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

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The title compound, [Rh($C_{17}H_{15}Cl_2N_2$)(CO)$_2$], is a rhodium(I) derivative of a β-diketiminato moiety. It is an example of a new type of β-diketiminate 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 C—H⋯O contact is observed.

Related literature

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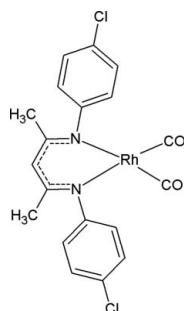
Experimental

Crystal data

[Rh($C_{17}H_{15}Cl_2N_2$)(CO)$_2$]	$V = 957.08$ (5) Å^3
$M_r = 477.14$	$Z = 2$
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation
$a = 9.6726$ (3) Å	$\mu = 1.19$ mm^{-1}
$b = 7.5911$ (2) Å	$T = 100$ K
$c = 13.6484$ (4) Å	$0.36 \times 0.31 \times 0.30$ mm
$\beta = 107.247$ (1)°	

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	$R_{int} = 0.026$
2491 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.017$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.050$	
$S = 0.93$	
2555 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
149 parameters	$\Delta\rho_{min} = -0.85$ 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⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C22—H22⋯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).

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

References

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supporting information

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

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S1. 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 moeities. (Table 1)

A staggared 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).

S2. 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 [Rh(μ-Cl)(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%).

S3. Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2$U_{eq(parent)}$ of the parent atom with a C—H distance of 0.99 (methyl) and 0.95 (aromatic).
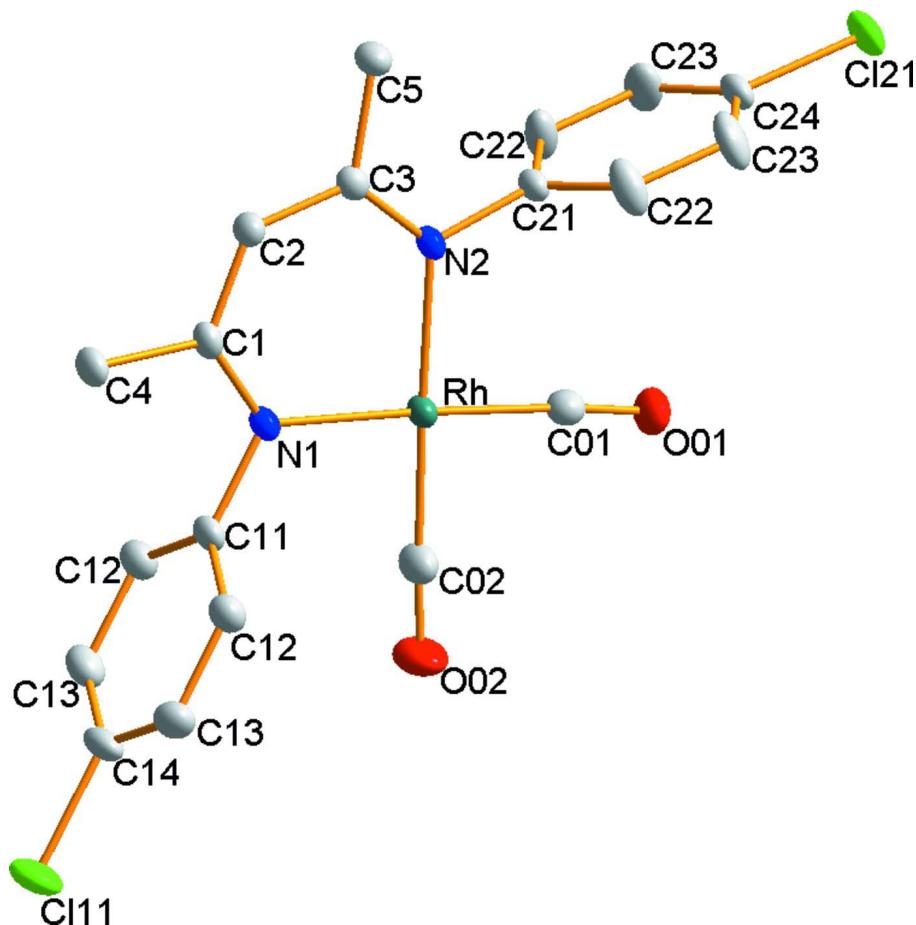
Figure 1

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)$_2$]	$F(000) = 476$
$M_r = 477.14$	$D_x = 1.656$ Mg m^{-3}
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yb	Cell parameters from 7852 reflections
$a = 9.6726$ (3) Å	$\theta = 2.7–28.3°$
$b = 7.5911$ (2) Å	$\mu = 1.19$ mm^{-1}
$c = 13.6484$ (4) Å	$T = 100$ K
$\beta = 107.247$ (1)°	Cuboid, colourless
$V = 957.08$ (5) Å^3	$0.36 \times 0.31 \times 0.3$ mm
$Z = 2$	

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer	Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
Radiation source: sealed tube	$T_{min} = 0.675$, $T_{max} = 0.717$
Graphite monochromator	27556 measured reflections
Detector resolution: 512 pixels mm^{-1}	2555 independent reflections
ω and φ scans	2491 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.026$

$\theta_{max} = 28.3°$, $\theta_{min} = 2.2°$

$h = -12 \rightarrow 12$

$k = -10 \rightarrow 10$

$l = -18 \rightarrow 15$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent
Least-squares matrix: full	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 $P = (F_o^2 + 2F_c^2)/3$
$S = 0.93$	$(\Delta/\sigma)_{max} = 0.001$
2555 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
149 parameters	$\Delta\rho_{min} = -0.85$ e Å^{-3}
0 restraints	

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$	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 (Å^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
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 (Å, º)

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—C23ⁱ	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—C12ⁱ	120.01 (16)
C1—N1—C11	116.46 (14)	C12—C11—N1	119.99 (8)
C1—N1—Rh1	126.83 (12)	C12ⁱ—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)	C13ⁱ—C14—C13	122.04 (18)
C2—C1—C4	117.29 (16)	C13ⁱ—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—C22ⁱ	119.37 (16)
C1—C2—H2	115.7	C22—C21—N2	120.24 (8)
C3—C2—H2	115.7	C22ⁱ—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	C23ⁱ—C24—C23	121.59 (17)
H4A—C4—H4C	109.5	C23ⁱ—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—C12ⁱ	−90.64 (14)
C01—Rh1—N2—C3	180	Rh1—N1—C11—C12ⁱ	89.36 (14)
N1—Rh1—N2—C3	0	C12ⁱ—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—C13ⁱ	−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 code: (i) x, $-y+1/2$, z.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C22—H22···O02[ii]	0.95	2.54	3.1723 (18)	124

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