

cis-Bis(2,2'-bipyridyl)dichlorido-ruthenium(II) dichloromethane solvate

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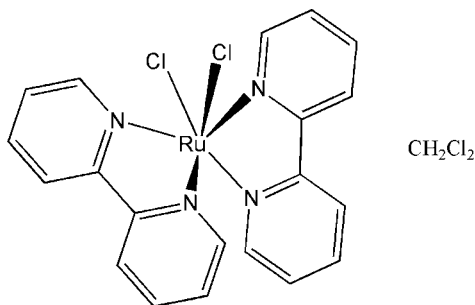
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.027; wR factor = 0.064; data-to-parameter ratio = 19.4.

In the crystal structure of the title compound, $[\text{RuCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)_2]\cdot\text{CH}_2\text{Cl}_2$, the complex consists of two bidentate 2,2'-bipyridyl N-atom donors and two chloride ions coordinated to an Ru^{II} centre which lies on a crystallographic twofold rotation axis. Equivalent ligands are *cis*, each related by the twofold rotation. One dichloromethane solvent molecule per Ru complex is trapped in the crystal structure.

Related literature

For related literature, see: Eggleston *et al.* (1985); Lackner *et al.* (2004); Nag *et al.* (2006).



Experimental

Crystal data

$[\text{RuCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)_2]\cdot\text{CH}_2\text{Cl}_2$
 $M_r = 569.26$
 Orthorhombic, *Ab*a2
 $a = 12.5658$ (9) Å
 $b = 15.4595$ (11) Å
 $c = 11.8864$ (9) Å
 $V = 2309.1$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.16$ mm⁻¹
 $T = 298$ (2) K
 $0.25 \times 0.22 \times 0.03$ mm

Data collection

Bruker/Siemens SMART APEX diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
 $T_{\text{min}} = 0.761$, $T_{\text{max}} = 0.966$
 16972 measured reflections
 2654 independent reflections
 2310 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.064$
 $S = 1.04$
 2654 reflections
 137 parameters
 1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.49$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³
 Absolute structure: Flack (1983), 1260 Freidel pairs
 Flack parameter: -0.01 (5)

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 2006); data reduction: *SAINT-Plus*; program(s) used to solve structure: *XS* in *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *XL* in *SHELXTL*; molecular graphics: *XP* in *SHELXTL*; software used to prepare material for publication: *XCIF* in *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2362).

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supplementary materials

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cis-Bis(2,2'-bipyridyl)dichloridoruthenium(II) dichloromethane solvate

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Comment

The structure of the title compound, (I), is shown in Fig. 1. All bond lengths and angles are normal. The complex has been reported earlier in lower symmetry space groups as the hydrate in *C2/c* (Eggleston *et al.*, 1985), as an acetone solvate in *P21/n* (Lackner *et al.*, 2004), and as an I₂ adduct in *C2/c* (Nag *et al.*, 2006).

The structure of (I) displays distorted octahedral coordination to the metal cation, without any significant hydrogen bonding. The solvent molecule is free in the lattice and displays larger thermal displacements than seen in the Ru complex.

The 2,2'-bipyridyl ligand is planar. The five membered chelate ring is folded about the N1...N12 line with a dihedral angle between the plane of the ligand, and the N1/Ru1/N12 plane of 5.0 (2)°. The dihedral angle between the least square planes of the two fold rotation related 2,2'-bipyridyl ligands is 87.78°. The Cl1—Ru1—Cl1ⁱ [symmetry code: (i) $-x, -y + 2, z$] angle is 93.31 (5)°.

Experimental

1.0 mmole ruthenium trichloride and 2.0 mmol 2,2'-dipyridyl were refluxed in absolute ethanol for 4 h followed with the addition of 11.8 mmol LiCl and further reflux for additional 1 h. The solvent was then removed by rotary evaporator and the crude product dissolved in dichloromethane, filtered and washed by water and reduced to 20 ml. Chromatography on alumina with 10:1 dichloromethane: methanol as eluent yielded 0.2 g of the neutral complex from the second dark-pink band. Suitable crystals of (I) were obtained upon recrystallization from dichloromethane and slow evaporation of solvent.

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H distances fixed at 0.93 Å (aromatic) and 0.97 Å (methylene) and with U_{iso} constrained to be 1.2 U_{eq} of the carrier atom.

Figures

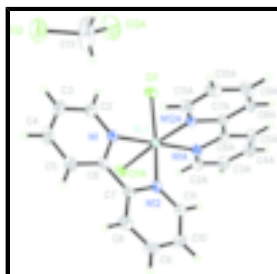


Fig. 1. The molecular structure of the title compound (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

cis-Bis(2,2'-bipyridyl)dichloridoruthenium(II) dichloromethane solvate

Crystal data

[RuCl₂(C₁₀H₈N₂)₂] \cdot CH₂Cl₂

$M_r = 569.26$

Orthorhombic, *Aba2*

Hall symbol: A 2 -2ac

$a = 12.5658$ (9) Å

$b = 15.4595$ (11) Å

$c = 11.8864$ (9) Å

$V = 2309.1$ (3) Å³

$Z = 4$

$F_{000} = 1136$

$D_x = 1.638$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 7381 reflections

$\theta = 2.6$ – 29.6°

$\mu = 1.16$ mm⁻¹

$T = 298$ (2) K

Plate, red

$0.25 \times 0.22 \times 0.03$ mm

Data collection

Bruker/Siemens SMART APEX
diffractometer

Monochromator: graphite

Detector resolution: 8.3 pixels mm⁻¹

$T = 298$ (2) K

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2004)

$T_{\min} = 0.761$, $T_{\max} = 0.966$

16972 measured reflections

2654 independent reflections

2310 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\text{max}} = 27.5^\circ$

$\theta_{\text{min}} = 2.6^\circ$

$h = -16 \rightarrow 16$

$k = -20 \rightarrow 20$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.064$

$S = 1.04$

2654 reflections

137 parameters

1 restraint

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0344P)^2 + 0.0898P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.49$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Extinction correction: none

Absolute structure: Flack (1983), 1260 Freidel pairs

Flack parameter: -0.01 (5)

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C2	0.2168 (3)	0.9265 (2)	0.3118 (3)	0.0486 (8)	
H2	0.2240	0.9752	0.3572	0.058*	
C3	0.2952 (3)	0.8648 (3)	0.3151 (4)	0.0588 (10)	
H3	0.3537	0.8715	0.3622	0.071*	
C4	0.2863 (2)	0.7930 (2)	0.2481 (4)	0.0594 (9)	
H4	0.3387	0.7505	0.2485	0.071*	
C5	0.1983 (3)	0.7851 (2)	0.1802 (3)	0.0507 (9)	
H5	0.1906	0.7368	0.1343	0.061*	
C6	0.1209 (2)	0.84917 (18)	0.1803 (3)	0.0361 (7)	
C7	0.0243 (3)	0.84691 (19)	0.1115 (3)	0.0369 (7)	
C8	0.0016 (3)	0.7830 (2)	0.0331 (3)	0.0488 (9)	
H8	0.0487	0.7373	0.0225	0.059*	
C9	-0.0896 (3)	0.7873 (2)	-0.0284 (3)	0.0569 (9)	
H9	-0.1048	0.7451	-0.0818	0.068*	
C10	-0.1594 (3)	0.8549 (2)	-0.0110 (3)	0.0505 (9)	
H10	-0.2224	0.8587	-0.0519	0.061*	
C11	-0.1340 (3)	0.9165 (2)	0.0681 (3)	0.0424 (7)	
H11	-0.1810	0.9621	0.0798	0.051*	
C13	0.5000	1.0000	0.1365 (7)	0.127 (5)	
H13A	0.5485	1.0318	0.0883	0.152*	0.50
H13B	0.4515	0.9682	0.0883	0.152*	0.50
Cl1	0.07119 (7)	1.09792 (5)	0.38787 (7)	0.0464 (2)	
Cl2	0.57183 (12)	0.92754 (9)	0.21546 (12)	0.1012 (5)	
N1	0.13028 (16)	0.91993 (13)	0.2463 (3)	0.0345 (5)	
N12	-0.0450 (2)	0.91362 (16)	0.1286 (2)	0.0334 (6)	
Ru1	0.0000	1.0000	0.24825 (6)	0.02831 (8)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0427 (18)	0.0461 (19)	0.057 (2)	0.0029 (15)	-0.0107 (17)	-0.0032 (16)
C3	0.0386 (18)	0.065 (2)	0.072 (3)	0.0103 (17)	-0.0118 (18)	0.010 (2)

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C4	0.0447 (17)	0.0552 (19)	0.078 (2)	0.0240 (15)	0.004 (3)	0.008 (3)
C5	0.050 (2)	0.0377 (17)	0.064 (2)	0.0135 (15)	0.0081 (18)	-0.0020 (16)
C6	0.0382 (16)	0.0276 (14)	0.0424 (17)	0.0005 (12)	0.0058 (13)	0.0014 (13)
C7	0.0469 (18)	0.0267 (15)	0.0372 (16)	0.0018 (12)	0.0049 (13)	-0.0023 (13)
C8	0.061 (2)	0.0390 (19)	0.0468 (19)	0.0030 (16)	0.0043 (17)	-0.0140 (16)
C9	0.069 (2)	0.054 (2)	0.048 (2)	-0.0087 (18)	-0.0080 (19)	-0.0144 (17)
C10	0.052 (2)	0.056 (2)	0.0440 (19)	-0.0116 (17)	-0.0157 (17)	0.0007 (17)
C11	0.0425 (17)	0.0387 (17)	0.0460 (18)	0.0010 (14)	-0.0048 (14)	0.0035 (14)
C13	0.190 (11)	0.130 (8)	0.061 (5)	0.101 (7)	0.000	0.000
Cl1	0.0544 (5)	0.0369 (4)	0.0479 (4)	-0.0043 (4)	-0.0010 (4)	-0.0102 (4)
Cl2	0.1190 (12)	0.0895 (8)	0.0952 (11)	0.0280 (8)	-0.0265 (8)	0.0055 (8)
N1	0.0339 (11)	0.0295 (10)	0.0403 (12)	0.0028 (8)	-0.0011 (15)	0.0003 (14)
N12	0.0352 (13)	0.0269 (14)	0.0380 (14)	0.0006 (11)	0.0017 (12)	0.0019 (11)
Ru1	0.03049 (13)	0.02159 (12)	0.03284 (14)	0.00206 (12)	0.000	0.000

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

C2—N1	1.342 (4)	C9—H9	0.9300
C2—C3	1.372 (5)	C10—C11	1.376 (5)
C2—H2	0.9300	C10—H10	0.9300
C3—C4	1.370 (6)	C11—N12	1.330 (4)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.374 (5)	C13—Cl2 ⁱ	1.718 (5)
C4—H4	0.9300	C13—Cl2	1.718 (5)
C5—C6	1.388 (4)	C13—H13A	0.9700
C5—H5	0.9300	C13—H13B	0.9700
C6—N1	1.351 (4)	Cl1—Ru1	2.4179 (9)
C6—C7	1.464 (4)	N1—Ru1	2.052 (2)
C7—N12	1.365 (4)	N12—Ru1	2.031 (3)
C7—C8	1.388 (5)	Ru1—N12 ⁱⁱ	2.031 (3)
C8—C9	1.362 (5)	Ru1—N1 ⁱⁱ	2.052 (2)
C8—H8	0.9300	Ru1—Cl1 ⁱⁱ	2.4179 (9)
C9—C10	1.379 (5)		
N1—C2—C3	123.0 (3)	C10—C11—H11	118.6
N1—C2—H2	118.5	Cl2 ⁱ —C13—Cl2	113.7 (5)
C3—C2—H2	118.5	Cl2 ⁱ —C13—H13A	108.8
C4—C3—C2	119.3 (3)	Cl2—C13—H13A	108.8
C4—C3—H3	120.4	Cl2 ⁱ —C13—H13B	108.8
C2—C3—H3	120.4	Cl2—C13—H13B	108.8
C3—C4—C5	118.6 (3)	H13A—C13—H13B	107.7
C3—C4—H4	120.7	C2—N1—C6	117.9 (3)
C5—C4—H4	120.7	C2—N1—Ru1	126.4 (2)
C4—C5—C6	120.0 (3)	C6—N1—Ru1	115.21 (19)
C4—C5—H5	120.0	C11—N12—C7	118.8 (3)
C6—C5—H5	120.0	C11—N12—Ru1	126.2 (2)
N1—C6—C5	121.2 (3)	C7—N12—Ru1	115.0 (2)
N1—C6—C7	114.5 (2)	N12—Ru1—N12 ⁱⁱ	91.08 (15)

C5—C6—C7	124.3 (3)	N12—Ru1—N1	79.47 (11)
N12—C7—C8	120.4 (3)	N12 ⁱⁱ —Ru1—N1	99.59 (11)
N12—C7—C6	115.3 (3)	N12—Ru1—N1 ⁱⁱ	99.59 (11)
C8—C7—C6	124.2 (3)	N12 ⁱⁱ —Ru1—N1 ⁱⁱ	79.47 (11)
C9—C8—C7	119.9 (3)	N1—Ru1—N1 ⁱⁱ	178.68 (19)
C9—C8—H8	120.0	N12—Ru1—Cl1	174.38 (8)
C7—C8—H8	120.0	N12 ⁱⁱ —Ru1—Cl1	88.07 (7)
C8—C9—C10	119.5 (3)	N1—Ru1—Cl1	95.19 (8)
C8—C9—H9	120.3	N1 ⁱⁱ —Ru1—Cl1	85.72 (8)
C10—C9—H9	120.3	N12—Ru1—Cl1 ⁱⁱ	88.07 (7)
C11—C10—C9	118.6 (3)	N12 ⁱⁱ —Ru1—Cl1 ⁱⁱ	174.38 (8)
C11—C10—H10	120.7	N1—Ru1—Cl1 ⁱⁱ	85.72 (8)
C9—C10—H10	120.7	N1 ⁱⁱ —Ru1—Cl1 ⁱⁱ	95.19 (8)
N12—C11—C10	122.8 (3)	Cl1—Ru1—Cl1 ⁱⁱ	93.31 (5)
N12—C11—H11	118.6		

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

Fig. 1

