, λ = 0.71069 Å, μ =

99.4 cm⁻¹, *F*(000) = 966.8, *T* = 298 K, *R* = 0.0584 for 3861 unique reflections and 379 parameters. *B* is triclinic, *P* $\bar{1}$, *a* = 8.611 (2), *b* = 9.118 (2), *c* = 11.186 (3) Å, α = 94.86 (2), β = 112.22 (2), γ = 94.96 (2)°, *V* = 803.4 Å³, *Z* = 2, *D_m* = 2.13 (2), *D_x* = 2.06 g cm⁻³, graphite-monochromated Mo *K* α radiation, λ = 0.71069 Å, μ = 97.4 cm⁻¹, *F*(000) = 483.4,

$T = 298$ K, $R = 0.0399$ for 3486 unique reflections and 190 parameters. The molecular structures in *A* and *B* are very similar. The P—Au—Cl unit is essentially linear and Au—Cl distances [2.277 (5), 2.272 (5), 2.274 (1) Å] are normal. The Au—P distances [2.214 (4), 2.218 (4), 2.220 (1) Å] are shorter than in most other gold—phosphine complexes.

Introduction. The complex was obtained by the reaction of tris(2-pyridyl)phosphine with chloroauric acid in aqueous ethanol (1:2) with the use of thiodiglycol as a reducing agent. Crystals of *A* were obtained by slow crystallization from aqueous ethanol (1:2) and crystals of *B* were obtained by accident from a mixture of the title compound and cobalt(II) nitrate in aqueous ethanol (1:2) which failed to react.

Experimental. Densities of crystals obtained by suspension in an aqueous zinc bromide solution. Crystals $0.097 \times 0.210 \times 0.258$ mm, *A*, $0.129 \times 0.226 \times 0.613$ mm, *B*. Space groups both $P\bar{1}$, chosen and justified by successful solution. Delaunay reduction revealed no hidden symmetry. Unit-cell parameters refined by least-squares fit of positional angles of 15 strong independent reflections measured on Syntex $P2_1$, (*A*) and Nicolet $P3$ (*B*) diffractometers at 298 K, monochromated $Mo K\alpha$ radiation. 4349 reflections measured for $2\theta < 45^\circ$, *A*, 3889 reflections measured for $2\theta < 55^\circ$, *B*. Intensities of h , $\pm k$, $\pm l$ measured by the θ - 2θ scan technique. Scan rate 5.0 to $29.3^\circ \text{ min}^{-1}$ in 2θ . The ratio of total background time to scan time is 1:1. Two standard reflections monitored every 48 scans showed that no correction for instrument instability or crystal decay was required. Data averaged to give 3861 reflections and $R_{\text{int}} = 0.026$, *A*, and 3486 reflections and $R_{\text{int}} = 0.009$, *B*. Reflections with $3\sigma_I > I > -3\sigma_I$ were treated by the method of French & Wilson (1978). L_p and absorption corrections were made (absorption correction factors 2.20–7.73, *A*, 3.19–11.2, *B*). Structures solved by heavy-atom method. Anisotropic least-squares refinement minimizing $\sum w(|F_o| - |F_c|)^2$, $w = (\sigma_F^2 + 0.001493F_o^2)^{-1}$, *A*, $w = (\sigma_F^2 + 0.000528F_o^2)^{-1}$, *B*. Final refinement for *A* blocks, each molecule refined alternately. H atoms not located. For *B* H atoms were located and included but not refined. Final $R = 0.0584$, $wR = 0.0645$, *A*, $R = 0.0399$, $wR = 0.0436$, *B*. Secondary-extinction correction not applied. In final refinement cycles $(\Delta/\sigma)_{\text{max}} = 0.036$, *A*, 0.022, *B*. Final difference maps revealed no significant regions of electron density with max. 1.0, min. $-0.84 \text{ e } \text{Å}^{-3}$, *A*, 1.0, $-0.87 \text{ e } \text{Å}^{-3}$, *B*. Scattering factors for atoms from Cromer & Waber (1974). Corrections for anomalous dispersion were made for Au, P and Cl (Cromer & Ibers, 1974). Calculations employed *XTAL* (Stewart & Hall, 1983), *SHELX76* (Sheldrick, 1976) and *SNOOPI* (Davies, 1983) program systems. Atomic

positional parameters and U_{eq} for non-hydrogen atoms in *A* and *B* are given in Tables 1 and 2, respectively.*

Discussion. Molecule 1 from *A* is shown in Fig. 1 (the other molecules are very similar) and selected bond lengths and angles are given in Table 3. The same numbering scheme is maintained in all three molecules, except that atoms in the second molecule of *A* have a prime affix. The general geometry of the molecule is very like that observed for chlorotriphenylphosphine-gold(I), *C* (Baenziger, Bennett & Soboroff, 1976), and chloro(2-pyridyl)diphenylphosphinegold(I), *D* (Alcock, Moore & Lampe, 1982). The P—Au—Cl unit is essentially linear in all three molecules. The Au—Cl bond distances are similar to those in *C* and *D*, but are shorter than the bridging Au—Cl distances in $[(C_6H_5)_3PAu]_2Cl^+$ [2.328 (5), 2.340 (5) Å (Muir, Muir & Lorca, 1980)] and marginally shorter than the terminal Au—Cl distances in $Cl-Au[(C_6H_5)_2P(CH_2)_2P(C_6H_5)_2]Au-Cl$ [2.299 (5), 2.289 (5) Å (Jones, 1980*a*)]. The distances and angles within the phosphine do not differ from corresponding values in $(C_6H_5)_2(C_5H_4N)P$ complexes of silver and gold (Baenziger *et al.*, 1976). The Au—P distances are short. They lie below Au—P values found for gold—phosphine clusters [range 2.267 (2)–2.42 (1) Å] (Demartin, Manassero, Naldini, Ruggeri & Sansoni, 1981; Van der Velden, Bour, Vollenbroek, Beurskens & Smits, 1979; Van der Velden, Bour, Otterloo, Bosman & Noordik, 1981; Vollenbroek, Bosman, Bour, Noordik & Beurskens, 1979), two-coordinate Au^I complexes [range 2.226 (4)–2.389 (6) Å] (Jones, 1980*a*; Jones & Sheldrick, 1980*a,b*; Muir *et al.*, 1980; Nesmeyanov, Perevalova, Struchkov, Antipin, Grandberg & Dyadchenko, 1980; Riley & Davis, 1980; Schmidbaur, Mandl, Bassett, Blaschke & Zimmer-Gasser, 1981; Simon & Lauher, 1980; Smyslova, Perevalova, Dyadchenko, Grandberg, Slovokhotov & Struchkov, 1981; Solans, Miravittles, Arrieta, Germain & Declercq, 1982) and the long distances in three- and four-coordinate Au^I complexes [range 2.359 (6)–2.610 (9) Å] (Guggenberger, 1974; Jones, 1980*b,c*; Jones, Sheldrick, Fugner, Gotzfried & Beck, 1981).

The 2-pyridyl rings in the phosphines of *A* (first molecule) and *B* are arranged so that the geometry is markedly distorted from C_3 [dihedral angles, (Au—P—Cl) i —(Ring i), *A*, $i = 1$, 17 (11); 2, 13 (4); 3, 62.5 (6)°; *A*, $i = 1'$, 71.0 (6); 2', 53.4 (6); 3', 55.6 (7)°; *B*, $i = 1$, 60.0 (2); 2, 18.4 (2); 3, 18.1 (3)°]. This is

* Lists of structure factors, anisotropic temperature factors, H-atom positions, least-squares-planes data and analytical data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44204 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters and U_{eq} ($\times 10^3$) for A, with e.s.d.'s in parentheses* Table 2. *Positional parameters ($\times 10^4$) and U_{eq} ($\times 10^3$) for B, with e.s.d.'s in parentheses*

	x	y	z	$U_{eq}(\text{\AA}^2)$
Au	369.50 (5)	696.93 (5)	655.57 (5)	24.3
Cl	445.1 (4)	789.4 (4)	-469.9 (4)	39
P	296.1 (3)	605.7 (3)	-223.7 (3)	23
C(11)	376 (1)	708 (1)	-35 (1)	25
C(21)	436 (1)	838 (1)	23 (1)	34
C(31)	485 (1)	908 (1)	170 (2)	38
C(41)	482 (1)	853 (2)	243 (2)	37
C(51)	420 (2)	728 (2)	174 (2)	43
N(1)	370 (1)	657 (1)	38 (1)	31
C(12)	134 (1)	549 (1)	-238 (1)	23
C(22)	96 (1)	578 (1)	-122 (2)	37
C(32)	-31 (2)	533 (2)	-145 (2)	49
C(42)	-108 (1)	462 (2)	-283 (2)	45
C(52)	-59 (1)	435 (2)	-390 (2)	41
N(2)	65 (1)	480 (1)	-367 (1)	39
C(13)	304 (1)	470 (1)	-279 (1)	22
C(23)	208 (1)	356 (1)	-301 (1)	29
C(33)	224 (1)	257 (1)	-353 (2)	36
C(43)	331 (1)	275 (1)	-372 (2)	35
C(53)	425 (1)	392 (1)	-344 (2)	39
N(3)	411 (1)	489 (1)	-295 (1)	37
Au'	149.04 (5)	883.12 (5)	970.34 (5)	27.1
Cl'	222.2 (4)	837.3 (4)	-212.7 (4)	39
P'	79.3 (3)	937.8 (3)	148.6 (3)	22.6
C(11')	-91 (1)	842 (1)	98 (1)	25
C(21')	-163 (1)	888 (1)	108 (2)	34
C(31')	-293 (1)	801 (2)	56 (2)	43
C(41')	-336 (2)	676 (2)	7 (2)	42
C(51')	-252 (1)	638 (2)	1 (2)	42
N(1')	-130 (1)	724 (1)	46 (1)	37
C(12')	124 (1)	916 (1)	286 (1)	25
C(22')	210 (2)	885 (2)	283 (2)	37
C(32')	244 (2)	876 (2)	398 (2)	50
C(42')	186 (2)	895 (2)	497 (2)	41
C(52')	107 (1)	929 (1)	493 (1)	32
N(2')	73 (1)	940 (1)	387 (1)	33
C(13')	139 (1)	1107 (1)	237 (1)	28
C(23')	260 (2)	1184 (2)	251 (2)	48
C(33')	306 (2)	1314 (2)	319 (2)	67
C(43')	231 (2)	1353 (2)	360 (2)	48
C(53')	111 (2)	1262 (2)	343 (2)	51
N(3')	70 (1)	1142 (1)	276 (1)	44

	x	y	z	$U_{eq}(\text{\AA}^2)$
Au	1722.3 (3)	3185.4 (2)	1489.2 (2)	38.8
Cl	2609 (3)	4463 (2)	154 (2)	57.7
P	902 (2)	1915 (2)	2802 (1)	36.8
C(11)	1649 (7)	90 (6)	2952 (5)	38
C(21)	1173 (10)	-828 (8)	1835 (6)	58
C(31)	1667 (11)	-2217 (9)	1921 (9)	70
C(41)	2695 (10)	-2629 (8)	3102 (9)	66
C(51)	3170 (11)	-1595 (9)	4162 (8)	68
N(1)	2653 (9)	-230 (7)	4102 (6)	63
C(12)	-1381 (7)	1488 (6)	2269 (6)	41
C(22)	-2447 (9)	2273 (8)	1389 (8)	56
C(32)	-4162 (10)	1953 (10)	1037 (9)	68
C(42)	-4768 (10)	873 (11)	1575 (10)	72
C(52)	-3609 (11)	118 (11)	2446 (10)	81
N(2)	-1931 (8)	410 (7)	2803 (6)	64
C(13)	1669 (8)	2873 (6)	4453 (6)	39
C(23)	981 (12)	2491 (10)	5334 (8)	76
C(33)	1676 (12)	3246 (11)	6589 (8)	89
C(43)	3023 (11)	4350 (9)	6884 (7)	62
C(53)	3614 (10)	4626 (8)	5958 (7)	59
N(3)	2957 (7)	3922 (6)	4737 (5)	50

$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2\cos\alpha U_{23} + 2\cos\beta U_{13} + 2\cos\gamma U_{12}).$$

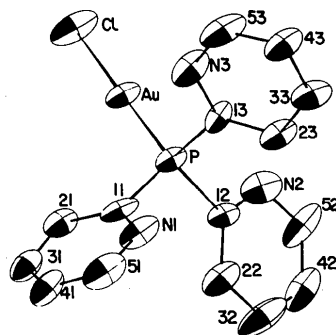


Fig. 1. The first molecule from A showing the atom numbering.

$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2\cos\alpha U_{23} + 2\cos\beta U_{13} + 2\cos\gamma U_{12}).$$

Table 3. *Selected interatomic distances (\AA) and angles ($^\circ$)*

	A	A'	B		A	A'	B
P-C(11)	1.84 (1)	1.86 (1)	1.834 (6)	C(42)-C(52)	1.38 (3)	1.35 (3)	1.38 (1)
C(11)-C(21)	1.42 (2)	1.36 (3)	1.344 (8)	C(52)-N(2)	1.39 (2)	1.36 (3)	1.34 (1)
C(21)-C(31)	1.42 (2)	1.42 (2)	1.37 (1)	N(2)-C(12)	1.30 (2)	1.34 (2)	1.333 (8)
C(31)-C(41)	1.37 (3)	1.40 (3)	1.39 (1)	Au-P	2.214 (4)	2.218 (4)	2.220 (1)
C(41)-C(51)	1.34 (3)	1.43 (4)	1.36 (1)	P-C(13)	1.82 (2)	1.86 (2)	1.821 (6)
C(51)-N(1)	1.32 (2)	1.35 (2)	1.36 (1)	C(13)-C(23)	1.39 (2)	1.37 (2)	1.381 (9)
N(1)-C(11)	1.30 (3)	1.32 (2)	1.324 (8)	C(23)-C(33)	1.40 (3)	1.42 (3)	1.39 (1)
Au-Cl	2.277 (5)	2.272 (5)	2.274 (1)	C(33)-C(43)	1.34 (3)	1.35 (4)	1.39 (1)
P-C(12)	1.84 (2)	1.84 (2)	1.820 (6)	C(43)-C(53)	1.39 (2)	1.41 (3)	1.35 (1)
C(12)-C(22)	1.39 (2)	1.38 (3)	1.370 (9)	C(53)-N(3)	1.36 (3)	1.32 (3)	1.343 (9)
C(22)-C(32)	1.42 (3)	1.43 (4)	1.37 (1)	N(3)-C(13)	1.31 (2)	1.23 (3)	1.321 (7)
C(32)-C(42)	1.40 (3)	1.37 (3)	1.36 (1)				
Au-P-C(11)	114.4 (5)	112.8 (4)	113.0 (2)				
P-C(11)-C(21)	118 (1)	124 (1)	115.8 (5)	Au-P-C(12)	114.0 (6)	115.0 (6)	114.4 (2)
C(11)-C(21)-C(31)	113 (2)	118 (2)	117.2 (6)	P-C(12)-C(22)	121 (1)	120 (1)	120.6 (5)
C(21)-C(31)-C(41)	122 (2)	117 (2)	121.4 (7)	C(12)-C(22)-C(32)	116 (1)	116 (2)	119.1 (7)
C(31)-C(41)-C(51)	117 (2)	121 (2)	116.7 (7)	C(22)-C(32)-C(42)	119 (2)	117 (2)	119.6 (8)
C(41)-C(51)-N(1)	125 (2)	120 (2)	122.8 (7)	C(32)-C(42)-C(52)	119 (2)	122 (2)	117.8 (7)
C(51)-N(1)-C(11)	119 (2)	119 (2)	117.8 (6)	C(42)-C(52)-N(2)	122 (2)	123 (2)	123.7 (8)
N(1)-C(11)-C(21)	124 (1)	126 (1)	124.1 (6)	C(52)-N(2)-C(12)	117 (2)	116 (2)	116.9 (7)
N(1)-C(11)-P	119 (1)	109 (1)	120.0 (4)	N(2)-C(12)-C(22)	127 (1)	126 (2)	122.9 (6)
P-C(13)-C(23)	124 (1)	115 (2)	122.2 (5)	N(2)-C(12)-P	112 (1)	114 (1)	116.5 (5)
C(13)-C(23)-C(33)	118 (2)	116 (2)	118.5 (7)	C(43)-C(53)-N(3)	122 (2)	121 (2)	124.3 (6)
C(23)-C(33)-C(43)	120 (2)	120 (2)	117.9 (7)	C(53)-N(3)-C(13)	119 (1)	121 (2)	116.7 (5)
C(33)-C(43)-C(53)	119 (2)	117 (2)	118.9 (6)	N(3)-C(13)-C(23)	123 (2)	125 (2)	123.6 (6)
Cl-Au-P	179.5 (1)	176.5 (2)	178.9 (1)	N(3)-C(13)-P	114 (1)	120 (1)	114.3 (4)

caused by the intermolecular interactions of the packing.

The packings of *A* and *B* are shown in Figs. 2 and 3, respectively. In *B* the molecular P–Au–Cl axis lies roughly along $[11\bar{1}]$ and because of the inversion centre all are parallel and dipolar interactions cancel. The small dihedral angle of ring 2, noted above, is a consequence of a π – π interaction between equivalent rings in molecules related by the inversion centre at $\frac{1}{2}, 0, 0$. Apart from this interaction the intermolecular ring–ring contacts are normal van der Waals interactions and are arranged primarily about the $z = \frac{1}{2}$ plane, whereas the Cl atoms lie close to the $z = 0$ plane.

The prime difference between the packing in *A* and *B* is that in *A* the P–Au–Cl axes are not all parallel and this is caused by the much greater extent of π – π ring–ring interactions. The second (primed) molecular axis is roughly parallel to *c* while the molecular axis of the first molecule lies roughly along $[11\bar{1}]$. There is a double stack of phenyl rings centered around the *b* axis. Along this line the sequence of pairs of rings is: rings 1 and 3 from *A'*; rings 3 and 1 from the centrosymmetrically (0,0,0)-related *A'*; rings 3 from *A* and its centrosymmetrically (0,0, $\frac{1}{2}$)-related pair; rings 1 and 3 from *A'*; etc. This packing arrangement explains why rings 1 and 3 in *A'* are close to coplanar [dihedral angle $23.0(9)^\circ$].

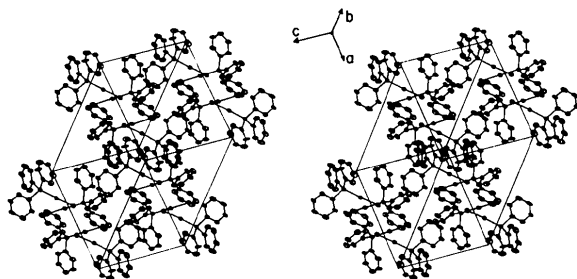


Fig. 2. The packing within the unit cell for *A*. The view is approximately down $[111]$.

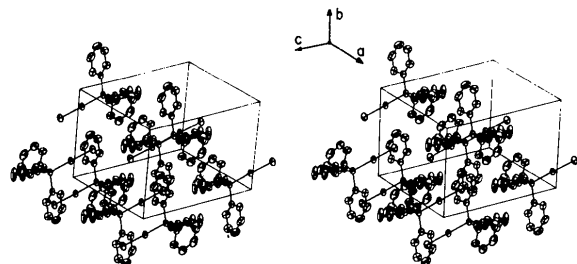


Fig. 3. The packing within the unit cell for *B*. The view is approximately down $[111]$.

We acknowledge with thanks financial assistance from the Natural Sciences and Engineering Research Council and help from Romolo Faggiani in acquiring the data.

References

- ALCOCK, N. W., MOORE, P. & LAMPE, P. A. (1982). *J. Chem. Soc. Dalton Trans.* pp. 207–210.
- BAENZIGER, N. C., BENNETT, W. E. & SOBOROFF, D. M. (1976). *Acta Cryst.* B32, 962–963.
- CROMER, D. T. & IBERS, J. A. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1, pp. 149–150. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B, pp. 99–100. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- DAVIES, K. (1983). CHEMGRAF suite, program SNOOPI. Chemical Design Ltd, Oxford, England.
- DEMARTIN, F., MANASSERO, H., NALDINI, L., RUGGERI, R. & SANSONI, M. (1981). *J. Chem. Soc. Chem. Commun.* pp. 222–223.
- FRENCH, S. & WILSON, K. (1978). *Acta Cryst.* A34, 517–525.
- GUGGENBERGER, L. J. (1974). *J. Organomet. Chem.* 81, 271–280.
- JONES, P. G. (1980a). *Acta Cryst.* B36, 2775–2776.
- JONES, P. G. (1980b). *Acta Cryst.* B36, 3105–3107.
- JONES, P. G. (1980c). *J. Chem. Soc. Chem. Commun.* pp. 1031–1033.
- JONES, P. G. & SHELDRIK, G. M. (1980a). *Acta Cryst.* B36, 1486–1488.
- JONES, P. G. & SHELDRIK, G. M. (1980b). *Acta Cryst.* B36, 2777–2779.
- JONES, P. G., SHELDRIK, G. M., FUGNER, A., GOTZFRIED, F. & BECK, W. (1981). *Chem. Ber.* 114, 1413–1417.
- MUIR, J. A., MUIR, M. M. & LORCA, E. (1980). *Acta Cryst.* B36, 931–933.
- NESMEYANOV, A. N., PEREVALOVA, E. G., STRUCHKOV, Y. T., ANTIPIN, M. Y., GRANDBERG, K. I. & DYADCHENKO, V. P. (1980). *J. Organomet. Chem.* 201, 343–349.
- RILEY, P. E. & DAVIS, R. E. (1980). *J. Organomet. Chem.* 192, 283–291.
- SCHMIDBAUR, H., MANDL, J. R., BASSETT, J.-M., BLASCHKE, G. & ZIMMER-GASSER, B. (1981). *Chem. Ber.* 114, 433–440.
- SHELDRIK, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.
- SIMON, F. E. & LAUHER, J. W. (1980). *Inorg. Chem.* 19, 2338–2343.
- SMYSLOVA, E. I., PEREVALOVA, E. G., DYADCHENKO, V. P., GRANDBERG, K. I., SLOVOKHOTOV, Y. L. & STRUCHKOV, Y. T. (1981). *J. Organomet. Chem.* 215, 269–279.
- SOLANS, X., MIRAVITLLES, C., ARRIETA, J. M., GERMAIN, G. & DECLERCQ, J.-P. (1982). *Acta Cryst.* B38, 1812–1815.
- STEWART, J. M. & HALL, S. R. (1983). The XTAL system of crystallographic programs. Tech. Rep. TR-1364. Univ. of Maryland, College Park, Maryland, USA.
- VAN DER VELDEN, J., BOUR, J. J., OTTERLOO, B. F., BOSMAN, W. P. & NOORDIK, J. H. (1981). *J. Chem. Soc. Chem. Commun.* pp. 583–584.
- VAN DER VELDEN, J., BOUR, J. J., VOLLENBROEK, F. A., BEURSKENS, P. T. & SMITS, J. M. M. (1979). *J. Chem. Soc. Chem. Commun.* pp. 1162–1163.
- VOLLENBROEK, F. A., BOSMAN, W. P., BOUR, J. J., NOORDIK, J. H. & BEURSKENS, P. T. (1979). *J. Chem. Soc. Chem. Commun.* pp. 387–388.