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## Structure of Bis(cyclopentadienyl)bis(pyridine)cadmium(II)

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**Abstract.**  $[\text{Cd}(\text{C}_5\text{H}_5)_2(\text{C}_5\text{H}_5\text{N})_2]$ ,  $M_r = 400.80$ , monoclinic,  $P2_1/n$ ,  $a = 12.79$  (1),  $b = 10.093$  (4),  $c = 14.38$  (1) Å,  $\beta = 107.20$  (8)°,  $V = 1773$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.501$  g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71073$  Å,  $\mu = 12.3$  cm<sup>-1</sup>,  $F(000) = 808$ ,  $T = 294$  K,  $R = 0.0326$  for 2407 observed reflections with  $I > 2\sigma(I)$ . The Cd atom is pseudo-tetrahedrally coordinated by the two N atoms of the pyridine molecules and by two C atoms of the  $\eta^1$ -bonded Cp moieties.

**Introduction.** Bis(cyclopentadienyl)cadmium  $[\text{Cd}(\text{Cp})_2]$  was prepared to compare its reactivity with the related  $[\text{Zn}(\text{Cp})_2]$  as part of an investigation on the reactions of (cyclopentadienyl)zinc compounds with  $\text{Ni}^0$  complexes (Budzelaar, Boersma, van der Kerk, Spek & Duisenberg, 1985*a,b*; Fischer, Boersma, Kojić-Prodić & Spek, 1985). Single crystals of the present pyridine complex were obtained by slow crystallization from pyridine. An X-ray study of this complex was undertaken in order to establish the molecular geometry of the first known example of a cyclopentadienyl–Cd

compound. A preliminary report of this study was given previously (Fischer, van Mier, Boersma, Smeets & Spek, 1987).

**Experimental.** Yellowish crystal  $0.58 \times 0.25 \times 0.20$  mm suitable for X-ray diffraction was sealed under nitrogen in a Lindemann-glass capillary. Enraf–Nonius CAD-4F diffractometer, Zr-filtered Mo  $K\alpha$  radiation, cell constants from the angular settings of 25 reflections with  $16.3 < \theta < 20.4^\circ$ . Intensity data of 4074 unique reflections were collected within a quarter of the reflection sphere:  $0 \leq h \leq 16$ ;  $0 \leq k \leq 13$ ;  $-18 \leq l \leq 17$ ;  $(\sin \theta)/\lambda < 0.65$  Å<sup>-1</sup>;  $\omega/2\theta$  scan mode with  $\Delta\omega = (0.70 + 0.35 \tan \theta)^\circ$ . Three reference reflections  $[200$  (e.s.d. = 0.64%);  $020$  (e.s.d. = 0.51%);  $002$  (e.s.d. = 0.68%)] measured every hour showed a linear decay of 0.2% during the 77 hours of X-ray exposure time. The intensity data were corrected for this decay and for Lorentz–polarization effects. No correction for absorption was applied in view of the small intensity variation of 6% about the mean of the  $360^\circ \psi$  scan of the 404 reflection. Standard deviations in the intensities were increased according to an analysis of the variance

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of the reference reflections:  $\sigma^2(I) = \sigma_{cs}^2(I) + (0.0091I)^2$  (McCandlish, Stout & Andrews, 1975) resulting in 2407 reflections with  $I > 2\sigma(I)$ . Space group  $P2_1/n$  derived from the systematic absences  $h0l$ ;  $h+l = 2n+1$  and  $0k0$ ;  $k = 2n+1$ .

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
Cd	0.24221 (2)	0.24646 (3)	0.00547 (1)	0.0502 (1)
N(1)	0.3968 (2)	0.1873 (3)	-0.0364 (2)	0.056 (1)
N(2)	0.1646 (2)	0.0325 (2)	-0.0085 (2)	0.047 (1)
C(1)	0.3106 (3)	0.2914 (4)	0.1731 (3)	0.061 (2)
C(2)	0.2007 (3)	0.3100 (4)	0.1705 (2)	0.066 (2)
C(3)	0.1781 (4)	0.4431 (4)	0.1642 (3)	0.070 (2)
C(4)	0.2729 (4)	0.5090 (4)	0.1645 (3)	0.070 (1)
C(5)	0.3541 (3)	0.4189 (5)	0.1702 (2)	0.067 (2)
C(6)	0.1320 (4)	0.3757 (6)	-0.1170 (3)	0.088 (2)
C(7)	0.2171 (5)	0.4626 (4)	-0.1075 (3)	0.093 (2)
C(8)	0.2660 (4)	0.4349 (6)	-0.1758 (4)	0.094 (2)
C(9)	0.2107 (5)	0.3334 (6)	-0.2305 (3)	0.093 (2)
C(10)	0.1285 (4)	0.2972 (4)	-0.1970 (4)	0.098 (2)
C(11)	0.4886 (4)	0.2549 (6)	-0.0035 (3)	0.089 (2)
C(12)	0.5749 (3)	0.2437 (7)	-0.0422 (4)	0.112 (2)
C(13)	0.5688 (3)	0.1583 (5)	-0.1149 (3)	0.082 (2)
C(14)	0.4766 (4)	0.0873 (4)	-0.1490 (3)	0.076 (2)
C(15)	0.3942 (3)	0.1029 (4)	-0.1071 (3)	0.070 (2)
C(16)	0.1300 (3)	-0.0314 (4)	-0.0926 (2)	0.060 (1)
C(17)	0.0908 (3)	-0.1568 (4)	-0.1023 (3)	0.070 (2)
C(18)	0.0849 (3)	-0.2221 (3)	-0.0200 (3)	0.069 (2)
C(19)	0.1178 (3)	-0.1591 (4)	0.0659 (3)	0.066 (2)
C(20)	0.1577 (3)	-0.0334 (4)	0.0697 (2)	0.055 (1)

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) for the non-hydrogen atoms

Cd—N(1)	2.309 (3)	C(6)—C(7)	1.373 (8)
Cd—N(2)	2.360 (3)	C(6)—C(10)	1.387 (7)
Cd—C(1)	2.353 (5)	C(7)—C(8)	1.341 (8)
Cd—C(6)	2.307 (5)	C(8)—C(9)	1.356 (8)
N(1)—C(11)	1.319 (6)	C(9)—C(10)	1.331 (8)
N(1)—C(15)	1.319 (5)	C(11)—C(12)	1.380 (7)
N(2)—C(16)	1.326 (4)	C(12)—C(13)	1.339 (8)
N(2)—C(20)	1.332 (4)	C(13)—C(14)	1.343 (7)
C(1)—C(2)	1.408 (6)	C(14)—C(15)	1.369 (7)
C(1)—C(5)	1.407 (6)	C(16)—C(17)	1.353 (6)
C(2)—C(3)	1.371 (6)	C(17)—C(18)	1.376 (6)
C(3)—C(4)	1.382 (7)	C(18)—C(19)	1.342 (6)
C(4)—C(5)	1.365 (7)	C(19)—C(20)	1.363 (6)
N(1)—Cd—N(2)	96.4 (1)	C(1)—C(5)—C(4)	108.1 (4)
N(1)—Cd—C(1)	103.3 (1)	Cd—C(6)—C(7)	89.9 (3)
N(1)—Cd—C(6)	108.9 (2)	Cd—C(6)—C(10)	100.0 (4)
N(2)—Cd—C(1)	106.5 (1)	C(7)—C(6)—C(10)	106.1 (4)
N(2)—Cd—C(6)	107.8 (2)	C(6)—C(7)—C(8)	108.8 (4)
C(1)—Cd—C(6)	129.1 (2)	C(7)—C(8)—C(9)	107.8 (5)
Cd—N(1)—C(11)	120.8 (3)	C(8)—C(9)—C(10)	109.3 (5)
Cd—N(1)—C(15)	122.6 (3)	C(6)—C(10)—C(9)	108.0 (5)
C(11)—N(1)—C(15)	115.3 (4)	N(1)—C(11)—C(12)	123.1 (5)
Cd—N(2)—C(16)	122.8 (2)	C(11)—C(12)—C(13)	119.9 (5)
Cd—N(2)—C(20)	121.1 (2)	C(12)—C(13)—C(14)	118.1 (4)
C(16)—N(2)—C(20)	116.1 (3)	C(13)—C(14)—C(15)	119.0 (4)
Cd—C(1)—C(2)	86.1 (2)	N(1)—C(15)—C(14)	124.5 (4)
Cd—C(1)—C(5)	100.2 (3)	N(2)—C(16)—C(17)	124.2 (3)
C(2)—C(1)—C(5)	106.1 (4)	C(16)—C(17)—C(18)	118.2 (4)
C(1)—C(2)—C(3)	108.8 (4)	C(17)—C(18)—C(19)	118.9 (3)
C(2)—C(3)—C(4)	107.7 (4)	C(18)—C(19)—C(20)	119.3 (4)
C(3)—C(4)—C(5)	109.3 (4)	N(2)—C(20)—C(19)	123.3 (3)

The structure was solved by standard Patterson (*SHELX84*; Sheldrick, 1984) and difference Fourier methods. All non-H atoms were refined anisotropically (on *F*) by blocked full-matrix least-squares techniques. H atoms were introduced on calculated positions and included in the refinement with individual positional parameters and one overall isotropic thermal parameter. Weights were introduced in the final refinement cycles. Convergence with 269 parameters was reached at  $R = 0.0326$ ,  $wR = 0.0185$ ,  $w = 1/\sigma^2(F)$ ,  $S = 1.67$ ,  $(\Delta/\sigma)_{\max} = 0.044$ . The final difference Fourier synthesis revealed max. and min. residual densities of 0.62 and  $-0.41 \text{ e \AA}^{-3}$  near the Cd atom.

Scattering factors from Cromer & Mann (1968); anomalous-dispersion factors from Cromer & Liberman (1970). Calculations performed with *SHELX76* (Sheldrick, 1976) and the *EUCLID* package (geometry calculations and illustrations) (Spek, 1982) on the CDC Cyber-855 of the University of Utrecht.

**Discussion.** Fractional atomic coordinates and equivalent isotropic thermal parameters of the non-hydrogen atoms are given in Table 1.\* Bond lengths and bond angles are listed in Table 2. The molecular structure of the title compound and the adopted atom numbering are presented in Fig. 1.

The crystal structure consists of four discrete molecules contained in the monoclinic unit cell with no unusual intermolecular contacts. The Cd atom is pseudo-tetrahedrally coordinated by the two N atoms of the pyridine molecules and by the two  $\eta^1$ -bonded C atoms of the Cp moieties. This is illustrated by the six relevant pseudo-tetrahedral angles which are in the range of  $96.4(1)$ – $129.1(2)^\circ$  [average =  $109(1)^\circ$ ].

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and geometrical data concerning H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43683 (32 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

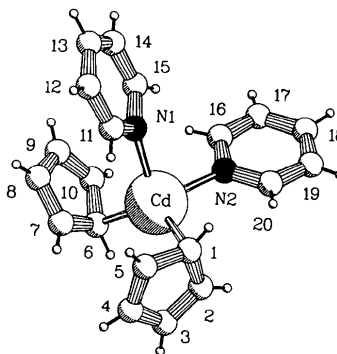


Fig. 1. *PLUTO* (*EUCLID* version; Spek, 1982) drawing of the title compound with the adopted atom numbering.

The Cd—N and Cd—C distances for the four coordinating atoms are 2.307 (5), 2.309 (3), 2.353 (5) and 2.360 (3) Å for C(6), N(1), C(1) and N(2) respectively. Comparable Cd—N distances have been found in other pyridine-containing Cd complexes such as *trans*-diiodotetrakis(pyridine)cadmium(II) with Cd—N distances of 2.368 (6) and 2.454 (9) Å (Ito, Shibata & Saito, 1984), in bis(*o*-hydroxybenzoate)tris(pyridine)-cadmium(II) with Cd—N distances in the range 2.350 (2)–2.372 (2) Å (Charles, Griffith, Rodesiler & Amma, 1983) and in bis(nitrato)tris(pyridine)-cadmium(II) with Cd—N distances of 2.301 (9) and 2.35 (1) Å (Cameron, Taylor & Nuttal, 1972). The deviation of the Cd atom from the least-squares planes through the N(1) and N(2) pyridine rings are 0.50 (1) and 0.119 (4) Å respectively.

Both Cp rings are  $\eta^1$ -bonded to the Cd atom; the angles between the Cd—C( $\eta^1$ ) bonds and the Cp ring planes are 80.1 (2) and 80.9 (3)° for the C(1) and C(6) rings respectively. The ring slippage [ $\equiv$  distance between ring centroid and the perpendicular projection of the Cd atom on the ring plane] is 1.424 (6) and 1.454 (7) Å for the C(1) and C(6) Cp rings respectively.

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(ZWO). The X-ray data were collected by Dr A. J. M. Duisenberg.

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## La Structure à 173 K d'un *tert*-Butyl-3 Cyclohexanediol-1,2 qui Cristallise avec Six Molécules Distinctes

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**Abstract.** C<sub>10</sub>H<sub>20</sub>O<sub>2</sub>,  $M_r = 172.27$ , triclinic,  $P\bar{1}$ ,  $a = 12.640$  (6),  $b = 14.404$  (5),  $c = 18.459$  (6) Å,  $\alpha = 96.99$  (6),  $\beta = 106.00$  (3),  $\gamma = 100.03$  (7)°,  $V = 3129.7$  Å<sup>3</sup>,  $D_x = 1.096$  Mg m<sup>-3</sup>,  $Z = 12$ ,  $F(000) = 1152$ ,  $\lambda(\text{Cu } K\alpha) = 1.54178$  Å,  $\mu(\text{Cu } K\alpha) = 0.55$  mm<sup>-1</sup>,  $T = 173$  K,  $R = 0.075$  for 6142 observed reflections. The cyclohexane ring is in the chair conformation and the *tert*-butyl radical is nearly staggered with respect to the cyclohexane. The average deviation from a perfectly staggered disposition is 15°. The six independent molecules in the asymmetric unit are interconnected through a complex network of hydrogen bonds. The O...O distances in the 12 hydrogen bonds range from 2.691 (4) to 2.832 (4) Å.

**Introduction.** Une première étude cristallographique de ce composé entreprise il y a une dizaine d'années, en capillaire, à la température ambiante, n'avait révélée clairement que cinq des six molécules dans l'unité asymétrique. Une succession de synthèses de Fourier différence et d'affinements faisait difficilement ressortir la sixième molécule. Cependant l'affinement des coordonnées fractionnaires qui se comportait raisonnablement bien pour les cinq premières molécules n'a jamais pu converger de manière satisfaisante pour la sixième molécule. Les longueurs de liaisons et les angles de valence de cette dernière prenaient des valeurs bien trop éloignées de leurs homologues dans les autres molécules. Ayant maintenant à notre disposition un