A Concise, Modular Synthesis of Chiral Peraza-macrocycles Using Chiral Aziridines

Supporting Information

B. Moon Kim*, Soon Mog So, and Hye Jin Choi

Center for Molecular Catalysis, School of Chemistry and Molecular Engineering,, Seoul National University, Seoul 151-747, Korea.

Experimental Section

(S)-N-Nosyl-2-isopropylaziridine (6). To a solution of L-valinol (2.00 g, 17 mmol) and TEA (12.0 mL, 85 mmol) in dry EtOAc (60 mL) was slowly added *p*-NsCl (7.53 g, 34 mmol) at 0 °C and stirred for 30 min. The reaction mixture was stirred at room temperature for 4 h. The reaction mixture was washed with brine (20 mL × 2). The organic layer was dried over anhydrous MgSO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified on a silica gel column chromatography (5:1 *n*-Hexane/EtOAc) to give a white solid (3.50 g, 76% yield): mp 64~65 °C; $[\alpha]_D^{18} +12.2$ (*c* 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) 0.84 (d, *J* = 6.7 Hz, 3H), 0.94 (d, *J* = 6.8 Hz, 3H), 1.41~1.59 (m, 1H), 2.21 (d, 1H), 2.64~2.79 (m, 2H), 8.17 (d, 2H), 8.41 (d, 2H); ¹³C NMR (75 MHz, CDCl₃) 19.43, 19.98, 30.51, 34.03, 47.48, 124.64, 129.79, 144.45, 150.98; HRMS (FAB) *m/z* calcd 271.0752 for C₁₁H₁₅N₂O₄S [M + H]⁺, found 271.0760

(2S,6S)-1,7-Dinosyl-2,6-diisopropyl-4-methoxybenzyl-1,4,7-triazaheptane (7). To a solution of compound **6** (2.30 g, 8.3 mmol) in dry acetonitrile (20 mL), was slowly added *p*-methoxybenzylamine (0.5 mL, 4.2 mmol) at room temperature. The reaction mixture was stirred at 80 °C for 12 h. Solvent was removed under reduced pressure and the residue dissolved in EtOAc (20 mL). The solution was washed with brine (10 mL × 2), dried over anhydrous MgSO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified on a silica gel column chromatography (1:1 *n*-Hexane/EtOAc) to give a pale yellow solid (2.40 g, 83% yield): mp 75~76 °C; $[\alpha]_D^{19} -7.2$ (*c* 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) 0.64 (d, *J* = 6.8 Hz, 6H), 0.72 (d, *J* = 6.9 Hz, 6H), 1.78~1.88 (m, 2H), 2.33 (dd, *J*_{AB} = 13.1 Hz, *J*_{AX} = 4.3 Hz, 2H), 2.59 (dd, *J*_{AB} = 13.1 Hz, *J*_{AX} = 10.3 Hz, 2H), 3.53 (AB_q, *J* = 13.1 Hz, 2H), 3.59~3.70 (m, 2H), 3.79 (s, 3H), 5.72 (br, 2H), 6.83 (d, 2H), 7.15 (d, 2H), 8.13(d, 4H), 8.34 (d, 4H); ¹³C NMR (75 MHz, CDCl₃) 18.16, 18.36, 31.07, 54.02, 55.67, 56.99, 57.98, 114.22, 124.69, 128.36, 129.54, 130.87, 147.98, 150.17, 159.34; HRMS (FAB) *m/z* calcd 678.2267 for C₃₀H₄₀N₅O₉S₂ [M + H]⁺, found 678.2280

(3S,7S,18S,22S)-2,5,8,17,20,23-Hexaaza[9,9]-3,7,18,22-tetraisopropyl-5,20-di(*p*-methoxybenzyl)paracyclophane (8) To a solution of compound **7** (2.60 g, 3.8 mmol) and

K₂CO₃ (5.20 g, 38 mmol) in dry CH₃CN (60 mL) was added α,α' -dibromo-*p*-xylene (1.50 g, 5.70 mmol) at room temperature. The reaction mixture was stirred at 50 °C for 36 h and to it was added LiOH.H₂O (1.60 g, 38 mmol) at room temperature followed by *n*-PrSH (3.4 mL, 38 mmol) slowly. The reaction mixture was stirred at room temperature for 12 h. The solvent was removed under reduced pressure and the residue dissolved in EtOAc (60 mL). The solution was washed with brine (15 mL $\times$ 2), dried over anhydrous MgSO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified on a silica gel column chromatography (9:1 CH₂Cl₂/MeOH) to give a yellow oil (820 mg, 53% yield): $[\alpha]_D^{22} +120.2$ (c 1.1, CHCl₃); ¹H NMR (300 MHz, CDCl₃) 0.72~0.93 (m, 24H), 1.48~1.65 (m, 4H), 1.90~2.12 (m, 4H), 2.28~2.62 (m, 8H), 3.05 (AB_q, *J* = 12.2 Hz, 4H), 3.72 (AB_q, *J* = 12.8 Hz, 8H), 3.83 (s, 6H), 6.87~7.41 (m, 16H); ¹³C NMR (75 MHz, CDCl₃) 17.11, 17.28, 20.28, 32.65, 54.76, 55.61, 58.99, 61.10, 114.07, 128.32, 129.03, 130.64, 140.06, 159.00; HRMS (FAB) *m/z* calcd 819.6264 for C₅₂H₇₉N₆O₂ [M + H]⁺, found 819.6251

(4*S*,8*S*)-3,6,9,15-Tetraazabicyclo[9,3,1]pentadeca-4,8-diisopropyl-6-(*p*-methoxybenzyl) -1(15),11,13-triene (9) To a solution of compound **7** (1.20 g, 1.8 mmol) and K₂CO₃ (2.48 g, 18 mmol) in dry CH₃CN (30 mL) was added 2,6-pyridinedimethanol bismethanesulfonate (800 mg, 2.7 mmol) at room temperature. The reaction mixture was stirred at 60 °C for 50 h. LiOH.H₂O (0.73 g, 18 mmol) was added to the reaction mixture at room temperature and to the mixture was added *n*-PrSH (3.2 mL, 36 mmol) slowly. The reaction mixture was stirred at room temperature for 12 h. Solvent was removed under reduced pressure and the residue was dissolved in EtOAc (30 mL), washed with brine (10 mL $\times$ 2), dried over anhydrous MgSO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified on a silica gel column chromatography (9:1 CH₂Cl₂/MeOH) to give a yellow oil (360 mg, 49% yield): $[\alpha]_D^{22} +101.4$ (c 1.3, CHCl₃); ¹H NMR (300 MHz, CDCl₃) 0.70~1.10 (m, 12H), 1.62~1.83 (m, 2H), 2.65~2.91 (m, 6H), 3.38 (AB_q, *J* = 12.5 Hz, 2H), 3.40~3.51 (m, 2H), 3.78 (s, 3H), 4.13~4.31 (m, 2H), 6.85~7.53 (m, 7H); HRMS (CI) *m/z* calcd 409.2968 for C₂₅H₃₇N₄O [M - H]⁺, found 409.2969

(2*S*)-1-Nosyl-2-isopropyl-1,4-diazabutane (10). To a solution of compound **6** (2.30 g, 8.30 mmol) in dry acetonitrile (20 mL) was slowly added LiN₃ (500 mg, 4.2 mmol) at room temperature. The reaction mixture was stirred at 60 °C for 12 h. The reaction mixture was removed under reduced pressure and filtered through Celite. The residue was dissolved in THF (40 mL) and to the solution PPh₃ (2.18 g, 8.3 mmol) was added at 50 °C. The mixture was stirred for 10 h before 1.5 mL of H₂O was added. Solvent was removed under reduced pressure and the residue was dissolved in CH₂Cl₂ (60 mL), washed with brine (10 mL $\times$ 2), dried over anhydrous MgSO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified on a silica gel column chromatography (9:1 CH₂Cl₂/MeOH) to give a yellow solid (1.82 g, 75% yield): mp 108~110 °C; $[\alpha]_D^{18} -8.4$ (c 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) 0.81 (d, 6H), 1.76~1.83 (m, 1H), 2.67 (dd, *J*_{AB} = 13.2 Hz, *J*_{AX} = 4.6 Hz, 1H), 2.82 (dd, *J*_{AB} = 13.2 Hz, *J*_{AX} = 6.0 Hz, 1H), 3.0 (br, 2H), 3.07 (dd, *J* = 4.6, 6.0 Hz, 1H), 5.4 (br, 1H), 8.11(d, *J* = 7.1 Hz, 2H), 8.36 (d, *J* = 7.1 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃) 18.71, 18.75, 28.89, 42.01, 61.11, 124.25, 128.23, 147.03, 149.79; HRMS (CI) *m/z* calcd 288.1018 for C₁₁H₁₈N₃O₄S [M + H]⁺, found 288.1017

(2*S*,6*S*)-1,4,7-Trinosyl-2,6-diisopropyl-1,4,7-triazaheptane (11). To a solution of compound **10** (1.40 g, 4.8 mmol) in dry acetonitrile (20 mL) was slowly added compound **6** (1.30 g, 4.8 mmol) at room temperature. The reaction mixture was stirred at 50 °C for 5 h. Solvent was removed under reduced pressure and the residue was dissolved in CH₂Cl₂ (30 mL). To the mixture were slowly added *p*-NsCl (1.06 g, 4.8 mmol) and TEA (0.7 mL, 5.0

mmol) at 0 °C. After 1 h, the mixture was washed with brine (5 mL × 2), dried over anhydrous MgSO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified on a silica gel column chromatography (4:1 *n*-Hexane/EtOAc) to give a white solid (3.04 g, 85% yield): mp 112~113 °C; $[\alpha]_D^{20}$ -78.8 (*c* 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) 0.63 (d, *J* = 6.8 Hz, 6H), 0.79 (d, *J* = 6.9 Hz, 6H), 1.75~1.91 (m, 2H), 3.29 (m, 4H), 3.48~3.60 (m, 2H), 5.13 (d, *J* = 7.8 Hz, 2H), 8.02~8.10 (m, 6H), 8.37~8.44 (m, 6H); ¹³C NMR (75 MHz, CDCl₃) 16.96, 18.85, 29.24, 51.49, 57.92, 124.88, 125.19, 128.65, 129.19, 144.09, 146.70, 150.49, 150.81; HRMS (FAB) *m/z* calcd 743.1476 for C₂₈H₃₅N₆O₁₂S₃ [M + H]⁺, found 743.1447

(4*S*,8*S*)-4,8-Diisopropyl-3,6,9,15-Tetraazabicyclo[9,3,1]pentadeca-1(15),11,13-triene (12) To a solution of compound **11** (1.52 g, 2.0 mmol) and K₂CO₃ (2.76 g, 20 mmol) in dry CH₃CN (40 mL) was added 2,6-pyridinemethanol bismethanesulfonate (880 mg, 3 mmol) at room temperature. The reaction mixture was stirred at 50 °C for 48 h. To the reaction mixture was added LiOH.H₂O (0.82 g, 20 mmol) at room temperature followed by *n*-PrSH (1.8 mL, 20 mmol) slowly. The reaction mixture was stirred at room temperature for 12 h. Solvent was removed under reduced pressure and the residue was dissolved in EtOAc (40 mL), washed with brine (10 mL × 2), dried over anhydrous MgSO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified on a silica gel column chromatography (9:1 CH₂Cl₂/MeOH) to give a yellow oil (270 mg, 47% yield): $[\alpha]_D^{23}$ +24.1 (*c* 0.9, CHCl₃); ¹H NMR (300 MHz, CDCl₃) 0.79~0.90 (m, 12H), 1.70~1.74 (m, 2H), 2.65~3.01 (m, 6H), 3.97 (AB_q, *J* = 17.4 Hz, 4H), 6.88 (d, 2H), 7.48 (t, 1H); ¹³C NMR (75 MHz, CDCl₃) 19.57, 19.78, 30.82, 49.96, 50.70, 60.90, 120.11, 137.92, 160.76; HRMS (CI) *m/z* calcd 291.2549 for C₁₇H₃₁N₄ [M + H]⁺, found 291.2556

(2*S*,6*S*)-1,7-Ditosyl-2,6-diisopropyl-1,4,7-triazaheptane (14). To a solution of compound **13** (600 mg, 2.5 mmol) and LiN₃ (380 mg, 7.5 mmol, containing 10 % MeOH) in CH₃CN (12 mL) was stirred for 12 h at 70 °C. Solvent was removed on a rotary evaporator, filtered and the filtrate was dissolved in MeOH (10 mL). To the solution was added Pd/C (50 mg) and the mixture was stirred under H₂ (1 atm) at room temperature for 4 h. The reaction mixture was filtered through a pad of Celite and methanol was evaporated on a rotary evaporator. The residue was dissolved in DMF (7 mL) and to the solution was added compound **13** (600 mg, 2.5 mmol) under atmosphere. The reaction mixture was stirred at 80 °C for 8 h. Solvent was removed under reduced pressure and the crude product was purified on a silica gel column chromatography (1:2 *n*-Hexane/EtOAc) to give a pale yellow oil (920 mg, 74% yield): mp 108 °C; $[\alpha]_D^{28}$ -18.7 (*c* 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) 0.72~0.79 (m, 12H), 1.62~1.68 (m, 2H), 2.04~2.23 (m, 2H), 2.34~2.39 (m, 8H), 2.95~2.96 (m, 2H), 6.05 (br, 2H) 7.28 (d, 4H), 7.77 (d, 4H); ¹³C NMR (75 MHz, CDCl₃) 18.09, 18.36, 21.29, 29.95, 49.06, 58.34, 126.83, 129.41, 137.98, 142.94; HRMS (FAB) *m/z* calcd 496.2304 for C₂₄H₃₈N₃O₄S₂ [M + H]⁺, found 496.2307

(2*S*,6*S*)-4-Carboxybenzyl-1,7-ditosyl-2,6-diisopropyl-1,4,7-triazaheptane (15). To a solution of compound **14** (1.60 g, 3.2 mmol) and TEA (0.5 mL, 3.5 mmol) in dry CH₂Cl₂ (15 mL) was slowly added CbzCl (0.46 mL, 3.2 mmol) at -25 °C and the mixture was stirred for 1 h. The reaction temperature was raised to room temperature and the stirring was continued for 1.5 h. The reaction mixture was washed with brine (10 mL × 2), dried over anhydrous MgSO₄, filtered and the filtrate was concentrated under reduced pressure. The crude product was purified on a silica gel column chromatography (2:1 *n*-Hexane/EtOAc) to give a white solid (1.73 g, 83% yield): mp 50 °C; $[\alpha]_D^{28}$ -16.9 (*c* 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) 0.64~0.77 (m, 12 H), 1.57~1.68 (m, 2H), 2.28 (s, 3H), 2.36~2.46 (m, 5H),

2.58~2.69 (m, 2H), 3.12~3.19 (m, 2H), 4.87 (d, $J = 8.4$ Hz, 1H), 5.06 (s, 2H), 5.41 (d, $J = 7.6$ Hz, 1H), 7.11 (d, 2H), 7.18 (d, 2H), 7.35~7.47 (m, 7H), 7.60 (d, 2H); ^{13}C NMR (75 MHz, CDCl_3) 17.31, 17.43, 17.79, 17.84, 21.20, 21.27, 30.55, 31.22, 47.07, 57.04, 57.50, 67.72, 126.52, 126.57, 128.43, 128.47, 128.61, 129.35, 129.52, 135.66, 137.92, 138.21, 142.71, 142.89, 156.31; HRMS (FAB) m/z calcd 630.2672 for $\text{C}_{32}\text{H}_{44}\text{N}_3\text{O}_6\text{S}_2$ $[\text{M} + \text{H}]^+$, found 630.2682

(2S,6S)-1,7-Ditosyl-2,6-diisopropyl-1,4,7-Triazacyclononane (16) To a solution of compound **15** (2.00 g, 3.2 mmol) and Cs_2CO_3 (3.88 g, 11 mmol) in DMF (80 mL) was added 1,2-ethanediol bis(*p*-toluenesulfonate) (1.52 g, 4.00 mmol) at room temperature. The reaction mixture was stirred for 48 h at 60 °C. Solvent was removed under reduced pressure, and the residue was dissolved in methanol (10 mL) and filtered through a pad of Celite. To the reaction mixture was added Pd/C (100 mg) and the mixture was stirred under H_2 (1 atm) at room temperature for 3 h. The reaction mixture was filtered through a pad of Celite and methanol was evaporated on a rotary evaporator. The crude product was purified on a silica gel column chromatography (1:2 *n*-Hexane/EtOAc) to give a white solid (0.98 g, 59% yield): mp 39 °C; $[\alpha]_D^{28} +82.1$ (c 1.0, CHCl_3); ^1H NMR (300 MHz, CDCl_3) 0.13 (d, $J = 6.7$ Hz, 6 H), 0.52 (d, $J = 6.5$ Hz, 6 H), 1.02~1.05 (m, 2H), 2.13 (s, 6 H), 2.62~2.69 (m, $J = 7.0$, 14.1 Hz, 2H), 2.87~2.91 (m, $J = 4.0$, 14.1 Hz, 2 H), 3.14~3.19 (m, 4H), 3.46~3.51 (m, 2 H), 7.02 (d, 4 H), 7.49 (d, 4 H); ^{13}C NMR (75 MHz, CDCl_3) 19.45, 19.67, 20.73, 27.47, 45.37, 50.51, 64.56, 126.60, 128.73, 137.57, 142.27; HRMS (FAB) m/z calcd 522.2460 for $\text{C}_{26}\text{H}_{40}\text{N}_3\text{O}_4\text{S}_2$ $[\text{M} + \text{H}]^+$, found 522.2460

(2S,6S)-2,6-Diisopropyl-1,4,7-Triazacyclononane (17) To a solution of compound **16** (3.00 g, 2.90 mmol) was slowly added conc H_2SO_4 (5.00 mL). The mixture was heated under nitrogen atmosphere for 30 h at 120 °C to give a dark solution. The mixture was poured into a conical flask and the flask was cooled to 0 °C in an ice bath. Water (10 mL) was added slowly and portions of potassium hydroxide pellets were carefully added, while cooling the flask in the ice bath. The pH of the viscous mixture was brought to below 13. Ethanol (60 mL) was added to the mixture and it was filtered through a Celite pad. The filter cake was washed with ethanol, and the combined aqueous alcoholic extracts were stripped of solvents *in vacuo* on a rotary evaporator. The yellow solid residue was dissolved in 6 N HCl (10 mL) and washed with dichloromethane (10 mL $\times$ 3). The pH of the aqueous layer was lowered to 13 by adding KOH pellets and the aqueous layer was extracted with dichloromethane (20 mL $\times$ 5). Combined organic layer was dried over anhydrous MgSO_4 , filtered and the filtrate was concentrated under reduced pressure to give a brownish oil (0.87g, 70% yield): $[\alpha]_D^{28} +55.8$ (c 1, CHCl_3); ^1H NMR (300 MHz, CDCl_3) 0.89 (d, $J = 6.7$ Hz, 6 H), 0.96 (d, $J = 6.7$ Hz, 6 H), 1.48~1.60 (m, 2 H), 2.32 ~2.49 (m, 4 H), 2.54~2.67 (m, 4 H), 2.72~2.97 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3) 18.27, 18.43, 30.29, 42.39, 47.07, 57.48

(2S,10S)-1,4,8,11-Tetratosyl-2,10-dimethyl-1,4,8,11-tetraazaundecane (19). A mixture of aziridine **18** (1.00 g, 4.7 mmol) and 1,3-diaminopropane (0.19 mL, 2.3 mmol) in dry DMF (10 mL) was stirred for 6 h at 50 °C. Solvent was removed under reduced pressure and the residue was dissolved in methylene chloride (20 mL). To the mixture were slowly added TEA (0.7 mL, 5 mmol) and *p*-TsCl (890 mg, 4.7 mmol) at 0 °C. Stirring was continued at room temperature for 5 h and the mixture was washed with brine (15 mL $\times$ 2), dried over anhydrous MgSO_4 , filtered and the filtrate was concentrated under reduced pressure. The crude product was purified on a silica gel column chromatography (1:1 *n*-Hexane/EtOAc) to give a colorless oil (1.22 g, 66% yield): mp 52 °C; $[\alpha]_D^{28} -14.1$ (c 1.0, CHCl_3); ^1H NMR (300 MHz, CDCl_3) 0.92 (m, 6H), 1.78~1.93 (m, 2H), 2.39~2.40 (m, 12H), 2.94~3.03 (m,

8H), 3.41~3.59 (m, 2H), 5.66 (d, $J = 7.2\text{Hz}$, 2H), 7.29 (d, 8H), 7.63 (d, 4H) 7.76 (d, 4H); ^{13}C NMR (75 MHz, CDCl_3) 14.61, 19.16, 21.96, 28.74, 49.06, 50.16, 55.24, 127.42, 127.77, 130.19, 130.28, 135.68, 138.04, 143.88, 144.15; HRMS (FAB) m/z calcd 805.2433 for $\text{C}_{37}\text{H}_{49}\text{N}_4\text{O}_8\text{S}_4$ $[\text{M} + \text{H}]^+$, found 805.2441

(2S,10S)-2,10-Dimethyl-1,4,8,11-tetraazacyclotetradecane (20) To a solution of compound **19** (1.30 g, 1.6 mmol) and Cs_2CO_3 (1.98 g, 5.6 mmol) in DMF (40 mL) was slowly added 1,3-propanediol bis(*p*-toluenesulfonate) (810 mg, 2.1 mmol). The mixture was stirred for 48 h at 60 °C. Solvent was removed under reduced pressure and the residue was dissolved in EtOAc (60 mL), washed with brine (15 mL $\times$ 3), dried over anhydrous MgSO_4 , filtered and the filtrate was concentrated under reduced pressure. The crude product was purified on a silica gel column chromatography (1:1 *n*-Hexane/EtOAc) to give a colorless oil (0.49 g, 72% yield): mp 102 °C; $[\alpha]_{\text{D}}^{28} +3.3$ (c 1.0, CHCl_3); ^1H NMR (300 MHz, CDCl_3) 1.06 (d, 6H), 1.79~1.84 (m, 2H), 1.94~1.98 (m, 2H), 2.42~2.43 (m, 12H), 2.98~ 3.37 (m, 12H), 3.87~3.89 (m, 2H), 7.27~7.34 (m, 8H), 7.70 (d, 8H); ^{13}C NMR (75 MHz, CDCl_3) 15.07, 21.46, 21.49, 26.92, 30.46, 43.74, 48.28, 54.67, 56.15, 127.26, 127.39, 129.68, 129.79, 135.12, 137.12, 143.41, 143.64; HRMS (FAB) m/z calcd 845.2747 for $\text{C}_{40}\text{H}_{53}\text{N}_4\text{O}_8\text{S}_4$ $[\text{M} + \text{H}]^+$, found 845.2742

Figure S1. X-ray structure of $[\text{Ni} \cdot (\mathbf{17})_2]\text{Cl}_2 \cdot \text{H}_2\text{O}$

Table S1. Crystallographic data for [Ni(C₁₂H₂₇N₃)₂]Cl₂•H₂O.

<i>formula</i>	NiC ₂₄ H ₅₆ N ₆ Cl ₂ O
fw	574.36
crystal system	orthorhombic
space group	P2 ₁ 2 ₁ 2 ₁
<i>a</i> , Å	8.988(5)
<i>b</i> , Å	17.056(5)
<i>c</i> , Å	19.521(5)
α , deg	90.000(5)
β , deg	90.000(5)
γ , deg	90.000(5)
<i>V</i> , Å ³	2992.6(20)
<i>Z</i> , formula units	4
<i>T</i> , K	293 (2)
λ , Å	0.71069
crystal size, mm ³	0.5 × 0.3 × 0.3
<i>D</i> _{calcd} , g•cm ⁻³	1.275
μ _{calcd} , mm ⁻¹	0.854
<i>F</i> (000)	1248
index range	-11 ≤ <i>h</i> ≤ 11, -22 ≤ <i>k</i> ≤ 22, -25 ≤ <i>l</i> ≤ 25
2 θ range, deg	3.2 – 45
no. of reflections collected	6820
no. of independent reflections	6767
no. of obsd data [<i>F</i> > 4 σ (<i>F</i>)]	5614
absorption correction	NONE
no. of restraints	0
no. of variable parameters	316
<i>R</i> ₁ ^a , <i>wR</i> ₂ ^b (4 σ)	0.0513, 0.1211
<i>R</i> ₁ ^a , <i>wR</i> ₂ ^b (all data)	0.0763, 0.1660
GOF (all data)	1.034
largest diff.peak and hole, eÅ ⁻³	0.403, - 0.449

$$^a R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|, \quad ^b wR(F^2) = [\Sigma w(F_o^2 - F_c^2)^2 / \Sigma w(F_o^2)^2]^{1/2}$$

$$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 3.4737P], \text{ where } P = (F_o^2 + 2F_c^2)/3$$

Table S2. Fractional atomic coordinates and equivalent displacement parameters($\text{\AA}^2$) of $[\text{Ni}(\text{C}_{12}\text{H}_{27}\text{N}_3)_2]\text{Cl}_2 \cdot \text{H}_2\text{O}$.

<i>atom</i>	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^*
Ni1	0.21800(4)	0.36818(2)	0.24791(3)	0.02363(11)
N1	0.1658(3)	0.2764(2)	0.3194(2)	0.0274(6)
N3	0.3951(3)	0.3888(2)	0.3181(2)	0.0283(6)
N2	0.0991(3)	0.4364(2)	0.3192(2)	0.0283(6)
N4	0.0477(3)	0.3397(2)	0.1756(2)	0.0264(6)
N5	0.2601(3)	0.4626(2)	0.1782(2)	0.0303(7)
N6	0.3506(3)	0.3075(2)	0.1769(2)	0.0272(6)
C1	0.0631(4)	0.3054(2)	0.3742(2)	0.0287(8)
C2	-0.0091(4)	0.3825(2)	0.3511(2)	0.0311(9)
C3	0.2036(4)	0.4732(2)	0.3691(2)	0.0340(9)
C4	0.3669(4)	0.4680(2)	0.3460(2)	0.0319(9)
C5	0.4015(4)	0.3248(2)	0.3691(2)	0.0339(9)
C6	0.3138(4)	0.2537(2)	0.3450(2)	0.0324(9)
C7	-0.1683(6)	0.2771(3)	0.4447(3)	0.0575(14)
C8	-0.0536(5)	0.2435(2)	0.3950(2)	0.0365(10)
C9	0.0175(6)	0.1711(3)	0.4258(3)	0.0482(12)
C10	0.4431(7)	0.5784(3)	0.4257(3)	0.065(2)
C11	0.4751(5)	0.4943(3)	0.4027(3)	0.0417(11)
C12	0.6363(6)	0.4887(4)	0.3781(3)	0.069(2)
C13	0.4107(4)	0.4471(2)	0.1502(2)	0.0317(9)
C14	0.4173(4)	0.3620(2)	0.1265(2)	0.0338(8)
C15	0.2533(4)	0.2476(2)	0.1464(2)	0.0304(9)
C16	0.1037(4)	0.2834(2)	0.1230(2)	0.0293(8)
C17	0.0038(4)	0.4171(2)	0.1483(2)	0.0344(9)
C18	0.1378(5)	0.4644(2)	0.1265(2)	0.0370(9)
C19	0.4324(7)	0.5901(3)	0.1115(3)	0.069(2)
C20	0.4646(6)	0.5043(3)	0.0935(3)	0.0431(11)
C21	0.6301(6)	0.4935(3)	0.0801(3)	0.0636(15)
C22	-0.1491(5)	0.2562(3)	0.0713(2)	0.0481(12)
C23	-0.0120(5)	0.2201(2)	0.1053(2)	0.0327(9)
C24	0.0532(6)	0.1558(3)	0.0604(3)	0.0520(13)
OW1	0.2331(4)	0.6163(2)	0.2561(2)	0.0766(11)
Cl1	-0.31298(10)	0.29994(7)	0.24391(7)	0.0500(3)
Cl2	0.10394(14)	0.08908(6)	0.25350(8)	0.0583(3)

$$^*U_{eq}=(1/3) \sum_{ij} U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

Table S3. Anisotropic displacement parameters($\text{\AA}^2$) of $[\text{Ni}(\text{C}_{12}\text{H}_{27}\text{N}_3)_2]\text{Cl}_2 \cdot \text{H}_2\text{O}$.

<i>atom</i>	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ni1	0.0228(2)	0.0252(2)	0.0229(2)	-0.0017(2)	-0.0002(2)	0.00093(15)
N1	0.0278(14)	0.0277(15)	0.027(2)	0.0009(12)	0.0004(12)	-0.0012(12)
N3	0.0249(14)	0.034(2)	0.026(2)	-0.0017(12)	-0.0023(12)	-0.0002(12)
N2	0.0259(14)	0.0284(14)	0.030(2)	-0.0031(13)	0.0006(13)	0.0056(12)
N4	0.0238(14)	0.0296(15)	0.026(2)	-0.0020(12)	-0.0019(12)	0.0008(12)
N5	0.032(2)	0.0252(14)	0.034(2)	0.0016(12)	0.0009(13)	-0.0021(12)
N6	0.0248(14)	0.029(2)	0.027(2)	-0.0030(13)	0.0009(12)	0.0025(12)
C1	0.028(2)	0.034(2)	0.024(2)	-0.005(2)	0.0031(15)	-0.004(2)
C2	0.023(2)	0.039(2)	0.032(2)	-0.003(2)	0.0037(14)	0.003(2)
C3	0.036(2)	0.031(2)	0.035(2)	-0.009(2)	0.003(2)	0.000(2)
C4	0.030(2)	0.031(2)	0.034(2)	-0.004(2)	0.000(2)	-0.004(2)
C5	0.028(2)	0.041(2)	0.033(2)	0.005(2)	-0.003(2)	0.001(2)
C6	0.034(2)	0.032(2)	0.032(2)	0.004(2)	-0.001(2)	0.005(2)
C7	0.047(3)	0.062(3)	0.063(3)	0.001(3)	0.024(2)	-0.014(2)
C8	0.036(2)	0.042(2)	0.032(2)	-0.001(2)	0.003(2)	-0.012(2)
C9	0.057(3)	0.044(2)	0.044(3)	0.004(2)	0.004(2)	-0.017(2)
C10	0.068(3)	0.056(3)	0.070(4)	-0.028(3)	-0.011(3)	-0.010(3)
C11	0.041(2)	0.046(2)	0.038(3)	-0.007(2)	-0.011(2)	-0.007(2)
C12	0.039(3)	0.077(4)	0.091(4)	-0.024(3)	-0.013(3)	-0.014(3)
C13	0.028(2)	0.037(2)	0.030(2)	0.004(2)	0.000(2)	-0.005(2)
C14	0.027(2)	0.039(2)	0.035(2)	-0.001(2)	0.005(2)	0.000(2)
C15	0.035(2)	0.025(2)	0.032(2)	-0.0031(14)	0.000(2)	0.0020(15)
C16	0.031(2)	0.031(2)	0.025(2)	0.001(2)	-0.002(2)	-0.003(2)
C17	0.027(2)	0.035(2)	0.041(3)	0.000(2)	-0.002(2)	0.005(2)
C18	0.040(2)	0.031(2)	0.039(2)	0.004(2)	-0.006(2)	0.002(2)
C19	0.078(4)	0.043(3)	0.087(4)	0.014(3)	0.022(3)	-0.013(3)
C20	0.046(3)	0.045(3)	0.038(3)	0.003(2)	0.002(2)	-0.013(2)
C21	0.050(3)	0.072(4)	0.069(4)	0.009(3)	0.018(3)	-0.017(3)
C22	0.041(2)	0.057(3)	0.047(3)	-0.005(2)	-0.011(2)	-0.011(2)
C23	0.038(2)	0.032(2)	0.028(2)	-0.001(2)	-0.005(2)	-0.007(2)
C24	0.061(3)	0.042(2)	0.053(3)	-0.014(2)	-0.006(2)	-0.011(2)
OW1	0.090(3)	0.044(2)	0.095(3)	0.002(2)	0.012(3)	0.017(2)
Cl1	0.0323(4)	0.0631(6)	0.0547(7)	-0.0063(6)	-0.0027(5)	0.0055(4)
Cl2	0.0803(7)	0.0400(5)	0.0548(7)	0.0041(6)	0.0054(9)	-0.0102(5)

Table S4. Selected bond lengths(Å) and angles(°) of [Ni(C₁₂H₂₇N₃)₂]Cl₂•H₂O.

Ni1-N6	2.100(3)	C1-C2	1.533(5)
Ni1-N2	2.106(3)	C1-C8	1.543(5)
Ni1-N3	2.130(3)	C3-C4	1.537(5)
Ni1-N4	2.138(3)	C4-C11	1.540(6)
Ni1-N5	2.141(3)	C5-C6	1.522(5)
Ni1-N1	2.149(3)	C7-C8	1.528(6)
N1-C6	1.473(5)	C8-C9	1.515(6)
N1-C1	1.498(5)	C10-C11	1.531(7)
N3-C5	1.477(5)	C11-C12	1.529(7)
N3-C4	1.479(5)	C13-C14	1.525(6)
N2-C2	1.477(5)	C13-C20	1.553(6)
N2-C3	1.492(5)	C15-C16	1.545(5)
N4-C17	1.478(5)	C16-C23	1.538(5)
N4-C16	1.494(5)	C17-C18	1.511(6)
N5-C13	1.484(5)	C19-C20	1.533(7)
N5-C18	1.493(5)	C20-C21	1.522(7)
N6-C15	1.472(4)	C22-C23	1.529(6)
N6-C14	1.480(5)	C23-C24	1.522(6)
N6-Ni1-N2	175.13(11)	C13-N5-Ni1	105.2(2)
N6-Ni1-N3	94.68(12)	C18-N5-Ni1	108.4(2)
N2-Ni1-N3	82.11(12)	C15-N6-C14	114.0(3)
N6-Ni1-N4	81.90(12)	C15-N6-Ni1	105.8(2)
N2-Ni1-N4	101.44(11)	C14-N6-Ni1	111.1(2)
N3-Ni1-N4	175.89(11)	N1-C1-C2	109.5(3)
N6-Ni1-N5	81.44(12)	N1-C1-C8	112.3(3)
N2-Ni1-N5	95.37(12)	C2-C1-C8	112.1(3)
N3-Ni1-N5	98.78(11)	N2-C2-C1	112.3(3)
N4-Ni1-N5	83.02(11)	N2-C3-C4	112.7(3)
N6-Ni1-N1	101.13(12)	N3-C4-C3	108.9(3)
N2-Ni1-N1	82.13(12)	N3-C4-C11	114.9(3)
N3-Ni1-N1	82.27(11)	C3-C4-C11	112.1(3)
N4-Ni1-N1	96.11(11)	N3-C5-C6	111.1(3)
N5-Ni1-N1	177.16(11)	N1-C6-C5	111.3(3)
C6-N1-C1	113.6(3)	C9-C8-C7	109.7(4)
C6-N1-Ni1	102.4(2)	C9-C8-C1	112.1(4)
C1-N1-Ni1	110.9(2)	C7-C8-C1	111.6(3)
C5-N3-C4	115.7(3)	C12-C11-C10	109.2(4)
C5-N3-Ni1	109.9(2)	C12-C11-C4	110.8(4)
C4-N3-Ni1	105.0(2)	C10-C11-C4	111.4(4)
C2-N2-C3	113.7(3)	N5-C13-C14	108.5(3)
C2-N2-Ni1	105.6(2)	N5-C13-C20	115.8(3)
C3-N2-Ni1	110.1(2)	C14-C13-C20	111.7(3)
C17-N4-C16	114.6(3)	N6-C14-C13	112.3(3)
C17-N4-Ni1	103.1(2)	N6-C15-C16	111.3(3)
C16-N4-Ni1	111.0(2)	N4-C16-C23	112.2(3)
C13-N5-C18	115.1(3)	N4-C16-C15	110.1(3)

C23-C16-C15	112.2(3)
N4-C17-C18	111.5(3)
N5-C18-C17	112.6(3)
C21-C20-C19	109.9(4)
C21-C20-C13	110.5(4)

C19-C20-C13	112.2(4)
C24-C23-C22	110.5(4)
C24-C23-C16	112.0(4)
C22-C23-C16	111.1(3)

Table S5. Fractional atomic coordinates and isotropic displacement parameters($\text{\AA}^2$) of $[\text{Ni}(\text{C}_{12}\text{H}_{27}\text{N}_3)_2]\text{Cl}_2 \cdot \text{H}_2\text{O}$.

<i>atom</i>	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{iso}</i>
H1A	0.1239(3)	0.2347(2)	0.2987(2)	0.033
H1B	0.1769(3)	0.3142(2)	0.2876(2)	0.033
H3A	0.3229(3)	0.3775(2)	0.2877(2)	0.034
H3B	0.4819(3)	0.3898(2)	0.2952(2)	0.034
H2A	0.0490(3)	0.4745(2)	0.2972(2)	0.034
H2B	0.1522(3)	0.4098(2)	0.2878(2)	0.034
H4A	-0.0314(3)	0.3182(2)	0.1967(2)	0.032
H4B	0.1190(3)	0.3467(2)	0.2074(2)	0.032
H5A	0.2618(3)	0.5090(2)	0.2002(2)	0.036
H5B	0.2389(3)	0.4254(2)	0.2094(2)	0.036
H6A	0.4243(3)	0.2834(2)	0.1999(2)	0.033
H6B	0.2976(3)	0.3355(2)	0.2074(2)	0.033
H1	0.1236(4)	0.3169(2)	0.4148(2)	0.034
H2C	-0.0876(4)	0.3710(2)	0.3185(2)	0.037
H2D	-0.0540(4)	0.4080(2)	0.3904(2)	0.037
H3C	0.1931(4)	0.4475(2)	0.4131(2)	0.041
H3D	0.1769(4)	0.5279(2)	0.3750(2)	0.041
H4	0.3788(4)	0.5049(2)	0.3079(2)	0.038
H5C	0.3612(4)	0.3432(2)	0.4123(2)	0.041
H5D	0.5044(4)	0.3100(2)	0.3766(2)	0.041
H6C	0.3684(4)	0.2273(2)	0.3089(2)	0.039
H6D	0.3024(4)	0.2172(2)	0.3828(2)	0.039
H7A	-0.2224(30)	0.3186(16)	0.4227(7)	0.086
H7B	-0.1184(7)	0.2974(21)	0.4844(9)	0.086
H7C	-0.2361(27)	0.2365(6)	0.4583(16)	0.086
H8	-0.1065(5)	0.2273(2)	0.3534(2)	0.044
H9A	-0.0588(6)	0.1352(9)	0.4400(15)	0.072
H9B	0.0763(29)	0.1859(4)	0.4648(10)	0.072
H9C	0.0799(29)	0.1464(11)	0.3923(6)	0.072
H10A	0.5222(26)	0.5963(10)	0.4549(19)	0.097
H10B	0.3509(26)	0.5798(5)	0.4505(20)	0.097
H10C	0.4361(49)	0.6118(6)	0.3863(3)	0.097
H11	0.4627(5)	0.4594(3)	0.4422(3)	0.050
H12A	0.7018(6)	0.5056(23)	0.4141(8)	0.104
H12B	0.6497(13)	0.5216(19)	0.3387(13)	0.104
H12C	0.6587(15)	0.4354(6)	0.3662(20)	0.104
H13	0.4806(4)	0.4520(2)	0.1884(2)	0.038
H14A	0.3649(4)	0.3570(2)	0.0833(2)	0.041
H14B	0.5203(4)	0.3474(2)	0.1188(2)	0.041
H15A	0.2345(4)	0.2066(2)	0.1798(2)	0.037
H15B	0.3029(4)	0.2240(2)	0.1074(2)	0.037
H16	0.1231(4)	0.3133(2)	0.0811(2)	0.035
H17A	-0.0506(4)	0.4457(2)	0.1832(2)	0.041
H17B	-0.0617(4)	0.4099(2)	0.1093(2)	0.041

H18A	0.1749(5)	0.4440(2)	0.0834(2)	0.044
H18B	0.1078(5)	0.5184(2)	0.1190(2)	0.044
H19A	0.4798(39)	0.6031(8)	0.1541(12)	0.104
H19B	0.3269(8)	0.5976(6)	0.1157(21)	0.104
H19C	0.4702(43)	0.6235(3)	0.0759(11)	0.104
H20	0.4110(6)	0.4918(3)	0.0512(3)	0.052
H21A	0.6851(6)	0.5061(22)	0.1208(7)	0.095
H21B	0.6606(11)	0.5275(17)	0.0436(14)	0.095
H21C	0.6491(8)	0.4400(7)	0.0675(19)	0.095
H22A	-0.2240(14)	0.2166(5)	0.0655(15)	0.072
H22B	-0.1221(9)	0.2771(17)	0.0274(7)	0.072
H22C	-0.1873(21)	0.2975(13)	0.0997(8)	0.072
H23	-0.0442(5)	0.1958(2)	0.1483(2)	0.039
H24A	-0.0247(9)	0.1209(12)	0.0461(15)	0.078
H24B	0.1265(29)	0.1271(13)	0.0858(6)	0.078
H24C	0.0988(34)	0.1789(3)	0.0207(9)	0.078

Table S6. Bond lengths(Å) and angles(°) of [Ni(C₁₂H₂₇N₃)₂]Cl₂•H₂O.

N1-H1A	0.90	C10-H10B	0.96
N1-H1B	0.90	C10-H10C	0.96
N3-H3A	0.90	C11-H11	0.98
N3-H3B	0.90	C12-H12A	0.96
N2-H2A	0.90	C12-H12B	0.96
N2-H2B	0.90	C12-H12C	0.96
N4-H4A	0.90	C13-H13	0.98
N4-H4B	0.90	C14-H14A	0.97
N5-H5A	0.90	C14-H14B	0.97
N5-H5B	0.90	C15-H15A	0.97
N6-H6A	0.90	C15-H15B	0.97
N6-H6B	0.90	C16-H16	0.98
C1-H1	0.98	C17-H17A	0.97
C2-H2C	0.97	C17-H17B	0.97
C2-H2D	0.97	C18-H18A	0.97
C3-H3C	0.97	C18-H18B	0.97
C3-H3D	0.97	C19-H19A	0.96
C4-H4	0.98	C19-H19B	0.96
C5-H5C	0.97	C19-H19C	0.96
C5-H5D	0.97	C20-H20	0.98
C6-H6C	0.97	C21-H21A	0.96
C6-H6D	0.97	C21-H21B	0.96
C7-H7A	0.96	C21-H21C	0.96
C7-H7B	0.96	C22-H22A	0.96
C7-H7C	0.96	C22-H22B	0.96
C8-H8	0.98	C22-H22C	0.96
C9-H9A	0.96	C23-H23	0.98
C9-H9B	0.96	C24-H24A	0.96
C9-H9C	0.96	C24-H24B	0.96
C10-H10A	0.96	C24-H24C	0.96
C6-N1-H1A	108.8(2)	C2-N2-H2B	108.8(2)
C1-N1-H1A	108.8(2)	C3-N2-H2B	108.8(2)
Ni1-N1-H1A	112.11(8)	Ni1-N2-H2B	3.30(8)
C6-N1-H1B	108.8(2)	H2A-N2-H2B	107.7
C1-N1-H1B	108.8(2)	C17-N4-H4A	108.6(2)
Ni1-N1-H1B	6.62(8)	C16-N4-H4A	108.6(2)
H1A-N1-H1B	107.7	Ni1-N4-H4A	110.86(9)
C5-N3-H3A	108.4(2)	C17-N4-H4B	108.6(2)
C4-N3-H3A	108.4(2)	C16-N4-H4B	108.6(2)
Ni1-N3-H3A	3.34(7)	Ni1-N4-H4B	5.63(8)
C5-N3-H3B	108.4(2)	H4A-N4-H4B	107.6
C4-N3-H3B	108.4(2)	C13-N5-H5A	108.5(2)
Ni1-N3-H3B	109.29(9)	C18-N5-H5A	108.5(2)
H3A-N3-H3B	107.4	Ni1-N5-H5A	111.15(9)
C2-N2-H2A	108.8(2)	C13-N5-H5B	108.5(2)
C3-N2-H2A	108.8(2)	C18-N5-H5B	108.5(2)
Ni1-N2-H2A	109.72(9)	Ni1-N5-H5B	4.09(8)

H5A-N5-H5B	107.5	H10A-C10-H10B	109.5
C15-N6-H6A	108.7(2)	C11-C10-H10C	109.5(3)
C14-N6-H6A	108.7(2)	H10A-C10-H10C	109.5
Ni1-N6-H6A	108.33(9)	H10B-C10-H10C	109.5
C15-N6-H6B	108.7(2)	C12-C11-H11	108.5(3)
C14-N6-H6B	108.7(2)	C10-C11-H11	108.5(3)
Ni1-N6-H6B	3.09(8)	C4-C11-H11	108.5(2)
H6A-N6-H6B	107.6	C11-C12-H12A	109.5(3)
N1-C1-H1	107.6(2)	C11-C12-H12B	109.5(3)
C2-C1-H1	107.6(2)	H12A-C12-H12B	109.5
C8-C1-H1	107.6(2)	C11-C12-H12C	109.5(3)
N2-C2-H2C	109.1(2)	H12A-C12-H12C	109.5
C1-C2-H2C	109.1(2)	H12B-C12-H12C	109.5
N2-C2-H2D	109.1(2)	N5-C13-H13	106.8(2)
C1-C2-H2D	109.1(2)	C14-C13-H13	106.8(2)
H2C-C2-H2D	107.9	C20-C13-H13	106.8(2)
N2-C3-H3C	109.1(2)	N6-C14-H14A	109.1(2)
C4-C3-H3C	109.1(2)	C13-C14-H14A	109.1(2)
N2-C3-H3D	109.1(2)	N6-C14-H14B	109.1(2)
C4-C3-H3D	109.1(2)	C13-C14-H14B	109.1(2)
H3C-C3-H3D	107.8	H14A-C14-H14B	107.9
N3-C4-H4	106.8(2)	N6-C15-H15A	109.4(2)
C3-C4-H4	106.8(2)	C16-C15-H15A	109.4(2)
C11-C4-H4	106.8(2)	N6-C15-H15B	109.4(2)
N3-C5-H5C	109.4(2)	C16-C15-H15B	109.4(2)
C6-C5-H5C	109.4(2)	H15A-C15-H15B	108.0
N3-C5-H5D	109.4(2)	N4-C16-H16	107.4(2)
C6-C5-H5D	109.4(2)	C23-C16-H16	107.4(2)
H5C-C5-H5D	108.0	C15-C16-H16	107.4(2)
N1-C6-H6C	109.4(2)	N4-C17-H17A	109.3(2)
C5-C6-H6C	109.4(2)	C18-C17-H17A	109.3(2)
N1-C6-H6D	109.4(2)	N4-C17-H17B	109.3(2)
C5-C6-H6D	109.4(2)	C18-C17-H17B	109.3(2)
H6C-C6-H6D	108.0	H17A-C17-H17B	108.0
C8-C7-H7A	109.5(3)	N5-C18-H18A	109.1(2)
C8-C7-H7B	109.5(3)	C17-C18-H18A	109.1(2)
H7A-C7-H7B	109.5	N5-C18-H18B	109.1(2)
C8-C7-H7C	109.5(2)	C17-C18-H18B	109.1(2)
H7A-C7-H7C	109.5	H18A-C18-H18B	107.8
H7B-C7-H7C	109.5	C20-C19-H19A	109.5(3)
C9-C8-H8	107.7(2)	C20-C19-H19B	109.5(3)
C7-C8-H8	107.7(3)	H19A-C19-H19B	109.5
C1-C8-H8	107.7(2)	C20-C19-H19C	109.5(3)
C8-C9-H9A	109.5(2)	H19A-C19-H19C	109.5
C8-C9-H9B	109.5(2)	H19B-C19-H19C	109.5
H9A-C9-H9B	109.5	C21-C20-H20	108.0(3)
C8-C9-H9C	109.5(3)	C19-C20-H20	108.0(3)
H9A-C9-H9C	109.5	C13-C20-H20	108.0(2)
H9B-C9-H9C	109.5	C20-C21-H21A	109.5(3)
C11-C10-H10A	109.5(3)	C20-C21-H21B	109.5(3)
C11-C10-H10B	109.5(3)	H21A-C21-H21B	109.5

C20-C21-H21C	109.5(3)	C24-C23-H23	107.7(3)
H21A-C21-H21C	109.5	C22-C23-H23	107.7(2)
H21B-C21-H21C	109.5	C16-C23-H23	107.7(2)
C23-C22-H22A	109.5(2)	C23-C24-H24A	109.5(3)
C23-C22-H22B	109.5(2)	C23-C24-H24B	109.5(3)
H22A-C22-H22B	109.5	H24A-C24-H24B	109.5
C23-C22-H22C	109.5(2)	C23-C24-H24C	109.5(3)
H22A-C22-H22C	109.5	H24A-C24-H24C	109.5
H22B-C22-H22C	109.5	H24B-C24-H24C	109.5