

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B _{eq}
Ru1	0.15105 (1)	0.25284 (1)	0.34868 (1)	2.549 (2)
P1	0.24983 (4)	0.20202 (3)	0.19457 (4)	2.718 (7)
P2	0.29715 (4)	0.27250 (3)	0.56057 (4)	2.746 (7)
N1	0.2157 (2)	0.4004 (1)	0.3879 (2)	3.69 (3)
N2	0.1603 (2)	0.4697 (1)	0.4077 (2)	3.39 (3)
N3	0.1133 (3)	0.5421 (2)	0.4299 (4)	6.09 (7)
C1	0.2727 (2)	0.0698 (1)	0.1254 (2)	3.21 (3)
C2	0.2369 (2)	0.0028 (2)	0.1703 (2)	3.84 (4)
C3	0.2603 (3)	-0.0964 (2)	0.1239 (3)	5.02 (6)
C4	0.3184 (3)	-0.1282 (2)	0.0326 (4)	5.39 (7)
C5	0.3527 (3)	-0.0619 (2)	-0.0140 (4)	5.44 (6)
C6	0.3295 (2)	0.0368 (2)	0.0310 (3)	4.66 (5)
C7	0.1511 (2)	0.2230 (2)	0.0416 (2)	3.21 (3)
C8	0.0867 (2)	0.1465 (2)	-0.0802 (2)	4.11 (4)
C9	0.0084 (3)	0.1669 (3)	-0.1911 (3)	5.21 (6)
C10	-0.0074 (3)	0.2629 (3)	-0.1820 (3)	5.38 (6)
C11	0.0554 (3)	0.3416 (2)	-0.0576 (3)	5.14 (5)
C12	0.1337 (2)	0.3218 (2)	0.0537 (3)	4.20 (4)
C13	0.3997 (2)	0.2567 (1)	0.2250 (2)	3.13 (3)
C14	0.4185 (2)	0.3108 (2)	0.1531 (3)	4.88 (5)
C15	0.5351 (3)	0.3467 (2)	0.1792 (4)	5.94 (6)
C16	0.6324 (2)	0.3279 (2)	0.2752 (3)	4.77 (6)
C17	0.6136 (2)	0.2713 (3)	0.3437 (3)	4.75 (6)
C18	0.4973 (2)	0.2354 (2)	0.3174 (2)	4.17 (4)
C19	0.2979 (2)	0.1576 (1)	0.5924 (2)	3.28 (3)
C20	0.2421 (3)	0.1430 (2)	0.6742 (3)	5.02 (5)
C21	0.2318 (5)	0.0484 (3)	0.6783 (4)	7.12 (8)
C22	0.2808 (5)	-0.0289 (2)	0.6070 (4)	7.64 (9)
C23	0.3368 (4)	-0.0154 (2)	0.5255 (4)	5.94 (7)
C24	0.3433 (2)	0.0768 (2)	0.5159 (2)	4.05 (4)
C25	0.2716 (2)	0.3682 (1)	0.7069 (2)	3.13 (3)
C26	0.3525 (2)	0.3890 (2)	0.8410 (2)	4.04 (4)
C27	0.3389 (3)	0.4668 (2)	0.9500 (2)	4.39 (5)
C28	0.2440 (3)	0.5241 (2)	0.9272 (2)	4.62 (5)
C29	0.1641 (3)	0.5036 (2)	0.7945 (3)	4.53 (5)
C30	0.1780 (2)	0.4266 (2)	0.6859 (2)	3.62 (4)
C31	0.4572 (2)	0.3126 (1)	0.6136 (2)	3.07 (3)
C32	0.4846 (2)	0.4003 (2)	0.6009 (2)	3.95 (4)
C33	0.6031 (2)	0.4393 (2)	0.6457 (3)	4.45 (5)
C34	0.6959 (2)	0.3891 (2)	0.7010 (3)	4.48 (5)
C35	0.6692 (2)	0.3031 (2)	0.7142 (3)	4.31 (5)
C36	0.5507 (2)	0.2642 (2)	0.6724 (2)	3.73 (4)
C37	-0.0327 (2)	0.2856 (2)	0.3437 (3)	4.32 (5)
C38	0.0072 (2)	0.2221 (2)	0.4164 (2)	4.51 (5)
C39	0.0307 (2)	0.1306 (2)	0.3255 (2)	4.15 (4)
C40	0.0016 (2)	0.1396 (2)	0.1960 (2)	3.86 (4)
C41	-0.0351 (2)	0.2354 (2)	0.2084 (3)	4.29 (5)

Table 2. Selected geometric parameters (Å, °)

Ru1—P1	2.3293 (5)	P1—C1	1.843 (2)
Ru1—P2	2.3304 (5)	P1—C7	1.842 (3)
Ru1—N1	2.135 (3)	P1—C13	1.848 (2)
Ru1—C37	2.222 (2)	P2—C19	1.833 (2)
Ru1—C38	2.230 (4)	P2—C25	1.845 (2)
Ru1—C39	2.202 (3)	P2—C31	1.835 (2)
Ru1—C40	2.190 (2)	N1—N2	1.186 (3)
Ru1—C41	2.187 (2)	N2—N3	1.164 (3)
P1—Ru1—P2	105.22 (2)	Ru1—P1—C13	125.22 (6)
P1—Ru1—N1	85.39 (6)	C1—P1—C7	103.31 (8)
P2—Ru1—N1	86.65 (5)	C1—P1—C13	98.0 (1)
N1—Ru1—C37	96.17 (9)	C7—P1—C13	103.2 (2)
N1—Ru1—C38	122.3 (1)	Ru1—P2—C19	110.65 (6)
N1—Ru1—C39	158.93 (9)	Ru1—P2—C25	113.69 (7)
N1—Ru1—C40	137.70 (9)	Ru1—P2—C31	124.66 (7)
N1—Ru1—C41	103.0 (1)	C19—P2—C25	104.7 (2)
Ru1—P1—C1	119.01 (8)	C19—P2—C31	103.62 (9)
Ru1—P1—C7	105.37 (7)		

All computations were carried out on a PDP-11/73 computer using SDP software (Enraf-Nonius, 1985).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71597 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1048]

References

- Albers, M. O., Robinson, D. J. & Singleton, E. (1987). *Coord. Chem. Rev.* **79**, 1–96.
- Birkenbach, L. & Kellerman, K. (1925). *Chem. Ber.* **58**, 2377.
- Bruce, M. I., Cifuentes, M. P., Snow, M. R. & Tiekink, R. T. (1989). *J. Organomet. Chem.* **359**, 379–399.
- Bruce, M. I., Hambley, T. W., Rodgers, J. R., Snow, M. R. & Wong, F. S. (1982). *Aust. J. Chem.* **35**, 1323–1333.
- Dori, Z. & Ziolo, R. F. (1973). *Chem. Rev.* **73**, 247–254.
- Dunitz, J. D. & Seiler, P. (1973). *Acta Cryst.* **B29**, 589–595.
- Enraf-Nonius (1988). *Structure Determination Package. SDP/PDP User's Guide*. Version 3.0. Enraf-Nonius, Delft, The Netherlands.
- Frank, K. G. & Selegue, J. P. (1991). *Acta Cryst.* **C47**, 35–37.
- Gaughan, A. P., Ziolo, R. F. & Dori, Z. (1971). *Inorg. Chem.* **10**, 2776–2781.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Pauling, L. (1967). *The Nature of the Chemical Bond*. Ithaca: Cornell Univ. Press.
- Wisner, J. M., Bartczak, T. J. & Ibers, J. A. (1985). *Inorg. Chim. Acta*, **100**, 115–123.

Acta Cryst. (1994). **C50**, 504–507

cis-Bis(9-carbazolyl)(1,2-dimethoxyethane)-cis-bis(tetrahydrofuran)ytterbium(II)†

C. T. ABRAHAM, G. B. DEACON, B. M. GATEHOUSE* AND G. N. WARD

Chemistry Department, Monash University, Clayton, Victoria 3168, Australia

(Received 3 June 1993; accepted 1 September 1993)

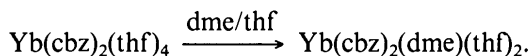
Abstract

The structure of [Yb(C₄H₁₀O₂)(C₄H₈O)₂(C₁₂H₈N)₂] is monomeric with a distorted octahedral environment at Yb. The metal centre is surrounded by two 9-carbazolyl ligands which are *cis* to each other, two *cis* tetrahydrofuran ligands and one chelating dimethoxyethane ligand. Yb—N distances are 2.45 (2) and 2.43 (3) Å with a *cis* N—Yb—N angle of 107.3 (7)°.

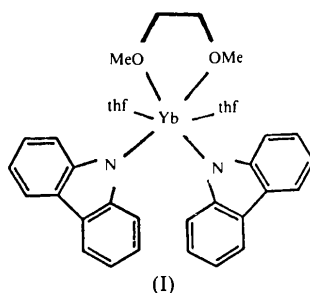
† Organoamido- and aryloxolanthenides (VIII).

Comment

The air-sensitive complex *cis*-bis(9-carbazolyl)-(1,2-dimethoxyethane)-*cis*-bis(tetrahydrofuran)ytterbium(II), $\text{Yb}(\text{cbz})_2(\text{dme})(\text{thf})_2$ (I), was prepared by the following reaction:



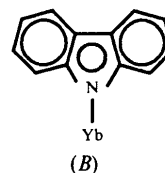
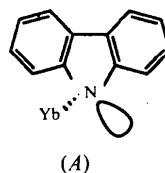
Bulky amides are used to generate low-coordinate (≤ 6) amidolanthanide complexes (Hart, 1987). An X-ray crystal structure study is often the only definitive way of determining how ligands are actually bound to the lanthanide metals and their coordination number. This is especially true for complexes with thf, since their crystals may also incorporate thf of solvation (Deacon, Feng, MacKinnon, Newnham, Nickel, Skelton & White, 1993).



The complex $\text{Yb}(\text{cbz})_2(\text{dme})(\text{thf})_2$ is monomeric with distorted octahedral stereochemistry about the Yb atom and *cis* cbz ligands; the distortion from octahedral geometry is evident from the large N—Yb—N angle of $107.3(7)^\circ$. This type of distortion was observed in a similar six-coordinate structure, $\text{Eu}(\text{cbz})_2(\text{thf})_4$ (Deacon, Forsyth, Gatehouse & White, 1990), containing *cis* cbz ligands, where the N—Eu—N angle was $109.8(2)^\circ$ and was considered to result from the steric repulsion between the bulky organoamido groups. The thf ligands in $\text{Yb}(\text{cbz})_2(\text{dme})(\text{thf})_2$ are also *cis* to each other. The dme has a small bite angle of $65.8(5)^\circ$ which compresses the edge of the octahedron. Comparisons between the M —N and M —O bond lengths in this, and in other similar organoamido La complexes can be made by subtracting the appropriate La ionic radius (Shannon, 1976) from the respective M —N/O bond length. The differences for the M —N distances in $\text{Yb}(\text{cbz})_2(\text{dme})(\text{thf})_2$ are 1.41 and 1.43 Å, and are comparable to those found in $\text{Eu}(\text{cbz})_2(\text{thf})_4$ of 1.38 and 1.40 Å. The range derived from the M —O bond lengths in $\text{Yb}(\text{cbz})_2(\text{dme})(\text{thf})_2$, 1.39–1.46 Å, is again similar to the range found for $\text{Eu}(\text{cbz})_2(\text{thf})_4$, 1.38–1.43 Å. These values are somewhat higher than those found for other organolanthanoid ether complexes [1.34(5) Å (Deacon, MacKinnon, Hambley & Taylor, 1983)], which is indicative of greater crowd-

ing in $\text{Yb}(\text{cbz})_2(\text{dme})(\text{thf})_2$, but are less than values (1.49–1.59 Å) for highly crowded thf complexes of bulky phenolatolanthanoid(III) compounds (Deacon, Feng, Nickel, Ogden & White, 1992). Both cbz complexes show the largest M —O distance for an O atom *trans* to a carbazole group, presumably due to the greater *trans* influence of the bulky charged cbz ligands compared to the thf ligands.

The Yb atom is displaced from the planes of the 9-carbazolyl ligands. Thus, the angle between the line joining N1 and Yb and the ligand plane defined by N1, C12, C11, C10 and C9 is 10.6° , and the angle between the line joining N2 and Yb and the ligand plane defined by N2, C21, C22, C23 and C24 is 17.4° . This deviation from planarity is markedly larger than that in $\text{Eu}(\text{cbz})_2(\text{thf})_4$, in which the angles are only 3.3 and 9.9° , respectively. The displacement of the Yb atom from the 9-carbazolyl planes may be indicative of pyramidal coordination at the N atoms [type (A)] as opposed to sp^2 hybridization of the N atoms in an aromatic cbz ligand (B), as was discussed for the structure of $\text{Eu}(\text{cbz})_2(\text{thf})_4$ (Deacon, Forsyth, Gatehouse & White, 1990).



Pyramidal geometry would also be reflected in a loss of aromaticity in the pyrrolyl rings. However, the C—C bond lengths in $\text{Yb}(\text{cbz})_2(\text{dme})(\text{thf})_2$ do not show alternation, the C—N distances are less than single-bond lengths and the Yb—N—C angles considerably exceed tetrahedral values (Table 2). Thus, the deviation of Yb from planarity with the cbz ring probably arises from the steric effects of the ligands. There is a widening of the angle between the

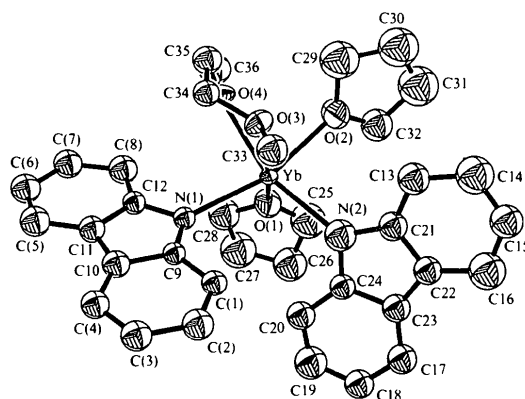


Fig. 1. Structure of $\text{Yb}(\text{cbz})_2(\text{dme})(\text{thf})_2$ showing the stereochemistry and the numbering scheme used.

carbazole planes to 72.7° compared to 59.2° for the Eu(cbz)₂(thf)₄ complex. This would relieve the steric crowding about the smaller Yb²⁺ ion. Even though the less sterically demanding dme ligand is present, the metal-ion size factor appears more important.

Experimental

Yb(cbz)₂(thf)₄ (Deacon, Forsyth, Gatehouse & White, 1990) was dissolved in hot dimethoxyethane and this solution was allowed to slowly mix with warm tetrahydrofuran under an N₂ atmosphere. This resulted in the formation of orange-yellow single crystals (yield 22%). Found Yb 23.4%; [Yb(C₄H₁₀O₂)(C₄H₈O)₂(C₁₂H₈N)₂] requires Yb 23.3%. IR spectrum: 1617m, 1580s, 1545w, 1340w, 1325s, 1290m, 1270s, 1230s, 1190w (sh), 1150m, 1120m, 1100m, 1055s, 1040s, 1025s, 995m, 920w, 890m, 850m (sh), 830w (sh), 760s, 725s, 665w cm⁻¹.

Crystal data

[Yb(C ₄ H ₁₀ O ₂)(C ₄ H ₈ O) ₂ (C ₁₂ H ₈ N) ₂]	<i>D</i> _r = 1.50 Mg m ⁻³
<i>M</i> _r = 739.79	Mo <i>K</i> α radiation
Monoclinic	λ = 0.7107 Å
<i>P</i> 2 ₁ / <i>a</i>	Cell parameters from 24 reflections
<i>a</i> = 13.844 (4) Å	θ = 7–10°
<i>b</i> = 25.734 (10) Å	μ = 2.90 mm ⁻¹
<i>c</i> = 9.590 (2) Å	<i>T</i> = 293 K
β = 107.58 (2)°	Tabular
<i>V</i> = 3257 (2) Å ³	0.38 × 0.15 × 0.06 mm
<i>Z</i> = 4	Orange-yellow

Data collection

Philips PW1100 four-circle diffractometer	1971 observed reflections
ω scans	[<i>I</i> ≥ 3σ(<i>I</i>)]
Absorption correction: analytical, indexed crystal faces	<i>R</i> _{int} = 0.07
<i>T</i> _{min} = 0.63, <i>T</i> _{max} = 0.84	θ _{max} = 30°
9617 measured reflections	<i>h</i> = -17 → 17
9141 independent reflections	<i>k</i> = 0 → 32
	<i>l</i> = 0 → 11
	3 standard reflections frequency: 240 min

Refinement

Refinement on <i>F</i>	<i>w</i> = 1/σ ² (<i>F</i> _o)
<i>R</i> = 0.0775	(Δ/σ) _{max} = 0.001
<i>wR</i> = 0.0626	Δρ _{max} = 2.0 e Å ⁻³
<i>S</i> = 2.48	Δρ _{min} = 1.5 e Å ⁻³
1971 reflections	Extinction correction: none
207 parameters	Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
Only H-atom <i>U</i> 's refined: one overall temperature factor of 0.114 (2) Å ²	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$				
	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Yb	0.15113 (9)	0.15582 (5)	0.45761 (13)	0.0319 (4)
O(1)	-0.0140 (13)	0.1239 (7)	0.3219 (19)	0.056 (9)

O(2)	0.2220 (14)	0.0737 (7)	0.3945 (21)	0.069 (10)
O(3)	0.3238 (11)	0.1898 (6)	0.5307 (17)	0.039 (7)
O(4)	0.2120 (12)	0.1816 (6)	0.2518 (16)	0.042 (7)
N(1)	0.1014 (13)	0.2468 (7)	0.4674 (19)	0.028 (5)
N(2)	0.1573 (18)	0.1163 (9)	0.6903 (25)	0.060 (7)
C(1)	0.1508 (17)	0.2603 (10)	0.7380 (23)	0.036 (7)
C(2)	0.1619 (20)	0.2952 (11)	0.8501 (29)	0.052 (8)
C(3)	0.1410 (17)	0.3475 (13)	0.8190 (25)	0.051 (7)
C(4)	0.1128 (18)	0.3664 (10)	0.6763 (26)	0.043 (7)
C(5)	0.0444 (20)	0.3777 (11)	0.3116 (29)	0.055 (8)
C(6)	0.0155 (22)	0.3681 (12)	0.1657 (32)	0.064 (10)
C(7)	0.0165 (21)	0.3185 (11)	0.1118 (30)	0.052 (9)
C(8)	0.0461 (19)	0.2738 (11)	0.2034 (27)	0.054 (8)
C(9)	0.1215 (16)	0.2773 (9)	0.5914 (24)	0.025 (6)
C(10)	0.1034 (18)	0.3303 (9)	0.5634 (27)	0.042 (8)
C(11)	0.0728 (17)	0.3331 (10)	0.4068 (25)	0.039 (7)
C(12)	0.0741 (17)	0.2826 (10)	0.3547 (25)	0.032 (7)
C(13)	0.3177 (21)	0.0674 (11)	0.7756 (28)	0.055 (8)
C(14)	0.3698 (25)	0.0263 (13)	0.8660 (32)	0.084 (11)
C(15)	0.3219 (22)	0.0030 (12)	0.9534 (29)	0.065 (9)
C(16)	0.2286 (25)	0.0137 (13)	0.9631 (34)	0.087 (11)
C(17)	-0.0084 (20)	0.0706 (11)	0.8967 (27)	0.053 (8)
C(18)	-0.0887 (21)	0.1021 (11)	0.8487 (29)	0.056 (9)
C(19)	-0.0958 (23)	0.1389 (11)	0.7471 (29)	0.069 (10)
C(20)	-0.0173 (18)	0.1463 (11)	0.6902 (25)	0.049 (8)
C(21)	0.2180 (21)	0.0785 (11)	0.7702 (28)	0.048 (8)
C(22)	0.1732 (20)	0.0537 (10)	0.8646 (28)	0.044 (7)
C(23)	0.0772 (20)	0.0746 (11)	0.8424 (27)	0.049 (8)
C(24)	0.0664 (20)	0.1144 (10)	0.7314 (26)	0.039 (7)
C(25)	-0.0599 (25)	0.0765 (14)	0.3450 (37)	0.100 (13)
C(26)	-0.1677 (24)	0.0897 (14)	0.3403 (35)	0.095 (12)
C(27)	-0.1892 (26)	0.1333 (13)	0.2292 (37)	0.113 (14)
C(28)	-0.0904 (21)	0.1552 (15)	0.2287 (30)	0.079 (9)
C(29)	0.3158 (26)	0.0690 (14)	0.3610 (37)	0.100 (13)
C(30)	0.3571 (26)	0.0149 (14)	0.4037 (39)	0.110 (14)
C(31)	0.2700 (27)	-0.0142 (14)	0.4159 (39)	0.120 (14)
C(32)	0.1864 (23)	0.0233 (12)	0.4019 (32)	0.073 (10)
C(33)	0.3723 (20)	0.2044 (11)	0.6848 (27)	0.057 (9)
C(34)	0.3370 (18)	0.2289 (9)	0.4334 (24)	0.037 (7)
C(35)	0.3119 (19)	0.2042 (11)	0.2858 (27)	0.052 (8)
C(36)	0.1786 (21)	0.1617 (14)	0.1107 (29)	0.078 (9)

Table 2. Selected geometric parameters (Å, °)

Yb—O(1)	2.41 (2)	C(9)—C(1)	1.41 (3)
Yb—O(2)	2.48 (2)	C(9)—C(10)	1.40 (3)
Yb—O(3)	2.44 (2)	C(10)—C(11)	1.43 (3)
Yb—O(4)	2.46 (1)	C(11)—C(5)	1.45 (3)
Yb—N(1)	2.45 (2)	C(11)—C(12)	1.40 (3)
Yb—N(2)	2.43 (3)	C(13)—C(14)	1.42 (4)
N(1)—C(9)	1.38 (2)	C(13)—C(21)	1.40 (3)
N(1)—C(12)	1.38 (3)	C(14)—C(15)	1.36 (4)
N(2)—C(21)	1.36 (3)	C(15)—C(16)	1.35 (4)
N(2)—C(24)	1.43 (3)	C(16)—C(22)	1.45 (4)
O(1)—C(25)	1.43 (4)	C(17)—C(18)	1.34 (3)
O(1)—C(28)	1.41 (3)	C(17)—C(23)	1.44 (3)
O(2)—C(29)	1.43 (3)	C(18)—C(19)	1.34 (4)
O(2)—C(32)	1.40 (3)	C(19)—C(20)	1.37 (3)
O(3)—C(33)	1.47 (3)	C(20)—C(24)	1.38 (3)
O(3)—C(34)	1.42 (2)	C(21)—C(22)	1.40 (3)
O(4)—C(35)	1.44 (3)	C(22)—C(23)	1.39 (3)
O(4)—C(36)	1.39 (3)	C(23)—C(24)	1.45 (3)
C(1)—C(2)	1.37 (3)	C(25)—C(26)	1.52 (4)
C(2)—C(3)	1.39 (4)	C(26)—C(27)	1.51 (4)
C(3)—C(4)	1.39 (3)	C(27)—C(28)	1.48 (4)
C(4)—C(10)	1.40 (3)	C(29)—C(30)	1.51 (4)
C(5)—C(6)	1.36 (3)	C(30)—C(31)	1.46 (4)
C(6)—C(7)	1.38 (3)	C(31)—C(32)	1.48 (4)
C(7)—C(8)	1.43 (3)	C(34)—C(35)	1.49 (3)
C(8)—C(12)	1.40 (3)		
N(1)—Yb—N(2)	107.3 (7)	C(13)—C(14)—C(15)	117 (3)
N(1)—Yb—O(1)	96.6 (6)	C(14)—C(15)—C(16)	128 (3)
N(1)—Yb—O(2)	163.7 (6)	C(15)—C(16)—C(22)	115 (3)
N(1)—Yb—O(3)	84.8 (5)	C(16)—C(22)—C(21)	120 (3)
N(1)—Yb—O(4)	86.6 (5)	C(16)—C(22)—C(23)	132 (3)
N(2)—Yb—O(1)	96.8 (7)	C(17)—C(18)—C(19)	123 (3)
N(2)—Yb—O(2)	87.8 (7)	C(18)—C(19)—C(20)	120 (3)

N(2)—Yb—O(3)	97.3 (7)	C(19)—C(20)—C(24)	120 (3)
N(2)—Yb—O(4)	157.5 (7)	C(20)—C(24)—C(23)	122 (2)
O(1)—Yb—O(2)	87.6 (6)	C(21)—C(13)—C(14)	119 (3)
O(1)—Yb—O(3)	164.8 (5)	C(21)—C(22)—C(23)	108 (2)
O(1)—Yb—O(4)	99.0 (6)	C(22)—C(21)—C(13)	121 (3)
O(2)—Yb—O(3)	87.1 (6)	C(22)—C(23)—C(17)	141 (3)
O(2)—Yb—O(4)	77.1 (6)	C(22)—C(23)—C(24)	106 (2)
O(3)—Yb—O(4)	65.8 (5)	C(23)—C(17)—C(18)	122 (3)
Yb—N(1)—C(9)	126 (1)	C(23)—C(24)—N(2)	109 (3)
Yb—N(1)—C(12)	128 (2)	C(24)—N(2)—C(21)	105 (2)
Yb—N(2)—C(21)	131 (2)	C(24)—C(23)—C(17)	114 (3)
Yb—N(2)—C(24)	119 (2)	C(25)—O(1)—C(28)	108 (2)
N(1)—C(9)—C(1)	127 (2)	C(25)—C(26)—C(27)	100 (3)
N(1)—C(9)—C(10)	114 (2)	C(26)—C(27)—C(28)	107 (3)
N(1)—C(12)—C(8)	129 (3)	C(27)—C(28)—O(1)	107 (3)
N(2)—C(21)—C(13)	127 (3)	C(4)—C(10)—C(11)	135 (2)
N(2)—C(21)—C(22)	112 (3)	C(5)—C(6)—C(7)	122 (3)
N(2)—C(24)—C(20)	130 (3)	C(6)—C(7)—C(8)	123 (3)
O(1)—C(25)—C(26)	107 (3)	C(7)—C(8)—C(12)	117 (3)
O(2)—C(29)—C(30)	108 (3)	C(8)—C(12)—C(11)	119 (2)
C(1)—C(2)—C(3)	120 (3)	C(9)—C(1)—C(2)	120 (3)
C(2)—C(3)—C(4)	122 (3)	C(9)—C(10)—C(11)	103 (2)
C(3)—C(4)—C(10)	117 (3)	C(10)—C(9)—C(1)	118 (2)
C(4)—C(10)—C(9)	122 (2)	C(10)—C(11)—C(5)	130 (2)
C(10)—C(11)—C(12)	107 (2)	C(29)—O(2)—C(32)	107 (2)
C(11)—C(5)—C(6)	117 (3)	C(29)—C(30)—C(31)	104 (3)
C(11)—C(12)—N(1)	112 (2)	C(30)—C(31)—C(32)	108 (3)
C(12)—N(1)—C(9)	103 (2)	C(31)—C(32)—O(2)	109 (3)
C(12)—C(11)—C(5)	123 (2)		

A representative crystal was mounted in a Lindemann glass capillary tube under Ar in a dry box. The crystal was fixed to the side of the tube with a small amount of dry liquid paraffin. Intensity data were measured on an automatic four-circle diffractometer equipped with a graphite monochromator, as described previously (Canty, Chaichit & Gatehouse, 1980). Three standard reflections decreased in intensity by 52 (2)%; a correction for decomposition was applied. It is believed that the high *R* value arises as a result of the decomposition. Data were processed using a program written specifically for the PW1100 diffractometer (Hornstra & Stubbe, 1972). Scattering factors for neutral atoms and corrections for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on the Monash University VAX11/780 computer system. Programs used: *SHELX76* (Sheldrick, 1976) and *ORFFE* (Busing, Martin & Levy, 1964).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71623 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1064]

References

- Busing, W. R., Martin, K. O. & Levy, H. A. (1964). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.
- Canty, A. J., Chaichit, N. & Gatehouse, B. M. (1980). *Acta Cryst.* **B36**, 786–789.
- Deacon, G. B., Feng, T., MacKinnon, P. I., Newnham, R. H., Nickel, S., Skelton, B. W. & White, A. H. (1993). *Aust. J. Chem.* **46**, 387–399.
- Deacon, G. B., Feng, T., Nickel, S., Ogden, M. I. & White, A. H. (1992). *Aust. J. Chem.* **45**, 671–683.
- Deacon, G. B., Forsyth, C. M., Gatehouse, B. M. & White, P. A. (1990). *Aust. J. Chem.* **43**, 1–12.

- Deacon, G. B., MacKinnon, P. I., Hambley, T. W. & Taylor, J. C. (1983). *J. Organomet. Chem.* **259**, 91–97.
- Hart, F. A. (1987). *Scandium, Yttrium and the Lanthanides*. In *Comprehensive Coordination Chemistry*, edited by G. Wilkinson, R. D. Gillard & J. A. McLeverly. Vol. 3. Oxford: Pergamon Press.
- Hornstra, J. & Stubbe, B. (1972). *PW1100 Data Processing Program*. Philips Research Laboratories, Eindhoven, The Netherlands.
- Shannon, A. C. (1976). *Acta Cryst.* **A32**, 751–767.
- Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Acta Cryst. (1994). **C50**, 507–510

A New Orthorhombic Crystal Form of Dichloro[(-)-(R,R)-2,2-dimethyl-4,5-bis(diphenylphosphinomethyl)-1,3-dioxolane-P,P']platinum, [{(R,R)-diop}PtCl₂]

CHRISTIAN GANTER AND A. GUY ORPEN*

School of Chemistry, University of Bristol, Bristol BS8 1TS, England

PAOLA BERGAMINI AND EMILIANA COSTA

Dipartimento di Chimica dell'Università di Ferrara e Centro di Fotochimica CNR, Via L. Borsari 46, 44100 Ferrara, Italy

(Received 1 February 1993; accepted 22 September 1993)

Abstract

In the orthorhombic form of the title compound, [PtCl₂(C₃₁H₃₂O₂P₂)], the geometry at Pt is approximately square planar and the molecular conformation observed is qualitatively similar to that of one of the two independent molecules in a triclinic polymorph determined previously. The crystal and molecular structures are pseudo-isomorphous with those of [(S,S)-diop}PtMeCl].

Comment

Crystals of dichloro[(-)-(R,R)-2,2-dimethyl-4,5-bis(diphenylphosphinomethyl)-1,3-dioxolane-P,P']-

