Supplementary Material (ESI) for Chemical Communications

An Efficient and General Synthesis of Oxazino[4,3-*a*]indoles by A Cascade Addition–Cyclization Reaction of (1*H*-Indol-2-yl)methanols and Vinyl Sulfonium Salts

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1. General methods

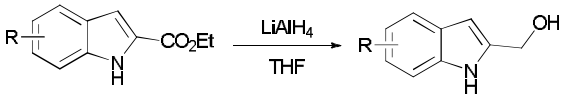
All reactions were monitored by TLC analysis with silica gel-coated plates. Flash column chromatography was performed using 200-300 mesh silica gel. ^{1}H NMR spectra were recorded on Varian Mercury 400 / 600 (400 / 600 MHz) spectrophotometers. Chemical shifts (δ) are reported in ppm from the solvent resonance as the internal standard ($CDCl_3$: 7.26 ppm). Data are reported as follows: chemical shift, multiplicity (s = single, d = doublet, t = triplet, dd = doublet of doublets, m = multiplet), coupling constants (Hz) and integration. ^{13}C NMR spectra were recorded on Varian Mercury 400/600 (100/150MHz) with complete proton decoupling spectrophotometers ($CDCl_3$: 77.0 ppm). Mass spectra were measured on a Finnigan Trace MS spectrometer or API 2000 LC/MS/MS (ESI-MS). Melting point was measured with BűCHI Melting Point B-545.

2. Materials

Commercial reagents were purified prior to use following the guidelines of Perrin and Armarego. All the solvents were treated according to general methods. Flash chromatography was conducted using 60 silica (mesh 230-400).

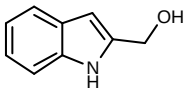
3. Experimental Procedures and Characterizations

3.1 General procedure for the synthesis of the (1*H*-indol-2-yl)methanol Compounds

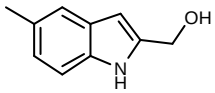


3a - 3e, 3g - 3i, 9

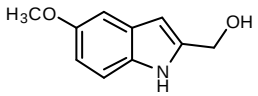
In a two-necked flask were charged with freshly distilled THF (20 mL) and $LiAlH_4$ (15.6 mmol, 0.59 g). It was cooled to 0 ℃ and the crude carboxylate (10.0 mmol) were carefully added in portionwise, and then warmed to room temperature. About 2 h later (monitored by TLC), the mixture was cooled to 0 ℃ and 20 mL of THF as well as 1.1 mL 20% of aq. KOH were added, repectively. After stirring for 10 min the mixture was filtered through a Buchner funnel and the salts was extracted again with 20 mL of reflux THF. The combined organic filtrates were washed with brine, dried over $MgSO_4$, filtered and concentrated. And the crude indolyl alcohol was purified by flash chromatography (petroleum ether/EtOAc = 5/1) to afford (1*H*-indol-2-yl)methanol compounds **3a**-**3e**, **3g**-**3i, 9** as a solid.



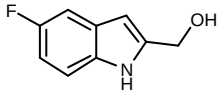
(1*H*-indol-2-yl)methanol (3a). White solid, 92% yield. mp 71-72 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) δ 8.26 (s, 1H), 7.53 (d, J = 7.6 Hz, 1H), 7.34 - 6.85 (m, 3H), 6.27 (s, 1H), 4.51 (s, 2H), 2.99 (d, J = 13.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 137.37, 136.29, 127.82, 122.11, 120.54, 119.86, 111.07, 100.54, 58.17.



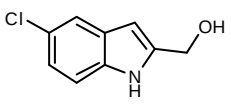
(5-methyl-1*H*-indol-2-yl)methanol (3b). White solid, 90% yield. mp 83-84 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (s, 1H), 7.35 (s, 1H), 7.19 (d, J = 8.3 Hz, 1H), 7.00 (d, J = 8.2 Hz, 1H), 6.30 (s, 1H), 4.74 (s, 2H), 2.43 (s, 3H), 2.10 (s, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 137.46, 134.58, 128.98, 128.02, 123.68, 120.13, 110.73, 100.07, 58.07, 21.37.



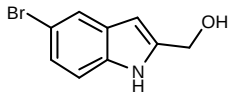
(5-methoxy-1*H*-indol-2-yl)methanol (3c). White solid, 93% yield. mp 81-82 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.30 (s, 1H), 7.08 (d, J = 8.8 Hz, 1H), 7.00 (d, J = 2.4 Hz, 1H), 6.80 (dd, J_1 = 8.8 Hz, J_2 = 2.5 Hz, 1H), 6.25 (s, 1H), 4.63 (d, J = 4.9 Hz, 2H), 3.80 (s, 3H), 2.74 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 154.04, 138.25, 131.47, 128.35, 112.25, 111.72, 102.29, 100.35, 58.42, 55.80.



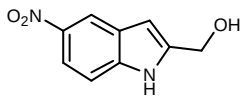
(5-fluoro-1*H*-indol-2-yl)methanol (3d). White solid, 90% yield. mp 91-92 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.46 (s, 1H), 7.18 (dd, J_1 = 9.5 Hz, J_2 = 2.3 Hz, 1H), 7.09 (dd, J_1 = 8.7 Hz, J_2 = 4.4 Hz, 1H), 6.86-6.90 (m, 1H), 6.28 (s, 1H), 4.66 (s, 2H), 2.72 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.03, 156.70, 139.11, 132.79, 128.27, 128.17, 111.60, 111.50, 110.61, 110.35, 105.41, 105.18, 100.65, 100.60, 58.37.



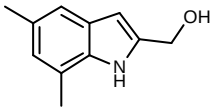
(5-chloro-1*H*-indol-2-yl)methanol (3e). White solid, 89% yield. mp 112-113 ℃. ^{1}H NMR (400 MHz, DMSO) δ (ppm) 11.27 (s, 1H), 7.84-7.22 (m, 2H), 7.06 (dd, J_1 = 8.5 Hz, J_2 = 1.9 Hz, 1H), 6.31 (s, 1H), 5.36 (t, J = 5.6 Hz, 1H), 4.65 (d, J = 5.5 Hz, 2H); ^{13}C NMR (100 MHz, DMSO) δ (ppm) 142.12, 134.63, 129.09, 123.19, 120.41, 118.77, 112.50, 98.18, 56.80.



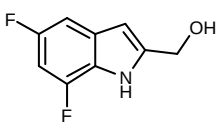
(5-bromo-1*H*-indol-2-yl)methanol (3f). In a three-necked flask were charged with freshly distilled toluene (130 mL) and ethyl 5-bromo-1*H*-indole-2-carboxylate (10.0 mmol, 2.68 g). It was cooled to -78 ℃ and DIBAL-H (20.0 mmol, 1M, 20 mL) were carefully added into by dropwise under the protection of N_2. About 2 h later (monitored by TLC), the reaction was quenched with saturated NH_4Cl solution at -78 ℃. The mixture was filtered through a Buchner funnel and the salts was washed with EtOAc. The combined organic filtrates were washed with brine, dried over $MgSO_4$, filtered and concentrated. And the crude indolyl alcohol was purified by flash chromatography (petroleum ether/EtOAc = 5/1) to afford **3f** as a white solid in 84% yield. mp 113-114 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 8.48 (s, 1H), 7.69 (s, 1H), 7.23 (d, J = 35.8 Hz, 2H), 6.33 (s, 1H), 4.81 (s, 2H), 2.05 (s, 1H); ^{13}C NMR (150 MHz, DMSO) δ (ppm) 141.96, 134.87, 129.84, 122.95, 121.81, 113.01, 111.20, 98.09, 56.79.



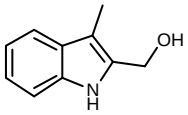
(5-nitro-1*H*-indol-2-yl)methanol (3g). Yellow solid, 84% yield. mp 114-115 ℃. ^{1}H NMR (400 MHz, DMSO) δ (ppm) 11.84 (s, 1H), 8.50 (s, 1H), 7.97 (d, J = 8.9 Hz, 1H), 7.50 (d, J = 8.9 Hz, 1H), 6.58 (s, 1H), 5.50 (s, 1H), 4.68 (d, J = 5.0 Hz, 2H); ^{13}C NMR (100 MHz, DMSO) δ (ppm) 144.36, 140.52, 139.54, 127.29, 116.77, 116.18, 111.34, 100.81, 56.71.



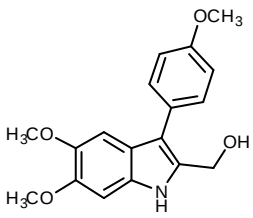
(5,7-dimethyl-1*H*-indol-2-yl)methanol (3h). White solid, 82% yield. mp 91-92 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 8.39 (s, 1H), 7.17 (s, 1H), 6.79 (s, 1H), 6.26 (s, 1H), 4.66 (s, 2H), 2.37 (s, 8H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 137.14, 134.32, 129.23, 127.75 , 124.42, 119.82, 117.79, 100.66, 58.63, 21.32, 16.52.



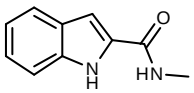
(5,7-difluoro-1*H*-indol-2-yl)methanol (3i). White solid, 84% yield. mp 78-79 ℃. ^{1}H NMR (400 MHz, DMSO) δ (ppm) 11.59 (s, 1H), 7.12 (dd, J_1 = 9.5 Hz, J_2 = 1.8 Hz, 1H), 6.99-6.77 (m, 1H), 6.41 (s, 1H), 5.33 (s, 1H), 4.62 (s, 2H); ^{13}C NMR (100 MHz, DMSO) δ (ppm) 156.93, 156.83, 154.62, 154.53, 149.28, 146.84, 146.69, 143.66, 130.77, 130.65, 130.58, 120.66, 120.53, 100.75, 100.52, 100.17, 95.91, 95.70, 95.61, 95.40, 56.67.



(3-methyl-1*H*-indol-2-yl)methanol (3j).[1] White solid, 73% yield. mp 111-112 ℃. ^{1}H NMR (400 MHz, DMSO) δ (ppm) 10.74 (s, 1H), 7.42 (d, J = 7.6 Hz, 1H), 7.30 (d, J = 7.7 Hz, 1H), 7.03 (t, J = 6.9 Hz, 1H), 6.96 (d, J = 7.3 Hz, 1H), 5.18-5.00 (m, 1H), 4.60 (d, J = 5.4 Hz, 2H), 2.22 (s, 3H); ^{13}C NMR (100 MHz, DMSO) δ (ppm) 135.40, 135.24, 128.50, 120.73, 118.04, 110.91, 105.70, 54.70, 8.32.



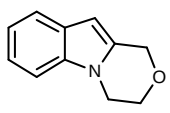
(5,6-dimethoxy-3-(4-methoxyphenyl)-1*H*-indol-2-yl)methanol (9). White solid, 75% yield. mp 84-85 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 8.27 (s, 1H), 7.39 (d, J = 8.6 Hz, 2H), 7.09 (s, 1H), 7.03 (d, J = 8.5 Hz, 2H), 6.89 (s, 1H), 4.85 (d, J = 5.6 Hz, 2H), 3.93 (s, 3H), 3.88 (d, J = 4.2 Hz, 6H), 1.75 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 158.30, 147.66, 145.57, 131.86, 130.36, 130.03, 127.09, 120.34, 115.28, 114.26, 101.64, 94.63, 57.14, 56.52, 56.30, 55.34.



N-methyl-1*H*-indole-2-carboxamide (3l). To a solution of ethanolic $MeNH_2$ (6.75 M, 4.0 mL) was added methyl 1*H*-indole-2-carboxylate (5.7 mmol, 0.99 g) and the resulting solution was stirred at room temperature until the carboxylate was consumed as determined by TLC. The mixture was filtered through a Buchner funnel and the filter was washed with Et_2O to give the pure product as a white solid in 72% yield. mp 222-223 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 9.51 (s, 1H), 7.65 (d, J = 8.0 Hz, 1H), 7.45 (d, J = 8.3 Hz, 1H), 7.29 (t, J = 7.6 Hz, 1H), 7.14 (t, J = 7.5 Hz, 1H), 6.81 (s, 1H), 6.22 (s, 1H), 3.07 (d, J = 4.9 Hz, 3H); ^{13}C NMR (100 MHz, DMSO) δ (ppm) 161.62, 136.36, 131.88, 127.16, 123.16, 121.45, 119.68, 112.31, 102.04, 25.82.

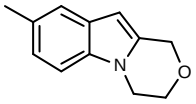
3.2 General procedure for the synthesis of the oxazino[4,3-*a*]indoles compounds

A stirred solution of (1*H*-indol-2-yl)methanol compounds **3a-3k** (0.5 mmol) or N-methyl-1*H*-indole-2-carboxamide **3l** (0.5 mmol, 0.0871 g) or 1*H*-indole-2-carboxamide **3m** (0.5 mmol, 0.0801 g) in CH_2Cl_2 (40 mL) was treated with KOH (1.25 mmol, 0.0702 g) at 0 ℃ under N_2. After 10 min a solution of diphenylvinylsulfonium salt **4**[2] (0.6 mmol, 0.2174 g) in CH_2Cl_2 (10 mL) was added dropwise and the reaction was stirred at 0 ℃, then the reaction mixture was warmed to r.t. and the reaction was stirred at this temperature until determined to be complete by TLC analysis. The solvent was concentrated under vacuum. The product was then purified using flash column chromatography on silica gel (PE/EA = 30/1 as eluant) to afford the desired product **5**.

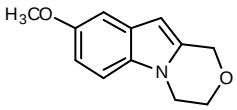


3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5a). Prepared according to the general procedure from **3a** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (93% yield). mp 129-130 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.56 (d, J = 7.8 Hz, 1H), 7.24 (d, J = 8.1 Hz, 1H), 7.17 (t, J = 7.8 Hz, 1H), 7.11 (t, J = 7.3 Hz, 1H), 6.19 (s, 1H), 4.94 (s, 2H), 4.09 (d, J = 5.7 Hz, 2H), 4.00 (d, J = 5.7 Hz, 2H); ^{13}C NMR(150 MHz, $CDCl_3$) δ (ppm) 136.08, 132.91, 127.87, 120.84, 119.98, 108.42, 95.73,

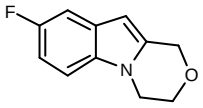
95.71, 64.90, 64.52, 41.61; HRMS: m/z (ESI) calculated $[M+H]^+$ 174.0913, measured 174.0917.



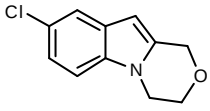
8-methyl-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5b). Prepared according to the general procedure from **3b** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (73% yield). mp 125-126 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.25 (s, 1H), 7.05 (d, J = 8.2 Hz, 1H), 6.91 (d, J = 8.2 Hz, 1H), 6.02 (s, 1H), 4.84 (s, 2H), 4.12-3.94 (m, 2H), 3.94-3.81 (m, 2H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 134.55, 132.96, 129.20, 128.16, 122.41, 119.94, 108.11, 95.26, 64.91, 64.54, 41.66, 21.42; HRMS: m/z (ESI) calculated $[M+H]^+$ 188.1070, measured 188.1071.



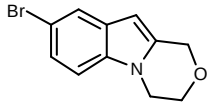
8-(methyloxy)-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5c). Prepared according to the general procedure from **3c** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (75% yield). mp 153-154 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.14 (d, J = 8.8 Hz, 1H), 7.03 (d, J = 2.4 Hz, 1H), 6.83 (dd, J_1 = 8.8 Hz, J_2 = 2.4 Hz, 1H), 6.12 (s, 1H), 4.93 (s, 2H), 4.11 (dd, J_1 = 6.2 Hz, J_2 = 4.4 Hz, 2H), 3.99 (dd, J_1 = 6.1 Hz, J_2 = 4.4 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 154.44, 133.57, 131.44, 128.32, 110.85, 109.10, 102.26, 95.51, 64.86, 64.52, 55.80, 41.68; HRMS: m/z (ESI) calculated $[M+Na]^+$ 226.0839, measured 226.0848.



8-fluoro-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5d). Prepared according to the general procedure from **3d** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (81% yield). mp 108-109 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.19 (dd, J_1 = 9.7 Hz, J_2 = 2.4 Hz, 1H), 7.13 (dd, J_1 = 8.8 Hz, J_2 = 4.3 Hz, 1H), 6.99-6.80 (m, 1H), 6.14 (s, 1H), 4.92 (s, 2H), 4.10 (dd, J_1 = 6.2 Hz, J_2 = 4.4 Hz, 2H), 3.98 (dd, J_1 = 6.0 Hz, J_2 = 4.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 141.93, 138.80, 136.23, 127.08, 117.22, 116.59, 108.24, 98.12, 64.61, 64.18, 41.93; HRMS: m/z (ESI) calculated M^+ 191.0746, measured 191.0744.

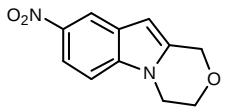


8-chloro-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5e). Prepared according to the general procedure from **3e** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (74% yield). mp 132-133 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.50 (d, J = 1.7 Hz, 1H), 7.18-7.02 (m, 2H), 6.12 (s, 1H), 4.93 (s, 2H), 4.11 (dd, J_1 = 6.2 Hz, J_2 = 4.5 Hz, 2H), 3.98 (dd, J_1 = 6.0 Hz, J_2 = 4.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 134.49, 134.30, 128.88, 125.58, 121.08, 119.59, 109.38, 95.47, 64.72, 64.35, 41.64; HRMS: m/z (ESI) calculated M^+ 207.0451, measured 207.0450.

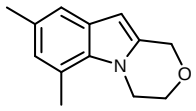


8-bromo-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5f). Prepared according to the general procedure from **3f** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (76% yield). mp 158-159 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.65 (d, J = 1.8 Hz, 1H), 7.22 (dd, J_1 = 8.5 Hz, J_2

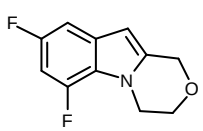
= 2.0 Hz, 1H), 7.08 (d, J = 8.6 Hz, 1H), 6.12 (s, 1H), 4.93 (s, 2H), 4.11 (dd, J_1 = 6.2 Hz, J_2 = 4.5 Hz, 2H), 3.98 (dd, J_1 = 6.1 Hz, J_2 = 4.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 134.76, 134.14, 129.54, 123.64, 122.65, 113.18, 109.84, 95.39, 64.70, 64.35, 41.62; HRMS: m/z (ESI) calculated M^+ 250.9946, measured 250.9947.



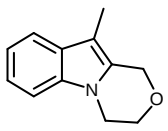
8-nitro-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5g). Prepared according to the general procedure from **3g** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a yellow solid (74% yield). mp 163-164 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.46 (d, J = 2.1 Hz, 1H), 8.04 (dd, J_1 = 9.0 Hz, J_2 = 2.2 Hz, 1H), 7.37-7.16 (m, 1H), 6.37 (s, 1H), 5.00 (s, 2H), 4.24-4.17 (m, 2H), 4.13 (dd, J_1 = 5.9 Hz, J_2 = 4.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 141.93, 138.80, 136.23, 127.08, 117.22, 116.59, 108.24, 98.12, 64.61, 64.18, 41.93; HRMS: m/z (ESI) calculated $[M+Na]^+$ 241.0584, measured 241.0582.



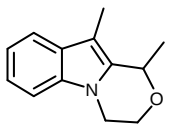
6,8-dimethyl-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5h). Prepared according to the general procedure from **3h** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (60% yield). mp 106-107 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.15 (s, 1H), 6.69 (s, 1H), 6.09 (d, J = 0.9 Hz, 1H), 4.91 (s, 2H), 4.44-4.32 (m, 2H), 4.11-4.01 (m, 2H), 2.64 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 133.84, 133.24, 129.21, 128.86, 125.58, 120.50, 117.88, 96.40, 65.17, 64.91, 45.24, 21.06, 19.55; HRMS: m/z (ESI) calculated $[M+H]^+$ 202.1226, measured 202.1236.



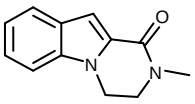
6,8-difluoro-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5i). Prepared according to the general procedure from **3i** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (61% yield). mp 89-90 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 6.95 (dd, J_1 = 9.0 Hz, J_2 = 2.0 Hz, 1H), 6.60-6.64 (m, 1H), 6.14 (s, 1H), 4.91 (s, 2H), 4.29 (d, J = 5.3 Hz, 2H), 4.09 (t, J = 5.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 157.67, 156.10, 150.03, 148.50, 148.40, 135.55, 130.59, 120.94, 100.87, 100.72, 97.09, 96.72, 96.67, 96.52, 64.63, 64.53, 44.35, 44.33; HRMS: m/z (ESI) calculated M^+ 209.0652, measured 209.0650.



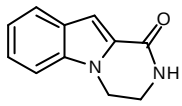
10-methyl-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5j). Prepared according to the general procedure from **3j** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (74% yield). mp 57-58 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.52 (d, J = 7.8 Hz, 1H), 7.23 (t, J = 5.2 Hz, 1H), 7.17 (t, J = 7.4 Hz, 1H), 7.12 (t, J = 7.3 Hz, 1H), 4.92 (s, 2H), 4.19-4.06 (m, 2H), 4.06 – 3.95 (m, 2H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 136.11, 128.67, 128.44, 120.93, 119.37, 118.24, 108.32, 104.15, 64.65, 63.92, 41.74, 7.90; HRMS: m/z (ESI) calculated M^+ 187.0997, measured 187.1000.



1,10-dimethyl-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (5k). Prepared according to the general procedure from **3k** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (62% yield). mp 63-64 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.54 (d, J = 7.8 Hz, 1H), 7.24 (t, J = 6.3 Hz, 1H), 7.18 (t, J = 7.4 Hz, 1H), 7.12 (t, J = 7.3 Hz, 1H), 5.13 (d, J = 6.5 Hz, 1H), 4.37-4.22 (m, 1H), 4.11-4.03 (m, 1H), 4.03-3.91 (m, 2H), 2.27 (s, 3H), 1.65 (d, J = 6.5 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 135.22, 132.67, 128.47, 120.94, 119.27, 118.16, 108.29, 103.85, 69.96, 61.91, 41.67, 20.44, 9.06; HRMS: m/z (ESI) calculated $[M+H]^+$ 202.1226, measured 202.1221.



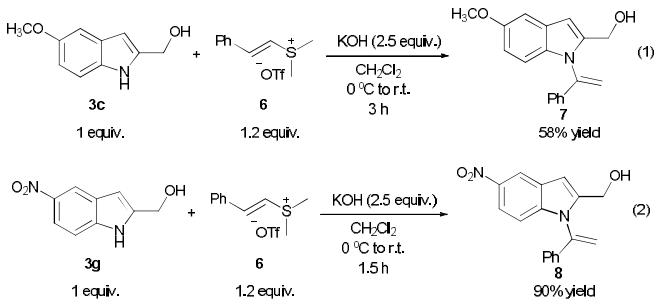
2-methyl-3,4-dihydropyrazino[1,2-*a*]indol-1(2*H*)-one (5l). Prepared according to the general procedure from **3l** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (80% yield). mp 245-246 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.71 (d, J = 8.0 Hz, 1H), 7.37-7.29 (m, 2H), 7.26 (d, J = 2.6 Hz, 1H), 7.16 (dd, J_1 = 10.5 Hz, J_2 = 3.9 Hz, 1H), 4.33-4.25 (m, 2H), 3.84-3.75 (m, 2H), 3.19 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 160.25, 136.31, 129.24, 127.36, 124.31, 122.60, 120.57, 109.47, 105.84, 47.98, 39.91, 34.21; HRMS: m/z (ESI) calculated $[M+H]^+$ 201.1022, measured 201.1025.



3,4-dihydropyrazino[1,2-*a*]indol-1(2*H*)-one (5m). Prepared according to the general procedure from indole-2-carboxamide **3m**[3] (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a white solid (90% yield). mp 197-198 ℃. ^{1}H NMR (400 MHz, DMSO) δ (ppm) 8.18 (s, 1H), 7.70 (d, J = 7.5 Hz, 1H), 7.53 (d, J = 7.9 Hz, 1H), 7.31 (s, 1H), 7.11 (d, J = 13.1 Hz, 2H), 4.27 (s, 2H), 3.67 (s, 2H); ^{13}C NMR (100 MHz, DMSO) δ (ppm) 160.38, 136.28, 129.70, 126.50, 123.96, 121.99, 120.32, 110.64, 104.19, 40.05, 39.75; HRMS: m/z (ESI) calculated $[M+Na]^+$ 209.0685, measured 209.0688.

3.3 Proposed reaction pathway for the cyclization process

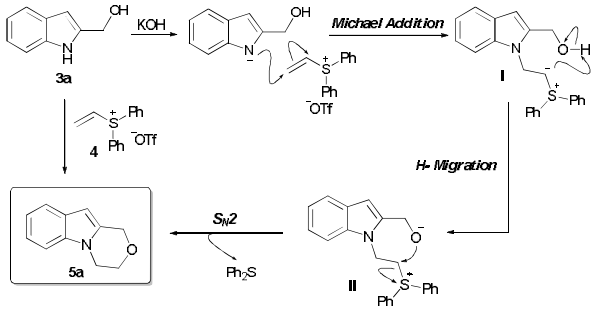
3.3.1 Initial experiment about mechanism investigation

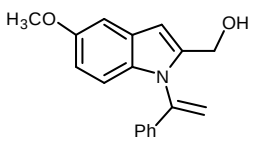


A stirred solution of (1*H*-indol-2-yl)methanol compounds **3c** and **3g** (0.5 mmol) in CH_2Cl_2 (40 mL) was treated with KOH (1.25 mmol, 0.0702 g) at 0 °C under N_2. After 10 min a solution of dimethyl[(E)-2-phenyl-1-ethenyl]sulfonium trifluoromethanesulfonate **6**[4] (0.6 mmol, 0.1572 g) in CH_2Cl_2 (10 mL) was added dropwise and the reaction was stirred at 0 °C, then the reaction mixture was warmed to r.t. and the reaction was stirred at this temperature until determined to be complete

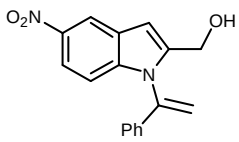
by TLC analysis. The solvent was concentrated under vacuum. The product was then purified using flash column chromatography on silica gel (PE/EA = 10/1 as eluant) to afford the desired product **7** and **8**.

3.3.2 Proposed reaction pathway for the cyclization process



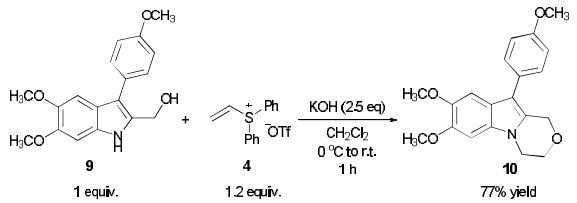


(5-methoxy-1-(1-phenylvinyl)-1*H*-indol-2-yl)methanol (7). Prepared according to the general procedure from **3c** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) to provide the title compound as a brown oil (58% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.32 – 7.23 (m, 3H), 7.11 (d, J = 7.5 Hz, 2H), 7.06 (s, 1H), 7.01 (d, J = 8.9 Hz, 1H), 6.79 – 6.72 (m, 1H), 6.54 (s, 1H), 5.96 (s, 1H), 5.45 (s, 1H), 4.53 (s, 2H), 3.82 (s, 3H), 1.81 (s, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 154.42, 142.64, 140.14, 136.80, 133.46, 129.10, 128.72, 127.87, 125.69, 113.44, 112.42, 111.74, 102.34, 57.40, 55.72; HRMS: m/z (ESI) calculated $[M+Na]^+$ 302.1152, measured 302.1158.



(5-nitro-1-(1-phenylvinyl)-1*H*-indol-2-yl)methanol (8). Prepared according to the general procedure from **3g** (0.5 mmol), **4** (0.6 mmol), KOH (1.25 mmol) and CH_2Cl_2 (50 mL) at 0 °C for 1.5 h to provide the title compound as a brown oil (90% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.49 (s, 1H), 7.96 (d, J = 9.0 Hz, 1H), 7.30 (dt, J_1 = 14.2 Hz, J_2 = 7.2 Hz, 3H), 7.11 (dd, J_1 = 14.9 Hz, J_2 = 8.4 Hz, 3H), 6.77 (s, 1H), 6.12 (s, 1H), 5.54 (s, 1H), 4.61 (s, 2H), 2.91 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 143.16, 141.83, 140.89, 135.53, 129.57, 128.94, 126.72, 125.39, 117.73, 114.72, 110.74, 104.14, 57.11; HRMS: m/z (ESI) calculated $[M+Na]^+$ 317.0897, measured 317.0902.

4. Synthesis of 7,8-bis(methyloxy)-10-[4-(methyloxy)phenyl]-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole 10



7,8-bis(methyloxy)-10-[4-(methyloxy)phenyl]-3,4-dihydro-1*H*-[1,4]oxazino[4,3-*a*]indole (10). A stirred solution of (5,6-dimethoxy-3-(4-methoxyphenyl)-1*H*-indol-2-yl)methanol **9** (0.5 mmol, 0.1567 g) in CH_2Cl_2 (40 mL) was treated with KOH (1.25 mmol, 0.0702 g) at 0 °C under N_2. After 10 min a solution of diphenylvinylsulfonium salt **4** (0.6 mmol, 0.2174 g) in CH_2Cl_2 (10 mL) was added dropwise and the reaction was stirred at 0 °C, then the reaction mixture was warmed to r.t. and the reaction was stirred at this temperature until determined to be complete by TLC analysis. The solvent was concentrated under vacuum. The product was then purified using flash column chromatography on silica gel (PE/EA = 30/1 as eluant) to afford the desired product **10** as a white solid in 77% yield. mp 147-148 ℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.33 (d, J = 8.5 Hz, 2H), 7.18 (s, 1H), 7.01 (d, J = 8.5 Hz, 2H), 6.81 (s, 1H), 5.02 (s, 2H), 4.20 (t, J = 5.0 Hz, 2H), 4.08 (t, J = 5.0 Hz, 2H), 3.97 (s, 3H), 3.91 (s, 3H), 3.87 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 157.76, 146.72, 145.57, 130.62, 129.46, 127.55, 127.11, 119.34, 114.22, 111.06, 101.13, 92.34, 64.62, 64.50, 56.44, 56.28, 55.29, 41.99; HRMS: m/z (ESI) calculated $[M+Na]^+$ 362.1363, measured 362.1364.

References

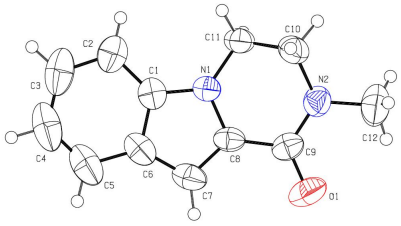
1 D. St C. Black, N. Kumar and L. C. H. Wong, *Synthesis*, 1986, 474.

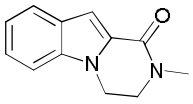
2 M. Yar, E. M. McGarrigle and V. K. Aggarwal, *Angew. Chem. Int. Ed.*, 2008, **47**, 3784.

3 P. Csomós, L. Fodor, I. Mándity and G. Bernáth, *Tetrahedron* 2007, **63**, 4983.

4 V. G. Nenajdenko, P. V. Vertelezkij, I. D. Gridnev, N. E. Shevchenko, E. S. Balenkova, *Tetrahedron*, 1997, **53**, 8173.

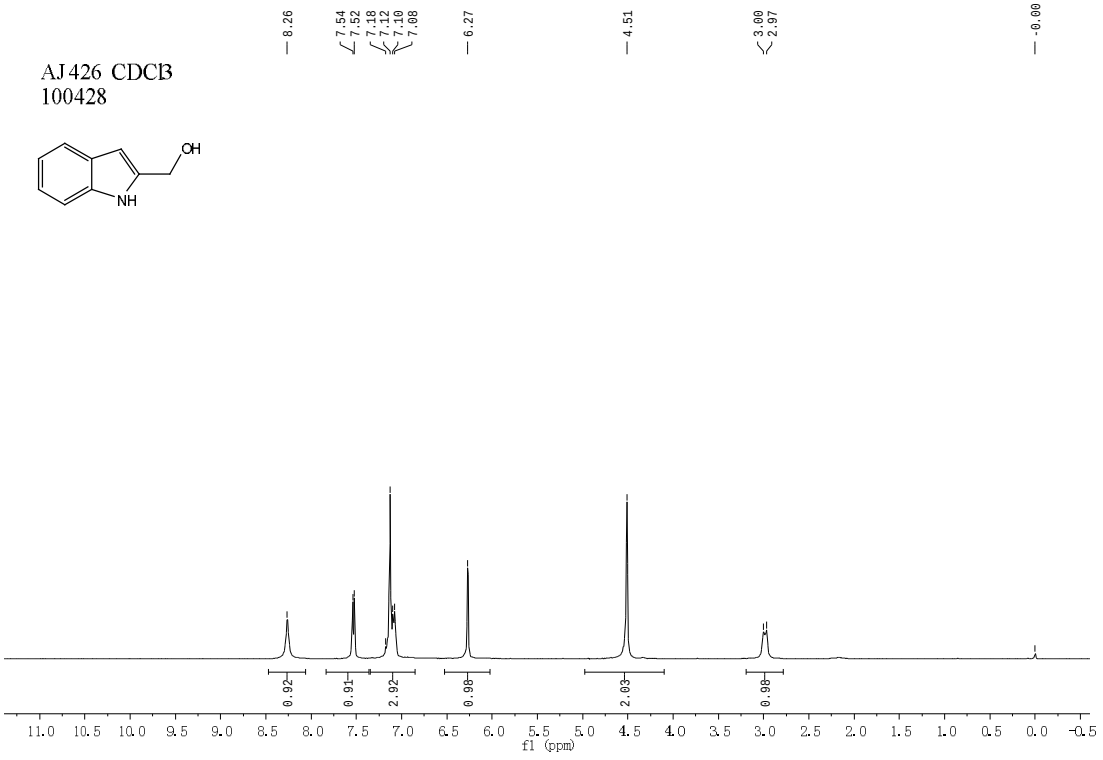
5. X-Ray structure of 2-methyl-3,4-dihydropyrazino[1,2-*a*]indol-1(2*H*)-one 5l

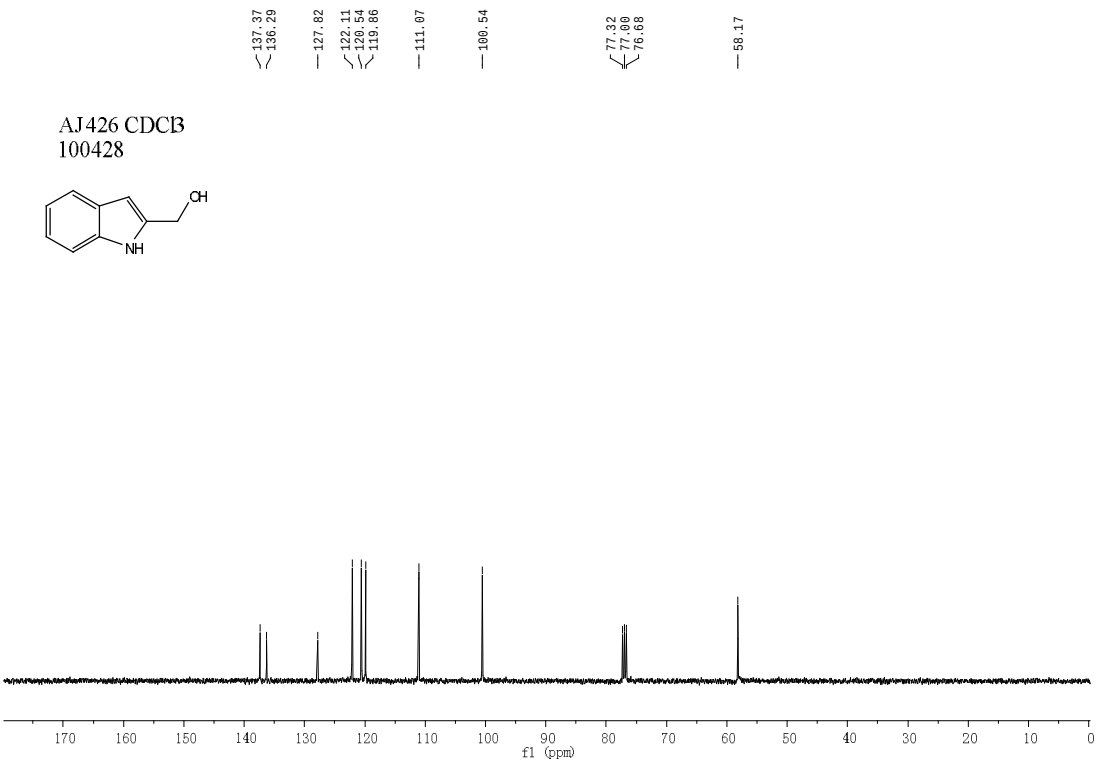


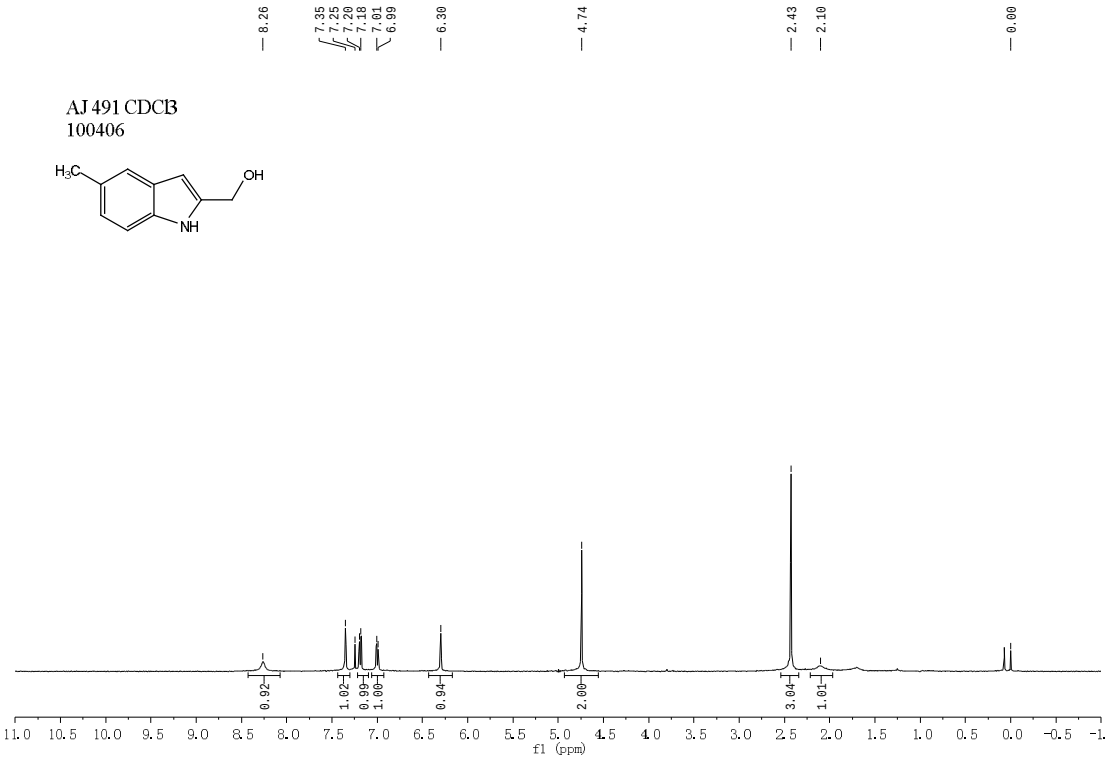


Crystal data for **5l**: $C_{12}H_{12}N_2O$, M = 200.24, monoclinic, $P2(1)/c$, a = 10.5035(12) Å, b = 8.0361(9) Å, c = 12.2743(14) Å, α = 90°, β = 98.375(2)°, γ = 90°, V = 1025.0(2) Å^3, Z = 4, T = 298(2), $F000$ = 424, final R indices [I>2σ(I)]: R_1 = 0.0489, wR_2 = 0.1371, R indices (all data): R_1 = 0.0679, wR_2 = 0.1458.

6. Copies of ^{1}H NMR and ^{13}C NMR Spectra



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7.20
7.18
7.01
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6.30
4.74
2.43
2.10
0.00
AJ491 CDCB
100406
H3C
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NH
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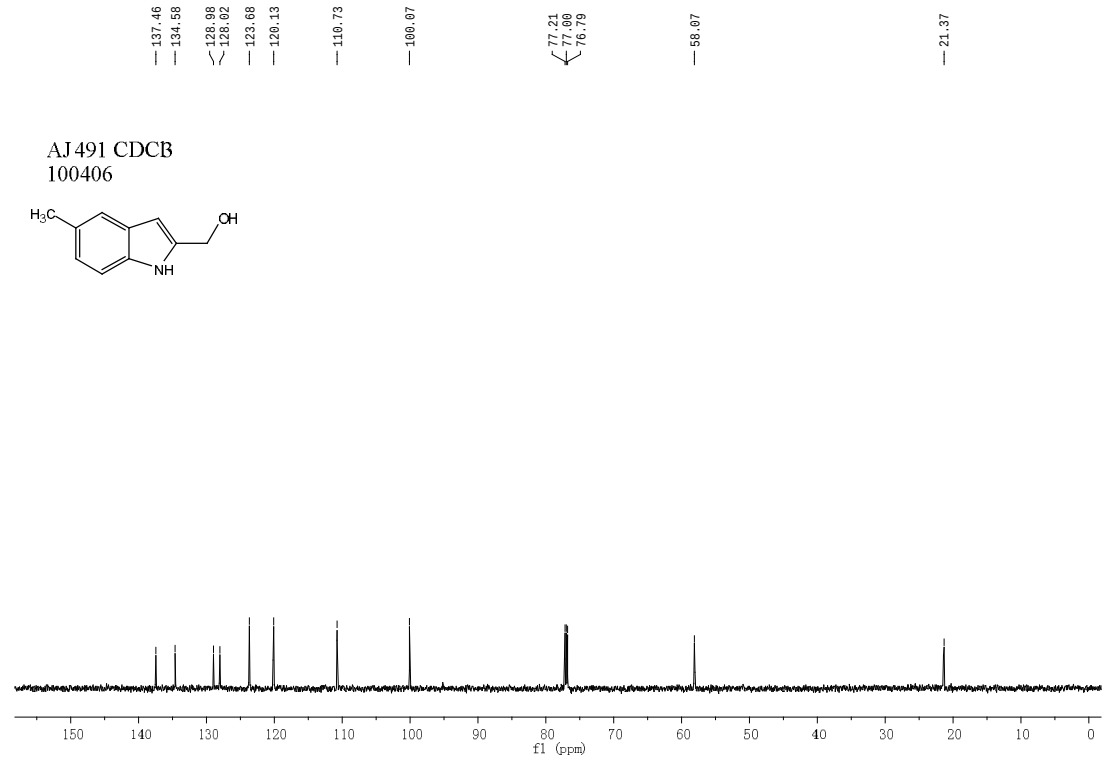
0.99
1.00
0.94
2.00
3.04
1.01
f1 (ppm)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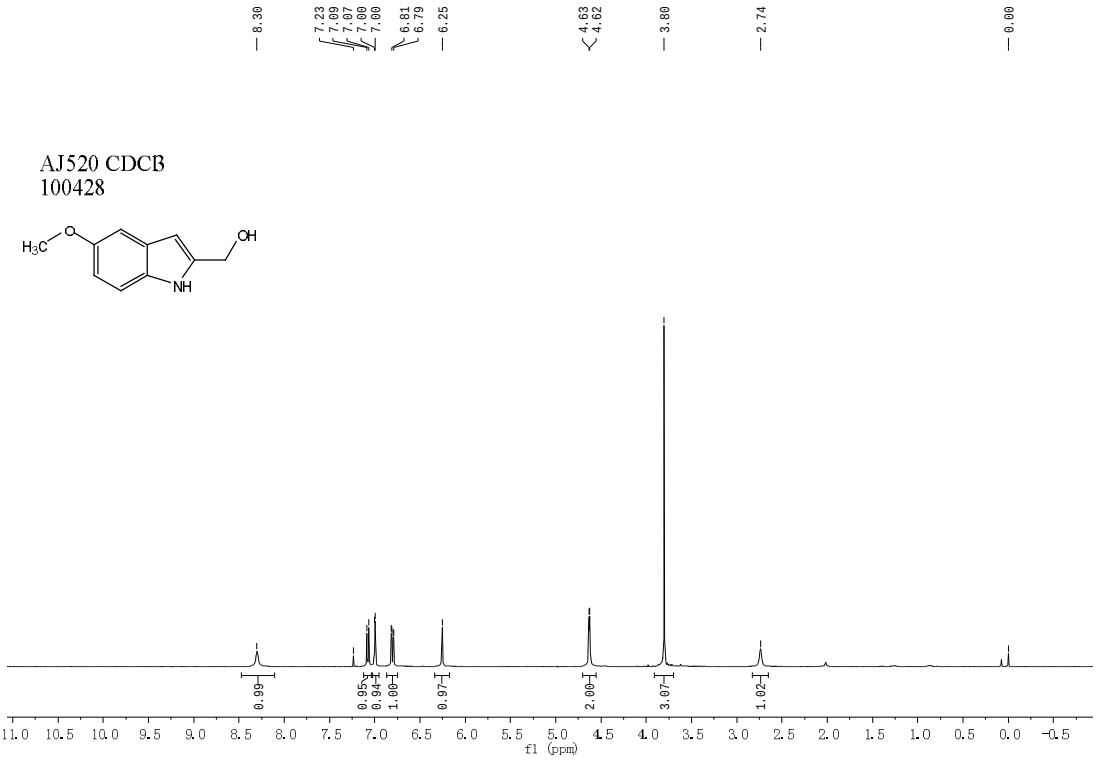
H3C
OH
NH
f1 (ppm)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0.00
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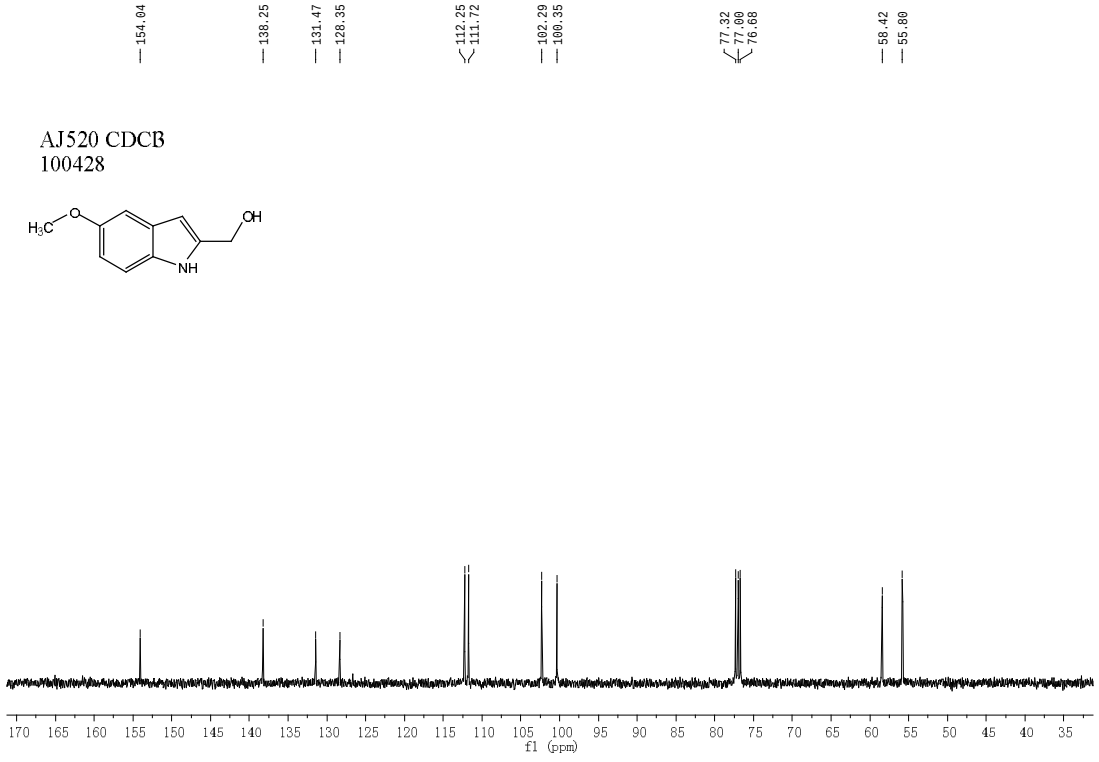
f1 (ppm)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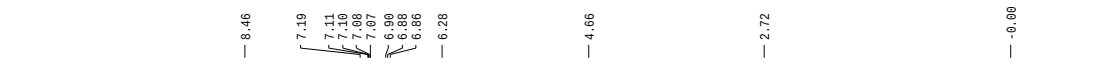
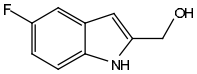
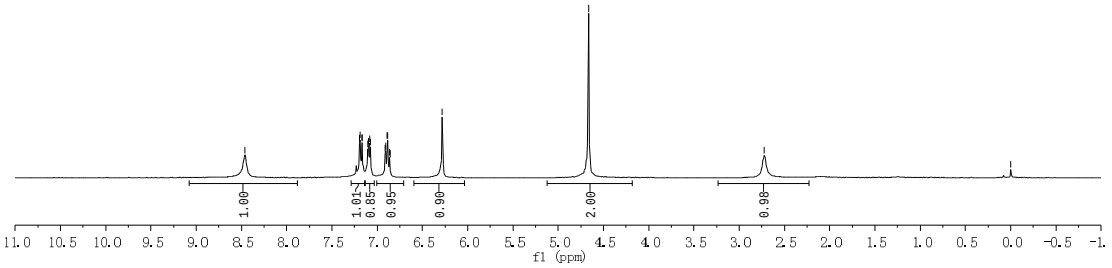
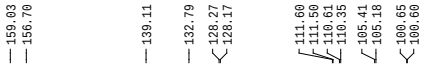
f1 (ppm)



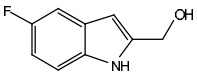
AJ499 CDCB
100428

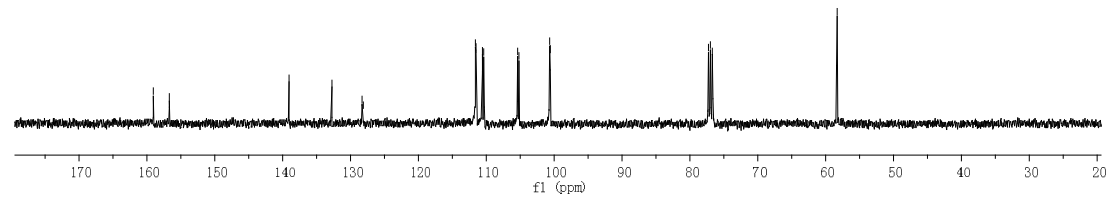


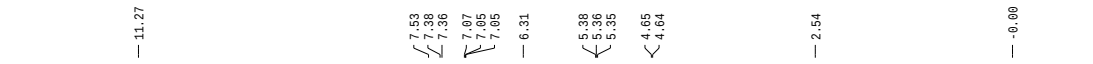




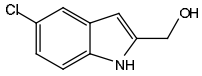
AJ499 CDCB
100428

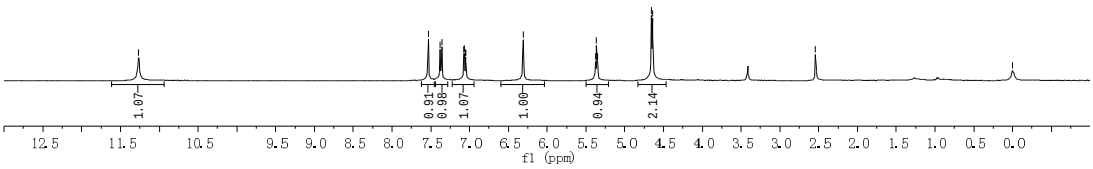




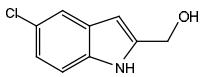


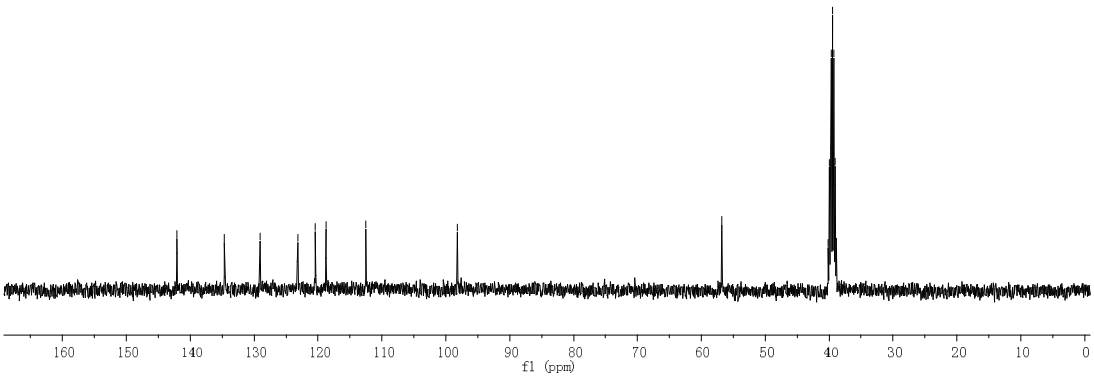
AJ490 DMSO
100428

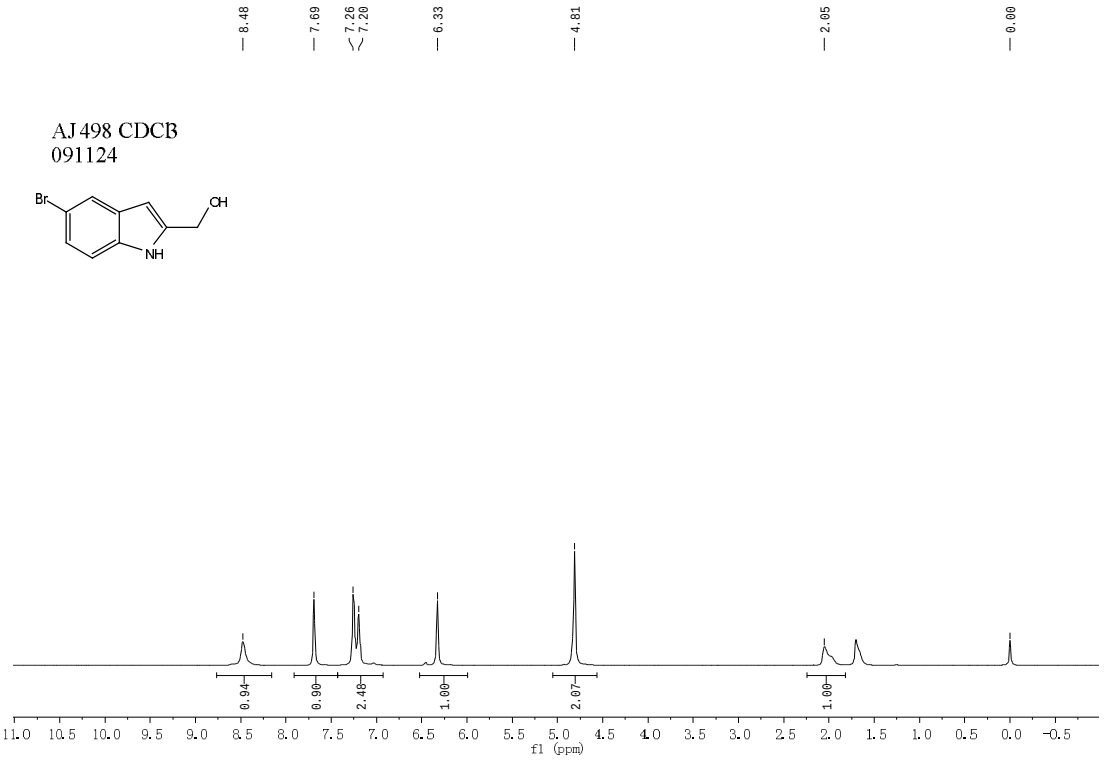


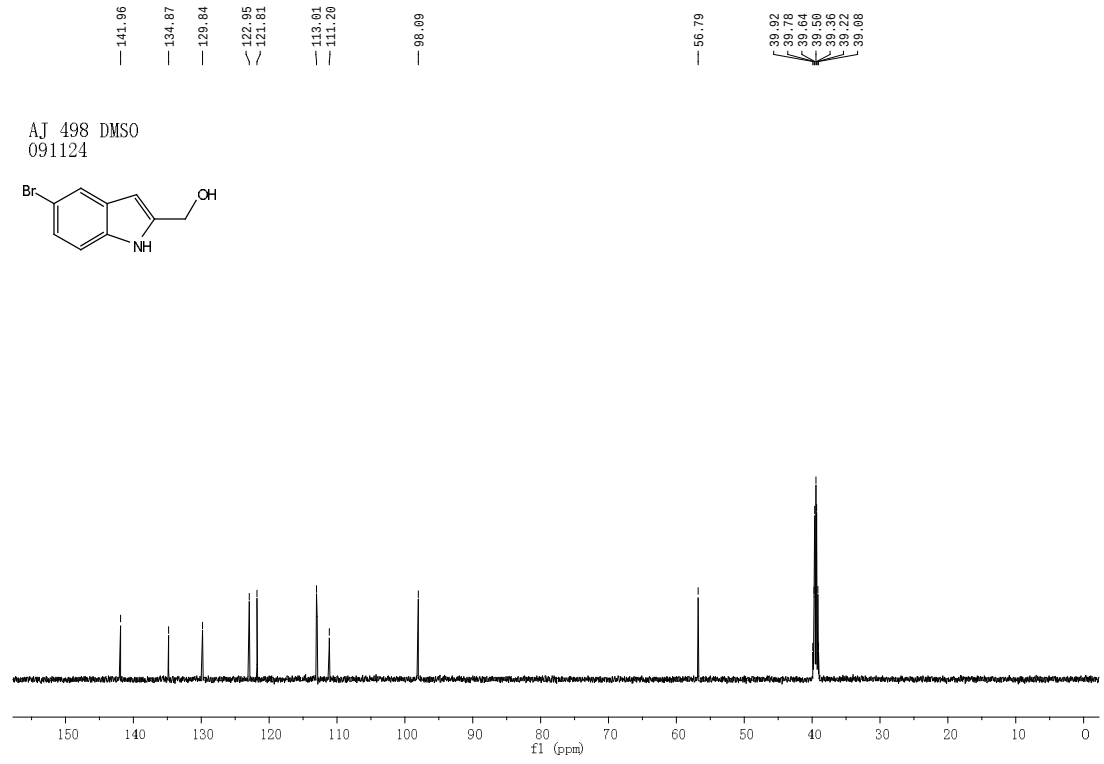


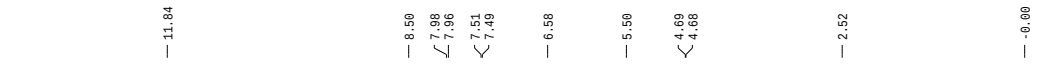
AJ490 DMSO
100428



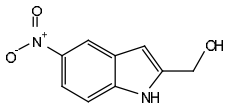


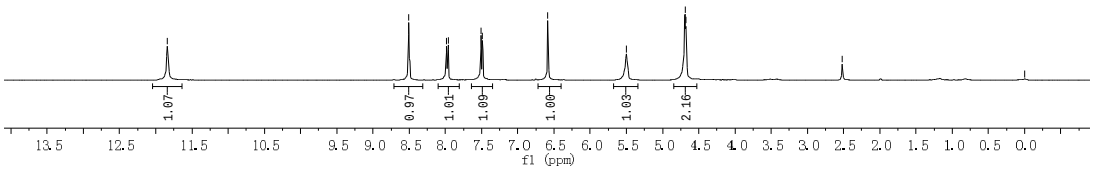

8.48
7.69
7.26
7.20
6.33
4.81
2.05
0.00
AJ 498 CDCB
091124
Br
OH
NH
0.94
0.90
2.48
1.00
2.07
1.00
f1 (ppm)


141.96
134.87
129.84
122.95
121.81
113.01
111.20
98.09
56.79
39.92
39.78
39.64
39.50
39.36
39.22
39.08
AJ 498 DMSO
091124
Br
OH
NH
f1 (ppm)

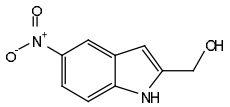


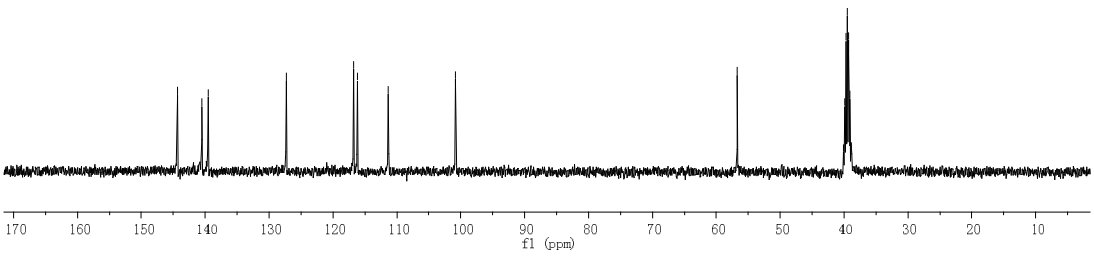
AJ494 DMSO
100428

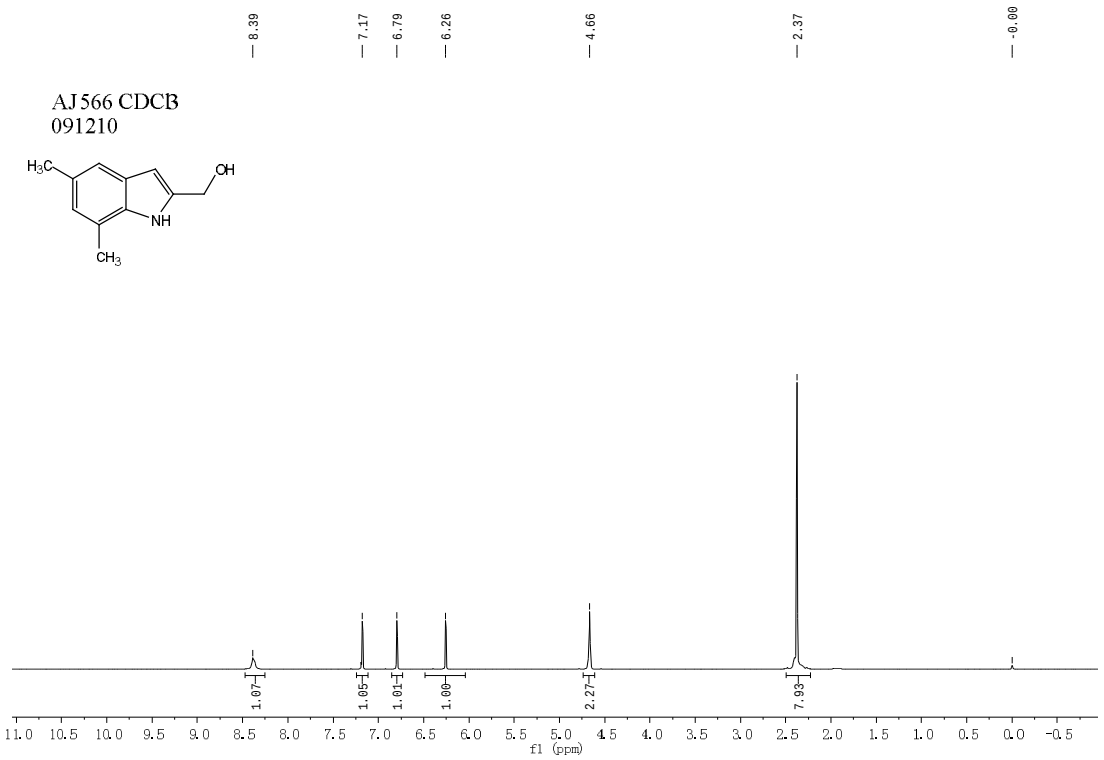


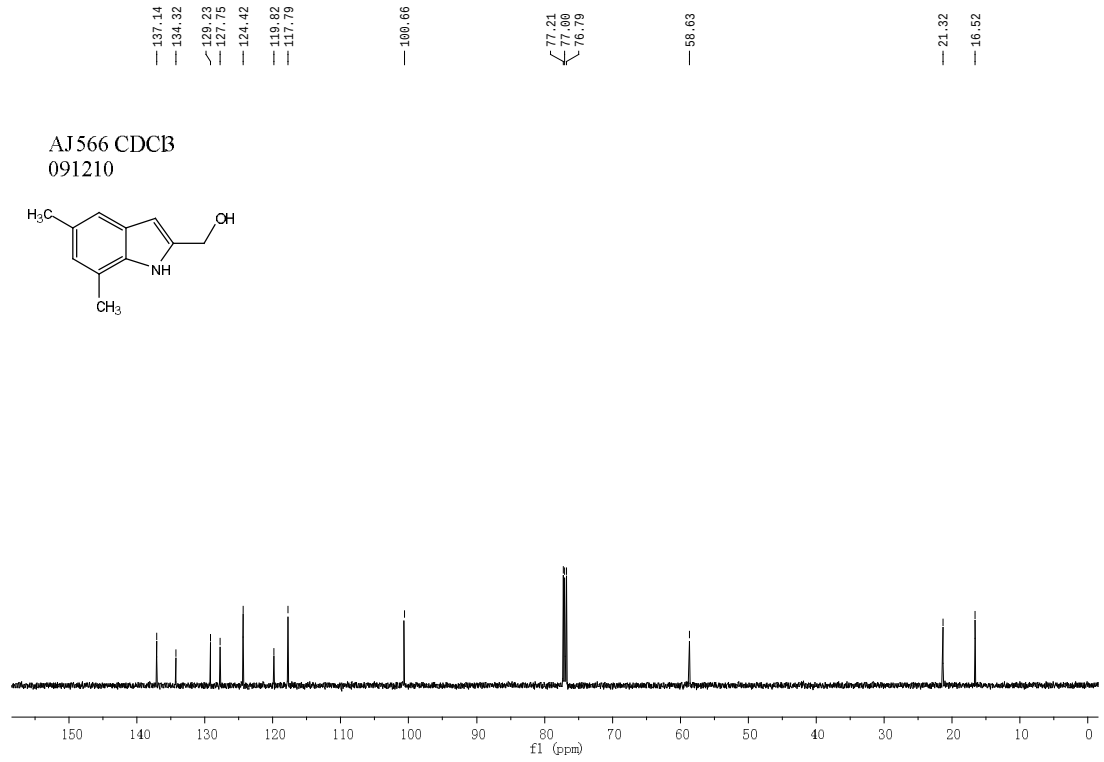


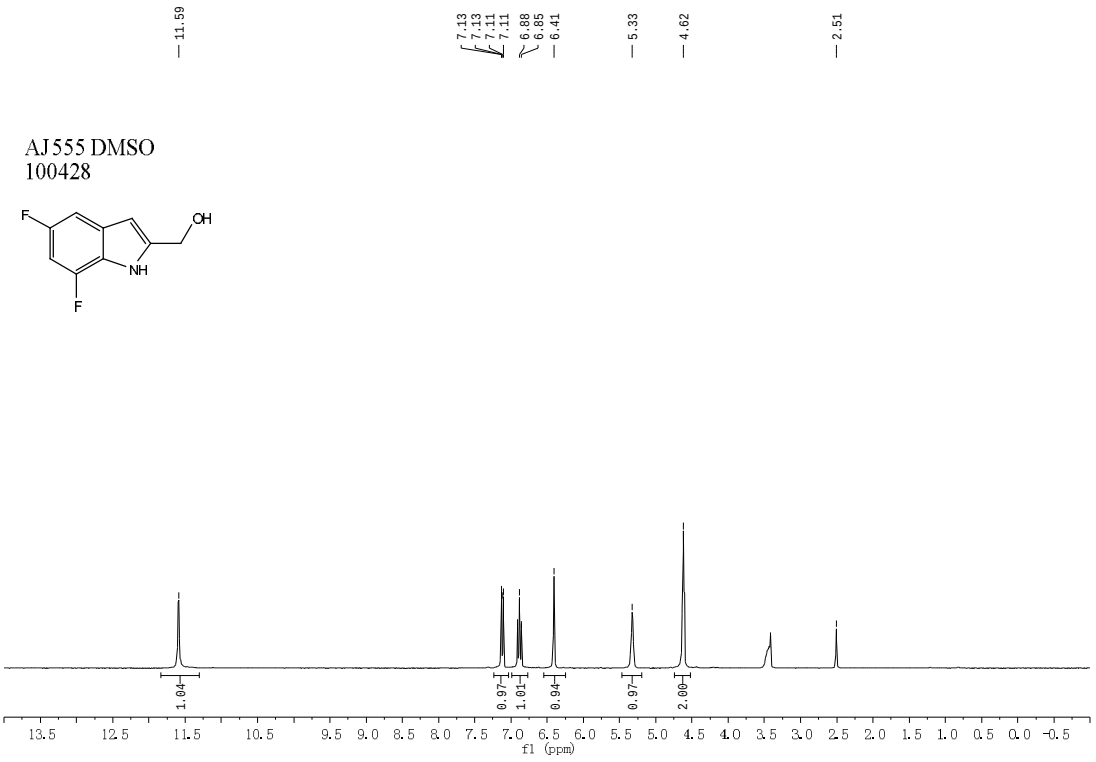
AJ494 DMSO
100428

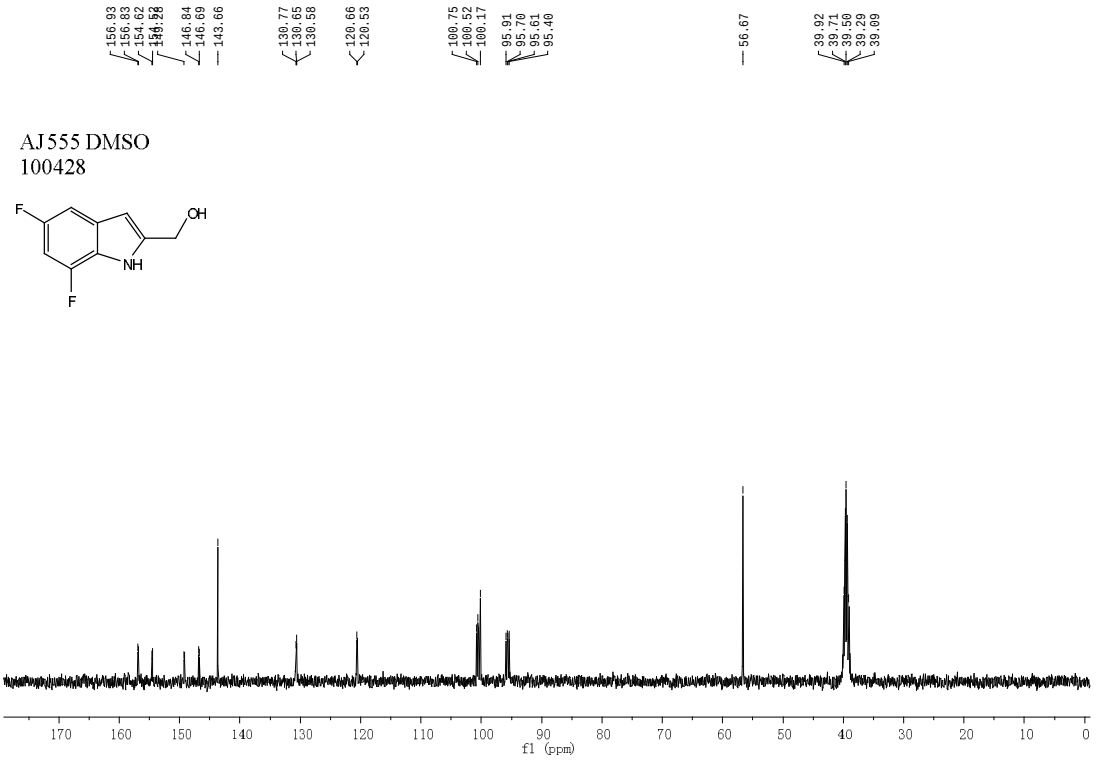


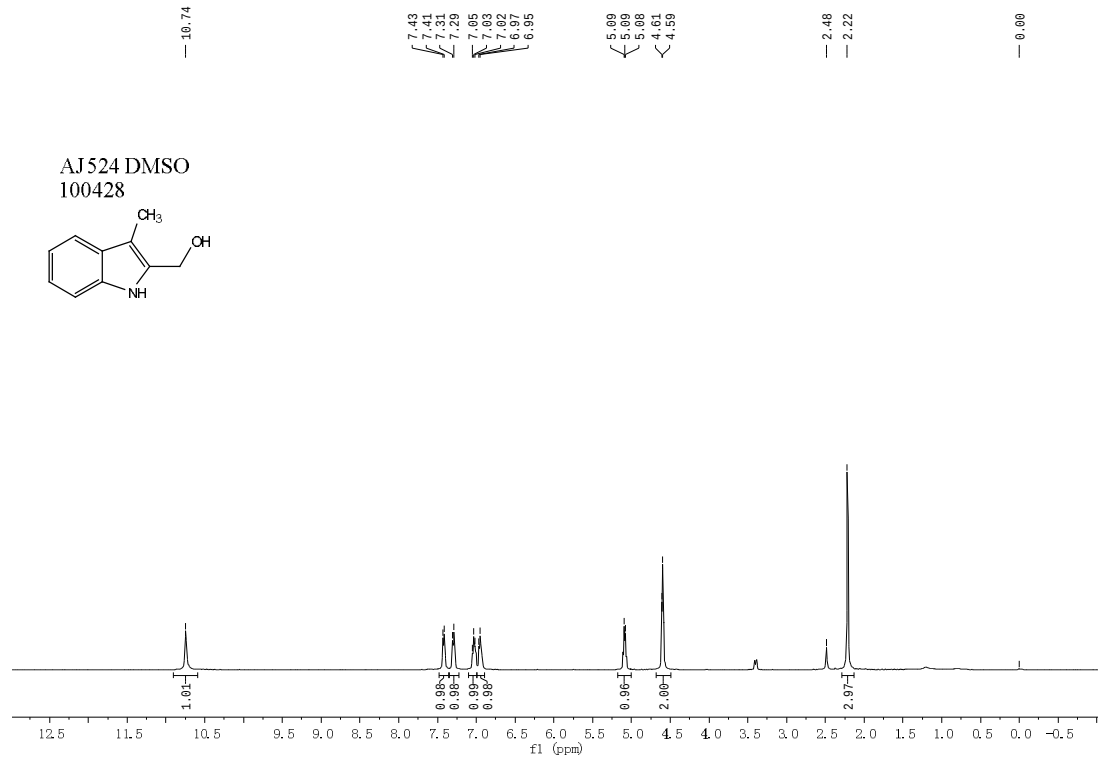


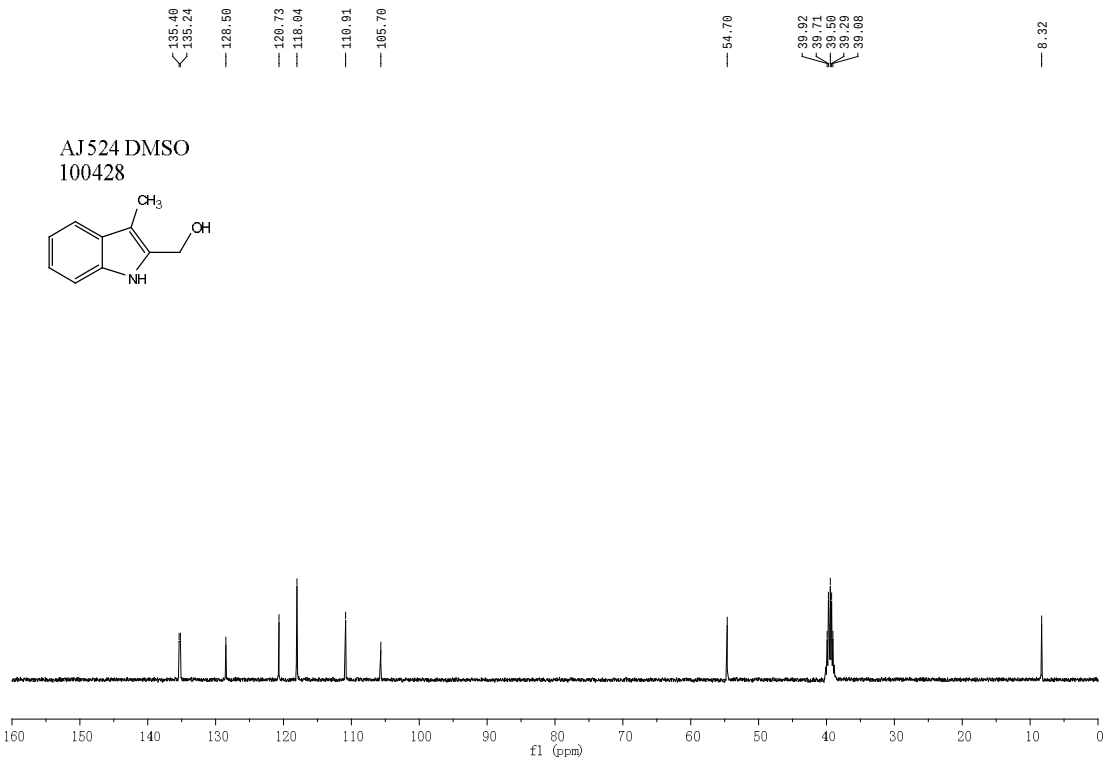

8.39
7.17
6.79
6.26
4.66
2.37
-0.00
AJ566 CDCB
091210
H3C
OH
NH
CH3
1.07
1.05
1.01
1.00
2.27
7.93
f1 (ppm)

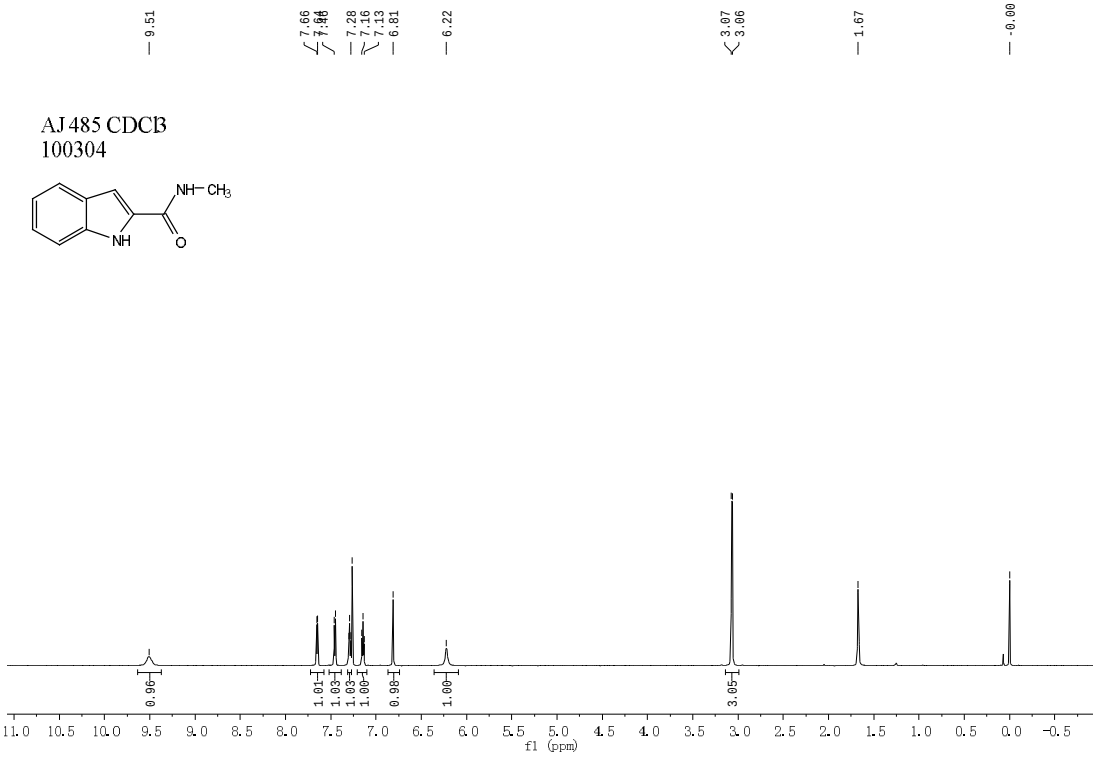

137.14
134.32
129.23
127.75
124.42
119.82
117.79
100.66
77.21
77.00
76.79
58.63
21.32
16.52
AJ566 CDCB
091210
H3C
OH
NH
CH3
f1 (ppm)

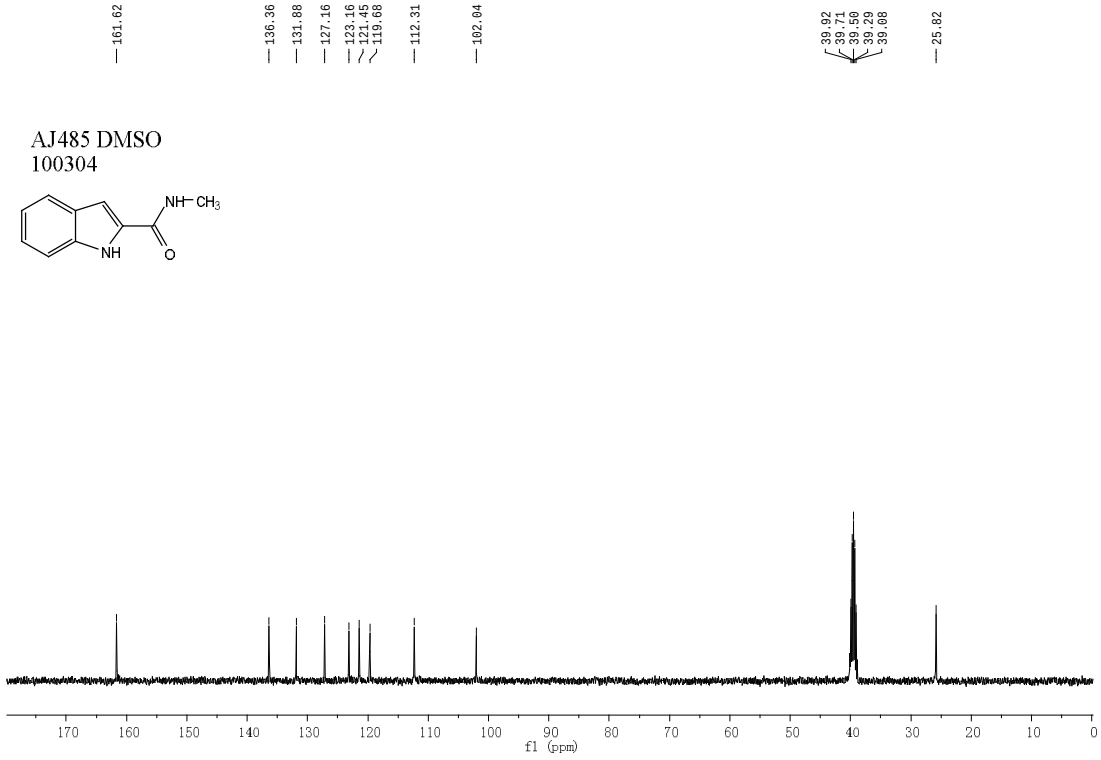

11.59
7.13
7.13
7.11
7.11
6.88
6.85
6.41
5.33
4.62
2.51
AJ555 DMSO
100428
F
OH
NH
F
1.04
0.97
1.01
0.94
0.97
2.00
f1 (ppm)


156.93
156.83
154.62
146.84
146.69
143.66
130.77
130.65
130.58
120.66
120.53
100.75
100.52
100.17
95.91
95.70
95.61
95.40
56.67
39.92
39.71
39.50
39.29
39.09
AJ555 DMSO
100428
F
OH
NH
F
f1 (ppm)


10.74
7.43
7.41
7.31
7.29
7.05
7.03
7.02
6.97
6.95
5.09
5.09
5.08
4.61
4.59
2.48
2.22
0.00
AJ524 DMSO
100428
CH3
OH
NH
1.01
0.98
0.98
0.99
0.98
0.96
2.00
2.97
f1 (ppm)


135.40
135.24
128.50
120.73
118.04
110.91
105.70
54.70
39.92
39.71
39.50
39.29
39.08
8.32
AJ524 DMSO
100428
CH3
OH
NH
f1 (ppm)


9.51
7.66
7.28
7.16
7.13
6.81
6.22
3.07
3.06
1.67
-0.00
AJ485 CDCl3
100304
NH-CH3
NH
O
0.96
1.01
1.03
1.03
1.00
0.98
1.00
3.05
f1 (ppm)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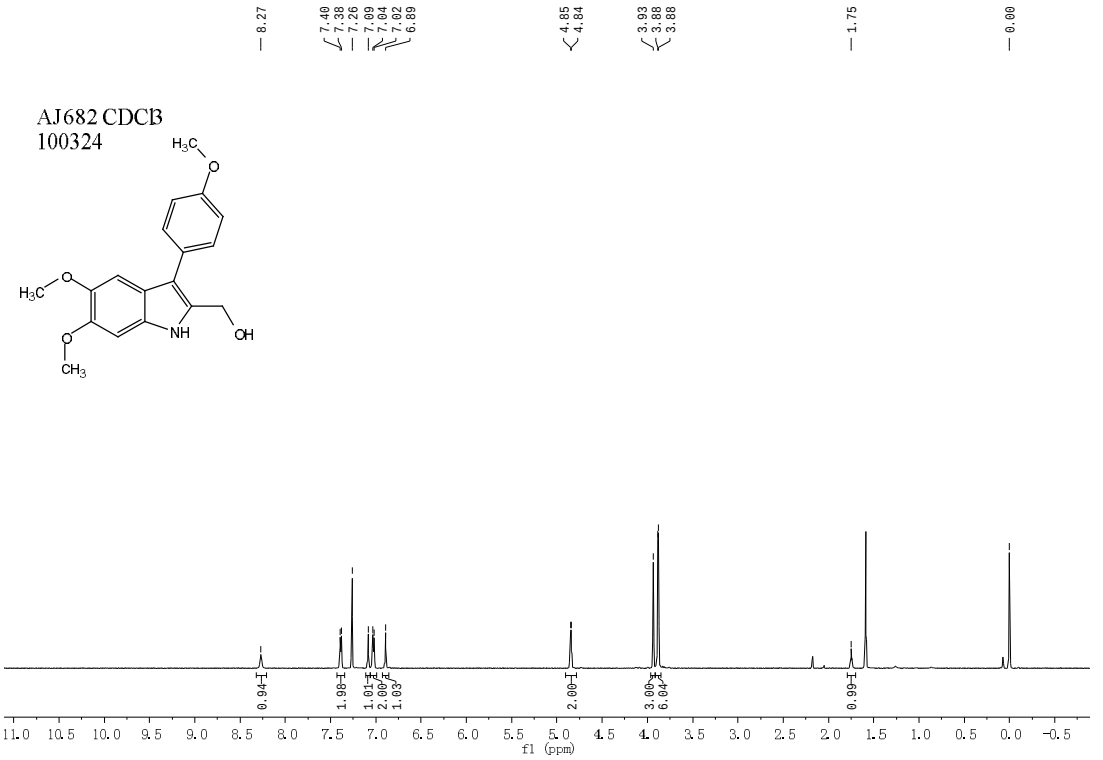
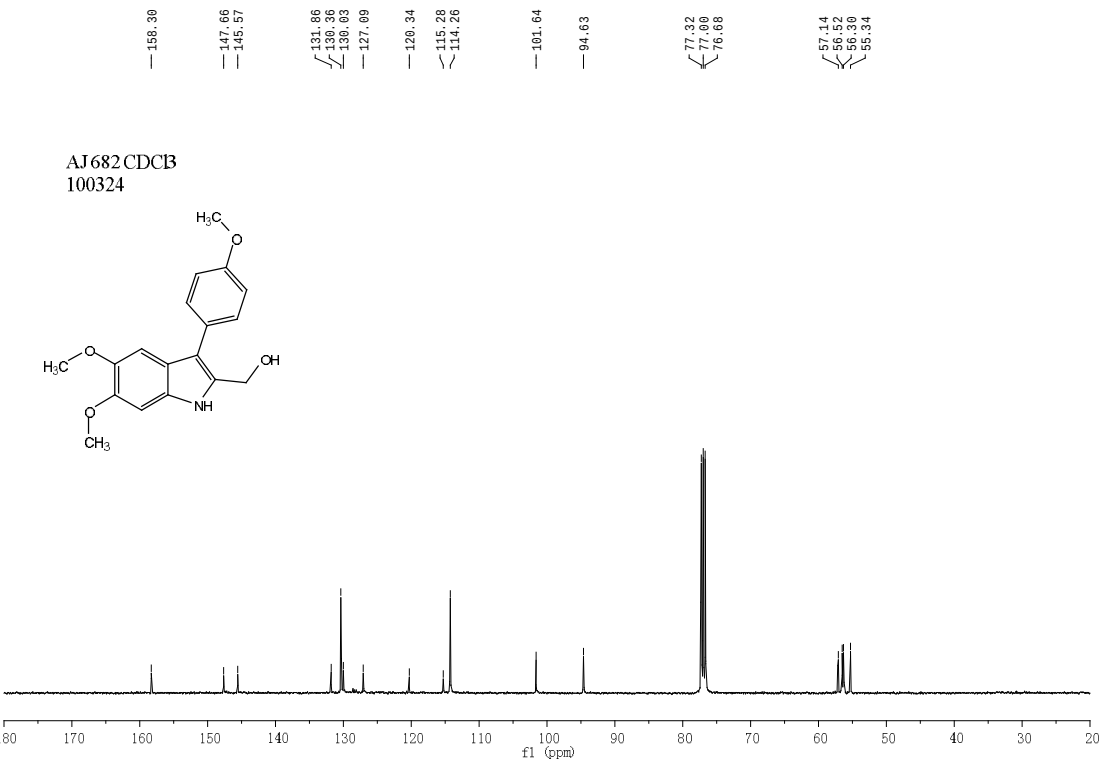
131.88
127.16
123.16
121.45
119.68
112.31
102.04
39.92
39.71
39.50
39.29
39.08
25.82
AJ485 DMSO
100304
NH-CH3
NH
O
f1 (ppm)


8.27
7.40
7.38
7.26
7.09
7.04
7.02
6.89
4.85
4.84
3.93
3.88
3.88
1.75
0.00
AJ682 CDCl3
100324
H3C
O
H3C
O
NH
OH
O
CH3
0.94
1.98
1.01
2.05
1.03
2.00
3.00
6.04
0.99
f1 (ppm)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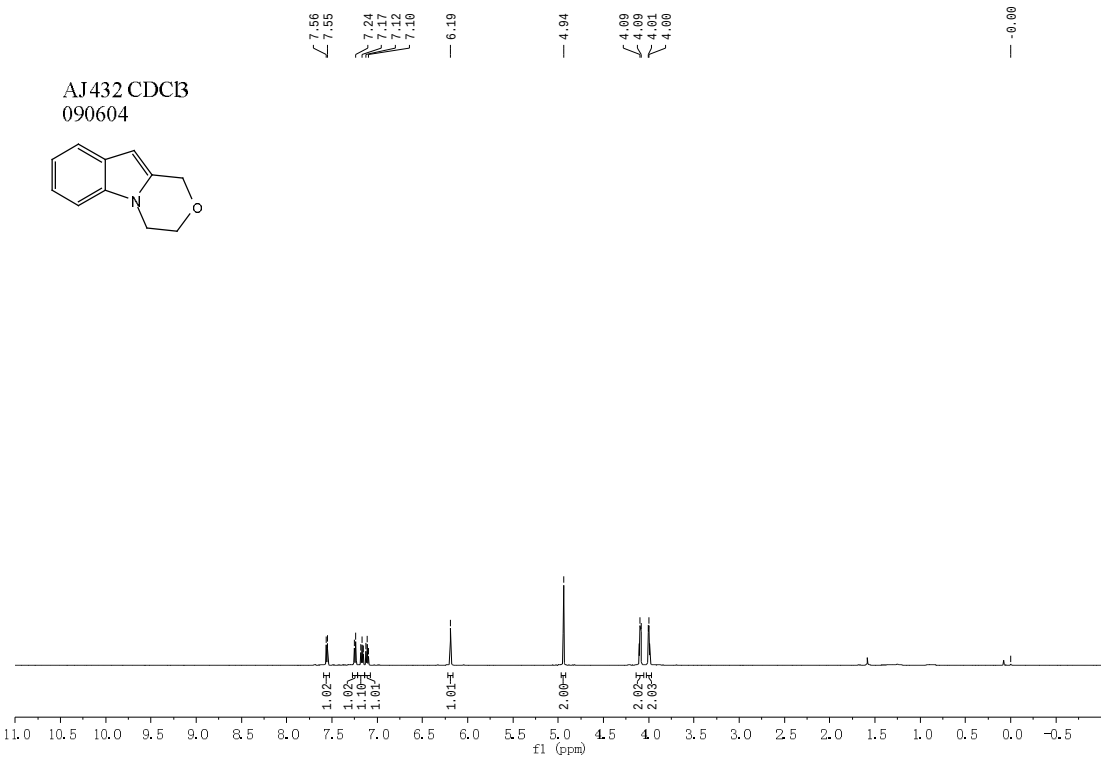
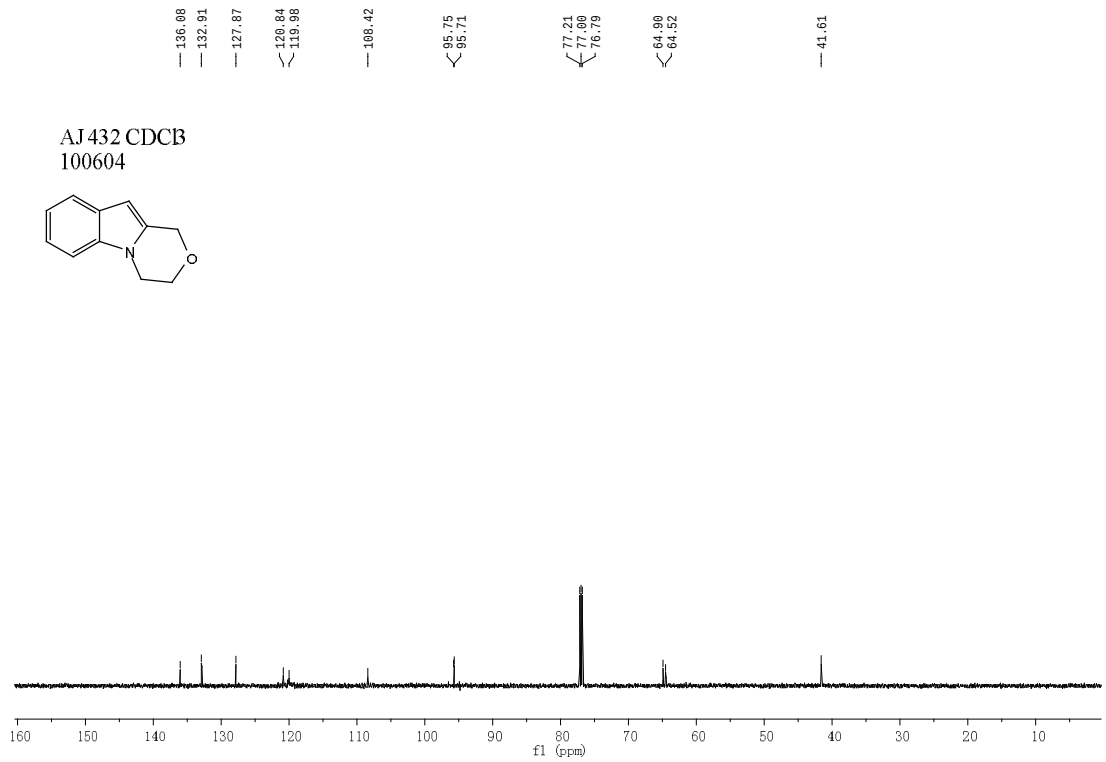
147.66
145.57
131.86
130.36
130.03
127.09
120.34
115.28
114.26
101.64
94.63
77.32
77.00
76.68
57.14
56.52
56.30
55.34
AJ682 CDCl3
100324
H3C
O
H3C
O
NH
OH
O
CH3
f1 (ppm)


7.56
7.55
7.24
7.17
7.12
7.10
6.19
4.94
4.09
4.09
4.01
4.00
0.00
AJ432 CDCl3
090604
1.02
1.02
1.10
1.01
1.01
2.00
2.02
2.03
f1 (ppm)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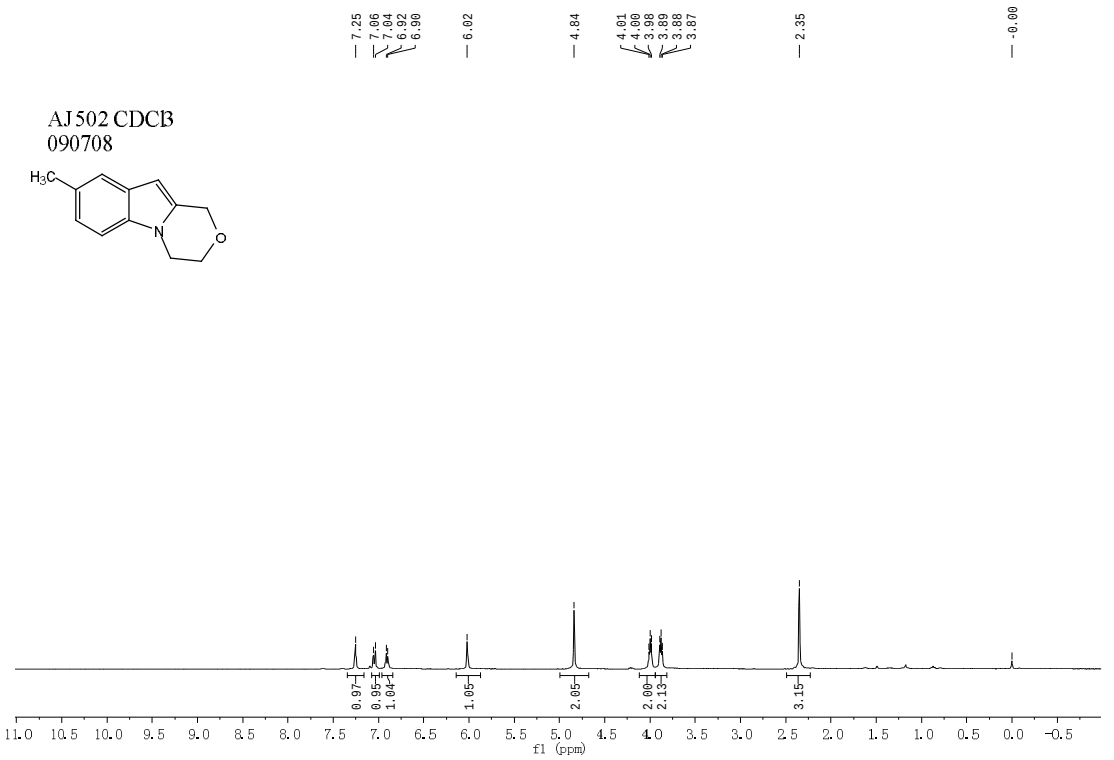
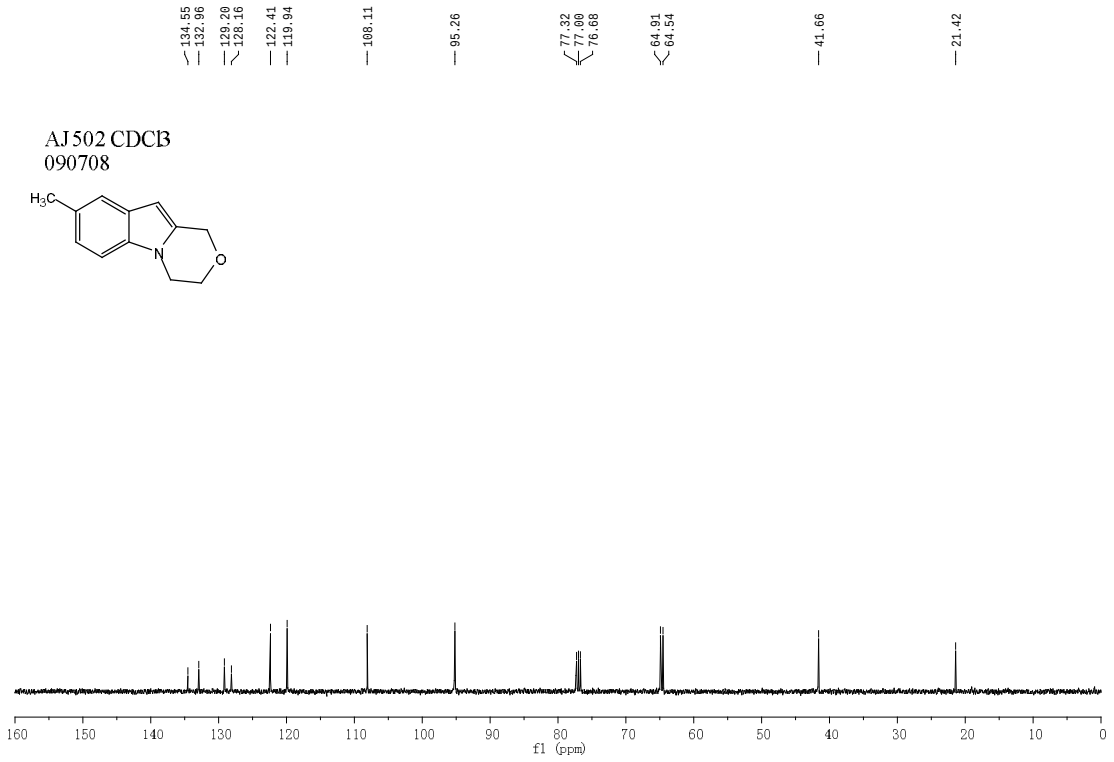
132.91
127.87
120.84
119.98
108.42
95.75
95.71
77.21
77.00
76.79
64.90
64.52
41.61
AJ432 CDCl3
100604
f1 (ppm)


7.25
7.06
7.04
6.92
6.90
6.02
4.84
4.01
4.00
3.98
3.89
3.88
3.87
2.35
0.00
AJ502 CDCl3
090708
H3C
N
O
0.97
0.95
1.04
1.05
2.05
2.00
2.13
3.15
f1 (ppm)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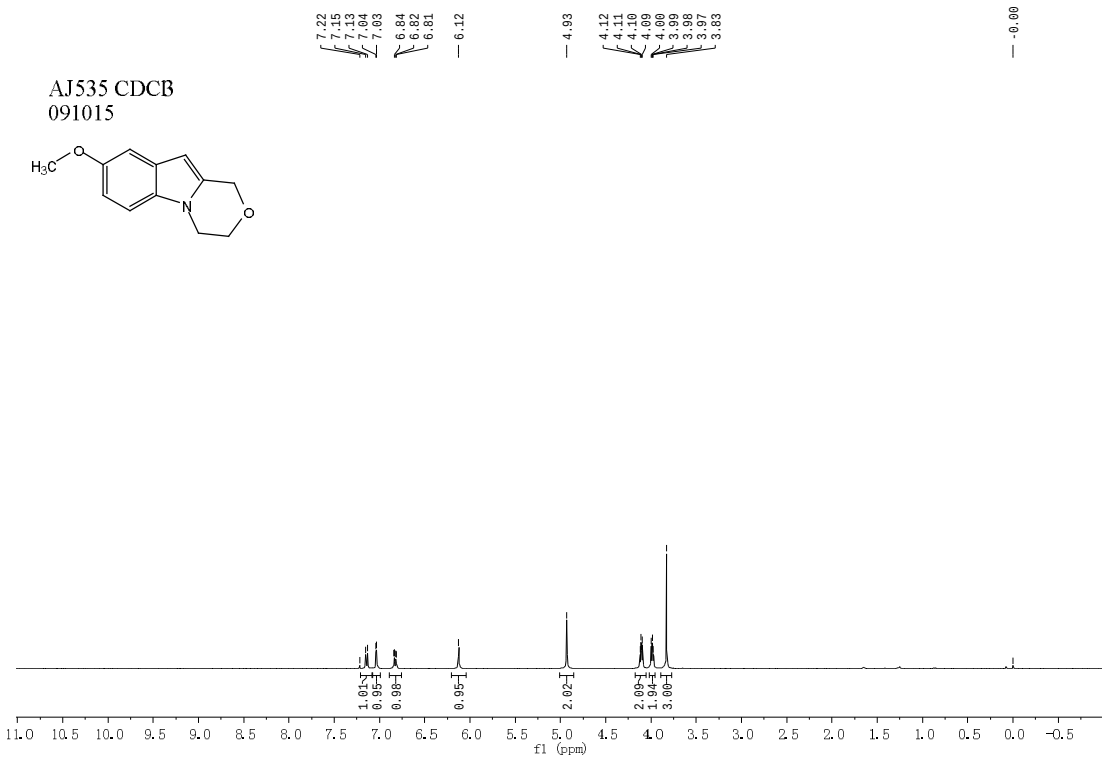
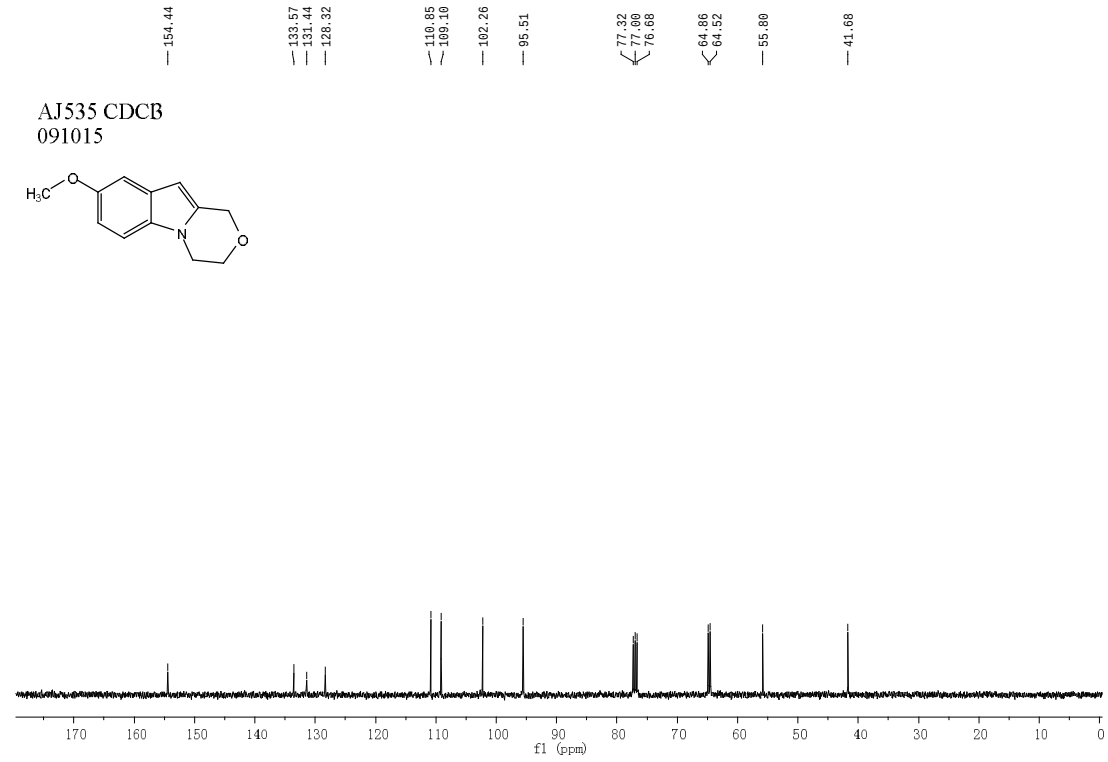
129.20
128.16
122.41
119.94
108.11
95.26
77.32
77.00
76.68
64.91
64.54
41.66
21.42
AJ502 CDCl3
090708
H3C
N
O
f1 (ppm)


7.22
7.15
7.13
7.04
7.03
6.84
6.82
6.81
6.12
4.93
4.12
4.11
4.10
4.00
4.00
3.99
3.98
3.97
3.83
0.00
AJ535 CDCB
091015
H3C
O
N
O
1.01
0.95
0.98
0.95
2.02
2.09
1.94
3.00
f1 (ppm)


154.44
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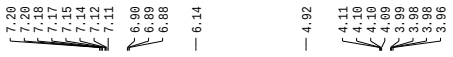
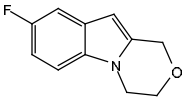
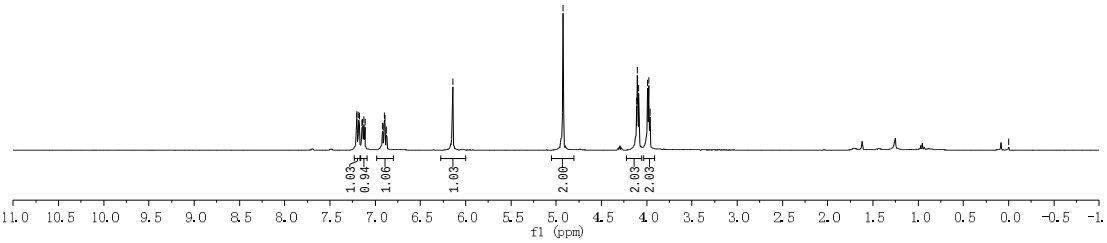
131.44
128.32
110.85
109.10
102.26
95.51
77.32
77.00
76.68
64.86
64.52
55.80
41.68
AJ535 CDCB
091015
H3C
O
N
O
f1 (ppm)

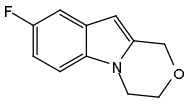


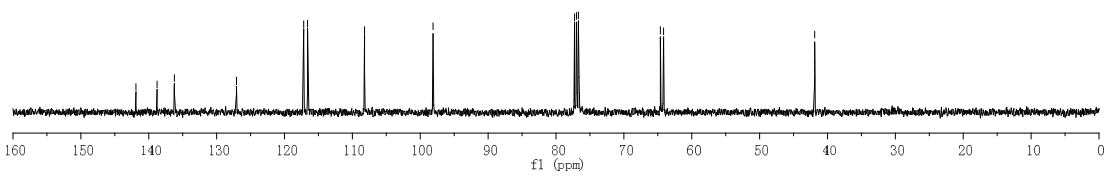
AJ507 CDCB
090708

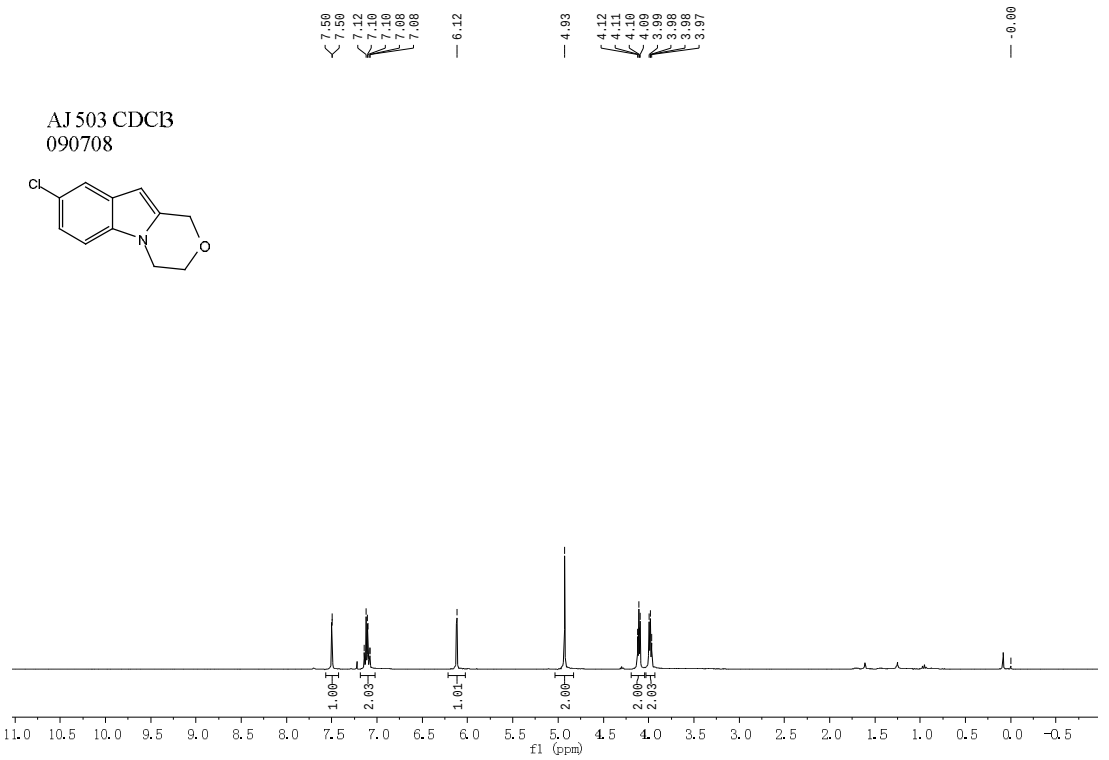


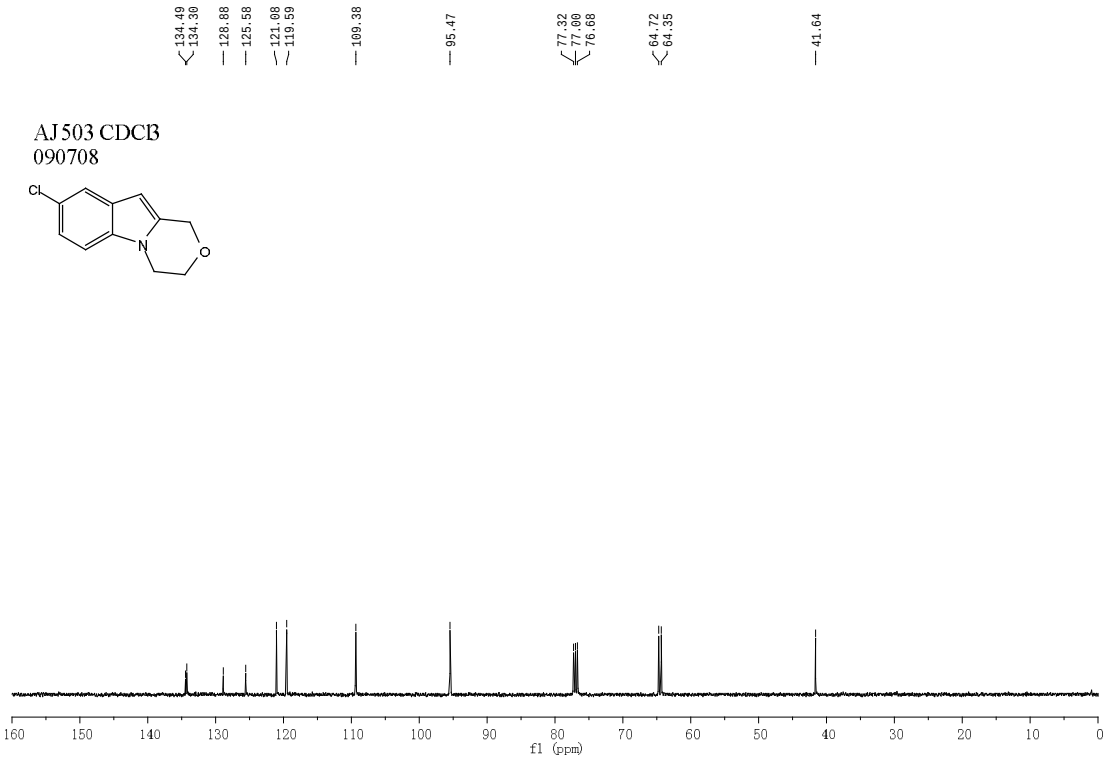


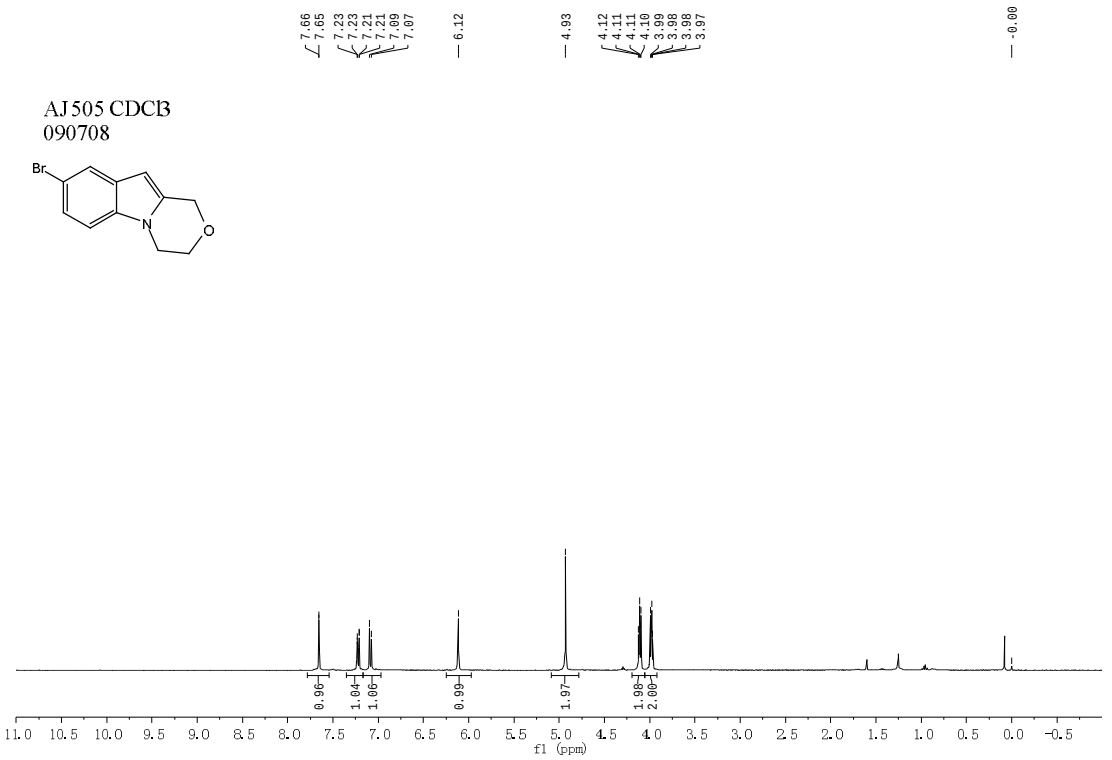
AJ507 CDCB
090708

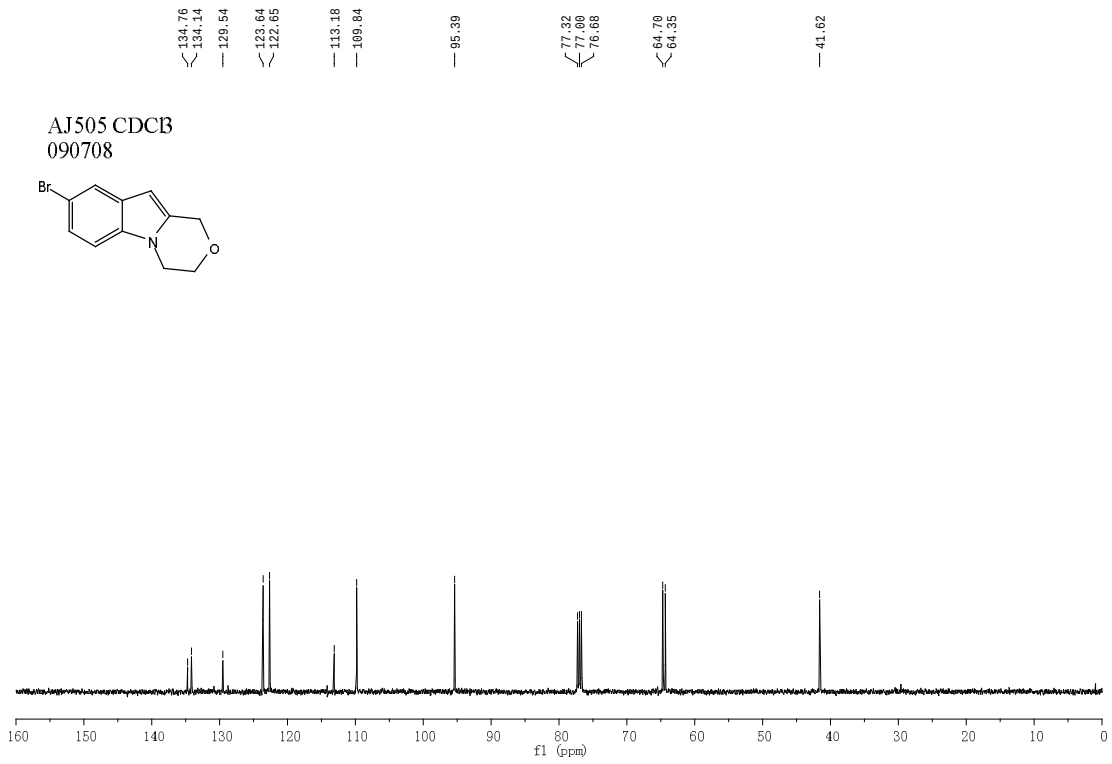


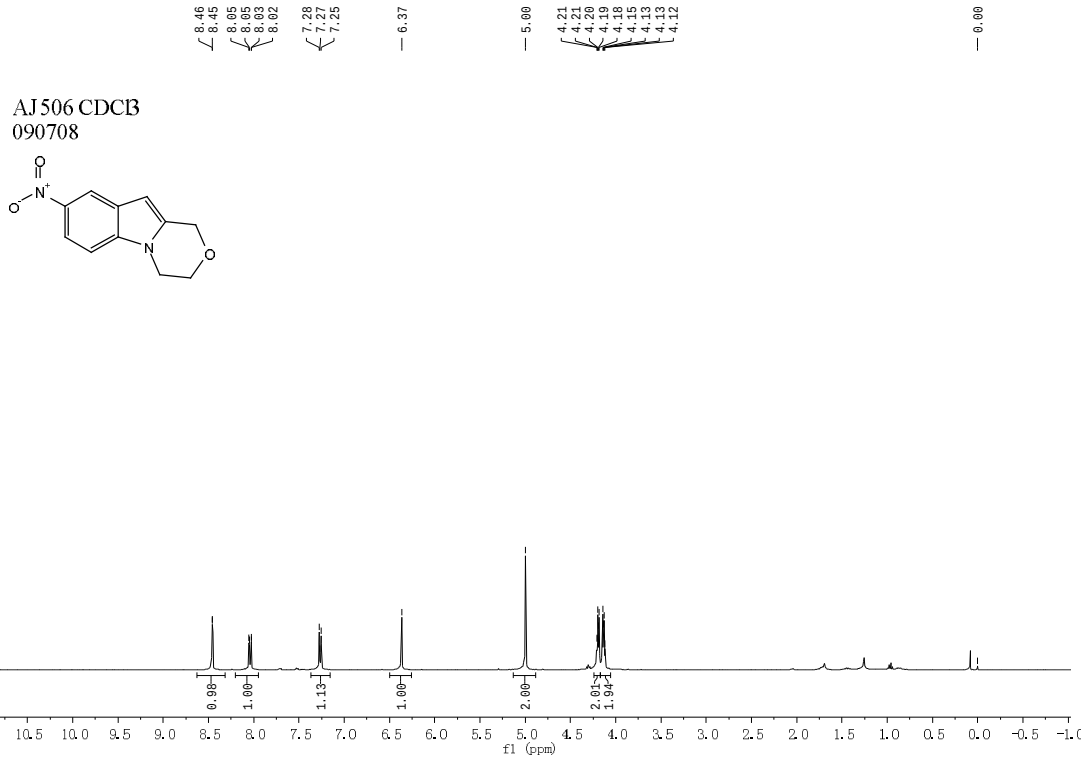


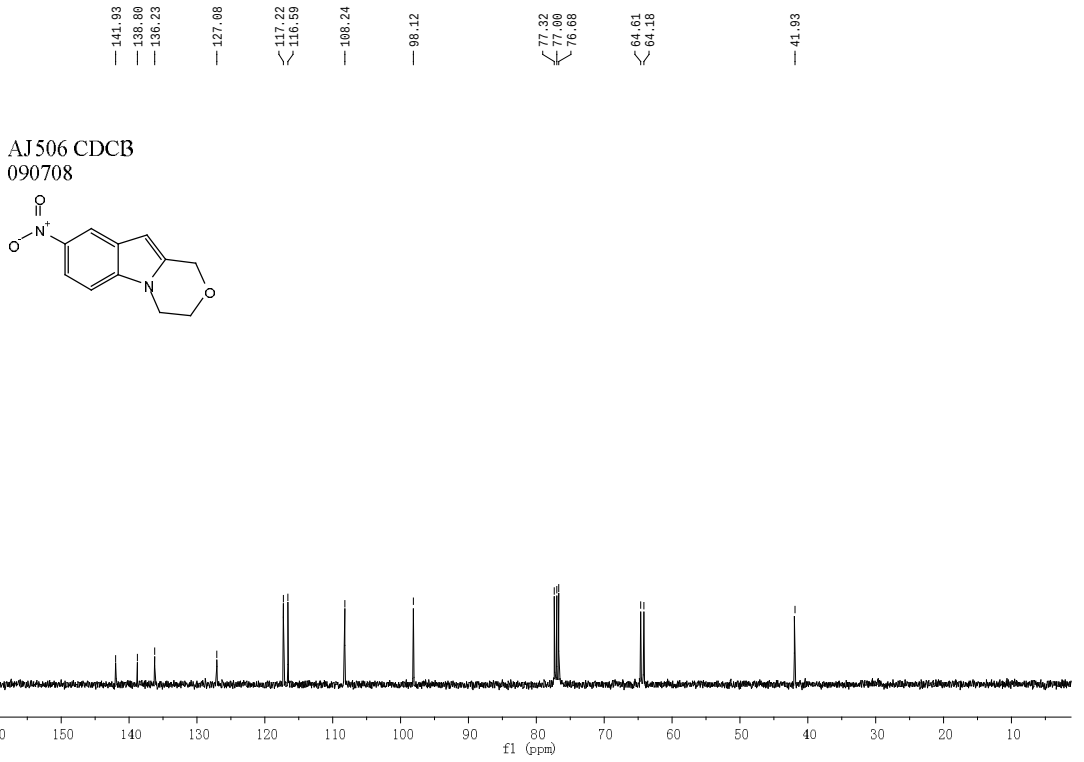

7.50
7.50
7.12
7.10
7.10
7.08
7.08
6.12
4.93
4.12
4.11
4.10
4.09
3.99
3.98
3.98
3.97
0.00
AJ 503 CDCB
090708
Cl
N
O
1.00
2.03
1.01
2.00
2.00
2.03
f1 (ppm)

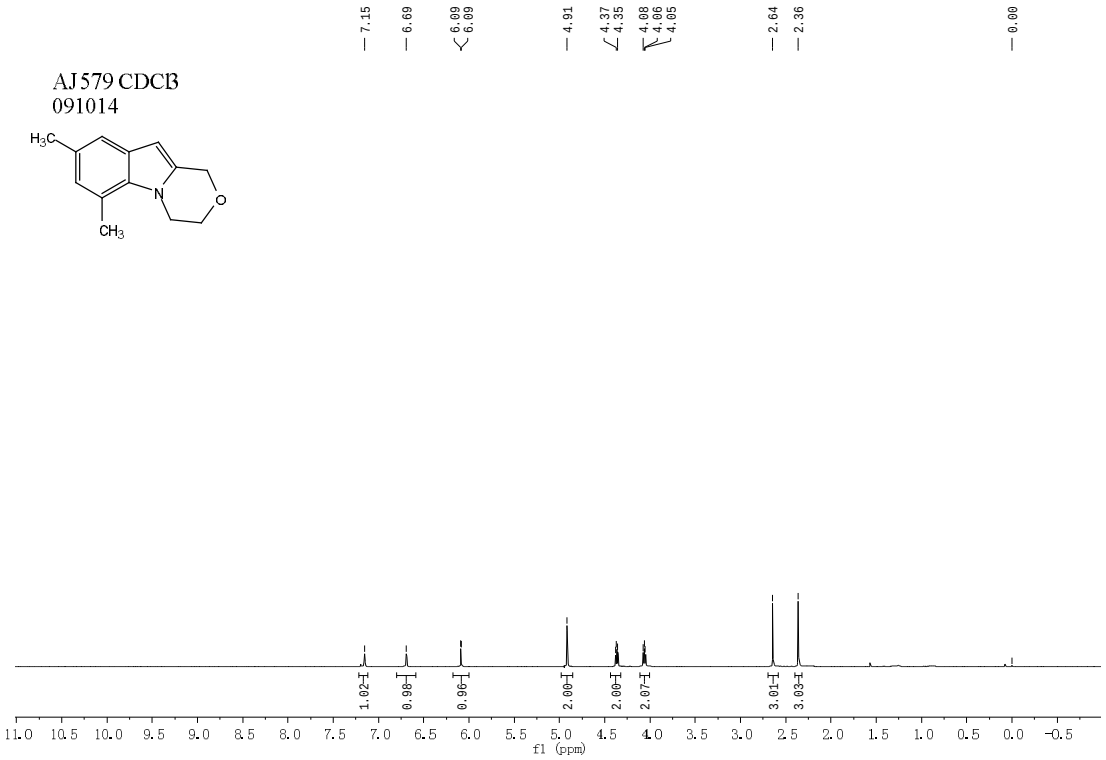

134.49
134.30
128.88
125.58
121.08
119.59
109.38
95.47
77.32
77.00
76.68
64.72
64.35
41.64
AJ 503 CDCB
090708
Cl
N
O
f1 (ppm)

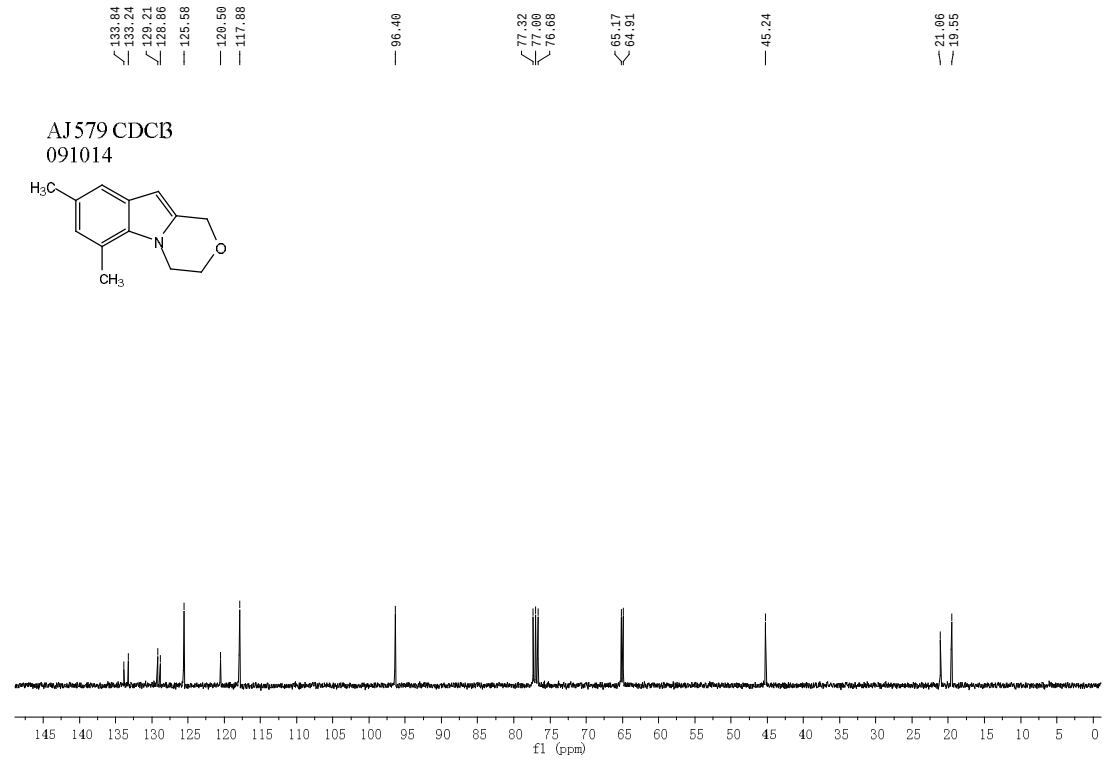

7.66
7.65
7.23
7.23
7.21
7.21
7.09
7.07
6.12
4.93
4.12
4.11
4.11
4.10
3.99
3.98
3.98
3.97
0.00
AJ505 CDCB
090708
Br
N
O
0.96
1.04
1.06
0.99
1.97
1.98
2.00
f1 (ppm)

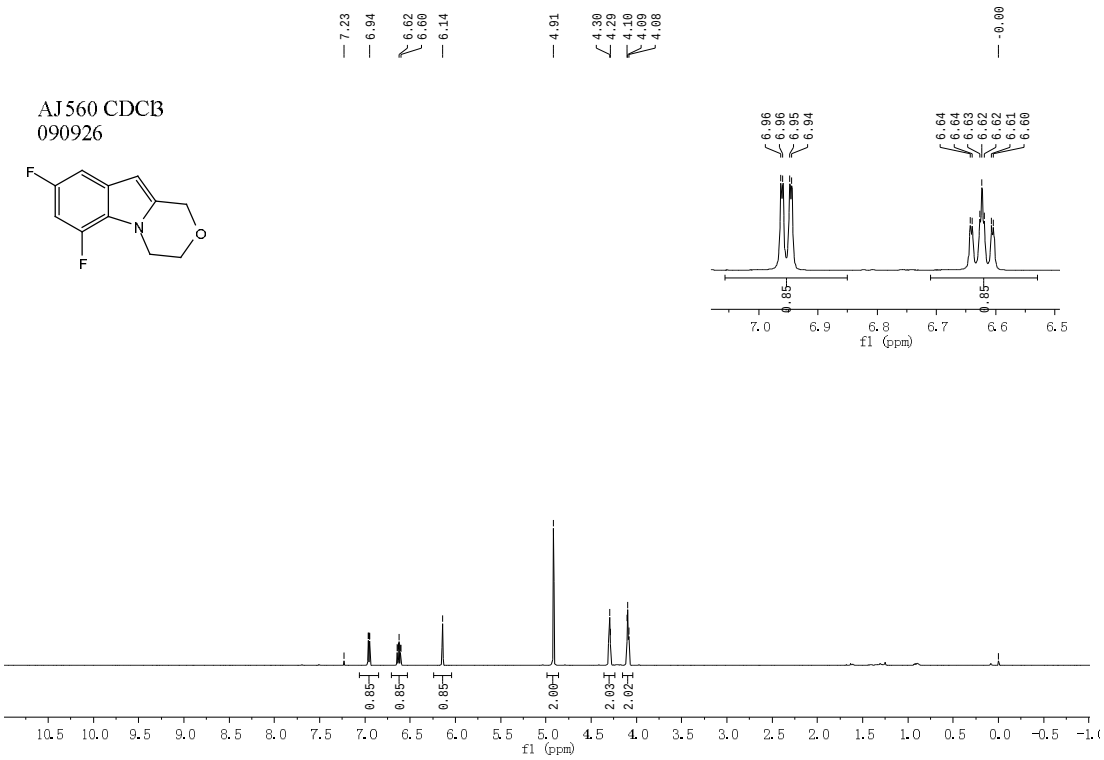

134.76
134.14
129.54
123.64
122.65
113.18
109.84
95.39
77.32
77.00
76.68
64.70
64.35
41.62
AJ505 CDCB
090708
Br
N
O
f1 (ppm)

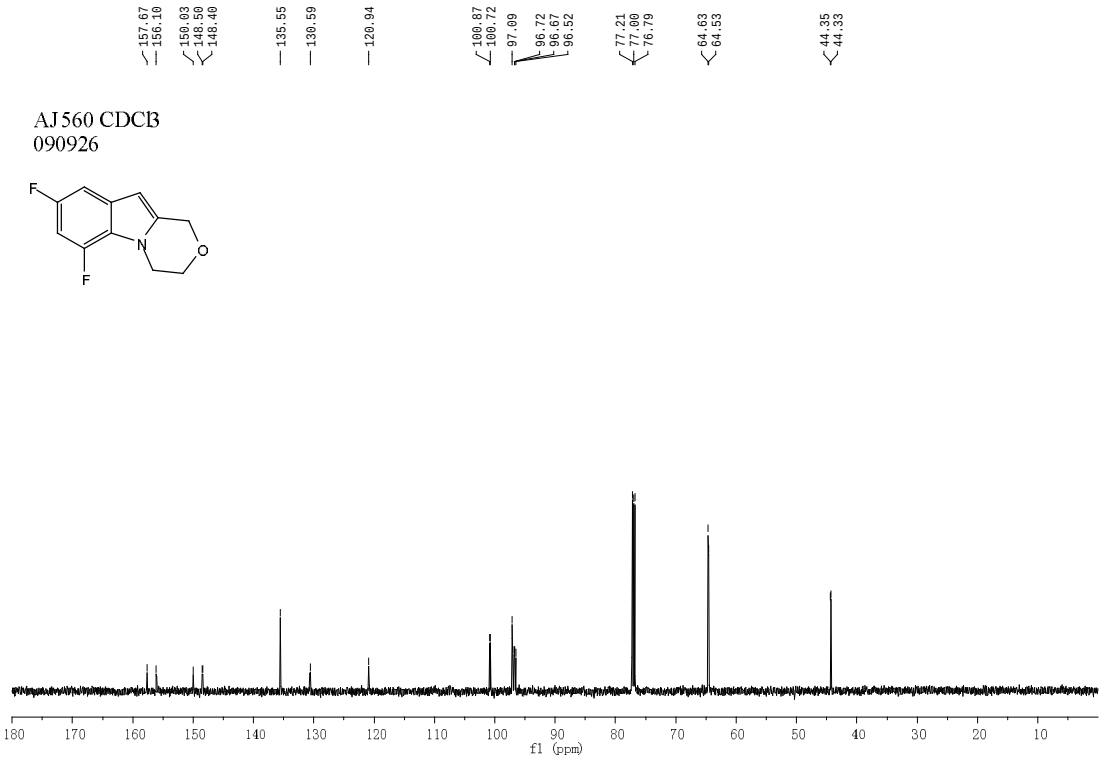

8.46
8.45
8.05
8.05
8.03
8.02
7.28
7.27
7.25
6.37
5.00
4.21
4.21
4.20
4.19
4.18
4.15
4.13
4.13
4.12
0.00
AJ506 CDCl3
090708
0.98
1.00
1.13
1.00
2.00
2.01
1.94
f1 (ppm)

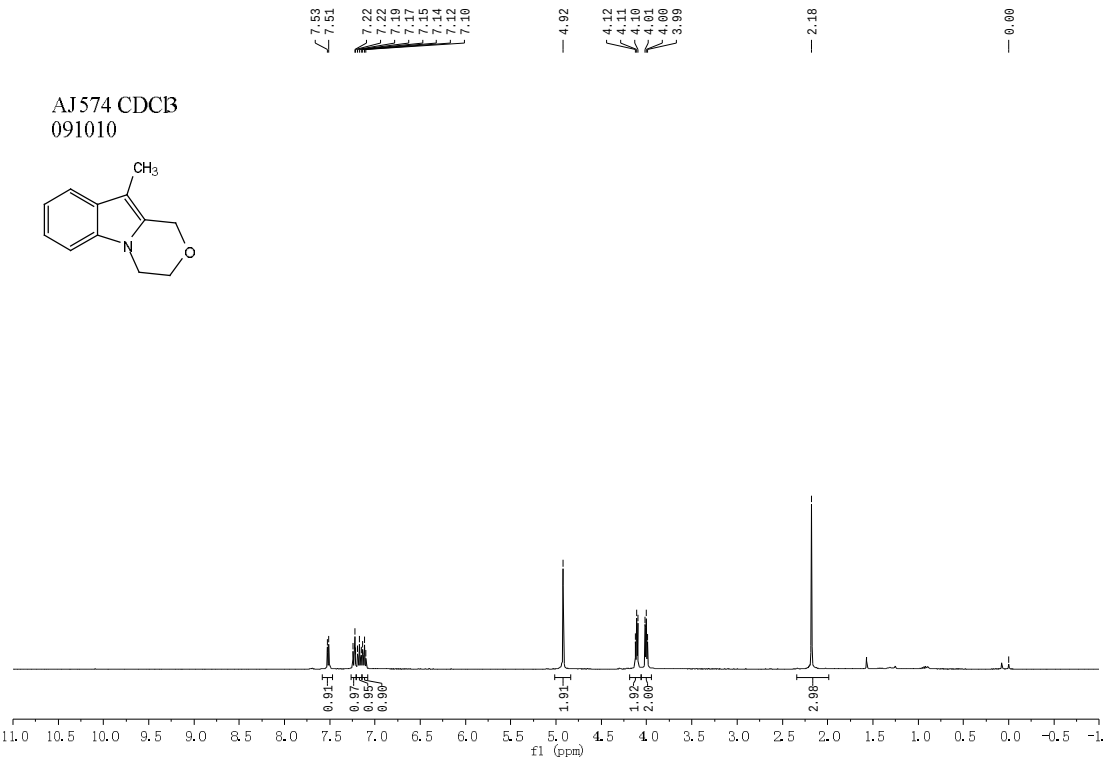

141.93
138.86
136.23
127.08
117.22
116.59
108.24
98.12
77.32
77.00
76.68
64.61
64.18
41.93
AJ506 CDCl3
090708
f1 (ppm)

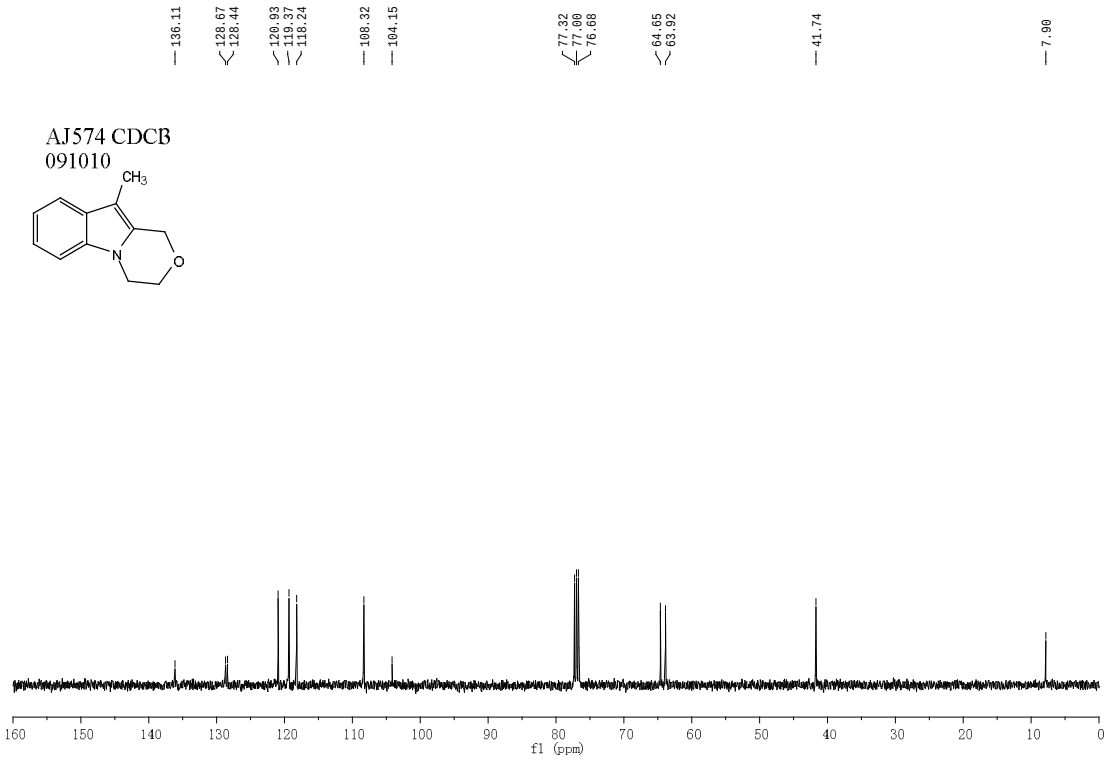

7.15
6.69
6.09
6.09
4.91
4.37
4.35
4.08
4.06
4.05
2.64
2.36
0.00
AJ579 CDCB
091014
H3C
N
O
CH3
1.02
0.98
0.96
2.00
2.00
2.07
3.01
3.03
f1 (ppm)

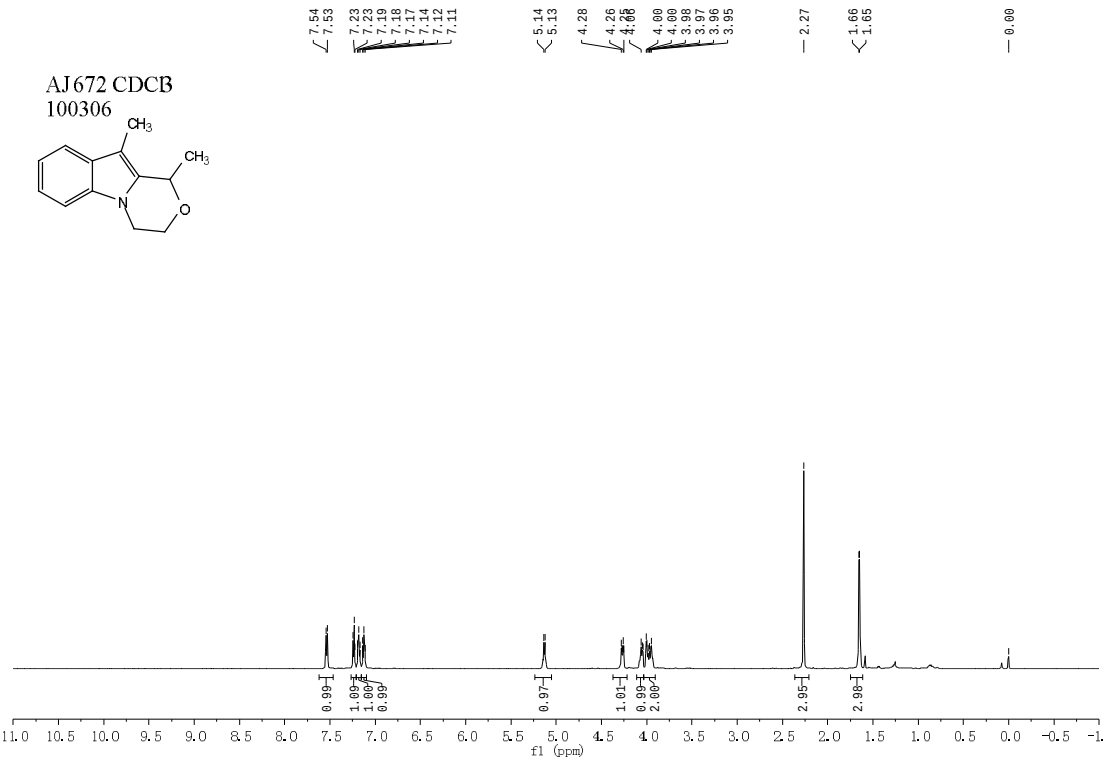

133.84
133.24
129.21
128.86
125.58
120.50
117.88
96.40
77.32
77.00
76.68
65.17
64.91
45.24
21.06
19.55
AJ579 CDCB
091014
H3C
N
O
CH3
f1 (ppm)

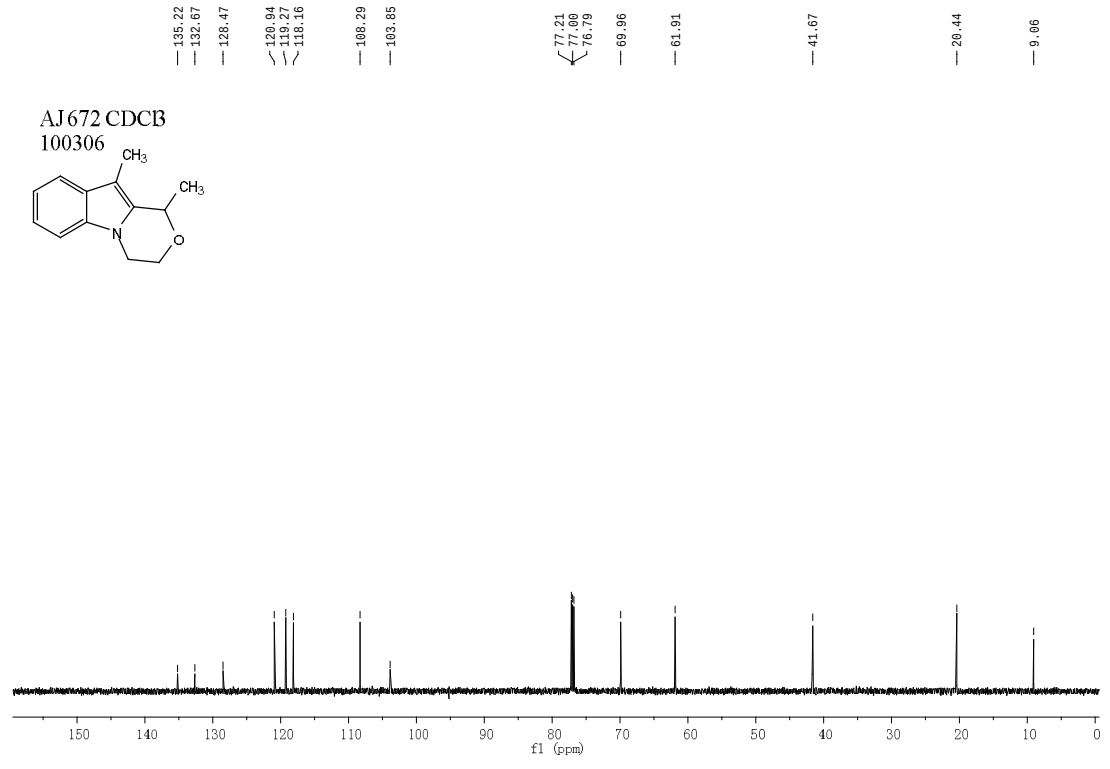

AJ560 CDCB
090926
F
N
O
F
7.23
6.94
6.62
6.60
6.14
4.91
4.30
4.29
4.10
4.09
4.08
0.00
6.96
6.96
6.95
6.94
6.64
6.64
6.63
6.62
6.62
6.61
6.60
0.85
0.85
7.0
6.9
6.8
6.7
6.6
6.5
f1 (ppm)
0.85
0.85
0.85
2.00
2.03
2.02
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

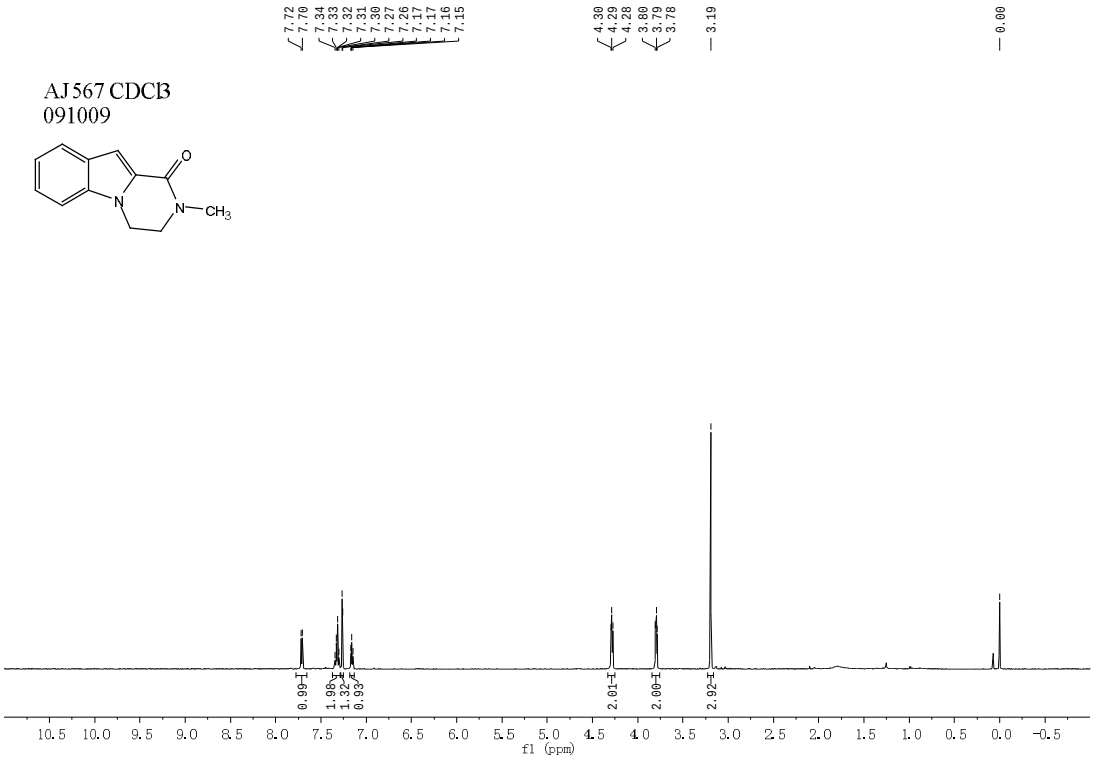

157.67
156.10
150.03
148.50
148.40
135.55
130.59
120.94
100.87
100.72
97.09
96.72
96.67
96.52
77.21
77.00
76.79
64.63
64.53
44.35
44.33
AJ560 CDCB
090926
F
N
O
F
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

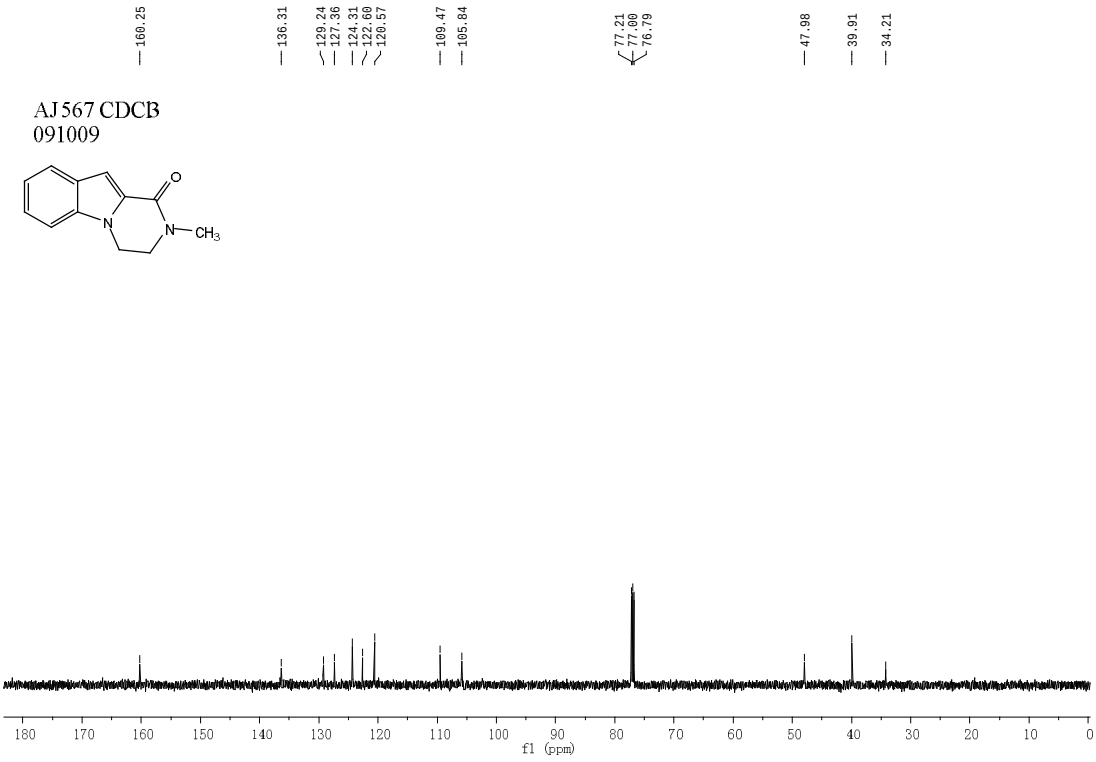

7.53
7.51
7.22
7.22
7.19
7.17
7.15
7.14
7.12
7.10
4.92
4.12
4.11
4.10
4.01
4.00
3.99
2.18
0.00
AJ574 CDCB
091010
CH3
N
O
0.91
0.97
0.95
0.90
1.91
1.92
2.00
2.98
f1 (ppm)

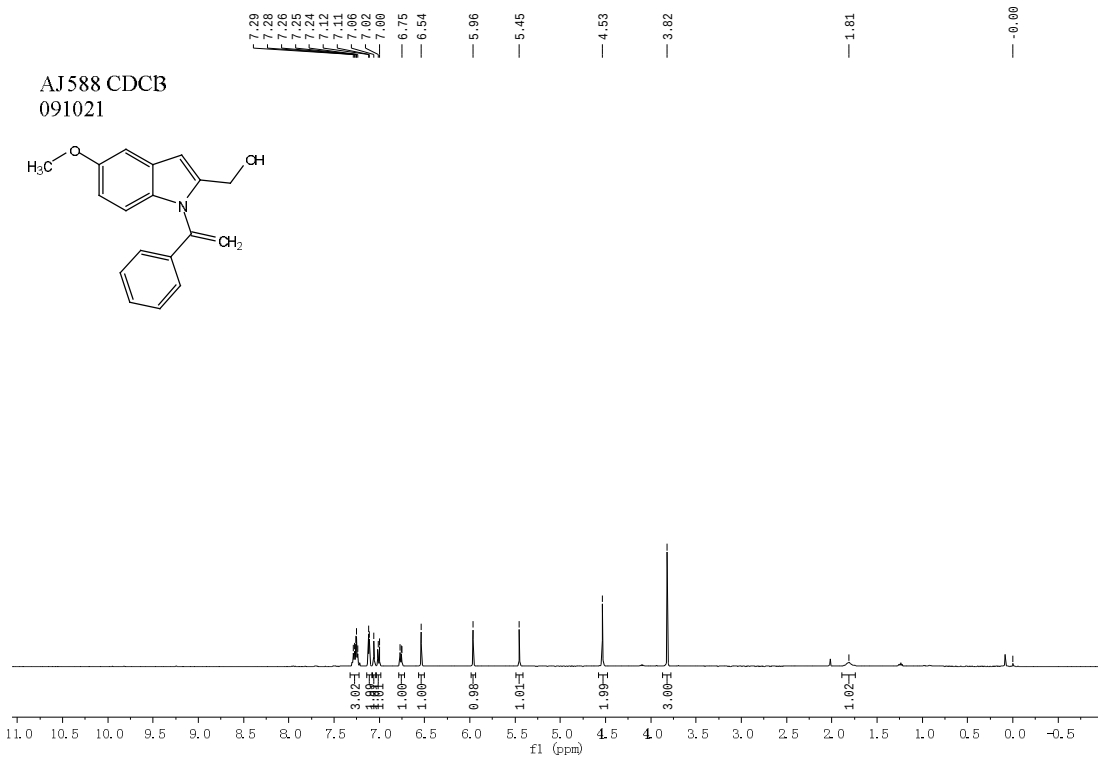

136.11
128.67
128.44
120.93
119.37
118.24
108.32
104.15
77.32
77.00
76.68
64.65
63.92
41.74
7.90
AJ574 CDCB
091010
CH3
N
O
f1 (ppm)

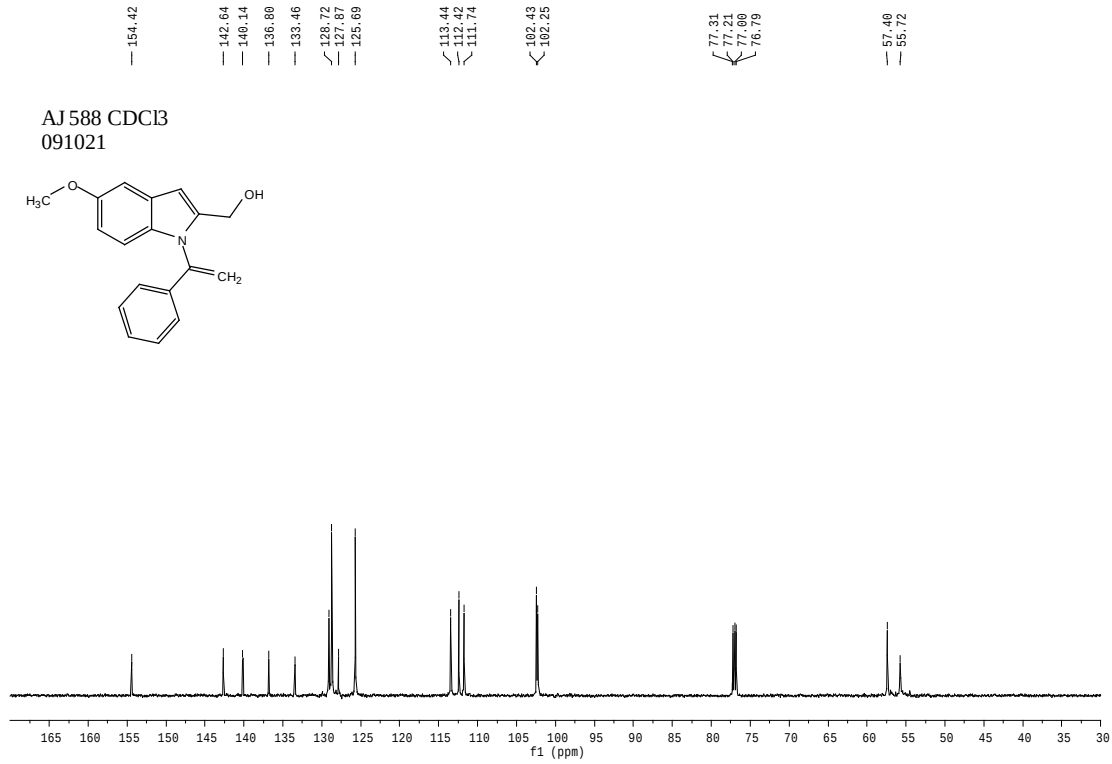

7.54
7.53
7.23
7.23
7.19
7.18
7.17
7.14
7.12
7.11
5.14
5.13
4.28
4.26
4.00
4.00
3.98
3.97
3.96
3.95
2.27
1.66
1.65
0.00
AJ672 CDCB
100306
CH3
CH3
N
O
0.99
1.09
1.00
0.99
0.97
1.01
0.99
2.00
2.95
2.98
f1 (ppm)

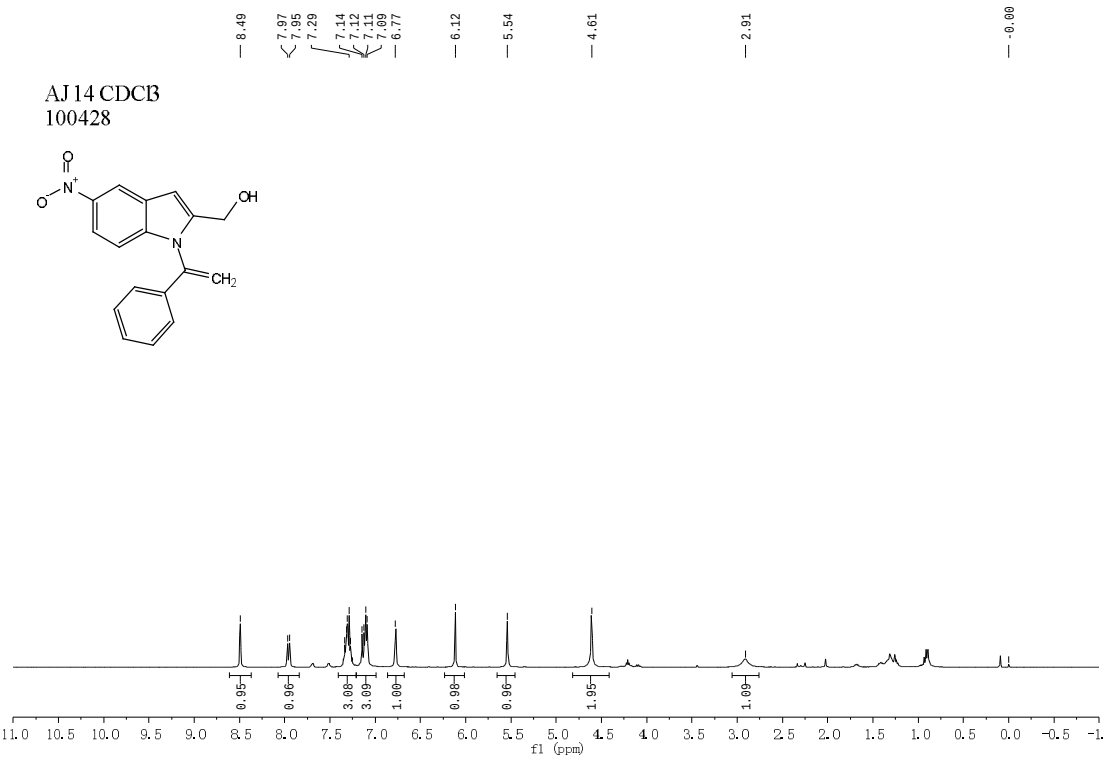

135.22
132.67
128.47
120.94
119.27
118.16
108.29
103.85
77.21
77.00
76.79
69.96
61.91
41.67
20.44
9.06
AJ672 CDCB
100306
CH3
CH3
N
O
f1 (ppm)

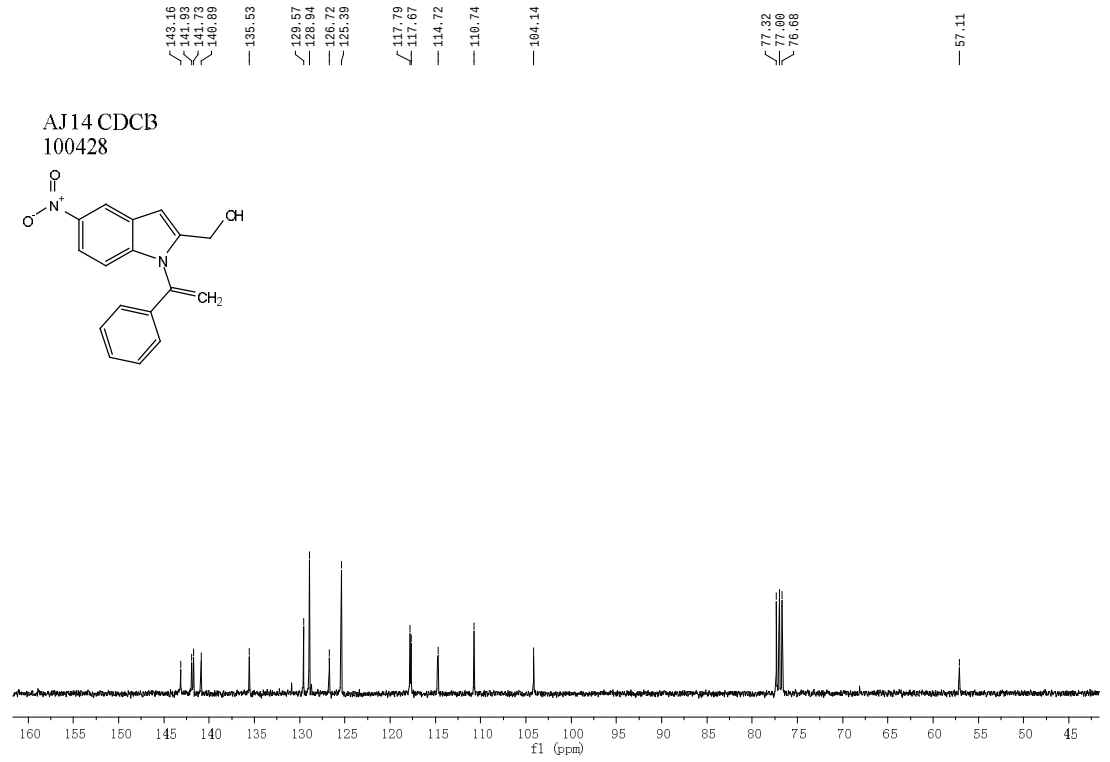

7.72
7.70
7.34
7.33
7.32
7.31
7.30
7.27
7.26
7.17
7.16
7.15
4.30
4.29
4.28
3.80
3.79
3.78
3.19
0.00
AJ567 CDCB
091009
O
N
N
CH3
0.99
1.96
1.32
0.93
2.01
2.00
2.92
f1 (ppm)

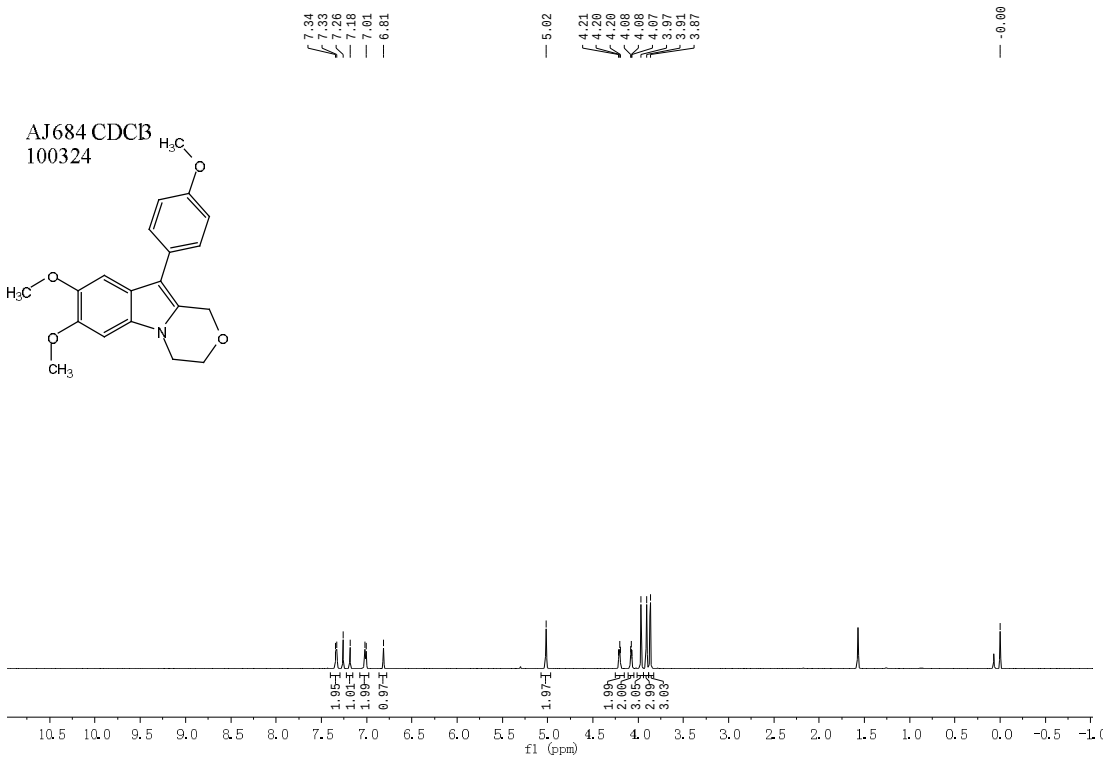

160.25
136.31
129.24
127.36
124.31
122.60
120.57
109.47
105.84
77.21
77.00
76.79
47.98
39.91
34.21
AJ567 CDCB
091009
O
N
N
CH3
f1 (ppm)

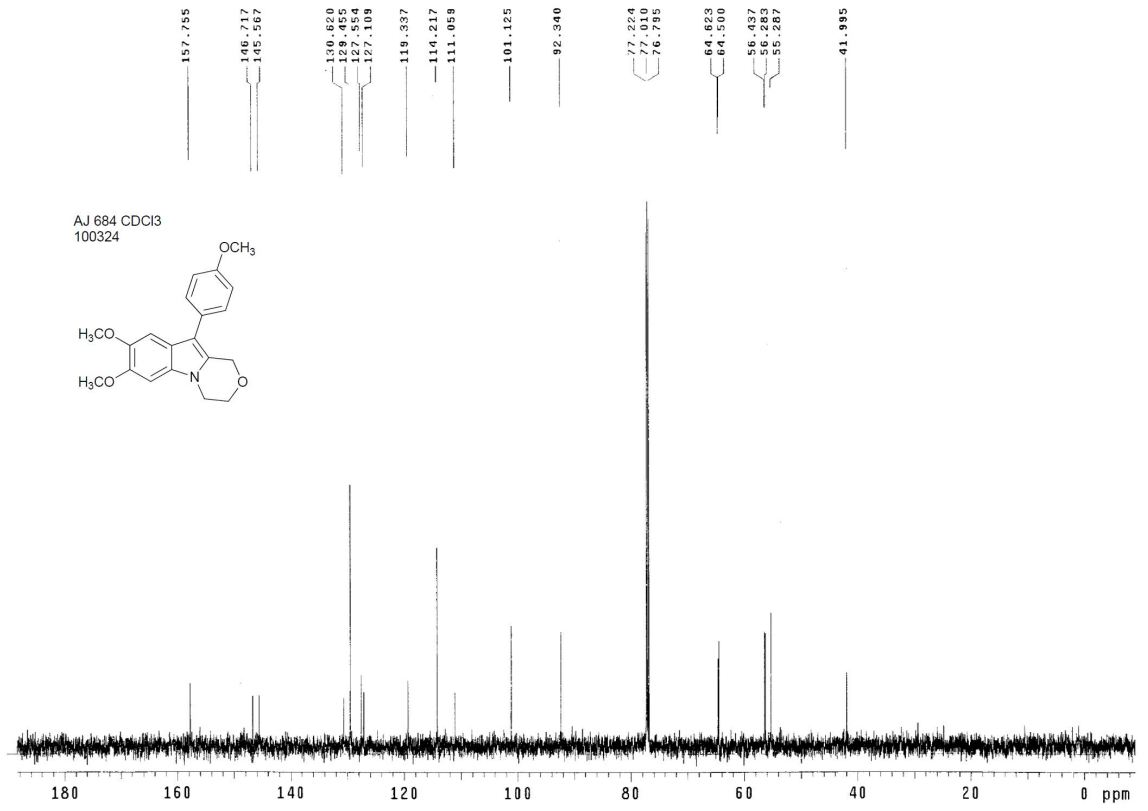

7.29
7.28
7.26
7.25
7.24
7.12
7.11
7.06
7.02
7.00
6.75
6.54
5.96
5.45
4.53
3.82
1.81
-0.00
AJ588 CDCB
091021
H3C
O
OH
N
CH2
3.02
1.00
1.00
0.98
1.01
1.99
3.00
1.02
f1 (ppm)

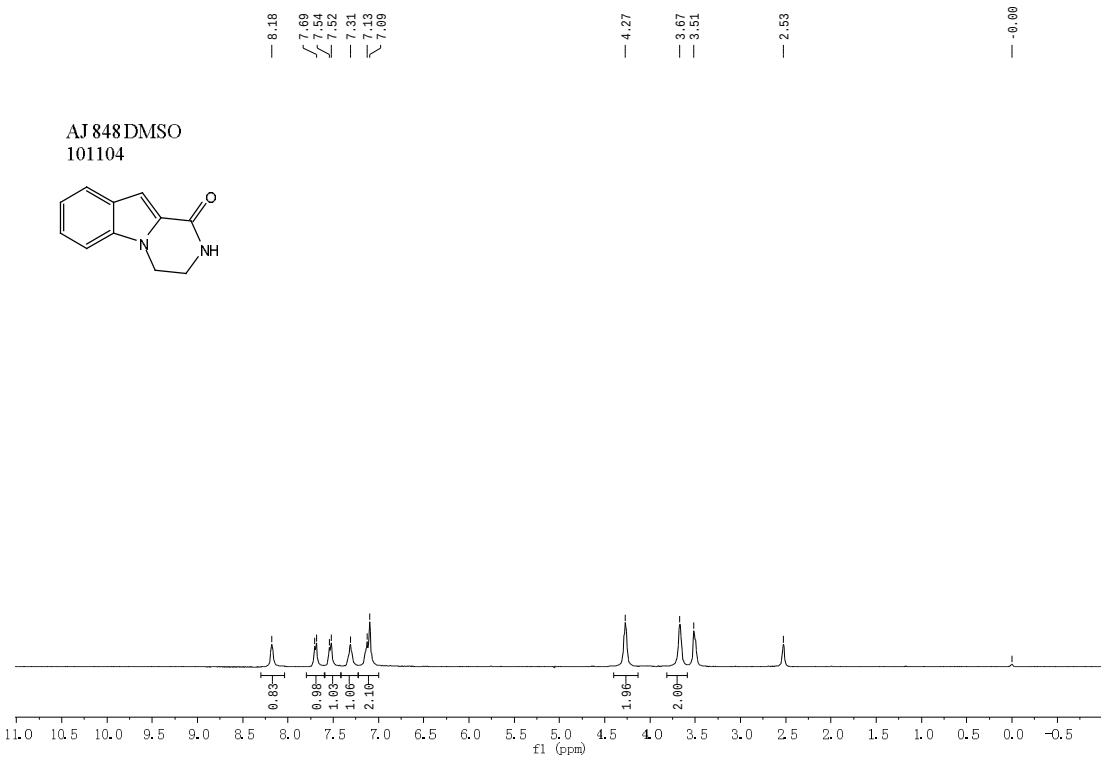

154.42
142.64
140.14
136.80
133.46
128.72
127.87
125.69
113.44
112.42
111.74
102.43
102.25
77.31
77.21
77.00
76.79
57.40
55.72
AJ588 CDCl3
091021
H3C
O
OH
N
CH2
f1 (ppm)

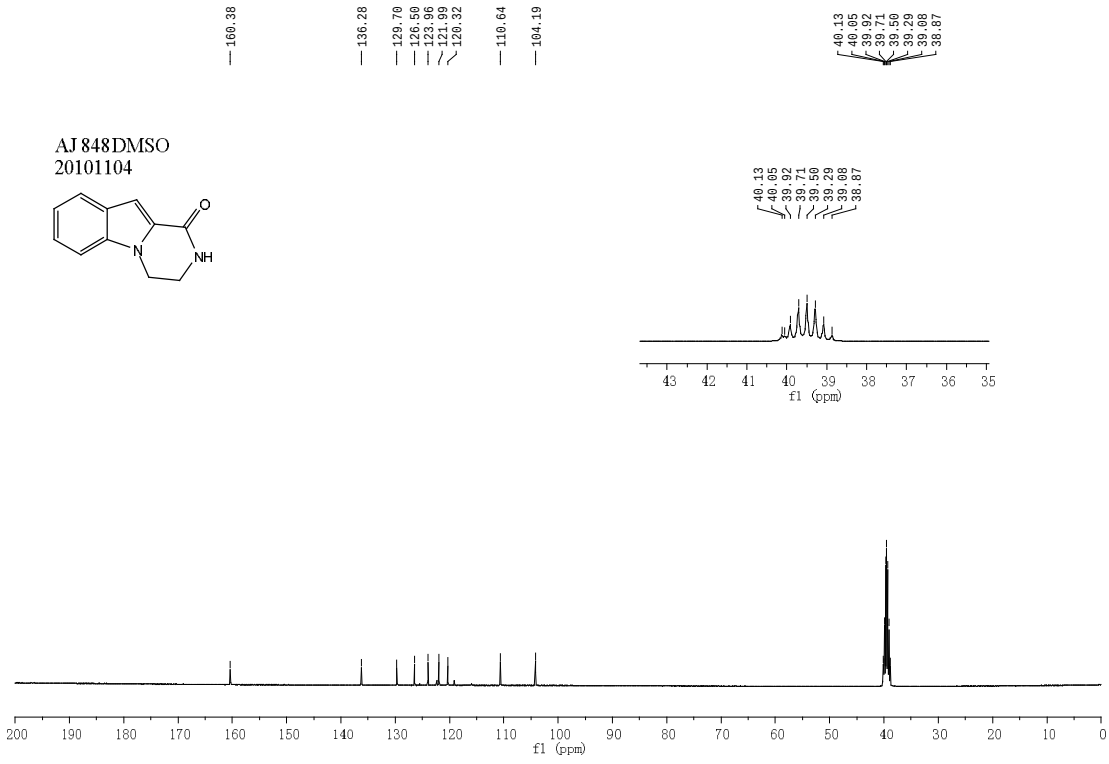

8.49
7.97
7.95
7.29
7.14
7.12
7.11
7.09
6.77
6.12
5.54
4.61
2.91
0.00
AJ14 CDCB
100428
OH
CH2
0.95
0.96
3.08
3.09
1.00
0.98
0.96
1.95
1.09
f1 (ppm)

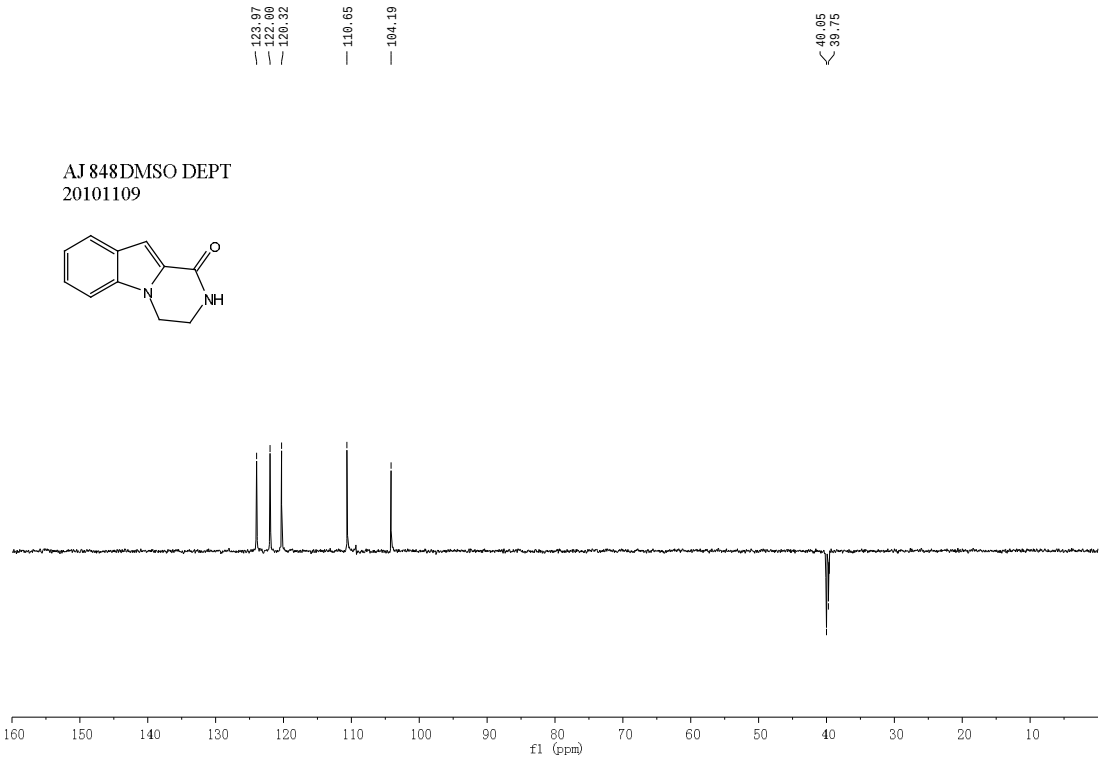

143.16
141.93
141.73
140.89
135.53
129.57
128.94
126.72
125.39
117.79
117.67
114.72
110.74
104.14
77.32
77.00
76.68
57.11
AJ14 CDCB
100428
OH
CH2
f1 (ppm)


AJ684 CDCl3
100324
7.34
7.33
7.26
7.18
7.01
6.81
5.02
4.21
4.20
4.20
4.08
4.08
4.07
3.97
3.91
3.87
-0.00
1.95
1.01
1.99
0.97
1.97
1.99
2.00
3.05
2.99
3.03
f1 (ppm)


157.755
146.717
145.567
130.620
129.455
127.554
127.109
119.337
114.217
111.059
101.125
92.340
77.224
77.010
76.795
64.623
64.500
56.437
56.283
55.287
41.995
AJ 684 CDCl3
100324
ppm


8.18
7.69
7.54
7.52
7.31
7.13
7.09
4.27
3.67
3.51
2.53
0.00
AJ 848 DMSO
101104
O
N
NH
0.83
0.98
1.03
1.06
2.10
1.96
2.00
f1 (ppm)


160.38
136.28
129.70
126.50
123.96
121.99
120.32
110.64
104.19
40.13
40.05
39.92
39.71
39.50
39.29
39.08
38.87
AJ 848 DMSO
20101104
O
N
NH
f1 (ppm)


123.97
122.00
120.32
110.65
104.19
40.05
39.75
AJ 848DMSO DEPT
20101109
O
N
NH
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)