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Note

Supporting Information

The Use of Chiral BINAM NHC-Rh(III) Complexes in Enantioselective Hydrosilylation of 3-Oxo-3-arylpropionic Acid Methyl or Ethyl Esters

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General remarks. Dichloromethane and 1,2-dichloroethane were freshly distilled from calcium hydride; THF and toluene were distilled from sodium (Na) under argon (Ar) atmosphere. Melting points were determined on a digital melting point apparatus and temperatures were uncorrected. Optical rotations were determined at 589 nm (sodium D line) by using a Perkin-Elmer-341 MC digital polarimeter; $[\alpha]_D$ -values are given in unit of $10 \text{ deg}^{-1} \text{ cm}_2 \text{ g}^{-1}$. ^1H NMR spectra were recorded on a Varian Mercury 300 MHz spectrometer for solution in CDCl_3 with tetramethylsilane (TMS) as an internal standard; coupling constants J are given in Hz. ^{13}C NMR spectra were recorded on a Varian Mercury 75 MHz spectrophotometers with complete proton decoupling spectrophotometers (CDCl_3 : 77.0 ppm). Infrared spectra were recorded on a Perkin-Elmer PE-983 spectrometer with absorption in cm^{-1} . Flash column chromatography was performed using 300-400 mesh silica gel. For thin-layer chromatography (TLC), silica gel plates (Huanghai GF₂₅₄) were used. Chiral HPLC was performed on a SHIMADZU SPD-10A *vp* series with chiral columns (Chiralpak AS-H, AD-H and Chiracel OD-H columns 4.6 x 250mm, (Daicel Chemical Ind., Ltd.)). Elementary analysis was taken on a Carlo-Erba 1106 analyzer. Mass spectra were recorded by EI, and HRMS was measured on a HP-5989 instrument. 3-Oxo-3-arylpropionic acid esters were prepared according to literature procedures.¹

General procedure for the synthesis of catalysts and the data of compounds SI-2, SI-3, SI-4, and NHC-Rh(III) complex 1 as well as SI-7, SI-8, SI-9, SI-10, and NHC-Rh(III) complex 2.

NHC-Rh(III) complex 1 was synthesized according to the literature.²

Compound SI-2. This is a known compound.² A red solid; Mp. $202.8\text{--}203.3^\circ\text{C}$; $[\alpha]_{\text{D}}^{20} -496.0$ (c 0.22, CHCl_3); ^1H NMR (300 MHz, CDCl_3 , TMS): δ 6.58–6.64 (m, 2H, ArH), 7.10–7.23 (m, 6H, ArH), 7.32–7.36 (m, 2H, ArH), 7.46–7.52 (m, 2H, ArH), 7.69 (d, $J = 8.7$ Hz, 2H, ArH), 7.92–7.97 (m, 4H, ArH), 8.02 (d, $J = 8.7$ Hz, 2H, ArH), 9.03 (s, 2H, NH).

Compound SI-3. This is a known compound.² A white solid; Mp. 257.2-258.4 °C; $[\alpha]_D^{20} +200.7$ (c 0.39, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 4.37 (br, 4H, NH₂), 5.20 (br, 2H, NH), 6.75-6.80 (m, 4H, ArH), 7.00-7.08 (m, 4H, ArH), 7.15 (d, J = 9.0 Hz, 2H, ArH), 7.24-7.29 (m, 6H, ArH), 7.82-7.84 (m, 4H, ArH).

Compound SI-4. This is a known compound.² A white solid; Mp. 294.5-294.8 °C; $[\alpha]_D^{20} +516.7$ (c 0.97, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 6.12 (d, 2H, J = 8.7 Hz, ArH),

6.51 (t, 2H, $J = 7.5$ Hz, ArH), 6.94-6.97 (m, 2H, ArH), 7.00 (s, 2H, CH), 7.45 (d, $J = 8.7$ Hz, 2H, ArH), 7.49-7.58 (m, 6H, ArH), 7.65-7.70 (m, 2H, ArH), 8.08 (d, $J = 8.7$ Hz, 4H, ArH).

NHC-Rh (III) complex 1. This is a known compound.² A yellow solid; Mp. > 300 °C (dec.); $[\alpha]_D^{20}$ -8.1 (c 1.06, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 2.02 (s, 3H, CH₃), 4.28 (s, 6H, CH₃), 6.70 (d, $J = 8.1$ Hz, 2H, ArH), 6.90-7.12 (m, 10H, ArH), 7.28-7.33 (m, 2H, ArH), 7.74 (d, $J = 8.1$ Hz, 2H, ArH), 7.94 (d, $J = 8.7$ Hz, 2H, ArH), 8.36 (d, $J = 8.7$ Hz, 2H, ArH).

Synthesis

of

(R)-(+)-N-(2-nitrophenyl)-1-(2-(2-nitrophenylamino)-5,6,7,8-tetrahydronaphthalen-1-yl)

-5,6,7,8-tetrahydronaphthalen-2-amine SI-7. Under argon atmosphere, a mixture of

(R)-1-(2-amino-5,6,7,8-tetrahydronaphthalen-1-yl)-5,6,7,8-tetrahydronaphthalen-2-amine

(R)-**SI-6** (146 mg, 0.50 mmol), 2-bromo-nitrobenzene (303 mg, 1.5 mmol), Pd₂(dba)₃ (12 mg,

0.0125 mmol), DPE-phos (20 mg, 0.0375 mmol), and Cs₂CO₃ (520 mg, 1.6 mmol) were

stirred in anhydrous toluene (4 mL) at 80 °C for 48 h. After the reaction mixture was cooled to

room temperature, the reaction was quenched by addition of 10 mL of H₂O. The organic

compound was extracted with EtOAc (2 x 20 mL) and dried over anhydrous Na₂SO₄. The solvent was removed under reduced pressure and the residue was purified by a silica gel flash column chromatography (eluent: petroleum ether/ethyl acetate = 20/1) to remove excess raw material, and then with eluent: petroleum ether/ethyl acetate = 4/1 to give **SI-7** as a red solid; Yield: 253 mg (95%). Mp. 192.8-193.5 °C; $[\alpha]_D^{20}$ -539.0 (c 0.25, CHCl₃); IR (CH₂Cl₂): ν 3342, 2932, 2858, 1616, 1498, 1573, 1505, 1254, 1146, 738 cm⁻¹; ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.72-1.81 (m, 8H, CH₂), 2.29-2.34 (m, 4H, CH₂), 2.82-2.88 (m, 4H, CH₂), 6.52-6.58 (m, 2H, ArH), 7.02-7.05 (m, 2H, ArH), 7.13 (d, *J* = 8.4 Hz, 2H, ArH), 7.21 (d, *J* = 8.4 Hz, 2H, ArH), 7.99 (d, *J* = 9.3 Hz, 2H, ArH), 8.90 (s, 2H, NH); ¹³C NMR (75 MHz, CDCl₃): δ 22.8, 23.0, 27.6, 29.6, 115.5, 117.0, 120.8, 126.3, 129.5, 131.3, 133.6, 135.4, 135.5, 137.0, 142.5; MS (EI) *m/e* (%): 534 (M⁺, 52), 499 (M⁺-35, 100), 465 (M⁺-69, 15), 341 (M⁺-185, 21); Anal. Calcd. for C₃₂H₃₀N₄O₄ requires: C 71.89, H 5.66, N 10.48%. Found: C 71.80, H 5.54, N 10.33%.

Synthesis of (R)-(-)-N¹-(1-(2-(2-aminophenylamino)-5,6,7,8-tetrahydro-naphthalen-1-yl))

-5,6,7,8-tetrahydronaphthalen-2-yl)benzene-1,2-diamine SI-8. A mixture of **SI-7** (134 mg, 0.25 mmol), 10% Pd-C (15 mg) in a mixed solution of EtOAc and EtOH (15 mL, 1/1) were stirred under H₂ atmosphere (15 atm) at 60 °C for 6 h. After cooling to room temperature, Pd-C was removed by filtration. The solvent was evaporated under reduced pressure. The residue was purified by a silica gel flash column chromatography (eluent: petroleum ether /ethyl acetate = 2/1~1/1) to give **SI-8** as a white solid; Yield: 109 mg (92%). Mp. 96.9-97.9 °C; IR (CH₂Cl₂): ν 3368, 2925, 2856 $\square$ 1913, 1613, 1501, 1301, 744 cm⁻¹; ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.72-1.78 (m, 8H, CH₂), 2.35-2.43 (m, 4H, CH₂), 2.74-2.76 (m, 4H, CH₂), 3.76 (br, 4H, NH₂), 4.88 (br, 2H, NH), 6.53 (d, *J* = 8.1 Hz, 2H, ArH), 6.70-6.77 (m, 4H, ArH), 6.97-7.04 (m, 6H, ArH); ¹³C NMR (75 MHz, CDCl₃): δ 23.2, 23.4, 27.4, 29.3, 111.4, 115.9, 118.9, 122.0, 126.0, 126.4, 128.28, 128.32, 129.5, 136.3, 141.1, 142.7; MS (EI) *m/e* (%): 474 (M⁺, 100), 367 (M⁺-107, 19), 325 (M⁺-149, 6); HRMS (EI) Calcd. for C₃₂H₃₄N₄ (M⁺) requires 474.2783, Found: 474.2740.

(R)-(+)-1-(5-(6-(1H-benzo[d]imidazol-1-yl)-1,2,3,4-tetra-hydronaphthalen-5-yl)-1,2,3,4-tetrahydronaphthalen-6-yl)-1H-benzo[d]imidazole SI-9. The compound **SI-8** (237 mg, 0.50 mmol) and triethyl orthoformate [$\text{HC}(\text{OC}_2\text{H}_5)_3$] (5.0 mL) containing a little TsOH were heated at 100 °C for 3 h. After the excess amount of triethyl orthoformate was removed under reduced pressure, the residue was purified by a silica gel flash column chromatography (eluent: petroleum ether/ethyl acetate = 2/3) to give **SI-9** as a white solid; Yield: 222 mg (90%). Mp. 330.0-330.8 °C; $[\alpha]_D^{20} +312.1$ (c 1.005, CHCl_3); IR (CH_2Cl_2): ν 3728, 3630, 2940, 1612, 1498, 1455, 1286, 1237, 737 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.82-1.93 (m, 8H, CH_2), 2.56-2.59 (m, 4H, CH_2), 2.92-2.96 (m, 4H, CH_2), 6.11 (d, 2H, $J = 8.4$ Hz, ArH), 6.50 (t, 2H, $J = 7.5$ Hz, ArH), 6.94 (t, 2H, $J = 7.5$ Hz, ArH), 7.02 (d, 2H, $J = 8.1$ Hz, ArH), 7.19 (d, 2H, $J = 8.1$ Hz, ArH), 7.24 (s, 2H, NCHN), 7.49 (d, $J = 8.4$ Hz, 2H, ArH); ^{13}C NMR (75 MHz, CDCl_3): δ 22.6, 22.7, 28.4, 29.5, 109.1, 119.0, 122.0, 123.3, 123.8, 130.4, 130.9, 132.0, 134.1, 138.30, 138.32, 141.3, 142.2; MS (EI) m/e (%): 494 (M^+ , 100), 493 (M^+-1 , 33), 452 (M^+-42 , 4), 375 (M^+-119 , 6); Anal. Calcd. for $\text{C}_{34}\text{H}_{30}\text{N}_4$ requires: C 82.56, H 6.11, N 11.33%. Found: C 82.41, H 5.96, N 11.09%.

Synthesis of (S)-1,1-(1,1'-binaphthyl)-3,3'-dimethyldibenzimidazolium diiodide **SI-10.** The compound **SI-9** (99 mg, 0.20 mmol) and CH₃I (0.24 mL, 4 mmol) in CH₃CN (4 mL) were stirred under reflux for 5 h. After cooling to room temperature, volatiles were removed under reduced pressure and the obtained solid compound **SI-10** was used for the next reaction without further purification. MS (ESI) *m/e*: 651.1 (M⁺-I), 262.1 (M⁺-2I)/2.

Synthesis of NHC-Rh(III) complex **2.** A mixture of **SI-10** (156 mg, 0.20 mmol), [RhCl(COD)]₂ (48 mg, 0.10 mmol), NaOAc (132 mg, 0.80 mmol), and KI (66 mg, 0.40 mmol) was stirred in CH₃CN (12 mL) under reflux for 24 h. After cooling, volatiles were removed under reduced pressure and the residue was purified by a silica gel flash column chromatography (eluent: hexane/ethyl acetate = 1/1) to an orange solid Rh(III) complex **2**. Yield: 51 mg (27%). A crystal suitable for X-ray analysis was obtained by recrystallization from hexane/THF (1/1). Mp. > 300 °C (dec.); [α]_D²⁰ 72.0 (c 0.25, CHCl₃); IR (CH₂Cl₂): ν 3727, 3630, 2933, 2858, 1721, 1465, 1335, 1090, 743 cm⁻¹; ¹H NMR (300 MHz, CDCl₃,

TMS): δ 1.35-1.57 (m, 8H, CH₂), 1.93-2.05 (m, 4H, CH), 2.00 (s, 3H, CH₃), 2.50-2.64 (m, 4H, CH), 4.36 (s, 6H, CH₃), 6.73 (d, J = 8.1 Hz, 2H, ArH), 7.04-7.11 (m, 2H, ArH), 7.21 (t, J = 7.2 Hz, 2H, ArH), 7.30 (t, J = 8.1 Hz, 2H, ArH), 7.97 (d, J = 8.1 Hz, 2H, ArH) ; ¹³C NMR (75 MHz, CDCl₃): δ 22.0, 22.2, 24.96, 24.98, 27.1, 29.1, 38.0, 109.8, 111.5, 122.6, 123.2, 127.7, 129.9, 133.1, 134.66, 134.69, 134.9, 135.7, 136.3, 139.0; MS (EI) m/z (%): 879 (M⁺-OAc, 38), 811 (M⁺-I, 100); Anal. Calcd. for C₃₈H₃₇I₂N₄O₂Rh requires: C 48.63, H 3.97, N 5.97%. Found: 49.02, H 4.18, N 5.82%.

General Procedure for NHC-Rh(III)-catalyzed Enantioselective Hydrosilylation

Reaction:

Under an Ar atmosphere, 3-oxo-3-arylpropionic acid esters (0.5 mmol) and Ph_2SiH_2 (138 mg, 0.75 mmol) were added to a solution of chiral Rh complex (0.01 mmol) in 2 mL of anhydrous THF. The reaction mixture was stirred at 15 °C for about 72 h. The reaction was quenched by addition of H_2O (1 mL) and 0.5 N HCl (0.5 mL). The resulting aqueous solution was stirred for 2~5 hr at room temperature and extracted with Et_2O (2 x 10 mL) and dried over anhydrous Na_2SO_4 . The solvent was removed under reduced pressure and the residue was purified by a silica gel flash column chromatography (eluent: pentane/ Et_2O = 10/1~4/1) to give the corresponding 3-hydroxy-3-arylpropionic acid methyl or ethyl esters. The enantiomeric excess of the obtained 3-hydroxy-3-arylpropionic acid methyl or ethyl esters was determined by chiral HPLC.

¹H NMR spectroscopic data of compounds of 5a-5i.

3-Hydroxy-3-phenylpropionic acid ethyl ester (5a).⁴ A colorless liquid; $[\alpha]_D^{20}$ -39.3 (c 1.01, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.27 (t, J = 7.2 Hz, 3H, CH₃), 2.68-2.73 (m, 2H, CH₂), 3.56 (br, 1H, OH), 4.14 (q, J = 7.2 Hz, 2H, CH₂), 5.13 (m, 1H, CH), 7.27-7.38 (m, 5H, ArH).

3-Hydroxy-3-(4-methylphenyl)propionic acid ethyl ester (5b).⁵ A colorless liquid; $[\alpha]_D^{20}$ -50.1 (c 1.00, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.27 (t, J = 7.2 Hz, 3H, CH₃), 2.35 (s, 3H, CH₃), 2.66-2.80 (m, 2H, CH₂), 2.94 (br, 1H, OH), 4.18 (q, J = 7.2 Hz, 2H, CH₂), 5.08-5.12 (m, 1H, CH), 7.17 (d, J = 7.8 Hz, 2H, ArH), 7.27 (d, J = 7.8 Hz, 2H, ArH).

3-Hydroxy-3-(2-methylphenyl)propionic acid ethyl ester (5c).⁵ A colorless liquid; $[\alpha]_D^{20}$ -87.9 (c 1.00, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.22 (t, J = 7.2 Hz, 3H, CH₃), 2.28 (s, 3H, CH₃), 2.59-2.62 (m, 2H, CH₂), 3.15 (br, 1H, OH), 4.14 (q, J = 7.2 Hz, 2H, CH₂), 5.27-5.31 (m, 1H, CH), 7.07 -7.20 (m, 3H, ArH), 7.43-7.46 (m, 1H, ArH).

3-Hydroxy-3-(3-methylphenyl)propionic acid ethyl ester (5d).⁶ A colorless liquid; $[\alpha]_D^{20}$

-51 (c 1.00, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.26 (t, *J* = 7.2 Hz, 3H, CH₃), 2.35 (s, 3H, CH₃), 2.68-2.77 (m, 2H, CH₂), 3.27 (br, 1H, OH), 4.18 (q, *J* = 7.2 Hz, 2H, CH₂), 5.08-5.11 (m, 1H, CH), 7.08-7.26 (m, 4H, ArH).

3-Hydroxy-3-(2-methoxyphenyl)propionic acid ethyl ester (5e).⁵ A colorless liquid; [α]_D²⁰ -47.0 (c 0.89, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.27 (t, *J* = 6.9 Hz, 3H, CH₃), 2.66-2.86 (m, 2H, CH₂), 3.50 (br, 1H, OH), 3.85 (s, 3H, CH₃), 4.18 (q, *J* = 6.9 Hz, 2H, CH₂), 5.34-5.38 (m, 1H, CH), 6.86-7.01 (m, 2H, ArH), 7.24-7.45 (m, 2H, ArH).

3-Hydroxy-3-(4-bromophenyl)propionic acid ethyl ester (5f).⁵ A colorless liquid; $[\alpha]_D^{20}$ -57.9 (c 1.12, CHCl_3); ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.26 (t, $J = 6.9$ Hz, 3H, CH_3), 2.67-2.70 (m, 2H, CH_2), 3.43 (br, 1H, OH), 4.16 (q, $J = 6.9$ Hz, 2H, CH_2), 5.08 (m, 1H, CH), 7.24 (d, $J = 7.8$ Hz, 2H, ArH), 7.47 (d, $J = 7.8$ Hz, 2H, ArH).

3-Hydroxy-3-(4-chlorophenyl)propionic acid ethyl ester (5g).⁵ A colorless liquid; $[\alpha]_D^{20}$ -55.7 (c 1.00, CHCl_3); ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.27 (t, $J = 7.2$ Hz, 3H, CH_3), 2.69-2.71 (m, 2H, CH_2), 3.41 (br, 1H, OH), 4.19 (q, $J = 7.2$ Hz, 2H, CH_2), 5.09-5.13 (m, 1H, CH), 7.32 (s, 4H, ArH).

3-Hydroxy-3-(2-chlorophenyl)propionic acid ethyl ester (5h).⁷ A colorless liquid; $[\alpha]_D^{20}$ -35.5 (c 1.00, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.21 (t, J = 7.2 Hz, 3H, CH₃), 2.46-2.55 (m, 1H, CH₂), 2.76-2.82 (m, 1H, CH₂), 3.46 (br, 1H, OH), 4.18 (q, J = 7.2 Hz, 2H, CH₂), 5.09 (m, 1H, CH), 7.22-7.28 (m, 3H, ArH), 7.38 (m, 1H, ArH).

3-Hydroxy-3-(4-chlorophenyl)propionic acid methyl ester (5i).⁸ A colorless liquid; $[\alpha]_D^{20}$ -28.4 (c 1.00, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 2.71-2.73 (m, 2H, CH₂), 3.30 (br,

1H, OH), 3.73 (s, 3H, CH₃), 5.09-5.14 (m, 1H, CH), 7.33 (s, 4H, ArH).

HPLC trace of compounds 5a, 5b, 5c, 5d, 5e, 5f, 5g, 5h, and 5i.

3-Hydroxy-3-phenyl-propionic acid ethyl ester (5a).

WH-500 色谱分析报告

WH-500 色谱分析报告

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		11.498	102404	2302017.5	89.6765	1.89	5214
2		15.537	8907	265007.3	10.3235	1.62	5435
Σ:			111311	2567024.8	100.0000		

3-Hydroxy-3-(4-methylphenyl)-propionic acid ethyl ester (5b).

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		31.577	14857	1220253.4	49.8101	3.00	2946
2		35.863	11953	1229559.1	50.1899	3.28	2423
Σ:			26810	2449812.5	100.0000		

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		30.200	25052	2739255.0	97.2013	3.83	1520
2		37.663	587	78869.5	2.7987	1.38	1566
Σ:			25639	2818124.5	100.0000		

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		23.993	25949	1754582.6	49.9393	3.23	2510
2		27.178	20070	1758844.7	50.0607	3.47	1917
Σ:			46019	3513427.3	100.0000		

WH-500 色谱分析报告

3-Hydroxy-3-(2-methylphenyl)-propionic acid ethyl ester (5c).

WH-500 色谱分析报告

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		24.913	1519	50026.6	3.9608	1.01	11405
1		29.053	26538	1213002.7	96.0392	0.80	8052
Σ:			28057	1263029.3	100.0000		

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		24.378	2040	62708.3	4.1566	1.31	12536
2		28.332	37463	1445916.7	95.8434	1.53	10740
Σ:			39503	1508625.0	100.0000		

3-Hydroxy-3-(3-methylphenyl)-propionic acid ethyl ester (5d).

WH-500 色谱分析报告

WH-500 色谱分析报告

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		9.952	110093	2048008.1	99.2866	1.79	5704
2		13.012	678	14716.5	0.7134	0.87	7162
Σ:			110771	2062724.5	100.0000		

3-Hydroxy-3-(2-methoxyphenyl)-propionic acid ethyl ester (5e).

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		26.575	28441	2128498.3	49.5263	2.29	2513
2		30.785	24092	2169218.5	50.4737	2.53	2330
Σ:			52533	4297716.8	100.0000		

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		25.943	54547	5242237.5	97.7739	2.66	1452
2		31.982	1775	119356.0	2.2261	1.32	4509
	Σ:		56322	5361593.5	100.0000		

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		26.130	37967	2221427.9	95.0323	2.26	3979
1		30.498	2271	116122.7	4.9677	1.16	7091
	Σ:		40258	2337550.6	100.0000		

3-Hydroxy-3-(4-bromophenyl)-propionic acid ethyl ester (5f).

WH-500 色谱分析报告

WH-500 色谱分析报告

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
1		32.217	33900	1533373.0	97.7560	1.34	10111
2		35.603	828	35198.6	2.2440	1.02	13980
Σ:			34728	1568571.4	100.0000		

3-Hydroxy-3-(4-chlorophenyl)-propionic acid ethyl ester (5g).

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
2		34.412	13420	634054.3	49.8606	1.13	10573
2		38.292	11774	637599.8	50.1394	0.84	9965
Σ:			25194	1271654.1	100.0000		

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
2		34.613	37038	1806673.8	98.5917	0.98	10036
2		38.200	502	25806.0	1.4083	1.16	11006
Σ:			37540	1832479.8	100.0000		

WH-500 色谱分析报告

ID	组分名	保留时间	峰高	峰面积	浓度	拖尾因子	理论塔板
2		34.408	31361	1472268.6	98.4666	1.04	10707
2		37.947	426	22927.4	1.5334	0.00	9908
Σ:			31787	1495196.0	100.0000		

3-Hydroxy-3-(2-chlorophenyl)-propionic acid ethyl ester (5h).

WH-500 色谱分析报告

WH-500 色谱分析报告

WH-500 色谱分析报告

3-Hydroxy-3-(4-chlorophenyl)-propionic acid methyl ester (5i).

WH-500 色谱分析报告

WH-500 色谱分析报告

WH-500 色谱分析报告

X-ray crystal data of NHC-Rh(III) complex **2**.

The crystal data of complex **2** has been deposited in CCDC with number 287294. Empirical Formula: $\text{C}_{38}\text{H}_{37}\text{I}_2\text{N}_4\text{O}_2\text{Rh}$; Formula Weight: 938.43; Crystal Color, Habit: colorless, prismatic; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: $a = 9.843(2)\text{\AA}$, $b = 19.500(4)\text{\AA}$, $c = 19.388(4)\text{\AA}$, $\alpha = 90^\circ$, $\beta = 96.280(5)^\circ$, $\gamma = 90^\circ$, $V = 3698.8(15)\text{\AA}^3$; Space group: $P2(1)$; $Z = 4$; $D_{\text{calc}} = 1.685\text{ g/cm}^3$; $F_{000} = 1840$; Diffractometer: Rigaku AFC7R; Residuals: R ; R_w : 0.1014, 0.2643.

Table 1. Crystal data and structure refinement for cd25468.

Identification code	cd25468
Empirical formula	C38 H37 I2 N4 O2 Rh
Formula weight	938.43
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)
Unit cell dimensions	a = 9.843(2) Å alpha = 90 deg. b = 19.500(4) Å beta = 96.280(5) deg. c = 19.388(4) Å gamma = 90 deg.
Volume	3698.8(15) Å ³
Z, Calculated density	4, 1.685 Mg/m ³
Absorption coefficient	2.169 mm ⁻¹
F(000)	1840
Crystal size	0.486 x 0.297 x 0.260 mm
Theta range for data collection	1.49 to 25.49 deg.
Limiting indices	-11<=h<=9, -22<=k<=23, -20<=l<=23
Reflections collected / unique	19004 / 12131 [R(int) = 0.1352]
Completeness to theta = 25.49	99.4 %
Absorption correction	Empirical
Max. and min. transmission	1.0000 and 0.7593
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	12131 / 19 / 809
Goodness-of-fit on F ²	1.019
Final R indices [I>2sigma(I)]	R1 = 0.1014, wR2 = 0.2643
R indices (all data)	R1 = 0.1202, wR2 = 0.2781
Absolute structure parameter	0.08(5)
Extinction coefficient	0.0047(6)
Largest diff. peak and hole	3.481 and -1.220 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd25468. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Rh(1)	12077(2)	10880(1)	-64(1)	37(1)
Rh(2)	2271(2)	2205(1)	5045(1)	41(1)
I(1)	10409(2)	11812(1)	-720(1)	57(1)
I(2)	14083(2)	10100(1)	556(1)	53(1)
I(3)	3699(2)	1280(1)	4396(1)	70(1)
I(4)	537(2)	2985(1)	5658(1)	60(1)
O(1)	13524(18)	11219(9)	-734(9)	59(4)
O(2)	13330(20)	11770(8)	241(9)	62(4)
O(3)	1195(19)	1320(8)	5369(9)	65(5)
O(4)	445(17)	1834(10)	4355(8)	64(5)
N(1)	11101(16)	9384(6)	-272(7)	30(3)
N(2)	11513(18)	9897(8)	-1197(8)	41(4)
N(3)	9490(20)	10729(9)	635(7)	49(5)
N(4)	11171(17)	11066(9)	1331(8)	42(4)
N(5)	2332(17)	3153(10)	3853(9)	51(5)
N(6)	3182(17)	3694(9)	4790(8)	40(4)
N(7)	5127(18)	2355(7)	5758(7)	35(4)
N(8)	3688(17)	1993(8)	6428(8)	38(4)
C(1)	11447(18)	9997(9)	-524(8)	30(4)
C(2)	11300(20)	9221(10)	-1377(7)	43(5)
C(3)	11300(30)	8849(11)	-1989(11)	73(8)
C(4)	11350(30)	8133(11)	-1962(11)	69(8)
C(5)	11020(30)	7842(12)	-1348(11)	67(7)
C(6)	10930(30)	8176(8)	-710(11)	59(7)
C(7)	11170(20)	8868(9)	-779(9)	48(5)
C(8)	11770(30)	10379(11)	-1765(10)	62(7)
C(9)	10833(17)	10854(9)	681(9)	30(4)
C(10)	8970(30)	10874(12)	1269(12)	58(6)
C(11)	7640(30)	10882(13)	1465(14)	72(8)
C(12)	7500(40)	11074(14)	2130(20)	95(12)
C(13)	8710(50)	11250(15)	2616(18)	99(13)
C(14)	9990(30)	11264(11)	2396(10)	59(6)
C(15)	10080(30)	11070(10)	1682(13)	55(6)
C(16)	12560(30)	11382(12)	1596(14)	72(8)
C(17)	10690(20)	9176(9)	411(9)	37(4)
C(18)	11710(30)	8798(13)	844(10)	57(6)
C(19)	11160(20)	8485(9)	1419(10)	48(6)
C(20)	9910(20)	8586(10)	1565(9)	35(4)
C(21)	9300(30)	8247(10)	2207(10)	51(6)
C(22)	7950(40)	8490(20)	2400(20)	114(19)
C(23)	7080(40)	8700(30)	1862(16)	111(13)
C(24)	7450(20)	9072(13)	1256(13)	58(6)
C(25)	8980(20)	8984(11)	1147(10)	46(5)
C(26)	9420(20)	9288(12)	555(10)	47(5)
C(27)	8408(19)	9709(9)	42(8)	32(4)
C(28)	8606(19)	10389(9)	84(9)	39(5)
C(29)	7749(16)	10851(9)	-250(8)	31(3)
C(30)	6780(20)	10543(12)	-747(10)	75(10)
C(31)	6700(20)	9818(13)	-884(10)	53(6)
C(32)	5430(30)	9506(15)	-1373(15)	72(8)
C(33)	5180(40)	8734(16)	-1340(20)	111(17)
C(34)	6480(40)	8340(20)	-1100(30)	110(30)
C(35)	7370(30)	8613(13)	-504(12)	57(6)
C(36)	7450(20)	9404(10)	-471(9)	36(4)
C(37)	13800(30)	11651(15)	-290(15)	71(7)
C(38)	14870(40)	12149(19)	-510(19)	107(11)
C(39)	2680(30)	3021(10)	4526(10)	49(5)
C(40)	2430(20)	3799(10)	3655(10)	39(5)
C(41)	2220(40)	4156(16)	2995(13)	82(9)
C(42)	2380(30)	4860(18)	3000(18)	93(10)
C(43)	2940(40)	5205(12)	3605(15)	77(9)

C(44)	3160(30)	4908(14)	4226(15)	74(8)
C(45)	2940(20)	4161(10)	4231(11)	44(5)
C(46)	1850(30)	2630(12)	3355(10)	54(6)
C(47)	3830(30)	2215(11)	5785(9)	48(5)
C(48)	5042(19)	1919(9)	6834(10)	41(4)
C(49)	5430(30)	1690(11)	7487(13)	59(6)
C(50)	6820(40)	1706(15)	7692(12)	91(12)
C(51)	7730(30)	1943(13)	7250(11)	77(9)
C(52)	7360(20)	2200(11)	6561(13)	51(5)
C(53)	5940(20)	2162(11)	6401(10)	46(5)
C(54)	2450(20)	1744(13)	6718(11)	55(6)
C(55)	3772(16)	3911(7)	5453(9)	34(3)
C(56)	2970(20)	4355(9)	5835(9)	49(5)
C(57)	3510(30)	4664(12)	6438(10)	64(7)
C(58)	4960(20)	4567(10)	6637(12)	51(5)
C(59)	5550(40)	4910(20)	7237(13)	96(12)
C(60)	6930(30)	4657(17)	7553(16)	97(13)
C(61)	7900(60)	4579(19)	6924(18)	110(20)
C(62)	7210(30)	4029(18)	6463(14)	82(10)
C(63)	5670(20)	4129(12)	6255(9)	51(5)
C(64)	5180(17)	3822(9)	5651(8)	32(4)
C(65)	5970(20)	3384(10)	5188(9)	43(5)
C(66)	5830(20)	2691(9)	5231(8)	40(5)
C(67)	6557(15)	2223(7)	4911(9)	43(5)
C(68)	7350(30)	2543(11)	4418(12)	62(7)
C(69)	7470(30)	3272(11)	4323(11)	54(6)
C(70)	8320(30)	3601(18)	3817(16)	85(10)
C(71)	8680(50)	4331(17)	3920(20)	99(12)
C(72)	7320(40)	4720(20)	4024(18)	102(11)
C(73)	6720(30)	4445(12)	4633(13)	74(9)
C(74)	6730(20)	3697(11)	4712(10)	42(5)
C(75)	430(30)	1327(15)	4837(15)	73(7)
C(76)	-760(40)	816(19)	4709(18)	102(11)

Table 3. Bond lengths [Å] and angles [deg] for cd25468.

Rh(1)-C(9)	1.994(15)
Rh(1)-C(1)	2.006(16)
Rh(1)-O(1)	2.135(16)
Rh(1)-O(2)	2.173(16)
Rh(1)-C(37)	2.34(3)
Rh(1)-I(2)	2.671(2)
Rh(1)-I(1)	2.676(2)
Rh(2)-C(39)	1.95(2)
Rh(2)-C(47)	1.98(2)
Rh(2)-O(3)	2.154(15)
Rh(2)-O(4)	2.240(15)
Rh(2)-C(75)	2.49(3)
Rh(2)-I(4)	2.663(2)
Rh(2)-I(3)	2.682(2)
O(1)-C(37)	1.21(3)
O(2)-C(37)	1.20(3)
O(3)-C(75)	1.21(3)
O(4)-C(75)	1.36(3)
N(1)-C(1)	1.35(2)
N(1)-C(7)	1.41(2)
N(1)-C(17)	1.48(2)
N(2)-C(1)	1.33(2)
N(2)-C(2)	1.37(2)
N(2)-C(8)	1.49(3)
N(3)-C(9)	1.34(3)
N(3)-C(10)	1.41(3)
N(3)-C(28)	1.46(3)
N(4)-C(15)	1.33(3)
N(4)-C(9)	1.33(2)
N(4)-C(16)	1.53(3)
N(5)-C(40)	1.32(3)
N(5)-C(39)	1.34(3)
N(5)-C(46)	1.45(3)
N(6)-C(55)	1.42(2)
N(6)-C(45)	1.42(2)
N(6)-C(39)	1.47(3)
N(7)-C(47)	1.32(3)
N(7)-C(66)	1.45(2)
N(7)-C(53)	1.46(3)
N(8)-C(47)	1.34(2)
N(8)-C(48)	1.48(3)
N(8)-C(54)	1.47(2)
C(2)-C(7)	1.367(17)
C(2)-C(3)	1.392(17)
C(3)-C(4)	1.397(18)
C(3)-H(3)	0.9300
C(4)-C(5)	1.391(18)
C(4)-H(4)	0.9300
C(5)-C(6)	1.409(18)
C(5)-H(5)	0.9300
C(6)-C(7)	1.377(17)
C(6)-H(6)	0.9300
C(8)-H(8A)	0.9600
C(8)-H(8B)	0.9600
C(8)-H(8C)	0.9600
C(10)-C(15)	1.33(4)
C(10)-C(11)	1.41(4)
C(11)-C(12)	1.37(4)
C(11)-H(11)	0.9300
C(12)-C(13)	1.47(6)
C(12)-H(12)	0.9300
C(13)-C(14)	1.38(5)
C(13)-H(13)	0.9300
C(14)-C(15)	1.45(3)
C(14)-H(14)	0.9300
C(16)-H(16A)	0.9600
C(16)-H(16B)	0.9600
C(16)-H(16C)	0.9600

C(17)-C(26)	1.33(3)
C(17)-C(18)	1.44(3)
C(18)-C(19)	1.43(3)
C(18)-H(18)	0.9300
C(19)-C(20)	1.31(3)
C(19)-H(19)	0.9300
C(20)-C(25)	1.39(3)
C(20)-C(21)	1.58(3)
C(21)-C(22)	1.50(4)
C(21)-H(21A)	0.9700
C(21)-H(21B)	0.9700
C(22)-C(23)	1.35(5)
C(22)-H(22A)	0.9700
C(22)-H(22B)	0.9700
C(23)-C(24)	1.47(4)
C(23)-H(23A)	0.9700
C(23)-H(23B)	0.9700
C(24)-C(25)	1.55(3)
C(24)-H(24A)	0.9700
C(24)-H(24B)	0.9700
C(25)-C(26)	1.40(3)
C(26)-C(27)	1.56(3)
C(27)-C(28)	1.34(3)
C(27)-C(36)	1.42(2)
C(28)-C(29)	1.350(16)
C(29)-C(30)	1.413(18)
C(29)-H(29)	0.9300
C(30)-C(31)	1.44(3)
C(30)-H(30)	0.9300
C(31)-C(36)	1.31(3)
C(31)-C(32)	1.60(3)
C(32)-C(33)	1.53(2)
C(32)-H(32A)	0.9700
C(32)-H(32B)	0.9700
C(33)-C(34)	1.53(2)
C(33)-H(33A)	0.9700
C(33)-H(33B)	0.9700
C(34)-C(35)	1.47(4)
C(34)-H(34A)	0.9700
C(34)-H(34B)	0.9700
C(35)-C(36)	1.55(3)
C(35)-H(35A)	0.9700
C(35)-H(35B)	0.9700
C(37)-C(38)	1.527(19)
C(38)-H(38A)	0.9600
C(38)-H(38B)	0.9600
C(38)-H(38C)	0.9600
C(40)-C(45)	1.37(3)
C(40)-C(41)	1.45(3)
C(41)-C(42)	1.38(5)
C(41)-H(41)	0.9300
C(42)-C(43)	1.41(4)
C(42)-H(42)	0.9300
C(43)-C(44)	1.33(4)
C(43)-H(43)	0.9300
C(44)-C(45)	1.47(3)
C(44)-H(44)	0.9300
C(46)-H(46A)	0.9600
C(46)-H(46B)	0.9600
C(46)-H(46C)	0.9600
C(48)-C(49)	1.36(3)
C(48)-C(53)	1.367(17)
C(49)-C(50)	1.39(4)
C(49)-H(49)	0.9300
C(50)-C(51)	1.385(19)
C(50)-H(50)	0.9300
C(51)-C(52)	1.44(3)
C(51)-H(51)	0.9300
C(52)-C(53)	1.40(3)
C(52)-H(52)	0.9300
C(54)-H(54A)	0.9600
C(54)-H(54B)	0.9600

C(54)-H(54C)	0.9600
C(55)-C(64)	1.41(2)
C(55)-C(56)	1.43(2)
C(56)-C(57)	1.371(17)
C(56)-H(56)	0.9300
C(57)-C(58)	1.45(3)
C(57)-H(57)	0.9300
C(58)-C(63)	1.37(3)
C(58)-C(59)	1.41(3)
C(59)-C(60)	1.51(4)
C(59)-H(59A)	0.9700
C(59)-H(59B)	0.9700
C(60)-C(61)	1.64(6)
C(60)-H(60A)	0.9700
C(60)-H(60B)	0.9700
C(61)-C(62)	1.51(4)
C(61)-H(61A)	0.9700
C(61)-H(61B)	0.9700
C(62)-C(63)	1.53(3)
C(62)-H(62A)	0.9700
C(62)-H(62B)	0.9700
C(63)-C(64)	1.358(17)
C(64)-C(65)	1.51(3)
C(65)-C(66)	1.36(3)
C(65)-C(74)	1.39(3)
C(66)-C(67)	1.355(16)
C(67)-C(68)	1.444(17)
C(67)-H(67)	0.9300
C(68)-C(69)	1.44(3)
C(68)-H(68)	0.9300
C(69)-C(74)	1.38(3)
C(69)-C(70)	1.50(4)
C(70)-C(71)	1.48(5)
C(70)-H(70A)	0.9700
C(70)-H(70B)	0.9700
C(71)-C(72)	1.57(5)
C(71)-H(71A)	0.9700
C(71)-H(71B)	0.9700
C(72)-C(73)	1.48(4)
C(72)-H(72A)	0.9700
C(72)-H(72B)	0.9700
C(73)-C(74)	1.47(3)
C(73)-H(73A)	0.9700
C(73)-H(73B)	0.9700
C(75)-C(76)	1.535(19)
C(76)-H(76A)	0.9600
C(76)-H(76B)	0.9600
C(76)-H(76C)	0.9600
C(9)-Rh(1)-C(1)	96.6(7)
C(9)-Rh(1)-O(1)	162.2(7)
C(1)-Rh(1)-O(1)	100.9(7)
C(9)-Rh(1)-O(2)	101.5(7)
C(1)-Rh(1)-O(2)	160.9(7)
O(1)-Rh(1)-O(2)	61.4(7)
C(9)-Rh(1)-C(37)	131.8(9)
C(1)-Rh(1)-C(37)	131.5(9)
O(1)-Rh(1)-C(37)	31.0(8)
O(2)-Rh(1)-C(37)	30.4(8)
C(9)-Rh(1)-I(2)	98.2(5)
C(1)-Rh(1)-I(2)	83.6(5)
O(1)-Rh(1)-I(2)	86.6(4)
O(2)-Rh(1)-I(2)	87.8(5)
C(37)-Rh(1)-I(2)	86.3(8)
C(9)-Rh(1)-I(1)	88.2(5)
C(1)-Rh(1)-I(1)	103.2(5)
O(1)-Rh(1)-I(1)	85.2(4)
O(2)-Rh(1)-I(1)	83.6(5)
C(37)-Rh(1)-I(1)	83.9(8)
I(2)-Rh(1)-I(1)	170.20(8)
C(39)-Rh(2)-C(47)	100.3(10)
C(39)-Rh(2)-O(3)	160.2(9)

C(47)-Rh(2)-O(3)	99.4(8)
C(39)-Rh(2)-O(4)	98.8(8)
C(47)-Rh(2)-O(4)	160.5(8)
O(3)-Rh(2)-O(4)	61.8(6)
C(39)-Rh(2)-C(75)	131.7(10)
C(47)-Rh(2)-C(75)	128.0(10)
O(3)-Rh(2)-C(75)	29.0(8)
O(4)-Rh(2)-C(75)	32.9(8)
C(39)-Rh(2)-I(4)	86.7(6)
C(47)-Rh(2)-I(4)	98.8(5)
O(3)-Rh(2)-I(4)	88.2(5)
O(4)-Rh(2)-I(4)	86.5(5)
C(75)-Rh(2)-I(4)	88.6(7)
C(39)-Rh(2)-I(3)	99.1(6)
C(47)-Rh(2)-I(3)	86.8(5)
O(3)-Rh(2)-I(3)	84.1(5)
O(4)-Rh(2)-I(3)	86.0(5)
C(75)-Rh(2)-I(3)	82.6(7)
I(4)-Rh(2)-I(3)	171.16(9)
C(37)-O(1)-Rh(1)	84.0(15)
C(37)-O(2)-Rh(1)	82.7(15)
C(75)-O(3)-Rh(2)	91.2(16)
C(75)-O(4)-Rh(2)	83.8(14)
C(1)-N(1)-C(7)	110.2(14)
C(1)-N(1)-C(17)	132.2(14)
C(7)-N(1)-C(17)	117.6(12)
C(1)-N(2)-C(2)	111.6(14)
C(1)-N(2)-C(8)	131.6(16)
C(2)-N(2)-C(8)	116.8(15)
C(9)-N(3)-C(10)	110.6(18)
C(9)-N(3)-C(28)	129.4(15)
C(10)-N(3)-C(28)	119(2)
C(15)-N(4)-C(9)	110.6(19)
C(15)-N(4)-C(16)	124(2)
C(9)-N(4)-C(16)	124.2(17)
C(40)-N(5)-C(39)	116.6(18)
C(40)-N(5)-C(46)	120.4(19)
C(39)-N(5)-C(46)	123(2)
C(55)-N(6)-C(45)	121.4(16)
C(55)-N(6)-C(39)	132.1(16)
C(45)-N(6)-C(39)	106.5(15)
C(47)-N(7)-C(66)	131.4(16)
C(47)-N(7)-C(53)	110.9(14)
C(66)-N(7)-C(53)	117.6(16)
C(47)-N(8)-C(48)	110.4(16)
C(47)-N(8)-C(54)	129.2(19)
C(48)-N(8)-C(54)	119.8(15)
N(2)-C(1)-N(1)	105.5(15)
N(2)-C(1)-Rh(1)	121.2(12)
N(1)-C(1)-Rh(1)	132.6(12)
N(2)-C(2)-C(7)	107.2(14)
N(2)-C(2)-C(3)	134.6(18)
C(7)-C(2)-C(3)	118(2)
C(2)-C(3)-C(4)	120(2)
C(2)-C(3)-H(3)	120.2
C(4)-C(3)-H(3)	120.3
C(5)-C(4)-C(3)	115(2)
C(5)-C(4)-H(4)	122.3
C(3)-C(4)-H(4)	122.2
C(4)-C(5)-C(6)	127(2)
C(4)-C(5)-H(5)	116.4
C(6)-C(5)-H(5)	116.4
C(7)-C(6)-C(5)	110.0(18)
C(7)-C(6)-H(6)	125.0
C(5)-C(6)-H(6)	125.0
C(2)-C(7)-C(6)	127.6(19)
C(2)-C(7)-N(1)	104.3(14)
C(6)-C(7)-N(1)	127.5(16)
N(2)-C(8)-H(8A)	109.4
N(2)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
N(2)-C(8)-H(8C)	109.5

H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
N(4)-C(9)-N(3)	105.2(14)
N(4)-C(9)-Rh(1)	124.6(13)
N(3)-C(9)-Rh(1)	129.6(13)
C(15)-C(10)-N(3)	104(2)
C(15)-C(10)-C(11)	124(2)
N(3)-C(10)-C(11)	132(2)
C(12)-C(11)-C(10)	117(3)
C(12)-C(11)-H(11)	121.7
C(10)-C(11)-H(11)	121.6
C(11)-C(12)-C(13)	121(3)
C(11)-C(12)-H(12)	119.7
C(13)-C(12)-H(12)	119.8
C(14)-C(13)-C(12)	121(2)
C(14)-C(13)-H(13)	119.6
C(12)-C(13)-H(13)	119.6
C(13)-C(14)-C(15)	116(3)
C(13)-C(14)-H(14)	121.8
C(15)-C(14)-H(14)	121.8
N(4)-C(15)-C(10)	110(2)
N(4)-C(15)-C(14)	129(3)
C(10)-C(15)-C(14)	121(2)
N(4)-C(16)-H(16A)	109.5
N(4)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
N(4)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(26)-C(17)-C(18)	125(2)
C(26)-C(17)-N(1)	119.9(17)
C(18)-C(17)-N(1)	114.7(18)
C(19)-C(18)-C(17)	112(2)
C(19)-C(18)-H(18)	124.1
C(17)-C(18)-H(18)	124.1
C(20)-C(19)-C(18)	123.6(19)
C(20)-C(19)-H(19)	118.2
C(18)-C(19)-H(19)	118.2
C(19)-C(20)-C(25)	122.0(18)
C(19)-C(20)-C(21)	123.5(18)
C(25)-C(20)-C(21)	114.4(18)
C(22)-C(21)-C(20)	119.3(18)
C(22)-C(21)-H(21A)	107.5
C(20)-C(21)-H(21A)	107.5
C(22)-C(21)-H(21B)	107.5
C(20)-C(21)-H(21B)	107.5
H(21A)-C(21)-H(21B)	107.0
C(23)-C(22)-C(21)	114(3)
C(23)-C(22)-H(22A)	108.8
C(21)-C(22)-H(22A)	108.8
C(23)-C(22)-H(22B)	108.8
C(21)-C(22)-H(22B)	108.8
H(22A)-C(22)-H(22B)	107.7
C(22)-C(23)-C(24)	125(4)
C(22)-C(23)-H(23A)	105.9
C(24)-C(23)-H(23A)	105.9
C(22)-C(23)-H(23B)	106.0
C(24)-C(23)-H(23B)	105.9
H(23A)-C(23)-H(23B)	106.3
C(23)-C(24)-C(25)	113(2)
C(23)-C(24)-H(24A)	109.1
C(25)-C(24)-H(24A)	109.1
C(23)-C(24)-H(24B)	109.1
C(25)-C(24)-H(24B)	109.0
H(24A)-C(24)-H(24B)	107.8
C(20)-C(25)-C(26)	118(2)
C(20)-C(25)-C(24)	124.5(19)
C(26)-C(25)-C(24)	117.1(18)
C(17)-C(26)-C(25)	119.0(19)
C(17)-C(26)-C(27)	120.1(17)
C(25)-C(26)-C(27)	120.7(19)
C(28)-C(27)-C(36)	122.5(16)

C(28)-C(27)-C(26)	113.7(16)
C(36)-C(27)-C(26)	123.6(17)
C(27)-C(28)-C(29)	123.4(17)
C(27)-C(28)-N(3)	124.4(15)
C(29)-C(28)-N(3)	109.7(16)
C(28)-C(29)-C(30)	112.5(17)
C(28)-C(29)-H(29)	123.7
C(30)-C(29)-H(29)	123.8
C(29)-C(30)-C(31)	124.4(18)
C(29)-C(30)-H(30)	117.8
C(31)-C(30)-H(30)	117.8
C(36)-C(31)-C(30)	118.3(18)
C(36)-C(31)-C(32)	119(2)
C(30)-C(31)-C(32)	121(2)
C(33)-C(32)-C(31)	118(3)
C(33)-C(32)-H(32A)	107.8
C(31)-C(32)-H(32A)	107.8
C(33)-C(32)-H(32B)	107.9
C(31)-C(32)-H(32B)	107.9
H(32A)-C(32)-H(32B)	107.2
C(34)-C(33)-C(32)	113(3)
C(34)-C(33)-H(33A)	109.1
C(32)-C(33)-H(33A)	109.1
C(34)-C(33)-H(33B)	109.1
C(32)-C(33)-H(33B)	109.1
H(33A)-C(33)-H(33B)	107.9
C(35)-C(34)-C(33)	117(3)
C(35)-C(34)-H(34A)	107.9
C(33)-C(34)-H(34A)	108.0
C(35)-C(34)-H(34B)	108.0
C(33)-C(34)-H(34B)	107.9
H(34A)-C(34)-H(34B)	107.2
C(34)-C(35)-C(36)	115(2)
C(34)-C(35)-H(35A)	108.5
C(36)-C(35)-H(35A)	108.5
C(34)-C(35)-H(35B)	108.5
C(36)-C(35)-H(35B)	108.5
H(35A)-C(35)-H(35B)	107.5
C(31)-C(36)-C(27)	117.3(18)
C(31)-C(36)-C(35)	124.5(19)
C(27)-C(36)-C(35)	118.3(16)
O(2)-C(37)-O(1)	132(3)
O(2)-C(37)-C(38)	117(3)
O(1)-C(37)-C(38)	111(3)
O(2)-C(37)-Rh(1)	66.9(14)
O(1)-C(37)-Rh(1)	65.0(14)
C(38)-C(37)-Rh(1)	175(2)
C(37)-C(38)-H(38A)	109.4
C(37)-C(38)-H(38B)	109.6
H(38A)-C(38)-H(38B)	109.5
C(37)-C(38)-H(38C)	109.4
H(38A)-C(38)-H(38C)	109.5
H(38B)-C(38)-H(38C)	109.5
N(5)-C(39)-N(6)	102.1(17)
N(5)-C(39)-Rh(2)	128.0(17)
N(6)-C(39)-Rh(2)	128.9(13)
N(5)-C(40)-C(45)	106.6(17)
N(5)-C(40)-C(41)	135(2)
C(45)-C(40)-C(41)	118(2)
C(42)-C(41)-C(40)	118(2)
C(42)-C(41)-H(41)	121.1
C(40)-C(41)-H(41)	121.1
C(41)-C(42)-C(43)	121(3)
C(41)-C(42)-H(42)	119.6
C(43)-C(42)-H(42)	119.6
C(44)-C(43)-C(42)	124(3)
C(44)-C(43)-H(43)	118.2
C(42)-C(43)-H(43)	118.2
C(43)-C(44)-C(45)	115(3)
C(43)-C(44)-H(44)	122.4
C(45)-C(44)-H(44)	122.4
C(40)-C(45)-N(6)	108.0(16)

C(40)-C(45)-C(44)	123(2)
N(6)-C(45)-C(44)	129(2)
N(5)-C(46)-H(46A)	109.5
N(5)-C(46)-H(46B)	109.4
H(46A)-C(46)-H(46B)	109.5
N(5)-C(46)-H(46C)	109.5
H(46A)-C(46)-H(46C)	109.5
H(46B)-C(46)-H(46C)	109.5
N(7)-C(47)-N(8)	107.7(18)
N(7)-C(47)-Rh(2)	130.6(13)
N(8)-C(47)-Rh(2)	121.4(18)
C(49)-C(48)-C(53)	124(2)
C(49)-C(48)-N(8)	132.4(17)
C(53)-C(48)-N(8)	104.1(16)
C(48)-C(49)-C(50)	115(2)
C(48)-C(49)-H(49)	122.3
C(50)-C(49)-H(49)	122.3
C(51)-C(50)-C(49)	121(2)
C(51)-C(50)-H(50)	119.5
C(49)-C(50)-H(50)	119.5
C(50)-C(51)-C(52)	125(2)
C(50)-C(51)-H(51)	117.5
C(52)-C(51)-H(51)	117.5
C(53)-C(52)-C(51)	110(2)
C(53)-C(52)-H(52)	125.2
C(51)-C(52)-H(52)	125.2
C(48)-C(53)-C(52)	125(2)
C(48)-C(53)-N(7)	106.6(17)
C(52)-C(53)-N(7)	128.0(17)
N(8)-C(54)-H(54A)	109.5
N(8)-C(54)-H(54B)	109.5
H(54A)-C(54)-H(54B)	109.5
N(8)-C(54)-H(54C)	109.5
H(54A)-C(54)-H(54C)	109.5
H(54B)-C(54)-H(54C)	109.5
C(64)-C(55)-N(6)	120.2(14)
C(64)-C(55)-C(56)	121.1(16)
N(6)-C(55)-C(56)	117.1(16)
C(57)-C(56)-C(55)	121(2)
C(57)-C(56)-H(56)	119.3
C(55)-C(56)-H(56)	119.3
C(56)-C(57)-C(58)	117(2)
C(56)-C(57)-H(57)	121.6
C(58)-C(57)-H(57)	121.6
C(63)-C(58)-C(59)	123(2)
C(63)-C(58)-C(57)	119(2)
C(59)-C(58)-C(57)	118(2)
C(58)-C(59)-C(60)	117(2)
C(58)-C(59)-H(59A)	108.2
C(60)-C(59)-H(59A)	108.1
C(58)-C(59)-H(59B)	108.1
C(60)-C(59)-H(59B)	108.2
H(59A)-C(59)-H(59B)	107.3
C(59)-C(60)-C(61)	107(3)
C(59)-C(60)-H(60A)	110.3
C(61)-C(60)-H(60A)	110.3
C(59)-C(60)-H(60B)	110.2
C(61)-C(60)-H(60B)	110.2
H(60A)-C(60)-H(60B)	108.5
C(62)-C(61)-C(60)	104(3)
C(62)-C(61)-H(61A)	111.0
C(60)-C(61)-H(61A)	111.0
C(62)-C(61)-H(61B)	110.9
C(60)-C(61)-H(61B)	110.9
H(61A)-C(61)-H(61B)	109.0
C(61)-C(62)-C(63)	116(3)
C(61)-C(62)-H(62A)	108.4
C(63)-C(62)-H(62A)	108.3
C(61)-C(62)-H(62B)	108.3
C(63)-C(62)-H(62B)	108.3
H(62A)-C(62)-H(62B)	107.4
C(64)-C(63)-C(58)	126(2)

C(64)-C(63)-C(62)	114.9(18)
C(58)-C(63)-C(62)	119.0(18)
C(63)-C(64)-C(55)	115.5(17)
C(63)-C(64)-C(65)	127.0(17)
C(55)-C(64)-C(65)	117.5(15)
C(66)-C(65)-C(74)	122.7(17)
C(66)-C(65)-C(64)	117.4(18)
C(74)-C(65)-C(64)	119.8(17)
C(67)-C(66)-C(65)	125.2(17)
C(67)-C(66)-N(7)	109.6(16)
C(65)-C(66)-N(7)	123.5(15)
C(66)-C(67)-C(68)	111.5(15)
C(66)-C(67)-H(67)	124.2
C(68)-C(67)-H(67)	124.3
C(67)-C(68)-C(69)	124.8(19)
C(67)-C(68)-H(68)	117.6
C(69)-C(68)-H(68)	117.7
C(74)-C(69)-C(68)	118(2)
C(74)-C(69)-C(70)	118(2)
C(68)-C(69)-C(70)	124(2)
C(71)-C(70)-C(69)	118(2)
C(71)-C(70)-H(70A)	107.8
C(69)-C(70)-H(70A)	107.8
C(71)-C(70)-H(70B)	107.9
C(69)-C(70)-H(70B)	107.9
H(70A)-C(70)-H(70B)	107.2
C(70)-C(71)-C(72)	107(3)
C(70)-C(71)-H(71A)	110.5
C(72)-C(71)-H(71A)	110.5
C(70)-C(71)-H(71B)	110.4
C(72)-C(71)-H(71B)	110.4
H(71A)-C(71)-H(71B)	108.6
C(73)-C(72)-C(71)	110(3)
C(73)-C(72)-H(72A)	109.7
C(71)-C(72)-H(72A)	109.6
C(73)-C(72)-H(72B)	109.6
C(71)-C(72)-H(72B)	109.6
H(72A)-C(72)-H(72B)	108.2
C(74)-C(73)-C(72)	116(3)
C(74)-C(73)-H(73A)	108.2
C(72)-C(73)-H(73A)	108.1
C(74)-C(73)-H(73B)	108.2
C(72)-C(73)-H(73B)	108.2
H(73A)-C(73)-H(73B)	107.3
C(69)-C(74)-C(65)	117(2)
C(69)-C(74)-C(73)	123(2)
C(65)-C(74)-C(73)	120.3(18)
O(3)-C(75)-O(4)	123(2)
O(3)-C(75)-C(76)	121(3)
O(4)-C(75)-C(76)	115(3)
O(3)-C(75)-Rh(2)	59.8(13)
O(4)-C(75)-Rh(2)	63.3(12)
C(76)-C(75)-Rh(2)	177(2)
C(75)-C(76)-H(76A)	109.4
C(75)-C(76)-H(76B)	109.5
H(76A)-C(76)-H(76B)	109.5
C(75)-C(76)-H(76C)	109.5
H(76A)-C(76)-H(76C)	109.5
H(76B)-C(76)-H(76C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd25468.
The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Rh(1)	41(1)	24(1)	45(1)	0(1)	3(1)	0(1)
Rh(2)	47(1)	30(1)	46(1)	-2(1)	-1(1)	-8(1)
I(1)	57(1)	39(1)	77(1)	18(1)	10(1)	8(1)
I(2)	47(1)	47(1)	64(1)	7(1)	-3(1)	1(1)
I(3)	76(1)	41(1)	93(1)	-23(1)	13(1)	-4(1)
I(4)	54(1)	59(1)	68(1)	-12(1)	16(1)	-9(1)
O(1)	56(10)	46(9)	76(10)	16(8)	11(8)	8(8)
O(2)	80(12)	28(8)	80(10)	-11(7)	19(9)	-13(8)
O(3)	64(11)	38(9)	91(11)	8(8)	1(9)	-24(8)
O(4)	48(9)	81(12)	60(9)	7(8)	-10(7)	-34(9)
N(1)	36(8)	3(6)	69(8)	16(5)	-1(7)	10(6)
N(2)	42(10)	34(9)	45(9)	7(7)	-5(7)	-12(7)
N(3)	78(14)	42(10)	28(8)	6(6)	14(8)	14(9)
N(4)	37(9)	47(10)	41(8)	-2(7)	4(7)	6(8)
N(5)	25(9)	66(13)	63(11)	-20(9)	10(8)	-23(8)
N(7)	48(10)	21(8)	38(8)	8(6)	7(7)	11(7)
N(8)	40(9)	28(8)	47(8)	-1(6)	9(7)	-31(7)
C(1)	28(9)	28(9)	31(8)	4(7)	-18(7)	-13(7)
C(2)	38(11)	56(13)	30(9)	-11(8)	-12(8)	0(10)
C(3)	90(20)	57(16)	68(15)	-25(12)	6(14)	-17(15)
C(4)	71(18)	69(18)	63(14)	-17(12)	-5(12)	36(14)
C(5)	73(17)	40(13)	92(18)	-24(12)	21(14)	-35(13)
C(6)	88(19)	13(9)	79(15)	20(9)	20(13)	-11(10)
C(7)	32(11)	53(14)	56(12)	-7(10)	-2(9)	12(10)
C(8)	110(20)	35(11)	39(11)	9(8)	-7(12)	13(13)
C(9)	20(8)	21(8)	51(10)	8(8)	15(7)	5(7)
C(10)	77(17)	31(11)	65(13)	-17(10)	5(12)	16(12)
C(11)	83(19)	51(14)	91(17)	2(14)	53(15)	-37(15)
C(12)	100(30)	53(17)	140(30)	-15(17)	90(30)	-11(17)
C(13)	170(40)	49(16)	90(20)	-19(15)	80(30)	-10(20)
C(14)	90(20)	40(12)	40(11)	-13(9)	-2(12)	-1(13)
C(15)	68(16)	17(10)	82(15)	16(9)	13(13)	-1(10)
C(16)	110(20)	28(12)	80(16)	-7(10)	9(15)	-35(14)
C(17)	54(13)	21(9)	31(9)	-20(7)	-16(8)	4(8)
C(18)	52(14)	72(16)	39(10)	7(10)	-24(9)	30(12)
C(19)	75(16)	17(9)	46(10)	16(7)	-24(10)	-25(10)
C(21)	79(17)	26(10)	48(11)	16(8)	15(11)	12(10)
C(22)	140(40)	160(40)	120(30)	100(30)	70(30)	90(30)
C(23)	110(30)	160(40)	70(20)	30(20)	24(19)	0(30)
C(24)	30(11)	54(14)	88(16)	19(12)	-1(11)	0(10)
C(25)	51(13)	44(12)	40(10)	1(8)	-7(9)	12(10)
C(26)	51(13)	45(12)	47(11)	9(9)	9(10)	19(10)
C(27)	31(10)	28(9)	33(9)	-1(7)	-9(7)	-2(8)
C(28)	44(11)	39(11)	32(9)	14(7)	-2(8)	-19(9)
C(30)	90(20)	90(20)	52(13)	34(13)	35(13)	67(17)
C(31)	40(12)	66(15)	46(11)	-6(10)	-26(9)	-3(11)
C(32)	33(13)	90(20)	90(18)	1(15)	-14(12)	-9(13)
C(34)	140(40)	100(30)	210(50)	-90(30)	-120(40)	50(30)
C(35)	51(14)	63(15)	58(13)	-2(11)	7(10)	33(12)
C(36)	36(10)	42(11)	29(9)	16(7)	-7(7)	4(8)
C(39)	75(16)	30(10)	43(11)	-3(8)	8(10)	15(11)
C(40)	36(11)	34(10)	49(10)	4(8)	6(8)	29(9)
C(41)	100(20)	80(20)	59(15)	-6(13)	-16(15)	-42(19)
C(42)	80(20)	90(20)	110(20)	13(18)	-33(18)	-25(18)
C(43)	120(30)	23(11)	98(19)	-10(12)	43(18)	-4(14)
C(44)	90(20)	59(17)	79(17)	31(13)	23(15)	14(15)
C(45)	45(12)	22(9)	66(13)	8(8)	8(10)	10(9)
C(46)	66(16)	55(14)	39(10)	-12(9)	-10(10)	-9(12)
C(47)	72(16)	46(12)	30(9)	5(8)	17(9)	30(12)
C(48)	40(11)	23(9)	60(12)	2(8)	4(9)	-16(8)
C(49)	65(17)	34(12)	83(16)	16(11)	21(13)	1(11)
C(50)	130(40)	64(18)	45(14)	22(12)	9(17)	-20(20)

C(51)	110(20)	56(15)	59(14)	-4(11)	-37(14)	-34(16)
C(52)	26(10)	34(11)	90(15)	-7(11)	-4(10)	2(9)
C(53)	42(11)	48(12)	49(11)	-1(9)	11(9)	-11(10)
C(54)	55(14)	67(16)	45(11)	4(10)	12(10)	-33(12)
C(55)	20(8)	5(7)	49(9)	-9(6)	6(7)	-3(6)
C(56)	68(15)	19(9)	59(12)	-9(8)	6(11)	25(10)
C(57)	77(17)	65(16)	45(11)	-8(11)	-8(11)	50(14)
C(58)	47(13)	28(11)	76(14)	13(9)	5(11)	7(9)
C(59)	110(30)	130(30)	47(13)	-58(17)	4(15)	0(20)
C(60)	100(30)	80(20)	90(20)	-43(17)	-56(19)	50(20)
C(61)	130(60)	40(20)	100(30)	-12(18)	-40(30)	-50(30)
C(62)	53(15)	120(20)	72(16)	-58(17)	-18(13)	43(16)
C(63)	53(14)	53(14)	45(11)	3(9)	-6(10)	6(11)
C(64)	12(8)	36(10)	46(10)	14(8)	1(7)	4(7)
C(65)	65(14)	39(11)	22(8)	0(7)	-5(8)	17(10)
C(66)	40(11)	59(13)	23(8)	21(8)	15(7)	-5(9)
C(67)	24(9)	0(7)	92(13)	-22(8)	-46(9)	15(7)
C(68)	81(19)	34(12)	76(15)	-17(11)	30(14)	-3(12)
C(69)	83(18)	33(11)	47(11)	-21(9)	14(11)	-8(11)
C(70)	70(20)	110(30)	86(19)	-5(18)	50(17)	2(18)
C(71)	130(30)	60(20)	110(20)	8(17)	40(20)	-20(20)
C(73)	110(20)	49(14)	73(15)	-15(11)	43(15)	-63(15)
C(74)	35(11)	52(12)	40(10)	-2(9)	11(8)	12(10)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd25468.

	x	y	z	U(eq)
H(3)	11275	9075	-2413	88
H(4)	11588	7870	-2330	82
H(5)	10828	7374	-1360	81
H(6)	10749	7962	-302	71
H(8A)	10979	10666	-1872	93
H(8B)	11932	10122	-2170	93
H(8C)	12547	10658	-1619	93
H(11)	6884	10762	1155	86
H(12)	6631	11094	2282	114
H(13)	8600	11353	3075	119
H(14)	10759	11390	2691	70
H(16A)	12514	11871	1536	108
H(16B)	13251	11197	1339	108
H(16C)	12770	11277	2079	108
H(18)	12623	8762	760	68
H(19)	11721	8195	1705	58
H(21A)	9226	7758	2118	61
H(21B)	9972	8307	2608	61
H(22A)	7535	8120	2640	121
H(22B)	8112	8867	2729	121
H(23A)	6400	8976	2057	123
H(23B)	6606	8287	1682	123
H(24A)	7259	9555	1312	69
H(24B)	6890	8911	845	69
H(29)	7791	11319	-162	38
H(30)	6158	10828	-1003	90
H(32A)	4609	9737	-1262	87
H(32B)	5553	9623	-1849	87
H(33A)	4502	8645	-1027	129
H(33B)	4824	8573	-1798	129
H(34A)	6219	7874	-979	124
H(34B)	7021	8297	-1486	124
H(35A)	8285	8434	-520	68
H(35B)	7044	8447	-80	68
H(38A)	14476	12597	-575	121
H(38B)	15180	11995	-936	121
H(38C)	15633	12168	-155	121
H(41)	1982	3919	2583	98
H(42)	2122	5110	2598	112
H(43)	3173	5665	3568	92
H(44)	3428	5155	4628	89
H(46A)	1135	2816	3031	82
H(46B)	1498	2248	3594	82
H(46C)	2590	2479	3110	82
H(49)	4804	1534	7777	71
H(50)	7154	1554	8134	110
H(51)	8658	1934	7411	92
H(52)	7965	2368	6266	61
H(54A)	1657	1873	6416	83
H(54B)	2410	1943	7168	83
H(54C)	2492	1253	6759	83
H(56)	2060	4436	5670	58
H(57)	2977	4924	6707	76
H(59A)	4923	4885	7588	116
H(59B)	5645	5393	7122	116
H(60A)	6832	4217	7777	117
H(60B)	7330	4979	7898	117
H(61A)	7950	5008	6673	127
H(61B)	8822	4441	7103	127
H(62A)	7342	3592	6700	99
H(62B)	7660	4003	6043	99
H(67)	6547	1754	4998	51
H(68)	7826	2258	4142	74

H(70A)	9160	3342	3821	102
H(70B)	7830	3555	3357	102
H(71A)	9081	4510	3525	119
H(71B)	9340	4385	4331	119
H(72A)	7506	5206	4089	122
H(72B)	6675	4665	3612	122
H(73A)	7214	4643	5047	89
H(73B)	5784	4601	4611	89
H(76A)	-834	559	5125	124
H(76B)	-587	509	4341	124
H(76C)	-1592	1062	4581	124

Table 6. Torsion angles [deg] for cd25468.

C(9)-Rh(1)-O(1)-C(37)	18(3)
C(1)-Rh(1)-O(1)-C(37)	-171.2(17)
O(2)-Rh(1)-O(1)-C(37)	1.0(16)
I(2)-Rh(1)-O(1)-C(37)	-88.4(16)
I(1)-Rh(1)-O(1)-C(37)	86.2(16)
C(9)-Rh(1)-O(2)-C(37)	-175.8(17)
C(1)-Rh(1)-O(2)-C(37)	23(3)
O(1)-Rh(1)-O(2)-C(37)	-1.0(16)
I(2)-Rh(1)-O(2)-C(37)	86.3(17)
I(1)-Rh(1)-O(2)-C(37)	-89.0(17)
C(39)-Rh(2)-O(3)-C(75)	15(3)
C(47)-Rh(2)-O(3)-C(75)	-171.1(18)
O(4)-Rh(2)-O(3)-C(75)	3.3(17)
I(4)-Rh(2)-O(3)-C(75)	90.3(18)
I(3)-Rh(2)-O(3)-C(75)	-85.3(18)
C(39)-Rh(2)-O(4)-C(75)	-178.9(16)
C(47)-Rh(2)-O(4)-C(75)	14(3)
O(3)-Rh(2)-O(4)-C(75)	-2.9(15)
I(4)-Rh(2)-O(4)-C(75)	-92.7(15)
I(3)-Rh(2)-O(4)-C(75)	82.5(15)
C(2)-N(2)-C(1)-N(1)	-4(2)
C(8)-N(2)-C(1)-N(1)	176(2)
C(2)-N(2)-C(1)-Rh(1)	167.5(14)
C(8)-N(2)-C(1)-Rh(1)	-13(3)
C(7)-N(1)-C(1)-N(2)	10(2)
C(17)-N(1)-C(1)-N(2)	-170.6(18)
C(7)-N(1)-C(1)-Rh(1)	-160.5(15)
C(17)-N(1)-C(1)-Rh(1)	19(3)
C(9)-Rh(1)-C(1)-N(2)	151.6(15)
O(1)-Rh(1)-C(1)-N(2)	-25.6(17)
O(2)-Rh(1)-C(1)-N(2)	-47(3)
C(37)-Rh(1)-C(1)-N(2)	-32(2)
I(2)-Rh(1)-C(1)-N(2)	-110.9(15)
I(1)-Rh(1)-C(1)-N(2)	61.9(16)
C(9)-Rh(1)-C(1)-N(1)	-40(2)
O(1)-Rh(1)-C(1)-N(1)	143.2(18)
O(2)-Rh(1)-C(1)-N(1)	122(2)
C(37)-Rh(1)-C(1)-N(1)	137.2(19)
I(2)-Rh(1)-C(1)-N(1)	57.9(18)
I(1)-Rh(1)-C(1)-N(1)	-129.2(18)
C(1)-N(2)-C(2)-C(7)	-3(2)
C(8)-N(2)-C(2)-C(7)	177(2)
C(1)-N(2)-C(2)-C(3)	-178(3)
C(8)-N(2)-C(2)-C(3)	2(4)
N(2)-C(2)-C(3)-C(4)	166(3)
C(7)-C(2)-C(3)-C(4)	-8(4)
C(2)-C(3)-C(4)-C(5)	17(4)
C(3)-C(4)-C(5)-C(6)	-15(5)
C(4)-C(5)-C(6)-C(7)	3(4)
N(2)-C(2)-C(7)-C(6)	180(2)
C(3)-C(2)-C(7)-C(6)	-5(4)
N(2)-C(2)-C(7)-N(1)	9(2)
C(3)-C(2)-C(7)-N(1)	-176(2)
C(5)-C(6)-C(7)-C(2)	7(4)
C(5)-C(6)-C(7)-N(1)	176(2)
C(1)-N(1)-C(7)-C(2)	-11(2)
C(17)-N(1)-C(7)-C(2)	168.7(16)
C(1)-N(1)-C(7)-C(6)	178(2)
C(17)-N(1)-C(7)-C(6)	-2(3)
C(15)-N(4)-C(9)-N(3)	0(2)
C(16)-N(4)-C(9)-N(3)	-169.5(19)
C(15)-N(4)-C(9)-Rh(1)	171.7(13)
C(16)-N(4)-C(9)-Rh(1)	2(3)
C(10)-N(3)-C(9)-N(4)	1(2)
C(28)-N(3)-C(9)-N(4)	-169.5(17)
C(10)-N(3)-C(9)-Rh(1)	-170.1(15)
C(28)-N(3)-C(9)-Rh(1)	20(3)
C(1)-Rh(1)-C(9)-N(4)	143.0(16)

O(1)-Rh(1)-C(9)-N(4)	-46(3)
O(2)-Rh(1)-C(9)-N(4)	-30.9(16)
C(37)-Rh(1)-C(9)-N(4)	-34(2)
I(2)-Rh(1)-C(9)-N(4)	58.5(16)
I(1)-Rh(1)-C(9)-N(4)	-114.0(15)
C(1)-Rh(1)-C(9)-N(3)	-47.7(18)
O(1)-Rh(1)-C(9)-N(3)	123(2)
O(2)-Rh(1)-C(9)-N(3)	138.4(17)
C(37)-Rh(1)-C(9)-N(3)	135.5(18)
I(2)-Rh(1)-C(9)-N(3)	-132.1(17)
I(1)-Rh(1)-C(9)-N(3)	55.4(17)
C(9)-N(3)-C(10)-C(15)	-2(2)
C(28)-N(3)-C(10)-C(15)	169.9(17)
C(9)-N(3)-C(10)-C(11)	174(3)
C(28)-N(3)-C(10)-C(11)	-15(4)
C(15)-C(10)-C(11)-C(12)	-3(4)
N(3)-C(10)-C(11)-C(12)	-178(3)
C(10)-C(11)-C(12)-C(13)	-1(4)
C(11)-C(12)-C(13)-C(14)	4(5)
C(12)-C(13)-C(14)-C(15)	-2(4)
C(9)-N(4)-C(15)-C(10)	-1(2)
C(16)-N(4)-C(15)-C(10)	168(2)
C(9)-N(4)-C(15)-C(14)	-180(2)
C(16)-N(4)-C(15)-C(14)	-10(3)
N(3)-C(10)-C(15)-N(4)	2(2)
C(11)-C(10)-C(15)-N(4)	-174(2)
N(3)-C(10)-C(15)-C(14)	-179.6(18)
C(11)-C(10)-C(15)-C(14)	5(4)
C(13)-C(14)-C(15)-N(4)	177(2)
C(13)-C(14)-C(15)-C(10)	-2(3)
C(1)-N(1)-C(17)-C(26)	79(3)
C(7)-N(1)-C(17)-C(26)	-101(2)
C(1)-N(1)-C(17)-C(18)	-107(2)
C(7)-N(1)-C(17)-C(18)	73(2)
C(26)-C(17)-C(18)-C(19)	7(3)
N(1)-C(17)-C(18)-C(19)	-167.2(16)
C(17)-C(18)-C(19)-C(20)	-6(3)
C(18)-C(19)-C(20)-C(25)	3(3)
C(18)-C(19)-C(20)-C(21)	-179(2)
C(19)-C(20)-C(21)-C(22)	169(3)
C(25)-C(20)-C(21)-C(22)	-13(4)
C(20)-C(21)-C(22)-C(23)	32(6)
C(21)-C(22)-C(23)-C(24)	-38(7)
C(22)-C(23)-C(24)-C(25)	21(6)
C(19)-C(20)-C(25)-C(26)	-1(3)
C(21)-C(20)-C(25)-C(26)	-178(2)
C(19)-C(20)-C(25)-C(24)	175(2)
C(21)-C(20)-C(25)-C(24)	-3(3)
C(23)-C(24)-C(25)-C(20)	1(4)
C(23)-C(24)-C(25)-C(26)	176(3)
C(18)-C(17)-C(26)-C(25)	-5(3)
N(1)-C(17)-C(26)-C(25)	168.9(18)
C(18)-C(17)-C(26)-C(27)	179.8(18)
N(1)-C(17)-C(26)-C(27)	-7(3)
C(20)-C(25)-C(26)-C(17)	1(3)
C(24)-C(25)-C(26)-C(17)	-174(2)
C(20)-C(25)-C(26)-C(27)	176.7(18)
C(24)-C(25)-C(26)-C(27)	1(3)
C(17)-C(26)-C(27)-C(28)	-78(3)
C(25)-C(26)-C(27)-C(28)	106(3)
C(17)-C(26)-C(27)-C(36)	96(3)
C(25)-C(26)-C(27)-C(36)	-80(3)
C(36)-C(27)-C(28)-C(29)	14(3)
C(26)-C(27)-C(28)-C(29)	-171.7(17)
C(36)-C(27)-C(28)-N(3)	174.4(17)
C(26)-C(27)-C(28)-N(3)	-11(3)
C(9)-N(3)-C(28)-C(27)	87(3)
C(10)-N(3)-C(28)-C(27)	-83(3)
C(9)-N(3)-C(28)-C(29)	-111(2)
C(10)-N(3)-C(28)-C(29)	80(2)
C(27)-C(28)-C(29)-C(30)	-11(3)
N(3)-C(28)-C(29)-C(30)	-174.4(16)

C(28)-C(29)-C(30)-C(31)	1(3)
C(29)-C(30)-C(31)-C(36)	8(4)
C(29)-C(30)-C(31)-C(32)	171(2)
C(36)-C(31)-C(32)-C(33)	-1(4)
C(30)-C(31)-C(32)-C(33)	-163(3)
C(31)-C(32)-C(33)-C(34)	-25(5)
C(32)-C(33)-C(34)-C(35)	44(7)
C(33)-C(34)-C(35)-C(36)	-36(6)
C(30)-C(31)-C(36)-C(27)	-6(3)
C(32)-C(31)-C(36)-C(27)	-169(2)
C(30)-C(31)-C(36)-C(35)	174(2)
C(32)-C(31)-C(36)-C(35)	11(3)
C(28)-C(27)-C(36)-C(31)	-4(3)
C(26)-C(27)-C(36)-C(31)	-178(2)
C(28)-C(27)-C(36)-C(35)	176(2)
C(26)-C(27)-C(36)-C(35)	2(3)
C(34)-C(35)-C(36)-C(31)	7(4)
C(34)-C(35)-C(36)-C(27)	-173(3)
Rh(1)-O(2)-C(37)-O(1)	2(3)
Rh(1)-O(2)-C(37)-C(38)	176(3)
Rh(1)-O(1)-C(37)-O(2)	-2(3)
Rh(1)-O(1)-C(37)-C(38)	-177(3)
C(9)-Rh(1)-C(37)-O(2)	6(2)
C(1)-Rh(1)-C(37)-O(2)	-170.2(13)
O(1)-Rh(1)-C(37)-O(2)	178(3)
I(2)-Rh(1)-C(37)-O(2)	-92.1(16)
I(1)-Rh(1)-C(37)-O(2)	87.6(16)
C(9)-Rh(1)-C(37)-O(1)	-172.8(12)
C(1)-Rh(1)-C(37)-O(1)	11(2)
O(2)-Rh(1)-C(37)-O(1)	-178(3)
I(2)-Rh(1)-C(37)-O(1)	89.6(15)
I(1)-Rh(1)-C(37)-O(1)	-90.7(15)
C(9)-Rh(1)-C(37)-C(38)	-138(27)
C(1)-Rh(1)-C(37)-C(38)	47(27)
O(1)-Rh(1)-C(37)-C(38)	35(26)
O(2)-Rh(1)-C(37)-C(38)	-143(28)
I(2)-Rh(1)-C(37)-C(38)	125(27)
I(1)-Rh(1)-C(37)-C(38)	-56(27)
C(40)-N(5)-C(39)-N(6)	-5(2)
C(46)-N(5)-C(39)-N(6)	176.2(19)
C(40)-N(5)-C(39)-Rh(2)	164.1(16)
C(46)-N(5)-C(39)-Rh(2)	-15(3)
C(55)-N(6)-C(39)-N(5)	-176.4(18)
C(45)-N(6)-C(39)-N(5)	5(2)
C(55)-N(6)-C(39)-Rh(2)	15(3)
C(45)-N(6)-C(39)-Rh(2)	-163.3(17)
C(47)-Rh(2)-C(39)-N(5)	153(2)
O(3)-Rh(2)-C(39)-N(5)	-33(4)
O(4)-Rh(2)-C(39)-N(5)	-23(2)
C(75)-Rh(2)-C(39)-N(5)	-23(3)
I(4)-Rh(2)-C(39)-N(5)	-109(2)
I(3)-Rh(2)-C(39)-N(5)	65(2)
C(47)-Rh(2)-C(39)-N(6)	-41(2)
O(3)-Rh(2)-C(39)-N(6)	132(2)
O(4)-Rh(2)-C(39)-N(6)	143(2)
C(75)-Rh(2)-C(39)-N(6)	142.2(19)
I(4)-Rh(2)-C(39)-N(6)	57(2)
I(3)-Rh(2)-C(39)-N(6)	-129.6(19)
C(39)-N(5)-C(40)-C(45)	2(3)
C(46)-N(5)-C(40)-C(45)	-178.8(19)
C(39)-N(5)-C(40)-C(41)	177(3)
C(46)-N(5)-C(40)-C(41)	-4(4)
N(5)-C(40)-C(41)-C(42)	178(3)
C(45)-C(40)-C(41)-C(42)	-8(4)
C(40)-C(41)-C(42)-C(43)	10(5)
C(41)-C(42)-C(43)-C(44)	-10(6)
C(42)-C(43)-C(44)-C(45)	8(5)
N(5)-C(40)-C(45)-N(6)	2(2)
C(41)-C(40)-C(45)-N(6)	-174(2)
N(5)-C(40)-C(45)-C(44)	-178(2)
C(41)-C(40)-C(45)-C(44)	6(4)
C(55)-N(6)-C(45)-C(40)	177.0(16)

C(39)-N(6)-C(45)-C(40)	-4(2)
C(55)-N(6)-C(45)-C(44)	-3(3)
C(39)-N(6)-C(45)-C(44)	175(2)
C(43)-C(44)-C(45)-C(40)	-6(4)
C(43)-C(44)-C(45)-N(6)	174(3)
C(66)-N(7)-C(47)-N(8)	-170.8(17)
C(53)-N(7)-C(47)-N(8)	5(2)
C(66)-N(7)-C(47)-Rh(2)	16(3)
C(53)-N(7)-C(47)-Rh(2)	-168.3(16)
C(48)-N(8)-C(47)-N(7)	-6(2)
C(54)-N(8)-C(47)-N(7)	-177(2)
C(48)-N(8)-C(47)-Rh(2)	168.2(13)
C(54)-N(8)-C(47)-Rh(2)	-3(3)
C(39)-Rh(2)-C(47)-N(7)	-41(2)
O(3)-Rh(2)-C(47)-N(7)	141(2)
O(4)-Rh(2)-C(47)-N(7)	126(2)
C(75)-Rh(2)-C(47)-N(7)	135.7(19)
I(4)-Rh(2)-C(47)-N(7)	-129.2(19)
I(3)-Rh(2)-C(47)-N(7)	57.7(19)
C(39)-Rh(2)-C(47)-N(8)	146.4(17)
O(3)-Rh(2)-C(47)-N(8)	-31.5(17)
O(4)-Rh(2)-C(47)-N(8)	-46(3)
C(75)-Rh(2)-C(47)-N(8)	-37(2)
I(4)-Rh(2)-C(47)-N(8)	58.1(17)
I(3)-Rh(2)-C(47)-N(8)	-115.0(16)
C(47)-N(8)-C(48)-C(49)	-177(2)
C(54)-N(8)-C(48)-C(49)	-5(3)
C(47)-N(8)-C(48)-C(53)	4(2)
C(54)-N(8)-C(48)-C(53)	176.6(19)
C(53)-C(48)-C(49)-C(50)	-1(4)
N(8)-C(48)-C(49)-C(50)	-179(2)
C(48)-C(49)-C(50)-C(51)	0(4)
C(49)-C(50)-C(51)-C(52)	1(5)
C(50)-C(51)-C(52)-C(53)	-1(4)
C(49)-C(48)-C(53)-C(52)	1(4)
N(8)-C(48)-C(53)-C(52)	179(2)
C(49)-C(48)-C(53)-N(7)	-179.9(19)
N(8)-C(48)-C(53)-N(7)	-1(2)
C(51)-C(52)-C(53)-C(48)	0(3)
C(51)-C(52)-C(53)-N(7)	-179(2)
C(47)-N(7)-C(53)-C(48)	-2(2)
C(66)-N(7)-C(53)-C(48)	174.3(16)
C(47)-N(7)-C(53)-C(52)	177(2)
C(66)-N(7)-C(53)-C(52)	-7(3)
C(45)-N(6)-C(55)-C(64)	-98(2)
C(39)-N(6)-C(55)-C(64)	84(3)
C(45)-N(6)-C(55)-C(56)	68(2)
C(39)-N(6)-C(55)-C(56)	-110(2)
C(64)-C(55)-C(56)-C(57)	-5(3)
N(6)-C(55)-C(56)-C(57)	-171(2)
C(55)-C(56)-C(57)-C(58)	5(3)
C(56)-C(57)-C(58)-C(63)	-6(3)
C(56)-C(57)-C(58)-C(59)	177(3)
C(63)-C(58)-C(59)-C(60)	-14(5)
C(57)-C(58)-C(59)-C(60)	163(3)
C(58)-C(59)-C(60)-C(61)	48(4)
C(59)-C(60)-C(61)-C(62)	-64(4)
C(60)-C(61)-C(62)-C(63)	50(4)
C(59)-C(58)-C(63)-C(64)	-175(3)
C(57)-C(58)-C(63)-C(64)	7(4)
C(59)-C(58)-C(63)-C(62)	-3(4)
C(57)-C(58)-C(63)-C(62)	180(3)
C(61)-C(62)-C(63)-C(64)	154(3)
C(61)-C(62)-C(63)-C(58)	-19(4)
C(58)-C(63)-C(64)-C(55)	-7(3)
C(62)-C(63)-C(64)-C(55)	-180(2)
C(58)-C(63)-C(64)-C(65)	175(2)
C(62)-C(63)-C(64)-C(65)	2(3)
N(6)-C(55)-C(64)-C(63)	171.5(17)
C(56)-C(55)-C(64)-C(63)	6(3)
N(6)-C(55)-C(64)-C(65)	-10(2)
C(56)-C(55)-C(64)-C(65)	-175.6(17)

C(63)-C(64)-C(65)-C(66)	101(3)
C(55)-C(64)-C(65)-C(66)	-77(2)
C(63)-C(64)-C(65)-C(74)	-83(3)
C(55)-C(64)-C(65)-C(74)	99(2)
C(74)-C(65)-C(66)-C(67)	11(3)
C(64)-C(65)-C(66)-C(67)	-172.8(16)
C(74)-C(65)-C(66)-N(7)	174.8(18)
C(64)-C(65)-C(66)-N(7)	-9(3)
C(47)-N(7)-C(66)-C(67)	-109(2)
C(53)-N(7)-C(66)-C(67)	76(2)
C(47)-N(7)-C(66)-C(65)	85(3)
C(53)-N(7)-C(66)-C(65)	-90(2)
C(65)-C(66)-C(67)-C(68)	-10(3)
N(7)-C(66)-C(67)-C(68)	-175.6(18)
C(66)-C(67)-C(68)-C(69)	6(3)
C(67)-C(68)-C(69)-C(74)	-2(4)
C(67)-C(68)-C(69)-C(70)	179(2)
C(74)-C(69)-C(70)-C(71)	20(5)
C(68)-C(69)-C(70)-C(71)	-161(3)
C(69)-C(70)-C(71)-C(72)	-50(4)
C(70)-C(71)-C(72)-C(73)	60(4)
C(71)-C(72)-C(73)-C(74)	-43(4)
C(68)-C(69)-C(74)-C(65)	2(3)
C(70)-C(69)-C(74)-C(65)	-179(2)
C(68)-C(69)-C(74)-C(73)	-177(3)
C(70)-C(69)-C(74)-C(73)	2(4)
C(66)-C(65)-C(74)-C(69)	-6(3)
C(64)-C(65)-C(74)-C(69)	177.6(19)
C(66)-C(65)-C(74)-C(73)	173(2)
C(64)-C(65)-C(74)-C(73)	-3(3)
C(72)-C(73)-C(74)-C(69)	12(4)
C(72)-C(73)-C(74)-C(65)	-168(3)
Rh(2)-O(3)-C(75)-O(4)	-6(3)
Rh(2)-O(3)-C(75)-C(76)	-177(3)
Rh(2)-O(4)-C(75)-O(3)	6(3)
Rh(2)-O(4)-C(75)-C(76)	177(3)
C(39)-Rh(2)-C(75)-O(3)	-173.1(15)
C(47)-Rh(2)-C(75)-O(3)	11(2)
O(4)-Rh(2)-C(75)-O(3)	-175(3)
I(4)-Rh(2)-C(75)-O(3)	-88.8(17)
I(3)-Rh(2)-C(75)-O(3)	91.2(17)
C(39)-Rh(2)-C(75)-O(4)	1(2)
C(47)-Rh(2)-C(75)-O(4)	-174.1(13)
O(3)-Rh(2)-C(75)-O(4)	175(3)
I(4)-Rh(2)-C(75)-O(4)	85.8(15)
I(3)-Rh(2)-C(75)-O(4)	-94.1(14)
C(39)-Rh(2)-C(75)-C(76)	-58(44)
C(47)-Rh(2)-C(75)-C(76)	126(44)
O(3)-Rh(2)-C(75)-C(76)	115(44)
O(4)-Rh(2)-C(75)-C(76)	-60(43)
I(4)-Rh(2)-C(75)-C(76)	26(44)
I(3)-Rh(2)-C(75)-C(76)	-154(44)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd25468 [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
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