

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

[N,N'-Bis(4-chlorophenyl)pentane-2,4-diiminato]dicarbonylrhodium(I)

T. N. Hill* and G. Steyl

Department of Chemistry, University of the Free State, Bloemfontein 9300, South Africa

Correspondence e-mail: tania.hill@gmail.com

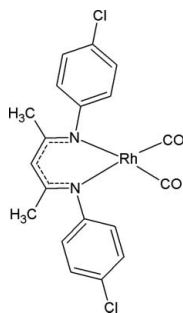
Received 30 October 2009; accepted 5 January 2010

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.017; wR factor = 0.050; data-to-parameter ratio = 17.1.

The title compound, $[\text{Rh}(\text{C}_{17}\text{H}_{15}\text{Cl}_2\text{N}_2)(\text{CO})_2]$, is a rhodium(I) derivative of a β -diketiminato moiety. It is an example of a new type of β -diketiminato derivative that has not yet been characterized *via* solid-state methods. The complex crystallizes with a distorted square-planar geometry about the Rh^{I} atom (m symmetry). A weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ contact is observed.

Related literature

For related diketiminato complexes, see: Smith *et al.* (2002, 2006).



Experimental

Crystal data

$[\text{Rh}(\text{C}_{17}\text{H}_{15}\text{Cl}_2\text{N}_2)(\text{CO})_2]$
 $M_r = 477.14$
 Monoclinic, $P2_1/m$
 $a = 9.6726$ (3) Å
 $b = 7.5911$ (2) Å
 $c = 13.6484$ (4) Å
 $\beta = 107.247$ (1)°

$V = 957.08$ (5) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.19$ mm⁻¹
 $T = 100$ K
 $0.36 \times 0.31 \times 0.30$ mm

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.675$, $T_{\max} = 0.717$
 27556 measured reflections

2555 independent reflections
 2491 reflections with $I > 2\sigma(I)$

 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.017$
 $wR(F^2) = 0.050$
 $S = 0.93$
 2555 reflections
 149 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.36$ e Å⁻³
 $\Delta\rho_{\min} = -0.85$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Rh1—C02	1.854 (2)	Rh1—N2	2.0523 (14)
Rh1—C01	1.8741 (19)	O01—C01	1.134 (2)
Rh1—N1	2.0453 (14)	O02—C02	1.137 (3)
C02—Rh1—C01	85.48 (8)	N1—Rh1—N2	89.87 (6)
C02—Rh1—N1	91.89 (7)	O01—C01—Rh1	176.21 (17)
C01—Rh1—N2	92.76 (7)	O02—C02—Rh1	177.45 (18)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C22—H22 \cdots O02 ⁱ	0.95	2.54	3.1723 (18)	124

Symmetry code: (i) $-x + 1, y + \frac{1}{2}, -z + 1$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT-Plus (Bruker, 2004); data reduction: SAINT-Plus and XPREP (Bruker, 2004); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Putz, 2006); software used to prepare material for publication: WinGX (Farrugia, 1999).

Financial assistance from the University of the Free State and Professor A. Roodt is gratefully acknowledged. Mr. L. Kirsten is acknowledged for the data collection. Part of this material is based on work supported by the South African National Research Foundation (NRF) under grant No. GUN 2068915. Opinions, findings, conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the NRF.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2073).

References

- Brandenburg, K. & Putz, H. (2006). DIAMOND. Crystal Impact GbR, Bonn, Germany.
 Bruker (2004). SAINT-Plus (including XPREP) and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2005). APEX2. Bruker AXS Inc., Madison, Wisconsin, USA.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Smith, J. M., Lachicotte, R. J. & Holland, P. L. (2002). *Organometallics*, **21**, 4808–4814.
 Smith, J. M., Sadique, A. R., Cundari, T. R., Rodgers, K. R., Lukat-Rodgers, G., Lachicotte, R. J., Flaschenriem, C. J., Vela, J. & Holland, P. L. (2006). *J. Am. Chem. Soc.* **128**, 756–769.

supplementary materials

Acta Cryst. (2010). E66, m205 [doi:10.1107/S1600536810000449]

[*N,N'*-Bis(4-chlorophenyl)pentane-2,4-diiminato]dicarbonylrhodium(I)

T. N. Hill and G. Steyl

Comment

In literature similar iron complexes have been reported containing tertiary butyl, isopropyl and methyl derivatives (Smith *et al.*, 2002; Smith *et al.*, 2006). Using a comparable ligand system, with electron withdrawing substituents on the phenyl ring, a series of rhodium(I) dicarbonyl complexes were prepared. The title compound (I) is a novel example of a 4-Chlorophenyl derivative. (Figure 1)

Due to the highly symmetrical nature of the complex both the Rh—C and Rh—N bond distances are similar. The carbonyl oxygen bond distances are the same, with the carbonyl's themselves being close to linearity. The mirror plane bisects the complex passing through the metal centre, the ketimine backbone and carbonyl moieties. (Table 1)

A staggered head-to-tail stacking is observed with no Rh—Rh interaction. The closest contact is a weak hydrogen bond between the phenyl ring (C22) of the diketiminato ligand and the adjacent carbonyl oxygen O02 (Table 2).

Experimental

The title complex was synthesized by the addition of *N,N'*-bis-(4-chlorophenyl)pentane-2,4-di-imine (167 mg, 0.514 mmol) to an acetone solution of the $[\text{Rh}(\mu\text{-Cl})(\text{CO})_2]_2$ (100 mg, 0.257 mmol). On slow evaporation of the solvent; crystals suitable for X-Ray crystallography were obtained. Yield: 147 mg (60%).

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent})$ of the parent atom with a C—H distance of 0.99 (methyl) and 0.95 (aromatic).

Figures

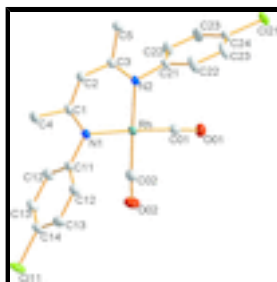


Fig. 1. View of (I) (50% probability displacement ellipsoids)

[*N,N'*-Bis(4-chlorophenyl)pentane-2,4-diiminato]dicarbonylrhodium(I)

Crystal data

[Rh(C ₁₇ H ₁₅ Cl ₂ N ₂)(CO) ₂]	$F(000) = 476$
$M_r = 477.14$	$D_x = 1.656 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2yb	Cell parameters from 7852 reflections
$a = 9.6726 (3) \text{ \AA}$	$\theta = 2.7\text{--}28.3^\circ$
$b = 7.5911 (2) \text{ \AA}$	$\mu = 1.19 \text{ mm}^{-1}$
$c = 13.6484 (4) \text{ \AA}$	$T = 100 \text{ K}$
$\beta = 107.247 (1)^\circ$	Cuboid, colourless
$V = 957.08 (5) \text{ \AA}^3$	$0.36 \times 0.31 \times 0.3 \text{ mm}$
$Z = 2$	

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer	2555 independent reflections
Radiation source: sealed tube graphite	2491 reflections with $I > 2\sigma(I)$
Detector resolution: 512 pixels mm^{-1}	$R_{\text{int}} = 0.026$
ω and φ scans	$\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.2^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.675$, $T_{\text{max}} = 0.717$	$k = -10 \rightarrow 10$
27556 measured reflections	$l = -18 \rightarrow 15$

Refinement

Refinement on F^2	0 restraints
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.017$	$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2 + 0.5478P]$
$wR(F^2) = 0.050$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.93$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2555 reflections	$\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
149 parameters	$\Delta\rho_{\text{min}} = -0.85 \text{ e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Rh1	0.637102 (13)	0.25	0.465229 (9)	0.01730 (6)	
N1	0.70723 (16)	0.25	0.33783 (11)	0.0184 (3)	
N2	0.84799 (16)	0.25	0.55685 (11)	0.0174 (3)	
O01	0.51242 (16)	0.25	0.64255 (11)	0.0306 (3)	
O02	0.32539 (17)	0.25	0.34080 (13)	0.0470 (5)	
Cl11	0.25234 (6)	0.25	-0.06636 (4)	0.04074 (14)	
Cl21	0.87899 (6)	0.25	0.99767 (3)	0.03319 (12)	
C01	0.5641 (2)	0.25	0.57800 (15)	0.0233 (4)	
C1	0.84413 (19)	0.25	0.33584 (13)	0.0183 (3)	
C02	0.4448 (2)	0.25	0.38608 (15)	0.0298 (4)	
C2	0.96380 (18)	0.25	0.42351 (13)	0.0185 (3)	
H2	1.0557	0.25	0.4116	0.022*	
C3	0.96652 (19)	0.25	0.52655 (13)	0.0178 (3)	
C4	0.8736 (2)	0.25	0.23319 (13)	0.0231 (4)	
H4A	0.820 (4)	0.152 (3)	0.1908 (14)	0.035*	0.5
H4B	0.9787 (16)	0.235 (5)	0.2437 (2)	0.035*	0.5
H4C	0.841 (4)	0.363 (3)	0.1977 (13)	0.035*	0.5
C5	1.11412 (19)	0.25	0.60505 (14)	0.0221 (3)	
H5A	1.1388 (9)	0.364 (2)	0.6276 (10)	0.033*	0.5
H5B	1.1816 (12)	0.206 (2)	0.5756 (6)	0.033*	0.5
H5C	1.1122 (5)	0.180 (2)	0.6599 (12)	0.033*	0.5
C11	0.59706 (19)	0.25	0.23963 (13)	0.0206 (3)	
C12	0.54311 (14)	0.09167 (18)	0.19293 (10)	0.0243 (3)	
H12	0.5792	-0.0165	0.2255	0.029*	
C13	0.43608 (14)	0.0908 (2)	0.09835 (10)	0.0278 (3)	
H13	0.3988	-0.0172	0.066	0.033*	
C14	0.3853 (2)	0.25	0.05267 (15)	0.0281 (4)	
C21	0.86559 (18)	0.25	0.66523 (12)	0.0185 (3)	
C22	0.86893 (17)	0.40678 (19)	0.71687 (10)	0.0303 (3)	
H22	0.8683	0.5151	0.6819	0.036*	
C23	0.87317 (17)	0.4078 (2)	0.81978 (10)	0.0322 (3)	
H23	0.8749	0.516	0.8552	0.039*	
C24	0.8748 (2)	0.25	0.86918 (13)	0.0232 (4)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Rh1	0.01599 (8)	0.02468 (8)	0.01164 (8)	0	0.00473 (5)	0
N1	0.0194 (7)	0.0248 (7)	0.0112 (6)	0	0.0046 (5)	0
N2	0.0191 (7)	0.0228 (7)	0.0102 (6)	0	0.0044 (5)	0
O01	0.0300 (7)	0.0437 (8)	0.0230 (7)	0	0.0154 (6)	0
O02	0.0194 (7)	0.0851 (14)	0.0326 (9)	0	0.0018 (6)	0
Cl11	0.0270 (2)	0.0652 (4)	0.0215 (2)	0	-0.00592 (18)	0
Cl21	0.0342 (2)	0.0550 (3)	0.01086 (19)	0	0.00754 (17)	0

supplementary materials

C01	0.0192 (8)	0.0295 (9)	0.0207 (8)	0	0.0052 (7)	0
C1	0.0230 (8)	0.0206 (7)	0.0123 (7)	0	0.0068 (6)	0
C02	0.0240 (9)	0.0461 (12)	0.0206 (9)	0	0.0087 (7)	0
C2	0.0175 (7)	0.0238 (8)	0.0151 (7)	0	0.0063 (6)	0
C3	0.0194 (8)	0.0187 (7)	0.0150 (7)	0	0.0047 (6)	0
C4	0.0241 (9)	0.0332 (9)	0.0138 (8)	0	0.0081 (7)	0
C5	0.0184 (8)	0.0298 (9)	0.0170 (8)	0	0.0038 (6)	0
C11	0.0201 (8)	0.0305 (9)	0.0116 (7)	0	0.0055 (6)	0
C12	0.0253 (6)	0.0302 (6)	0.0170 (5)	-0.0013 (5)	0.0056 (5)	-0.0004 (5)
C13	0.0255 (6)	0.0376 (7)	0.0196 (6)	-0.0056 (6)	0.0055 (5)	-0.0053 (5)
C14	0.0186 (8)	0.0500 (13)	0.0136 (7)	0	0.0013 (6)	0
C21	0.0171 (7)	0.0268 (8)	0.0116 (7)	0	0.0044 (6)	0
C22	0.0500 (8)	0.0255 (6)	0.0179 (6)	-0.0132 (6)	0.0140 (6)	-0.0030 (5)
C23	0.0489 (8)	0.0319 (7)	0.0182 (6)	-0.0150 (6)	0.0135 (6)	-0.0094 (5)
C24	0.0203 (8)	0.0391 (10)	0.0098 (7)	0	0.0041 (6)	0

Geometric parameters (\AA , $^\circ$)

Rh1—C02	1.854 (2)	C4—H4C	0.9902
Rh1—C01	1.8741 (19)	C5—H5A	0.9234
Rh1—N1	2.0453 (14)	C5—H5B	0.9234
Rh1—N2	2.0523 (14)	C5—H5C	0.9234
N1—C1	1.332 (2)	C11—C12	1.3877 (16)
N1—C11	1.444 (2)	C11—C12 ⁱ	1.3877 (16)
N2—C3	1.329 (2)	C12—C13	1.3949 (17)
N2—C21	1.438 (2)	C12—H12	0.95
O01—C01	1.134 (2)	C13—C14	1.3816 (18)
O02—C02	1.137 (3)	C13—H13	0.95
Cl11—C14	1.748 (2)	C14—C13 ⁱ	1.3816 (18)
Cl21—C24	1.7420 (18)	C21—C22	1.3787 (16)
C1—C2	1.397 (2)	C21—C22 ⁱ	1.3787 (16)
C1—C4	1.510 (2)	C22—C23	1.3929 (17)
C2—C3	1.399 (2)	C22—H22	0.95
C2—H2	0.95	C23—C24	1.3727 (17)
C3—C5	1.510 (2)	C23—H23	0.95
C4—H4A	0.9902	C24—C23 ⁱ	1.3727 (17)
C4—H4B	0.9902		
C02—Rh1—C01	85.48 (8)	C3—C5—H5B	109.5
C02—Rh1—N1	91.89 (7)	H5A—C5—H5B	109.5
C01—Rh1—N1	177.37 (6)	C3—C5—H5C	109.5
C02—Rh1—N2	178.24 (7)	H5A—C5—H5C	109.5
C01—Rh1—N2	92.76 (7)	H5B—C5—H5C	109.5
N1—Rh1—N2	89.87 (6)	C12—C11—C12 ⁱ	120.01 (16)
C1—N1—C11	116.46 (14)	C12—C11—N1	119.99 (8)
C1—N1—Rh1	126.83 (12)	C12 ⁱ —C11—N1	119.99 (8)
C11—N1—Rh1	116.72 (11)	C11—C12—C13	120.27 (13)
C3—N2—C21	118.04 (14)	C11—C12—H12	119.9
C3—N2—Rh1	127.13 (12)	C13—C12—H12	119.9

C21—N2—Rh1	114.83 (11)	C14—C13—C12	118.70 (14)
O01—C01—Rh1	176.21 (17)	C14—C13—H13	120.7
N1—C1—C2	123.98 (15)	C12—C13—H13	120.7
N1—C1—C4	118.73 (15)	C13 ⁱ —C14—C13	122.04 (18)
C2—C1—C4	117.29 (16)	C13 ⁱ —C14—Cl11	118.98 (9)
O02—C02—Rh1	177.45 (18)	C13—C14—Cl11	118.98 (9)
C1—C2—C3	128.69 (16)	C22—C21—C22 ⁱ	119.37 (16)
C1—C2—H2	115.7	C22—C21—N2	120.24 (8)
C3—C2—H2	115.7	C22 ⁱ —C21—N2	120.24 (8)
N2—C3—C2	123.51 (15)	C21—C22—C23	120.65 (13)
N2—C3—C5	120.04 (15)	C21—C22—H22	119.7
C2—C3—C5	116.45 (15)	C23—C22—H22	119.7
C1—C4—H4A	109.5	C24—C23—C22	118.87 (14)
C1—C4—H4B	109.5	C24—C23—H23	120.6
H4A—C4—H4B	109.5	C22—C23—H23	120.6
C1—C4—H4C	109.5	C23 ⁱ —C24—C23	121.59 (17)
H4A—C4—H4C	109.5	C23 ⁱ —C24—Cl21	119.20 (8)
H4B—C4—H4C	109.5	C23—C24—Cl21	119.20 (8)
C3—C5—H5A	109.5		
C02—Rh1—N1—C1	180	C1—C2—C3—C5	180
N2—Rh1—N1—C1	0	C1—N1—C11—C12	90.64 (14)
C02—Rh1—N1—C11	0	Rh1—N1—C11—C12	-89.36 (14)
N2—Rh1—N1—C11	180	C1—N1—C11—C12 ⁱ	-90.64 (14)
C01—Rh1—N2—C3	180	Rh1—N1—C11—C12 ⁱ	89.36 (14)
N1—Rh1—N2—C3	0	C12 ⁱ —C11—C12—C13	0.9 (3)
C01—Rh1—N2—C21	0	N1—C11—C12—C13	179.63 (13)
N1—Rh1—N2—C21	180	C11—C12—C13—C14	-0.1 (2)
C11—N1—C1—C2	180	C12—C13—C14—C13 ⁱ	-0.8 (3)
Rh1—N1—C1—C2	0	C12—C13—C14—Cl11	179.46 (11)
C11—N1—C1—C4	0	C3—N2—C21—C22	92.31 (15)
Rh1—N1—C1—C4	180	Rh1—N2—C21—C22	-87.69 (15)
N1—C1—C2—C3	0	C3—N2—C21—C22 ⁱ	-92.31 (15)
C4—C1—C2—C3	180	Rh1—N2—C21—C22 ⁱ	87.69 (15)
C21—N2—C3—C2	180	C22 ⁱ —C21—C22—C23	-1.1 (3)
Rh1—N2—C3—C2	0	N2—C21—C22—C23	174.34 (15)
C21—N2—C3—C5	0	C21—C22—C23—C24	0.4 (3)
Rh1—N2—C3—C5	180	C22—C23—C24—C23 ⁱ	0.4 (3)
C1—C2—C3—N2	0	C22—C23—C24—Cl21	-179.62 (13)

Symmetry codes: (i) $x, -y+1/2, z$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C22—H22 ⁱⁱ —O02 ⁱⁱ	0.95	2.54	3.1723 (18)	124

Symmetry codes: (ii) $-x+1, y+1/2, -z+1$.

Fig. 1

