Supporting Information

Axially Chiral NHC-Pd(II) Complexes in the Oxidative Kinetic Resolution of Secondary Alcohols Using Molecular Oxygen as a Terminal Oxidant

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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 Bruker AM-300 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 Bruker AM-300 spectrophotometers (75 MHz) 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. Percent conversion was analyzed by GC using a supelcowaxTM-10 fused silica capillary column (30.0 m x 0.25 mm x 0.25 μm) purchased from Supelco Industries. Chiral HPLC was performed on a SHIMADZU SPD-10A *vp* series with chiral columns (Chiralpak AS-H, OD-H and OJ-H columns 4.6x250mm, (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. Racemic alcohols were purchased from commercial company or prepared by corresponding aldehydes or reduced from corresponding ketones.¹

General procedure for the synthesis of catalysts and the data of compounds 2, 5-7, 10-13 and NHC-Pd(II) complex 1.

NHC-Pd(II) complex 1 was synthesized according to the literature.²

Compound 5. It is a known compounds.² A red solid; Mp. $202.8\text{-}203.3^\circ\text{C}$; $[\alpha]_{\text{D}}^{20} -496.0$ (c 0.22, CHCl_3); $^1\text{H NMR}$ (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 6. It is a known compounds.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 7. It is a known compounds.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).

MHC-Pd(II) complex 1. It is a known compounds.³ A yellow solid; Mp. > 300 °C (dec.); $[\alpha]_D^{20} +270.0$ (c 0.086, CHCl₃); ¹H NMR (300 MHz, CDCl₃, TMS): δ 3.82 (s, 6H, CH₃), 6.68 (d, $J = 8.7$ Hz, 2H, ArH), 6.74-6.78 (m, 4H, ArH), 6.82-6.85 (m, 2H, ArH), 6.87-6.94 (m, 4H, ArH), 7.19-7.25 (m, 2H, ArH), 7.72 (d, $J = 8.4$ Hz, 2H, ArH), 8.03-8.10 (m, 4H, ArH).

(*R*)-(+)-N-(2-nitrophenyl)-1-(2-(2-nitrophenylamino)-5,6,7,8-tetrahydronaphthalen-1-yl)-5,6,7,8-tetrahydronaphthalen-2-amine (10). 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*)-**9** (146 mg, 0.50 mmol), 2-bromonitrobenzene **4** (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 (4.0 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 **10** as a red solid; Yield: 253 mg (95%). Mp. 192.8-193.5 °C; $[\alpha]_D^{20}$ -539.0 (c 0.25, CHCl₃); IR (KBr): 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⁺, 51.71), 499 (M⁺-35, 100), 465 (M⁺-69, 14.88), 341 (M⁺-185, 20.95); 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 (11). A mixture of **10** (134 mg, 0.25 mmol), 10% Pd-C (15 mg) in 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 **11** as a white solid; Yield: 109 mg (92%). Mp. 96.9-97.9 °C; $[\alpha]_D^{20} +187.0$ (c 0.52, CHCl₃); IR (KBr): ν 3368, 2925, 2856, 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, 18.79), 325 (M⁺-149, 5.59); HRMS (EI) Calcd. for C₃₂H₃₄N₄ (M⁺) requires 474.2783, Found: 474.2740.

Synthesis

of

(R)-(+)-1-(5-(6-(1H-benzo[d]imidazol-1-yl)-1,2,3,4-tetrahydronaphthalen-5-yl)-1,2,3,4-tetrahydronaphthalen-6-yl)-1H-benzo[d]imidazole (12). The compound **11** (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 **12** 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 (KBr): 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 ($\text{M}^+ - 1$, 32.96), 452 ($\text{M}^+ - 42$, 3.60), 375 ($\text{M}^+ - 119$, 6.37); 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 **13.** The compound **12** (99 mg, 0.20 mmol) and CH₃I (0.24 mL, 4 mmol) in CH₃CN (4.0 mL) were stirred under reflux for 5 h. After cooling to room temperature, volatiles were removed under reduced pressure and the obtained solid compound **13** 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-Pd(II) complex 2. The compound **13** (156 mg, 0.2 mmol) and Pd(OAc)₂ (44.8 mg, 0.2 mmol) was refluxed in THF (10 mL) for 16 h. The volatiles were then removed under reduced pressure and the residue was purified by a silica gel flash column chromatography (eluent: petroleum ether/ethyl acetate, 2/1 - 0/1) to give **2** as a yellow solid (132.6 mg, 75%). A single crystal suitable for X-ray crystal analysis was obtained by recrystallization from a saturated solution of CH₂Cl₂/PE. Mp. > 300 °C (dec.); [α]_D²⁰ +53 (c 0.24, CHCl₃); IR (CH₂Cl₂): ν 3054, 2930, 2306, 1480, 1436, 1340, 1132, 897, 740, 705, 557 cm⁻¹; ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.17-1.41 (6H, m, CH₂), 1.55-1.64 (4H, m, CH₂), 1.81-1.91 (2H, m, CH₂), 2.20-2.31 (2H, m, CH₂), 2.56-2.66 (2H, m, CH₂), 3.92 (6H, s, CH₃), 6.89 (2H, d, *J* = 8.1 Hz, ArH), 7.11-7.15 (4H, m, ArH), 7.18-7.20 (1H, m, ArH), 7.22-7.24 (3H, m, ArH), 7.60 (2H, d, *J* = 8.1 Hz, ArH); MS (EI) *m/z* (%): 882 (M⁺, 0.01), 755 (M⁺-127, 1.33), 508 (M⁺-374, 1.45), 492 (M⁺-390, 100), 142 (M⁺-740, 23.04); Anal. Calcd. for C₃₆H₃₄I₂N₄Pd requires: C 48.97, H 3.88, N 6.35%. Found: C 49.40, H 4.03, N 6.31%.

General procedure for the oxidation of secondary alcohols.

Base screening trials.

A 25 mL Schlenk flask equipped with a magnetic stir bar was charged with powdered molecular sieves (MS 3A, 250 mg) and flame-dried under vacuum. After cooling under dry Ar, racemic NHC-Pd(II) complex **1** (21.8 mg, 0.025 mmol, 0.05 equiv), and base (0.25 mmol, 0.5 equiv) was added followed by toluene (5.0 mL). The flask was vacuum evacuated and filled with O₂ (3 times, balloon), then the alcohol (0.50 mmol, 1.0 equiv) was introduced. The reaction mixture was allowed stirred at 80 °C for 48 h. After cooling to room temperature, the reaction mixture was filtered through a small plug of silica gel (eluent: ethyl acetate), and biphenyl (38.5 mg, 0.25 mmol, 0.5 equiv) was added as internal standard, then analyzed by GC for percent conversion.

Solvent screening trials.

A 25 mL Schlenk flask equipped with a magnetic stir bar was charged with powdered molecular sieves (MS3A, 250 mg) and flame-dried under vacuum. After cooling under dry Ar, racemic NHC-Pd(II) complex **1** (21.8 mg, 0.025 mmol, 0.05 equiv), and Cs₂CO₃ (81.5 mg, 0.25 mmol, 0.5 equiv) was added followed by solvent (5.0 mL). The flask was vacuum evacuated and filled with O₂ (3 times, balloon), then the alcohol (0.50 mmol, 1.0 equiv) was introduced. The reaction mixture was allowed stirred at 80 °C for 48 h. After cooling to room temperature, the reaction mixture was filtered through a small plug of silica gel (eluent: ethyl acetate), and biphenyl (38.5 mg, 0.25 mmol, 0.5 equiv) was added as internal standard, then analyzed by GC for percent conversion. During examination of the solvent effects under oxygen atmosphere, the reaction should be carried out with safety caution at elevated temperature.

General procedure for the oxidative kinetic resolution of secondary alcohols.

A typical procedure is given below for the reaction expressed in entry 7 of Table SI-3. A 25 mL Schlenk flask equipped with a magnetic stir bar was charged with powdered molecular sieves (MS 4A, 250 mg) and flame-dried under vacuum. After cooling under dry Ar, chiral NHC-Pd(II) complex **1** (43.6 mg, 0.05 mmol, 0.1 equiv), and Cs₂CO₃ (81.5 mg, 0.25 mmol, 0.5 equiv) was added followed by toluene (5.0 mL). The flask was vacuum evacuated and filled with O₂ (3 times, balloon), then the alcohol (0.50 mmol, 1.0 equiv) was introduced. The reaction mixture was allowed stirred at 80 °C for 48 h. After cooling to room temperature, the reaction mixture was filtered through a small plug of silica gel (eluent: ethyl acetate), then analyzed by GC for percent conversion. After that, the solvent was removed under reduced pressure and the residue was purified by a silica gel flash column chromatography (eluent: petroleum ether/ethyl acetate, 15/1 - 10/1) to give 1-phenylethanol as a colorless oil, which was analyzed by HPLC for enantiomeric excess.

^1H NMR spectra.

1-phenylethanol (14a). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.50 (d, $J = 6.6$ Hz, 3H, CH_3), 1.89 (br, 1H, OH), 4.90 (q, $J = 6.6$ Hz, 1H, CH), 7.27-7.40 (m, 5H, ArH).

Acetophenone (14b). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 2.61 (s, 3H, CH_3), 7.44-7.50 (m, 2H, ArH), 7.54-7.57 (m, 1H, ArH), 7.95-7.98 (m, 2H, ArH).

1-(4-fluorophenyl)ethanol (15a). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.47 (d, $J = 6.3$ Hz, 3H, CH_3), 1.86 (br, 1H, OH), 4.88 (q, $J = 6.3$ Hz, 1H, CH), 7.00-7.06 (m, 2H, ArH), 7.31-7.36 (m, 2H, ArH).

1-(4-fluorophenyl)ethanone (15b). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 2.59 (s, 3H, CH_3), 7.10-7.15 (m, 2H, ArH), 7.96-8.00 (m, 2H, ArH).

1-(4-chlorophenyl)ethanol (16a). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.44 (d, $J = 6.6$ Hz, 3H, CH_3), 2.36 (br, 1H, OH), 4.83 (q, $J = 6.6$ Hz, 1H, CH), 7.25-7.31 (m, 4H, ArH).

1-(4-chlorophenyl)ethanone (16b). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 2.52 (s, 3H, CH_3), 7.36 (d, $J = 8.7$ Hz, 2H, ArH), 7.82 (d, $J = 8.7$ Hz, 2H, ArH).

1-(4-bromophenyl)ethanol (17a). A yellow solid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.44 (d, $J = 6.6$ Hz, 3H, CH_3), 2.31 (br, 1H, OH), 4.81 (q, $J = 6.6$ Hz, 1H, CH), 7.21 (d, $J = 8.4$ Hz, 2H, ArH), 7.45 (d, $J = 8.4$ Hz, 2H, ArH).

1-(4-bromophenyl)ethanone (17b). A white solid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 2.57 (s, 3H, CH_3), 7.58 (d, $J = 8.7$ Hz, 2H, ArH), 7.80 (d, $J = 8.7$ Hz, 2H, ArH).

1-p-tolylethanol (18a). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.48 (d, J = 6.9 Hz, 3H, CH_3), 2.38 (s, 3H, CH_3), 2.60 (br, 1H, OH), 4.83 (q, J = 6.9 Hz, 1H, CH), 7.17 (d, J = 7.1 Hz, 2H, ArH), 7.27 (d, J = 7.1 Hz, 2H, ArH).

1-p-tolylethanone (18b). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 2.39 (s, 3H, CH_3), 2.55 (s, 3H, CH_3), 7.23 (d, J = 7.8 Hz, 2H, ArH), 7.84 (d, J = 7.8 Hz, 2H, ArH).

1-(4-Methoxy-phenyl)-ethanol (19a). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.48 (d, $J = 6.6$ Hz, 3H, CH_3), 1.80 (br, 1H, OH), 3.81 (s, 3H, CH_3), 4.86 (q, $J = 6.6$ Hz, 1H, CH), 6.87-6.91 (m, 2H, ArH), 7.29-7.33 (m, 2H, ArH).

1-(4-Methoxy-phenyl)-ethanone (19b). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 2.56 (s, 3H, CH_3), 3.88 (s, 3H, CH_3), 6.94 (d, $J = 8.7$ Hz, 2H, ArH), 7.94 (d, $J = 8.7$ Hz, 2H, ArH).

1-(4-chlorophenyl)ethanone (20a). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 0.88 (t, $J = 6.9$ Hz, 3H, CH_3), 1.63-1.81 (m, 2H, CH_2), 2.26 (br, 1H, OH), 4.50-4.55 (m, 1H, CH), 7.18 (d, $J = 8.4$ Hz, 2H, ArH), 7.45 (d, $J = 8.4$ Hz, 2H, ArH).

1-(4-chlorophenyl)ethanone (20b). A white solid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.22 (t, $J = 7.2$ Hz, 3H, CH_3), 2.98 (q, $J = 7.2$ Hz, 2H, CH_2), 7.60 (d, $J = 8.7$ Hz, 2H, ArH), 7.83 (d, $J = 8.7$ Hz, 2H, ArH).

1-(3-fluorophenyl)ethanol (21a). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.46 (d, $J = 6.6$ Hz, 3H, CH_3), 2.21 (br, 1H, OH), 4.87 (q, $J = 6.6$ Hz, 1H, CH), 6.91-6.98 (m, 1H, ArH), 7.06-7.12 (m, 2H, ArH), 7.26-7.33 (m, 1H, ArH).

1-(3-fluorophenyl)ethanone (21b). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 2.61 (s, 3H, CH_3), 7.24-7.31 (m, 1H, ArH), 7.42-7.49 (m, 1H, ArH), 7.63-7.66 (m, 1H, ArH), 7.73-7.76 (m, 1H, ArH).

1-(3-Fluoro-phenyl)-propan-1-ol (22a). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 0.90 (t, $J = 7.2$ Hz, 3H, CH_3), 1.66-1.80 (m, 2H, CH_2), 2.34 (br, 1H, OH), 4.55-4.59 (m, 1H, CH), 6.91-6.98 (m, 1H, ArH), 7.02-7.09 (m, 2H, ArH), 7.25-7.33 (m, 1H, ArH).

1-(3-Fluoro-phenyl)-propan-1-one (22b). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.23 (t, $J = 7.2$ Hz, 3H, CH_3), 2.99 (q, 2H, $J = 7.2$ Hz, 2H, CH_2), 7.22-7.29 (m, 1H, ArH), 7.41-7.48 (m, 1H, ArH), 7.62-7.67 (m, 1H, ArH), 7.73-7.76 (m, 1H, ArH).

1-(3-Fluorophenyl)pentan-1-ol (23a). A colorless liquid; ¹H NMR (300 MHz, CDCl₃, TMS): δ 0.89 (t, *J* = 6.9 Hz, 3H, CH₃), 1.19-1.43 (m, 4H, CH₂), 1.62-1.83 (m, 2H, CH₂), 2.10 (br, 1H, OH), 4.65 (t, *J* = 6.6 Hz, 1H, CH), 6.92-6.98(m, 1H, ArH), 7.04-7.10 (m, 2H, ArH), 7.26-7.33 (m, 1H, ArH).

1-(3-Fluoro-phenyl)-pentan-1-one (23b). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 0.96 (t, $J = 7.5$ Hz, 3H, CH_3), 1.35-1.47 (m, 2H, CH_2), 1.67-1.77 (m, 2H, CH_2), 2.95 (t, $J = 7.2$ Hz, 2H, CH_2), 7.22-7.28(m, 1H, ArH), 7.4.-7.48 (m, 1H, ArH), 7.61-7.66 (m, 1H, ArH), 7.73-7.76 (m, 1H, ArH).

1-m-Tolyl-ethanol (24a). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.49 (d, $J = 6.0$ Hz, 3H, CH_3), 1.88 (br, 1H, OH), 2.36 (s, 3H, CH_3), 4.83-4.89 (m, 1H, CH), 7.08-7.27(m, 4H, ArH).

1-m-Tolyl-ethanone (24b). A colorless liquid; ¹H NMR (300 MHz, CDCl₃, TMS): δ 2.41 (s, 3H, CH₃), 2.59 (s, 3H, CH₃), 7.32-7.39 (m, 2H, ArH), 7.74-7.77 (m, 2H, ArH).

1-Naphthalen-1-yl-ethanol (25a). A white solid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 1.58 (d, $J = 6.3$ Hz, 3H, CH_3), 2.43 (br, 1H, OH), 5.54-5.56 (m, 1H, CH), 7.39-7.49 (m, 3H, ArH), 7.60 (d, $J = 6.6$ Hz, 1H, ArH), 7.72 (d, $J = 8.1$ Hz, 1H, ArH), 7.81-7.84 (m, 1H, ArH), 8.01-8.04 (m, 1H, ArH).

1-Naphthalen-1-yl-ethanone (25b). A colorless liquid; ^1H NMR (300 MHz, CDCl_3 , TMS): δ 2.70 (s, 3H, CH_3), 7.42-7.58 (m, 3H, ArH), 7.82-7.96 (m, 3H, ArH), 8.75 (d, $J = 8.7$ Hz, 1H, ArH).

1-Naphthalen-2-yl-ethanol (26a). A white solid; ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.58 (d, *J* = 6.3 Hz, 3H, CH₃), 1.99 (br, 1H, OH), 5.04-5.10 (m, 1H, CH), 7.43-7.54 (m, 3H, ArH), 7.81-7.85 (m, 4H, ArH).

1-Naphthalen-2-yl-ethanone (26b). A white solid; ¹H NMR (300 MHz, CDCl₃, TMS): δ 2.72

(s, 3H, CH₃), 7.52-7.62 (m, 2H, ArH), 7.86-7.90 (m, 2H, ArH), 7.96 (d, *J* = 7.5 Hz, 1H, ArH), 8.01-7.05 (m, 1H, ArH), 8.46 (s, 1H, ArH).

1-Benzofuran-2-yl-ethanol (27a). A colorless liquid; ¹H NMR (300 MHz, CDCl₃, TMS): δ 1.62 (d, *J* = 6.6 Hz, 3H, CH₃), 2.32 (d, *J* = 4.8 Hz, 1H, OH), 4.96-5.04 (m, 1H, CH), 6.59 (s, 1H, ArH), 7.18-7.29 (m, 2H, ArH), 7.45 (d, *J* = 8.4 Hz, 1H, ArH), 7.53 (d, *J* = 8.4 Hz, 1H, ArH).

1-Benzofuran-2-yl-ethanone (27b). A white solid; ¹H NMR (300 MHz, CDCl₃, TMS): δ 2.61 (s, 3H, CH₃), 7.29-7.34 (m, 1H, ArH), 7.45-7.51 (m, 2H, ArH), 7.58 (d, *J* = 8.4 Hz, 1H, ArH), 7.71 (d, *J* = 8.4 Hz, 1H, ArH).

3-Methyl-4-phenyl-but-3-en-2-ol (28a). A colorless liquid; ¹H NMR (300 MHz, CDCl₃,

TMS): δ 1.35 (d, $J = 6.6$ Hz, 3H, CH₃), 1.87 (s, 3H, CH₃), 2.05 (br, 1H, OH), 4.36 (q, $J = 6.6$ Hz, 1H, CH), 6.50 (s, 1H, =CH), 7.18-7.34 (m, 5H, ArH).

3-Methyl-4-phenyl-but-3-en-2-one (28b). A colorless liquid; ¹H NMR (300 MHz, CDCl₃, TMS): δ 2.05 (s, 3H, CH₃), 2.47 (s, 3H, CH₃), 7.34-7.43 (m, 5H, ArH), 7.53 (s, 1H, =CH).

1,2,3,4-Tetrahydro-naphthalen-1-ol (29a). A white solid; ¹H NMR (300 MHz, CDCl₃, TMS):

δ 1.71-1.78 (m, 1H, CH₂), 1.81-2.03 (m, 4H, CH₂), 2.66-2.87 (m, 2H, CH₂), 4.75 (m, 1H, CH), 7.08-7.12 (m, 1H, ArH), 7.17-7.24 (m, 2H, ArH), 7.40-7.43 (m, 1H, ArH).

3,4-Dihydro-2H-naphthalen-1-one (29b). A colorless liquid; ¹H NMR (300 MHz, CDCl₃, TMS): δ 2.10-2.19 (m, 2H, CH₂), 2.64-2.68 (m, 2H, CH₂), 2.95-2.99 (m, 2H, CH₂), 7.24-7.33 (m, 2H, ArH), 7.45-7.50 (m, 1H, ArH), 8.02-8.05 (m, 1H, ArH).

HPLC trace.

WH-500 HPLC report
WH-500 色谱分析报告

Translation: Content, R time, Peak height, Peak area, Percent, Tailing factor, Theoretic separation

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		8.193	81272	1200181.1	49.8213	1.22	6135
2		9.708	71224	1208791.9	50.1787	1.14	6522
Σ:			152496	2408973.0	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		8.288	4945	59885.2	3.2544	1.04	9336
2		9.682	127965	1780250.7	96.7456	1.31	9653
Σ:			132910	1840135.8	100.0000		

金属有机HPLC分析报告

样品文件名: lxx-f-1. che

分析日期: 2006-06-07

流动相:

检测波长:

分析者:

色谱柱:

流速:

柱压:

No.	R. Time	PeakHeight	PeakArea	PerCent
1	15.210	240062.3	5254316.8	48.0599
2	15.960	226718.0	5319783.4	48.6587
3	16.877	4203.1	95983.0	0.8779
4	17.877	10572.7	262760.1	2.4034
Total		481586.2	10932843.3	100.0000

金属有机HPLC分析报告

样品文件名: lxx-f-2. che

分析日期: 2006-06-07

流动相:

检测波长:

catalyst-1

分析者:

色谱柱:

流速:

柱压:

No.	R. Time	PeakHeight	PeakArea	PerCent
1	15.043	183300.1	4095157.2	91.8715
2	15.793	11716.5	259275.9	5.8166
3	17.710	4168.9	103048.4	2.3118
Total		199185.4	4457481.5	100.0000

金属有机HPLC分析报告

样品文件名: lxx-4-f. che

分析日期: 2006-06-07

流动相:

检测波长:

catalyst-2

分析者:

色谱柱:

流速:

柱压:

色谱分析报告

样品名称: 分析谱图

样品批号:

分析日期: 2006-05-11

色谱柱:

流速:

样品文件名: lxx-b. che

分析者:

分析时间: 09:10

流动相:

检测波长:

色谱分析报告

样品名称:分析谱图

样品文件名:lxg-a.che

样品批号:

分析者:

分析日期:2006-05-11

分析时间:09:55

色谱柱:

流动相:

流速:

catalyst-1

检测波长:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	21.377	273029.6	9698358.6	88.5226
2	2	Unknown	23.293	34716.8	1257439.1	11.4774
Total				307746.4	10955797.7	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		20.910	170514	4991124.7	49.5956	1.14	10171
2		22.833	158784	5072516.7	50.4044	1.16	10182
Σ:			329298	10063641.4	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		21.005	144283	4159315.1	99.0189	1.14	10582
2		23.055	1504	41211.0	0.9811	0.99	14110
Σ:			145787	4200526.0	100.0000		

金属有机HPLC分析报告

样品文件名: lxx-1. che

分析日期: 2006-04-28

流动相:

检测波长:

分析者:

色谱柱:

流速:

柱压:

No.	R. Time	PeakHeight	PeakArea	PerCent
1	28.210	43511.0	1929232.4	49.9467
2	30.293	40487.8	1933346.8	50.0533
Total		83998.8	3862579.2	100.0000

金属有机HPLC分析报告

样品文件名: lxx-2. che

分析日期: 2006-04-28

流动相:

检测波长:

catalyst 1

分析者:

色谱柱:

流速:

柱压:

No.	R. Time	PeakHeight	PeakArea	PerCent
1	27.960	789719.6	37171172.2	89.9865
2	30.293	82815.3	4136322.8	10.0135
Total		872534.9	41307495.0	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		39.825	68213	4194033.3	50.1387	1.06	8362
2		42.873	64519	4170827.7	49.8613	1.06	8767
Σ:			132732	8364861.0	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		39.873	91780	5725131.7	96.5224	1.06	8144
2		42.735	2914	206271.8	3.4776	1.04	7264
Σ:			94694	5931403.6	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		9.163	63871	2268074.5	49.8322	0.85	1327
2		10.735	63007	2283350.3	50.1678	0.96	1749
Σ:			126878	4551424.8	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		9.138	9567	282700.2	13.5325	0.81	1906
2		10.692	51877	1806343.8	86.4675	0.94	1879
Σ:			61444	2089044.0	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		23.425	9155	341351.8	49.8152	1.21	7867
2		25.292	8483	343884.2	50.1848	1.19	7758
Σ:			17638	685236.0	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		23.775	858	32202.8	5.2658	1.10	7998
2		25.450	14071	579337.4	94.7342	1.28	7615
Σ:			14929	611540.2	100.0000		

色谱分析报告

样品名称:分析谱图

样品文件名:lgx-11-14-2.che

样品批号:

分析者:

分析日期:2006-11-17

分析时间:11:34

色谱柱:

流动相:

流速:

检测波长:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	13.052	4454.8	110903.7	4.2918
2	2	Unknown	15.085	4397.3	117910.7	4.5630
3	3	Unknown	16.485	37418.3	1179241.9	45.6353
4	4	Unknown	19.018	34333.8	1176000.1	45.5098
Total				80604.2	2584056.4	100.0000

色谱分析报告

样品名称:分析谱图
 样品批号:
 分析日期:2006-11-17
 色谱柱: catalyst 2
 流速:

样品文件名:lxg-19-34
 分析者:
 分析时间:13:32
 流动相:
 检测波长:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	12.293	14813.0	6893183.5	45.3669
2	2	Unknown	14.377	807.4	21298.7	0.1402
3	3	Unknown	16.710	17626.7	602846.9	3.9676
4	4	Unknown	18.793	186131.4	7676984.0	50.5254
Total				219378.5	15194313.1	100.0000

HPLC report from State Key Lab. of Organometallic Chem.

金属有机HPLC分析报告

样品文件名: lxxg-j-1. che

分析日期: 2006-06-01

流动相:

检测波长:

分析者:

色谱柱:

流速:

柱压:

金属有机HPLC分析报告

样品文件名: lxxg-j-2. che

分析日期: 2006-06-01

流动相:

检测波长:

分析者:

色谱柱:

流速:

柱压:

金属有机HPLC分析报告

样品文件名: lxx-3-f-1. che

分析日期: 2006-06-09

流动相:

检测波长:

分析者:

色谱柱:

流速:

柱压:

No.	R. Time	PeakHeight	PeakArea	PerCent
1	14.960	190825.6	4238855.7	49.6751
2	16.293	178372.9	4294305.4	50.3249
Total		369198.5	8533161.1	100.0000

金属有机HPLC分析报告

样品文件名: lxx-3-f-2. che

分析日期: 2006-06-09

流动相:

检测波长:

分析者:

色谱柱:

流速:

柱压:

No.	R. Time	PeakHeight	PeakArea	PerCent
1	15.377	510865.4	11229210.8	93.1651
2	16.710	32984.6	785131.1	6.5349
Total		543850.1	12014341.9	100.0000

HPLC report

色谱分析报告

样品名称:分析谱图

样品文件名:1xg-11-14-3 che

样品批号:

分析者:

分析日期:2006-11-16

分析时间:15:20

色谱柱:

流动相:

流速:

检测波长:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	5.210	118.8	36885.0	0.2373
2	2	Unknown	19.793	265001.0	7783294.7	50.0748
3	3	Unknown	21.377	242688.8	7723157.8	49.6879
Total			507808.7	15543337.5	100.0000	

色谱分析报告

样品名称:分析谱图

样品文件名:1xg-9-36-1. che

样品批号:

分析者:

分析日期:2006-11-17

分析时间:10:01

色谱柱:

流动相:

流速:

检测波长:

No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1	Unknown	20.710	5980.0	149226.7	15.3806
2	2	Unknown	22.377	30594.7	821001.2	84.6194
Total			36574.7	970227.9	100.0000	

HPLC report from State Key Lab. of Organometallic Chem.

金属有机HPLC分析报告

样品文件名: lxx-9-39-1-rac. che

分析日期: 2006-12-06

流动相:

检测波长:

分析者:

色谱柱:

流速:

柱压:

No.	R. Time	PeakHeight	PeakArea	PerCent
1	8.960	156207.7	2278277.5	49.5610
2	9.960	135190.0	2318636.6	50.4390
Total		291397.7	4596914.1	100.0000

金属有机HPLC分析报告

样品文件名: lxx-9-39-1. che

分析日期: 2006-12-06

流动相:

检测波长:

分析者:

色谱柱:

流速:

柱压:

No.	R. Time	PeakHeight	PeakArea	PerCent
1	8.185	833.1	34008.5	6.3094
2	9.127	26344.6	403617.2	74.8804
3	10.043	3892.4	66394.5	12.3177
4	11.127	573.5	34995.9	6.4926
Total		31643.6	539016.1	100.0000

HPLC report

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		19.808	169154	17158008.1	49.9822	0.79	760
2		32.275	137672	17170256.7	50.0178	0.78	1335
Σ:			306826	34328264.7	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		19.608	2518	251714.8	5.7414	0.78	767
2		31.958	33522	4132468.3	94.2586	0.77	1339
Σ:			36040	4384183.0	100.0000		

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		22.525	10998	937424.0	50.2327	0.80	1392
2		27.342	10452	928740.1	49.7673	0.86	1887
Σ:			21450	1866164.1	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
1		22.542	1451	206512.3	3.1452	1.15	500
2		27.225	82964	6359473.8	96.8548	0.96	2514
Σ:			84415	6565986.0	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		29.425	57986	2420795.6	49.9768	1.33	9901
2		31.458	54532	2423042.2	50.0232	1.27	9990
Σ:			112518	4843837.8	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		29.133	97127	4086152.3	18.3656	1.35	9558
2		31.050	385280	18162787.5	81.6344	1.40	8646
Σ:			482407	22248939.8	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		16.108	841597	58875659.4	49.9043	0.82	1057
2		18.833	796798	59101365.9	50.0957	0.83	1285
Σ:			1638395	117977025.3	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		16.800	126703	8435577.4	11.4800	0.79	1269
2		19.942	886513	65045343.8	88.5200	0.86	1472
Σ:			1013216	73480921.2	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		16.000	13219	802227.7	12.1353	0.80	1385
2		18.608	92131	5808464.0	87.8647	0.83	1736
Σ:			105350	6610691.7	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		12.575	448831	17514937.0	50.2507	0.96	2070
2		17.008	405937	17340176.5	49.7493	1.17	3160
Σ:			854768	34855113.5	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		12.475	563162	11498515.5	99.8503	1.36	7440
2		16.567	625	17239.5	0.1497	1.06	7190
Σ:			563787	11515755.0	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		12.683	618743	12467965.8	99.8099	1.39	7896
2		17.192	844	23741.5	0.1901	1.00	7444
Σ:			619587	12491707.3	100.0000		

Examples of GC trace of the oxidative kinetic resolution.

GC report

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		26.403	4539	23109.6	66.5290	0.62	536018
2		29.552	2805	11626.5	33.4710	0.73	1013109
Σ:			7344	34736.1	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
1		25.765	3527	20623.2	61.2135	0.62	386976
2		30.747	3094	13067.4	38.7865	0.78	1056290
Σ:			6621	33690.6	100.0000		

References.

- [1] Koul, S.; Koul, J. L.; Taneja, S. C.; Dhar, K. L.; Jamwal, D. S.; Singh, K.; Reen, R.K.; Singh, J. *Bioorg. Med. Chem.* **2000**, 8, 251-268.
- [2] Duan, W.-L.; Shi, M.; Rong, G.-B. *Chem. Commun.* **2003**, 2916-2917.
- [3] Xu, Q.; Duan, W. -L.; Lei, Z. Y.; Zhu, Z. B. Shi, M. *Tetrahedron* **2005**, 61, 11225-11229.

The crystal data of complex **2** has been deposited in CCDC with number 603240. Empirical Formula: $\text{C}_{37}\text{H}_{36}\text{Cl}_2\text{N}_4\text{I}_2\text{Pd}$; Formula Weight: 967.80; Crystal Color, Habit: colorless, prismatic; Crystal System: Orthorhombic; Lattice Type: Primitive; Lattice Parameters: $a = 12.8832(8)\text{\AA}$, $b = 14.0578(9)\text{\AA}$, $c = 209.2839(13)\text{\AA}$, $\alpha = 90^\circ$, $\beta = 90^\circ$, $\gamma = 90^\circ$, $V = 3673.6(4)\text{\AA}^3$; Space group: $P2(1)2(1)2(1)$; $Z = 4$; $D_{\text{calc}} = 1.750\text{ g/cm}^3$; $F_{000} = 1888$; Diffractometer: Rigaku AFC7R; Residuals: R ; R_w : 0.543, 0.1246.

Table 1. Crystal data and structure refinement for cd25504.

Identification code	cd25504
Empirical formula	C37 H36 Cl2 I2 N4 Pd
Formula weight	967.80
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 12.8832(8) Å alpha = 90 deg. b = 14.0578(9) Å beta = 90 deg. c = 20.2839(13) Å gamma = 90 deg.
Volume	3673.6(4) Å ³
Z, Calculated density	4, 1.750 Mg/m ³
Absorption coefficient	2.363 mm ⁻¹
F(000)	1888
Crystal size	0.505 x 0.375 x 0.213 mm
Theta range for data collection	1.76 to 27.00 deg.
Limiting indices	-16<=h<=15, -17<=k<=15, -25<=l<=25
Reflections collected / unique	21852 / 8002 [R(int) = 0.0854]
Completeness to theta = 27.00	100.0 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.63407
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	8002 / 2 / 402
Goodness-of-fit on F ²	0.947
Final R indices [I>2sigma(I)]	R1 = 0.0543, wR2 = 0.1246
R indices (all data)	R1 = 0.0741, wR2 = 0.1327
Absolute structure parameter	-0.02(3)
Largest diff. peak and hole	1.045 and -0.706 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd25504. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Pd	7009(1)	9161(1)	9433(1)	45(1)
I(1)	5192(1)	9981(1)	9685(1)	74(1)
I(2)	7832(1)	9759(1)	10573(1)	69(1)
Cl(1)	6869(6)	7163(5)	5297(3)	213(2)
Cl(2)	8989(6)	6981(6)	5046(4)	239(3)
N(1)	9242(5)	9485(4)	9121(3)	48(2)
N(2)	8896(5)	7988(4)	9087(3)	40(1)
N(3)	5518(5)	7884(5)	8729(3)	47(2)
N(4)	6552(5)	8583(4)	8018(3)	41(1)
C(1)	8451(6)	8855(5)	9170(3)	41(2)
C(2)	10179(7)	9027(6)	9056(3)	49(2)
C(3)	11207(7)	9376(7)	9067(4)	64(2)
C(4)	11957(8)	8725(8)	9006(4)	69(3)
C(5)	11737(7)	7758(8)	9002(4)	61(2)
C(6)	10772(7)	7388(6)	9003(4)	53(2)
C(7)	9978(6)	8075(6)	9031(3)	44(2)
C(8)	9073(7)	10518(6)	9118(5)	67(3)
C(9)	6332(6)	8466(5)	8678(3)	42(2)
C(10)	5147(7)	7648(5)	8099(4)	49(2)
C(11)	4305(7)	7106(6)	7891(5)	58(2)
C(12)	4146(8)	7063(8)	7234(6)	73(3)
C(13)	4794(8)	7499(7)	6788(5)	71(3)
C(14)	5642(7)	8045(7)	6972(5)	62(2)
C(15)	5804(7)	8086(6)	7649(4)	46(2)
C(16)	5096(7)	7505(6)	9322(5)	66(2)
C(17)	8311(6)	7188(5)	8837(4)	40(2)
C(18)	8017(7)	6472(6)	9280(4)	54(2)
C(19)	7428(6)	5737(6)	9026(5)	59(2)
C(20)	7211(6)	5643(5)	8362(4)	46(2)
C(21)	6527(7)	4851(6)	8133(5)	68(2)
C(22)	6655(8)	4655(6)	7416(6)	85(3)
C(23)	6561(8)	5565(7)	7007(5)	77(3)
C(24)	7425(6)	6231(6)	7197(4)	57(2)
C(25)	7550(5)	6356(5)	7929(4)	42(2)
C(26)	8082(5)	7165(5)	8177(3)	36(2)
C(27)	8304(6)	8021(5)	7770(3)	40(2)
C(28)	7579(6)	8758(6)	7747(3)	45(2)
C(29)	7768(7)	9604(6)	7452(4)	59(2)
C(30)	8708(8)	9728(7)	7137(4)	71(3)
C(31)	9425(7)	8996(7)	7108(4)	59(2)
C(32)	10440(8)	9166(10)	6754(5)	91(4)
C(33)	11018(9)	8289(12)	6572(5)	111(5)
C(34)	11048(7)	7637(10)	7158(6)	98(4)
C(35)	9973(7)	7314(7)	7379(5)	62(2)
C(36)	9228(7)	8136(6)	7423(3)	48(2)
C(37)	8086(11)	7730(12)	5363(9)	168(7)

Table 3. Bond lengths [Å] and angles [deg] for cd25504.

Pd-C(1)	1.980(8)
Pd-C(9)	2.015(7)
Pd-I(1)	2.6587(8)
Pd-I(2)	2.6795(8)
Cl(1)-C(37)	1.764(14)
Cl(2)-C(37)	1.696(13)
N(1)-C(1)	1.354(9)
N(1)-C(2)	1.374(10)
N(1)-C(8)	1.468(10)
N(2)-C(1)	1.357(9)
N(2)-C(7)	1.403(10)
N(2)-C(17)	1.446(9)
N(3)-C(9)	1.334(9)
N(3)-C(10)	1.405(10)
N(3)-C(16)	1.424(10)
N(4)-C(9)	1.378(9)
N(4)-C(15)	1.407(10)
N(4)-C(28)	1.453(9)
C(2)-C(7)	1.364(11)
C(2)-C(3)	1.413(12)
C(3)-C(4)	1.336(13)
C(3)-H(3)	0.9300
C(4)-C(5)	1.389(13)
C(4)-H(4)	0.9300
C(5)-C(6)	1.348(12)
C(5)-H(5)	0.9300
C(6)-C(7)	1.409(11)
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)	1.388(12)
C(10)-C(11)	1.392(11)
C(11)-C(12)	1.350(14)
C(11)-H(11)	0.9300
C(12)-C(13)	1.374(14)
C(12)-H(12)	0.9300
C(13)-C(14)	1.387(13)
C(13)-H(13)	0.9300
C(14)-C(15)	1.390(11)
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)	1.370(9)
C(17)-C(18)	1.401(10)
C(18)-C(19)	1.381(11)
C(18)-H(18)	0.9300
C(19)-C(20)	1.382(11)
C(19)-H(19)	0.9300
C(20)-C(25)	1.402(10)
C(20)-C(21)	1.494(11)
C(21)-C(22)	1.489(12)
C(21)-H(21A)	0.9700
C(21)-H(21B)	0.9700
C(22)-C(23)	1.530(13)
C(22)-H(22A)	0.9700
C(22)-H(22B)	0.9700
C(23)-C(24)	1.505(12)
C(23)-H(23A)	0.9700
C(23)-H(23B)	0.9700
C(24)-C(25)	1.504(10)
C(24)-H(24A)	0.9700
C(24)-H(24B)	0.9700
C(25)-C(26)	1.420(9)
C(26)-C(27)	1.488(10)
C(27)-C(36)	1.392(10)
C(27)-C(28)	1.395(10)

C(28)-C(29)	1.354(11)
C(29)-C(30)	1.380(12)
C(29)-H(29)	0.9300
C(30)-C(31)	1.384(13)
C(30)-H(30)	0.9300
C(31)-C(36)	1.390(11)
C(31)-C(32)	1.511(12)
C(32)-C(33)	1.487(18)
C(32)-H(32A)	0.9700
C(32)-H(32B)	0.9700
C(33)-C(34)	1.503(17)
C(33)-H(33A)	0.9700
C(33)-H(33B)	0.9700
C(34)-C(35)	1.525(12)
C(34)-H(34A)	0.9700
C(34)-H(34B)	0.9700
C(35)-C(36)	1.505(12)
C(35)-H(35A)	0.9700
C(35)-H(35B)	0.9700
C(37)-H(37A)	0.9700
C(37)-H(37B)	0.9700

C(1)-Pd-C(9)	95.5(3)
C(1)-Pd-I(1)	166.4(2)
C(9)-Pd-I(1)	88.6(2)
C(1)-Pd-I(2)	85.97(19)
C(9)-Pd-I(2)	168.4(2)
I(1)-Pd-I(2)	92.63(3)
C(1)-N(1)-C(2)	111.2(6)
C(1)-N(1)-C(8)	122.4(7)
C(2)-N(1)-C(8)	126.4(7)
C(1)-N(2)-C(7)	110.6(6)
C(1)-N(2)-C(17)	121.5(6)
C(7)-N(2)-C(17)	123.9(6)
C(9)-N(3)-C(10)	110.0(6)
C(9)-N(3)-C(16)	126.6(7)
C(10)-N(3)-C(16)	123.4(7)
C(9)-N(4)-C(15)	108.4(6)
C(9)-N(4)-C(28)	125.1(6)
C(15)-N(4)-C(28)	120.5(6)
N(1)-C(1)-N(2)	105.1(6)
N(1)-C(1)-Pd	125.7(6)
N(2)-C(1)-Pd	128.6(6)
C(7)-C(2)-N(1)	107.3(7)
C(7)-C(2)-C(3)	121.3(9)
N(1)-C(2)-C(3)	131.2(8)
C(4)-C(3)-C(2)	116.0(9)
C(4)-C(3)-H(3)	122.0
C(2)-C(3)-H(3)	122.0
C(3)-C(4)-C(5)	121.6(9)
C(3)-C(4)-H(4)	119.2
C(5)-C(4)-H(4)	119.2
C(6)-C(5)-C(4)	124.4(9)
C(6)-C(5)-H(5)	117.8
C(4)-C(5)-H(5)	117.8
C(5)-C(6)-C(7)	113.9(8)
C(5)-C(6)-H(6)	123.0
C(7)-C(6)-H(6)	123.0
C(2)-C(7)-N(2)	105.7(7)
C(2)-C(7)-C(6)	122.4(8)
N(2)-C(7)-C(6)	131.7(7)
N(1)-C(8)-H(8A)	109.5
N(1)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
N(1)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
N(3)-C(9)-N(4)	108.1(6)
N(3)-C(9)-Pd	125.3(5)
N(4)-C(9)-Pd	126.2(6)
C(15)-C(10)-C(11)	121.3(8)
C(15)-C(10)-N(3)	106.6(7)

C(11)-C(10)-N(3)	132.1(8)
C(12)-C(11)-C(10)	116.2(9)
C(12)-C(11)-H(11)	121.9
C(10)-C(11)-H(11)	121.9
C(11)-C(12)-C(13)	122.5(10)
C(11)-C(12)-H(12)	118.7
C(13)-C(12)-H(12)	118.7
C(12)-C(13)-C(14)	123.3(9)
C(12)-C(13)-H(13)	118.4
C(14)-C(13)-H(13)	118.4
C(13)-C(14)-C(15)	114.0(9)
C(13)-C(14)-H(14)	123.0
C(15)-C(14)-H(14)	123.0
C(14)-C(15)-C(10)	122.6(9)
C(14)-C(15)-N(4)	130.5(9)
C(10)-C(15)-N(4)	106.8(7)
N(3)-C(16)-H(16A)	109.5
N(3)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
N(3)-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)	123.5(7)
C(26)-C(17)-N(2)	118.2(6)
C(18)-C(17)-N(2)	118.4(6)
C(19)-C(18)-C(17)	116.5(7)
C(19)-C(18)-H(18)	121.7
C(17)-C(18)-H(18)	121.7
C(18)-C(19)-C(20)	123.1(8)
C(18)-C(19)-H(19)	118.5
C(20)-C(19)-H(19)	118.5
C(19)-C(20)-C(25)	118.6(7)
C(19)-C(20)-C(21)	119.6(7)
C(25)-C(20)-C(21)	121.5(7)
C(22)-C(21)-C(20)	112.1(8)
C(22)-C(21)-H(21A)	109.2
C(20)-C(21)-H(21A)	109.2
C(22)-C(21)-H(21B)	109.2
C(20)-C(21)-H(21B)	109.2
H(21A)-C(21)-H(21B)	107.9
C(21)-C(22)-C(23)	111.5(8)
C(21)-C(22)-H(22A)	109.3
C(23)-C(22)-H(22A)	109.3
C(21)-C(22)-H(22B)	109.3
C(23)-C(22)-H(22B)	109.3
H(22A)-C(22)-H(22B)	108.0
C(24)-C(23)-C(22)	108.8(8)
C(24)-C(23)-H(23A)	109.9
C(22)-C(23)-H(23A)	109.9
C(24)-C(23)-H(23B)	109.9
C(22)-C(23)-H(23B)	109.9
H(23A)-C(23)-H(23B)	108.3
C(25)-C(24)-C(23)	113.9(8)
C(25)-C(24)-H(24A)	108.8
C(23)-C(24)-H(24A)	108.8
C(25)-C(24)-H(24B)	108.8
C(23)-C(24)-H(24B)	108.8
H(24A)-C(24)-H(24B)	107.7
C(20)-C(25)-C(26)	120.1(7)
C(20)-C(25)-C(24)	120.1(7)
C(26)-C(25)-C(24)	119.7(7)
C(17)-C(26)-C(25)	117.9(7)
C(17)-C(26)-C(27)	118.8(6)
C(25)-C(26)-C(27)	122.9(6)
C(36)-C(27)-C(28)	118.1(7)
C(36)-C(27)-C(26)	122.6(7)
C(28)-C(27)-C(26)	119.3(6)
C(29)-C(28)-C(27)	123.1(8)
C(29)-C(28)-N(4)	118.6(8)
C(27)-C(28)-N(4)	118.2(7)
C(28)-C(29)-C(30)	118.3(9)
C(28)-C(29)-H(29)	120.8

C(30)-C(29)-H(29)	120.9
C(29)-C(30)-C(31)	120.7(8)
C(29)-C(30)-H(30)	119.6
C(31)-C(30)-H(30)	119.6
C(30)-C(31)-C(36)	120.3(8)
C(30)-C(31)-C(32)	118.7(9)
C(36)-C(31)-C(32)	120.9(10)
C(33)-C(32)-C(31)	114.9(10)
C(33)-C(32)-H(32A)	108.5
C(31)-C(32)-H(32A)	108.5
C(33)-C(32)-H(32B)	108.6
C(31)-C(32)-H(32B)	108.6
H(32A)-C(32)-H(32B)	107.5
C(32)-C(33)-C(34)	108.8(9)
C(32)-C(33)-H(33A)	109.9
C(34)-C(33)-H(33A)	109.9
C(32)-C(33)-H(33B)	109.9
C(34)-C(33)-H(33B)	109.9
H(33A)-C(33)-H(33B)	108.3
C(33)-C(34)-C(35)	113.0(10)
C(33)-C(34)-H(34A)	109.0
C(35)-C(34)-H(34A)	109.0
C(33)-C(34)-H(34B)	109.0
C(35)-C(34)-H(34B)	109.0
H(34A)-C(34)-H(34B)	107.8
C(36)-C(35)-C(34)	111.6(8)
C(36)-C(35)-H(35A)	109.3
C(34)-C(35)-H(35A)	109.3
C(36)-C(35)-H(35B)	109.3
C(34)-C(35)-H(35B)	109.3
H(35A)-C(35)-H(35B)	108.0
C(31)-C(36)-C(27)	119.3(9)
C(31)-C(36)-C(35)	121.6(8)
C(27)-C(36)-C(35)	119.1(7)
Cl(2)-C(37)-Cl(1)	107.5(9)
Cl(2)-C(37)-H(37A)	110.4
Cl(1)-C(37)-H(37A)	110.4
Cl(2)-C(37)-H(37B)	110.1
Cl(1)-C(37)-H(37B)	110.1
H(37A)-C(37)-H(37B)	108.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd25504.
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
Pd	48(1)	45(1)	41(1)	-5(1)	2(1)	3(1)
I(1)	61(1)	79(1)	82(1)	-17(1)	5(1)	19(1)
I(2)	88(1)	68(1)	51(1)	-13(1)	-5(1)	-4(1)
N(1)	48(4)	41(4)	53(4)	3(3)	0(3)	-3(3)
N(2)	40(4)	41(4)	40(3)	5(3)	0(3)	-1(3)
N(3)	46(4)	47(4)	49(4)	4(3)	12(3)	1(3)
N(4)	36(4)	45(4)	43(3)	-3(3)	-3(3)	3(3)
C(1)	50(5)	45(4)	28(3)	-2(3)	-5(3)	-1(4)
C(2)	58(5)	55(5)	35(4)	-2(3)	-5(4)	-16(4)
C(3)	55(6)	83(7)	52(5)	22(5)	-6(4)	-16(5)
C(4)	47(5)	98(8)	63(5)	18(5)	1(5)	-8(6)
C(5)	50(6)	90(7)	42(5)	10(5)	0(4)	18(5)
C(6)	51(5)	53(5)	53(5)	2(4)	9(4)	-4(4)
C(7)	49(5)	52(5)	31(4)	1(3)	5(3)	-10(4)
C(8)	63(6)	45(5)	92(7)	3(5)	3(5)	-17(4)
C(9)	43(5)	47(4)	37(4)	-3(3)	-3(3)	4(4)
C(10)	44(5)	44(4)	58(5)	-10(4)	2(4)	3(4)
C(11)	38(5)	62(6)	74(6)	-3(5)	-3(4)	-9(4)
C(12)	58(6)	82(7)	78(7)	-19(6)	-5(5)	-2(6)
C(13)	62(6)	81(7)	70(6)	-23(5)	-21(6)	13(6)
C(14)	57(6)	68(6)	60(6)	-11(5)	-2(5)	5(5)
C(15)	50(5)	45(5)	44(4)	-6(4)	-16(4)	14(4)
C(16)	63(6)	68(5)	66(6)	-4(5)	20(5)	-7(5)
C(17)	38(4)	37(4)	46(4)	-6(3)	3(3)	3(3)
C(18)	50(5)	60(5)	52(5)	16(4)	1(4)	-13(4)
C(19)	49(5)	47(5)	81(6)	6(4)	17(4)	3(4)
C(20)	38(4)	36(4)	64(5)	-1(4)	2(4)	-2(3)
C(21)	68(6)	46(5)	89(7)	-4(5)	-4(5)	-11(5)
C(22)	69(7)	49(6)	137(10)	-17(6)	-20(6)	-24(5)
C(23)	86(7)	63(6)	81(6)	-17(5)	-21(6)	2(6)
C(24)	50(5)	58(5)	63(5)	-27(4)	-9(4)	3(4)
C(25)	34(4)	42(4)	49(4)	-9(3)	8(3)	1(3)
C(26)	31(4)	41(4)	37(4)	-5(3)	5(3)	-1(3)
C(27)	37(4)	46(4)	36(4)	-8(3)	4(3)	-9(3)
C(28)	48(5)	57(5)	30(4)	5(3)	0(3)	-8(4)
C(29)	59(6)	56(5)	62(5)	17(4)	-6(4)	-7(4)
C(30)	81(7)	70(7)	62(5)	31(5)	-10(5)	-27(6)
C(31)	60(6)	72(6)	46(4)	16(4)	-6(4)	-25(5)
C(32)	72(7)	128(10)	74(7)	26(7)	26(6)	-28(7)
C(33)	76(8)	208(16)	50(6)	-6(8)	26(6)	-45(10)
C(34)	48(7)	143(11)	103(9)	-45(8)	15(6)	-20(7)
C(35)	43(5)	84(7)	60(5)	-19(5)	8(4)	-13(5)
C(36)	49(5)	62(5)	35(4)	-10(4)	2(3)	-16(4)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd25504.

	x	y	z	U(eq)
H(3)	11353	10021	9114	76
H(4)	12642	8925	8966	83
H(5)	12294	7337	8998	73
H(6)	10642	6737	8987	63
H(8A)	8342	10648	9114	100
H(8B)	9389	10789	8733	100
H(8C)	9379	10792	9506	100
H(11)	3876	6790	8188	70
H(12)	3576	6726	7076	87
H(13)	4656	7424	6341	85
H(14)	6064	8357	6669	74
H(16A)	5486	7738	9692	98
H(16B)	5133	6824	9310	98
H(16C)	4385	7699	9364	98
H(18)	8210	6490	9721	65
H(19)	7164	5285	9316	71
H(21A)	6689	4280	8381	81
H(21B)	5809	5015	8221	81
H(22A)	6130	4204	7276	102
H(22B)	7330	4369	7340	102
H(23A)	6607	5414	6541	92
H(23B)	5895	5864	7088	92
H(24A)	8071	5992	7016	68
H(24B)	7294	6848	7000	68
H(29)	7278	10089	7461	71
H(30)	8860	10309	6941	85
H(32A)	10301	9526	6356	110
H(32B)	10881	9554	7033	110
H(33A)	11719	8452	6438	124
H(33B)	10677	7976	6205	124
H(34A)	11386	7962	7521	117
H(34B)	11461	7082	7050	117
H(35A)	9707	6849	7069	75
H(35B)	10026	7010	7807	75
H(37A)	8081	8324	5119	201
H(37B)	8241	7868	5821	201

Table 6. Torsion angles [deg] for cd25504.

C(2)-N(1)-C(1)-N(2)	-4.2(8)
C(8)-N(1)-C(1)-N(2)	173.8(8)
C(2)-N(1)-C(1)-Pd	168.1(5)
C(8)-N(1)-C(1)-Pd	-14.0(11)
C(7)-N(2)-C(1)-N(1)	4.0(7)
C(17)-N(2)-C(1)-N(1)	-154.0(6)
C(7)-N(2)-C(1)-Pd	-167.9(5)
C(17)-N(2)-C(1)-Pd	34.1(9)
C(9)-Pd-C(1)-N(1)	131.2(6)
I(1)-Pd-C(1)-N(1)	24.1(13)
I(2)-Pd-C(1)-N(1)	-60.4(6)
C(9)-Pd-C(1)-N(2)	-58.5(6)
I(1)-Pd-C(1)-N(2)	-165.6(5)
I(2)-Pd-C(1)-N(2)	110.0(6)
C(1)-N(1)-C(2)-C(7)	2.7(8)
C(8)-N(1)-C(2)-C(7)	-175.1(8)
C(1)-N(1)-C(2)-C(3)	-171.6(8)
C(8)-N(1)-C(2)-C(3)	10.6(14)
C(7)-C(2)-C(3)-C(4)	4.5(11)
N(1)-C(2)-C(3)-C(4)	178.1(8)
C(2)-C(3)-C(4)-C(5)	-6.5(12)
C(3)-C(4)-C(5)-C(6)	5.6(14)
C(4)-C(5)-C(6)-C(7)	-2.0(12)
N(1)-C(2)-C(7)-N(2)	-0.2(8)
C(3)-C(2)-C(7)-N(2)	174.8(7)
N(1)-C(2)-C(7)-C(6)	-176.2(7)
C(3)-C(2)-C(7)-C(6)	-1.2(11)
C(1)-N(2)-C(7)-C(2)	-2.5(8)
C(17)-N(2)-C(7)-C(2)	155.0(6)
C(1)-N(2)-C(7)-C(6)	173.1(7)
C(17)-N(2)-C(7)-C(6)	-29.5(12)
C(5)-C(6)-C(7)-C(2)	-0.1(11)
C(5)-C(6)-C(7)-N(2)	-175.0(7)
C(10)-N(3)-C(9)-N(4)	-3.4(8)
C(16)-N(3)-C(9)-N(4)	173.7(7)
C(10)-N(3)-C(9)-Pd	170.4(5)
C(16)-N(3)-C(9)-Pd	-12.5(11)
C(15)-N(4)-C(9)-N(3)	3.0(8)
C(28)-N(4)-C(9)-N(3)	-149.5(7)
C(15)-N(4)-C(9)-Pd	-170.7(5)
C(28)-N(4)-C(9)-Pd	36.8(10)
C(1)-Pd-C(9)-N(3)	134.6(6)
I(1)-Pd-C(9)-N(3)	-58.4(6)
I(2)-Pd-C(9)-N(3)	37.8(15)
C(1)-Pd-C(9)-N(4)	-52.8(7)
I(1)-Pd-C(9)-N(4)	114.2(6)
I(2)-Pd-C(9)-N(4)	-149.6(7)
C(9)-N(3)-C(10)-C(15)	2.4(8)
C(16)-N(3)-C(10)-C(15)	-174.8(7)
C(9)-N(3)-C(10)-C(11)	-177.1(8)
C(16)-N(3)-C(10)-C(11)	5.7(13)
C(15)-C(10)-C(11)-C(12)	-2.5(12)
N(3)-C(10)-C(11)-C(12)	177.0(9)
C(10)-C(11)-C(12)-C(13)	2.3(15)
C(11)-C(12)-C(13)-C(14)	-2.3(16)
C(12)-C(13)-C(14)-C(15)	2.3(14)
C(13)-C(14)-C(15)-C(10)	-2.5(12)
C(13)-C(14)-C(15)-N(4)	-177.8(8)
C(11)-C(10)-C(15)-C(14)	2.8(12)
N(3)-C(10)-C(15)-C(14)	-176.8(7)
C(11)-C(10)-C(15)-N(4)	179.0(7)
N(3)-C(10)-C(15)-N(4)	-0.6(8)
C(9)-N(4)-C(15)-C(14)	174.4(8)
C(28)-N(4)-C(15)-C(14)	-31.7(12)
C(9)-N(4)-C(15)-C(10)	-1.4(8)
C(28)-N(4)-C(15)-C(10)	152.5(7)
C(1)-N(2)-C(17)-C(26)	75.0(9)
C(7)-N(2)-C(17)-C(26)	-80.1(9)

C(1)-N(2)-C(17)-C(18)	-105.8(8)
C(7)-N(2)-C(17)-C(18)	99.2(8)
C(26)-C(17)-C(18)-C(19)	-3.0(12)
N(2)-C(17)-C(18)-C(19)	177.7(7)
C(17)-C(18)-C(19)-C(20)	6.2(12)
C(18)-C(19)-C(20)-C(25)	-4.1(12)
C(18)-C(19)-C(20)-C(21)	-177.3(8)
C(19)-C(20)-C(21)-C(22)	-160.9(8)
C(25)-C(20)-C(21)-C(22)	26.1(11)
C(20)-C(21)-C(22)-C(23)	-50.8(11)
C(21)-C(22)-C(23)-C(24)	62.9(11)
C(22)-C(23)-C(24)-C(25)	-48.1(11)
C(19)-C(20)-C(25)-C(26)	-1.5(11)
C(21)-C(20)-C(25)-C(26)	171.5(7)
C(19)-C(20)-C(25)-C(24)	173.8(7)
C(21)-C(20)-C(25)-C(24)	-13.1(11)
C(23)-C(24)-C(25)-C(20)	24.8(11)
C(23)-C(24)-C(25)-C(26)	-159.8(7)
C(18)-C(17)-C(26)-C(25)	-2.2(11)
N(2)-C(17)-C(26)-C(25)	177.1(6)
C(18)-C(17)-C(26)-C(27)	171.8(7)
N(2)-C(17)-C(26)-C(27)	-9.0(10)
C(20)-C(25)-C(26)-C(17)	4.5(10)
C(24)-C(25)-C(26)-C(17)	-170.9(7)
C(20)-C(25)-C(26)-C(27)	-169.2(7)
C(24)-C(25)-C(26)-C(27)	15.4(11)
C(17)-C(26)-C(27)-C(36)	93.7(9)
C(25)-C(26)-C(27)-C(36)	-92.7(9)
C(17)-C(26)-C(27)-C(28)	-84.1(9)
C(25)-C(26)-C(27)-C(28)	89.5(9)
C(36)-C(27)-C(28)-C(29)	-5.7(11)
C(26)-C(27)-C(28)-C(29)	172.2(7)
C(36)-C(27)-C(28)-N(4)	170.3(6)
C(26)-C(27)-C(28)-N(4)	-11.8(10)
C(9)-N(4)-C(28)-C(29)	-114.4(8)
C(15)-N(4)-C(28)-C(29)	96.2(9)
C(9)-N(4)-C(28)-C(27)	69.4(10)
C(15)-N(4)-C(28)-C(27)	-79.9(8)
C(27)-C(28)-C(29)-C(30)	2.6(12)
N(4)-C(28)-C(29)-C(30)	-173.4(7)
C(28)-C(29)-C(30)-C(31)	1.7(13)
C(29)-C(30)-C(31)-C(36)	-2.8(13)
C(29)-C(30)-C(31)-C(32)	179.7(8)
C(30)-C(31)-C(32)-C(33)	-162.5(9)
C(36)-C(31)-C(32)-C(33)	20.0(14)
C(31)-C(32)-C(33)-C(34)	-47.0(13)
C(32)-C(33)-C(34)-C(35)	62.2(13)
C(33)-C(34)-C(35)-C(36)	-47.1(13)
C(30)-C(31)-C(36)-C(27)	-0.4(12)
C(32)-C(31)-C(36)-C(27)	177.0(8)
C(30)-C(31)-C(36)-C(35)	177.6(8)
C(32)-C(31)-C(36)-C(35)	-4.9(12)
C(28)-C(27)-C(36)-C(31)	4.5(11)
C(26)-C(27)-C(36)-C(31)	-173.4(7)
C(28)-C(27)-C(36)-C(35)	-173.6(7)
C(26)-C(27)-C(36)-C(35)	8.6(11)
C(34)-C(35)-C(36)-C(31)	18.1(12)
C(34)-C(35)-C(36)-C(27)	-163.9(8)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd25504 [A and deg.].

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