

S(1)—W—S(3)	106.38 (8)	W—S(2)—Cu(1)	74.63 (7)
S(3)—W—S(2)	107.97 (8)	W—S(2)—Cu(3)	74.25 (7)
O(1)—W—S(1)	110.4 (2)	W—S(3)—Cu(2)	74.42 (7)
O(1)—W—S(2)	111.1 (2)	W—S(3)—Cu(3)	74.46 (7)
O(1)—W—S(3)	112.3 (2)	P(4)—S(4)—Cu(1)	107.1 (1)
S(1)—Cu(1)—S(4)	95.28 (9)	P(4)—S(5)—Cu(3)	103.4 (1)
S(2)—Cu(1)—S(1)	101.94 (8)	O(2)—P(4)—S(5)	112.5 (3)
S(2)—Cu(1)—S(4)	107.51 (8)	O(2)—P(4)—S(4)	106.4 (3)
S(3)—Cu(2)—S(1)	99.89 (9)	O(3)—P(4)—O(2)	99.1 (5)
S(3)—Cu(3)—S(2)	102.32 (8)	O(3)—P(4)—S(5)	108.3 (4)
S(3)—Cu(3)—S(5)	104.8 (1)	O(3)—P(4)—S(4)	110.6 (3)
S(5)—Cu(3)—S(2)	117.5 (1)	S(5)—P(4)—S(4)	118.3 (2)
P(1)—Cu(1)—S(1)	109.70 (9)	C(1)—O(2)—P(4)	124 (1)
P(1)—Cu(1)—S(2)	121.42 (9)	C(3)—O(3)—P(4)	127 (1)
P(1)—Cu(1)—S(4)	116.8 (1)	C(2)—C(1)—O(2)	118 (2)
P(2)—Cu(2)—S(1)	119.5 (1)	O(2)—C(3)—C(4)	120 (2)
P(2)—Cu(2)—S(3)	117.42 (9)	C(111)—P(1)—Cu(1)	111.3 (3)
P(3)—Cu(3)—S(3)	118.21 (9)	C(131)—P(1)—Cu(1)	118.1 (3)
P(3)—Cu(3)—S(5)	105.6 (1)	C(121)—P(1)—Cu(1)	114.1 (3)
P(3)—Cu(3)—S(2)	109.03 (9)	C(231)—P(2)—Cu(2)	115.5 (3)
Cu(1)—S(2)—Cu(3)	108.32 (9)	C(211)—P(2)—Cu(2)	111.7 (3)
Cu(2)—S(1)—Cu(1)	84.79 (8)	C(221)—P(2)—Cu(2)	116.6 (3)
C(321)—P(3)—Cu(3)	109.0 (3)	C(311)—P(3)—Cu(3)	116.2 (3)

Data collection used *CONTROL* software (Molecular Structure Corporation, 1988). The scan speed varied between 2.4 and 8° min⁻¹ (in ω) on the basis of *SEARCH* intensity. The scan width was $(1.207 + 0.350\text{tan}\theta)^\circ$ with maximum $(\sin\theta)/\lambda = 0.5946 \text{ \AA}^{-1}$. The structure was solved by conventional heavy-atom methods. The W atom was located from a three-dimensional Patterson synthesis and the remaining non-H atoms were located using the *DIRDIF* program (Beurskens, 1984). H atoms were placed in geometrically calculated positions (C—H 0.95 Å) but were not included in the refinement. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for the W, Cu, S, P and O atoms and isotropic displacement parameters for all C atoms. All calculations were performed on a VAX 785 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package. The view of the molecule (Fig. 1) was produced using *ORTEPII* (Johnson, 1976).

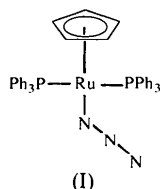
This research was supported by grants from the State Key Laboratory of Structural Chemistry, Fujian Institute of Research on Structure of Matter, Chinese Academy of Science, and from the National Science Foundation of China.

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 71595 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1053]

References

- Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – An Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors*. Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6526 ED Nijmegen, The Netherlands.
- Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Tables 2.2A and 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Lu, S., Zhu, N., Wu, X., Wu, Q. & Lu, J. (1989). *J. Mol. Struct.* **197**, 15–31.
- Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Müller, A., Bögge, H. & Schimanski, U. (1983). *Inorg. Chim. Acta*, **69**, 5–16, and references therein.
- Zhan, H., Zheng, Y., Wu, X. & Lu, J. (1989). *Inorg. Chim. Acta*, **156**, 277–280.
- Zhu, N., Zheng, Y. & Wu, X. (1990). *J. Chem. Soc. Chem. Commun.* pp. 780–781.
- Acta Cryst.* (1994). **C50**, 502–504
- Azido(η^5 -cyclopentadienyl)bis(triphenylphosphine)ruthenium(II)**
- MIRZA M. TAQUI KHAN, MOHAN M. BHADBHADDE, M. R. H. SIDDIQUI AND K. VENKATASUBRAMANIAN
- Discipline of Coordination Chemistry, Central Salt & Marine Chemicals Research Institute, Bhavnagar 364 002, India*
- J. A. TIKHONOVA
- Institute of Organoelement Compounds, Russian Academy of Sciences, Moscow, Russia*
- (Received 27 January 1993; accepted 9 July 1993)
- Abstract**
- The molecule [RuN₃(C₅H₅){P(C₆H₅)₃]₂ has a typical three-legged-piano-stool geometry; Ru—Cp⁰ (where Cp⁰ is the centre of the ring) is 1.843 (3) Å, the three legs are 2.3293 (5) and 2.3304 (5) (Ru—P) and 2.135 (3) Å (Ru—N) in length while the angles are P—Ru—P 105.22 (2), P—Ru—N 85.39 (6) and 86.65 (5)°. The azide group is almost linear [N—N—N 175.2 (3)°] and is coordinated to Ru with an Ru—N—N angle of 124.5 (2)°; there is a small difference between the N—N distances [1.186 (3) and 1.164 (3) Å], the longer being adjacent to the Ru atom.
- Comment**
- The complex CpRu(Cl)(PPh₃)₂ (Bruce, Cifuentes, Snow & Tiekink, 1989) has been used as the starting material for the synthesis of several complexes because of its substitutionally labile chloride and

phosphines (Albers, Robinson & Singleton, 1987). Structure determinations were undertaken to show the effect on $\text{CpRu}(L)(\text{PPh}_3)_2$ of changing the substituent L . The title compound (I) was prepared by refluxing a mixture of $\text{CpRu}(\text{Cl})(\text{PPh}_3)_2$ and NaN_3 in 95% ethanol. The dark-brown precipitate obtained on cooling was recrystallized from a CH_2Cl_2 - n -hexane mixture.



The coordination geometry of the Ru atom can be described as distorted tetrahedral if Cp is considered to occupy one coordination site. Alternatively, it may be viewed as distorted octahedral if Cp is allowed three facial coordination sites. We prefer to describe it as a three-legged piano stool (Frank & Selegue, 1991); this geometry is typical of $\text{CpRu}(L)(\text{PPh}_3)_2$ complexes (Wisner, Bartczak & Ibers, 1985). The dihedral angles between the Cp ring and the planes containing Ru, P1, P2 and P1, P2, N1 are $58.64(9)$ and $3.1(8)^\circ$, respectively.

The Ru—C distances in the present structure [2.187(2)–2.230(4) Å] compare well with those in $\text{CpRuCl}(\text{PPh}_3)_2$ (Bruce, Hambley, Rodgers, Snow & Wong, 1982) showing the pseudo-halide nature of the azide substituent (Birkenbach & Kellerman, 1925). The Cp ring coordination is influenced by the substituent L in the $\text{CpRu}(L)(\text{PPh}_3)_2$ system. When L is N_3^- , atoms C(39) and C(40) of the Cp ring, which are almost *trans* to the N atom, are 2.202(3) and 2.190(2) Å from Ru; when L is CO, the equivalent Ru—C distances are 2.256(3) and 2.227(3) Å, while those for L equals $-\text{C}\equiv\text{CPh}$ are 2.265(2) and 2.271(2) Å (Wisner *et al.*, 1985).

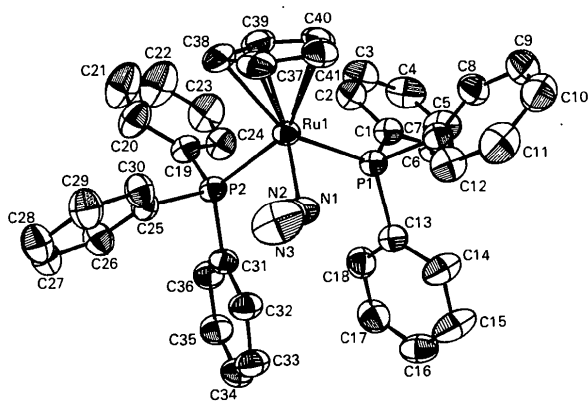
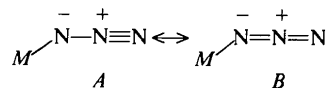


Fig. 1. Perspective view (ORTEP; Johnson, 1976) of the molecule showing the atom-numbering scheme.

As suggested by Pauling (1967), and later confirmed from several X-ray structure determinations (Dori & Ziolo, 1973), the canonical forms A and B are the main contributors to the ground state in a metal–azide coordination compound:



A copper–azide complex with $\text{N1}-\text{N2} = 1.196(18)$ and $\text{N2}-\text{N3} = 1.076(18)$ Å is a clear case of A being the dominant form (Gaughan, Ziolo & Dori, 1971). In the present structure, the difference between $\text{N1}-\text{N2}$ [1.186(3) Å] and $\text{N2}-\text{N3}$ [1.164(3) Å] is not as large as in the earlier example but is significant at about the 7σ level, suggesting that resonance form A is slightly more dominant than B .

Experimental

Crystal data

$[\text{RuN}_3(\text{C}_5\text{H}_5)(\text{C}_{18}\text{H}_{15}\text{P})_2]$

$M_r = 732.8$

Triclinic

$P\bar{1}$

$a = 11.969(3)$ Å

$b = 14.541(1)$ Å

$c = 11.284(1)$ Å

$\alpha = 111.51(1)^\circ$

$\beta = 110.69(1)^\circ$

$\gamma = 88.27(2)^\circ$

$V = 1698(13)$ Å³

$Z = 2$

$D_x = 1.433$ Mg m⁻³

Cu $K\alpha$ radiation

$\lambda = 1.5418$ Å

Cell parameters from 16 reflections

$\theta = 20-36^\circ$

$\mu = 4.979$ mm⁻¹

$T = 295$ K

Plate

$0.12 \times 0.10 \times 0.04$ mm

Dark red-brown

Data collection

Enraf-Nonius CAD-4 diffractometer

$\omega/2\theta$ scans

Absorption correction: empirical (North, Phillips & Mathews, 1968)

$T_{\min} = 0.65$, $T_{\max} = 0.93$

6084 measured reflections

(symmetry equivalents not collected)

5544 observed reflections [$I \geq 4\sigma(I)$]

$\theta_{\max} = 65^\circ$

$h = 0 \rightarrow 14$

$k = -17 \rightarrow 17$

$l = -13 \rightarrow 13$

3 standard reflections

frequency: 60 min

intensity variation: not significant

Refinement

Refinement on F

$R = 0.038$

$wR = 0.041$

$S = 0.51$

5544 reflections

424 parameters

H-atom parameters not refined

$w = 1/\sigma^2(F)$ with Dunitz–Seiler weighting factor (Dunitz & Seiler, 1973)

$(\Delta/\sigma)_{\max} = 0.01$

$\Delta\rho_{\max} = 0.37$ e Å⁻³

$\Delta\rho_{\min} = -0.69$ e Å⁻³

except for three peaks of

$ca -1.5$ e Å⁻³ near Ru

Extinction correction: none

Atomic scattering factors

from *International Tables*

for X-ray Crystallography

(1974, Vol. IV)

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).