Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information:

Pd-Catalyzed Asymmetric Hydrogenation of 3-(Toluenesulfonamidoalkyl)indoles

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1. General and Materials

General: All reactions were carried out under an atmosphere of nitrogen using standard schlenk techniques, unless otherwise noted. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker DRX-400 spectrometers. The chemical shifts for ^{1}H NMR were recorded in ppm downfield from tetramethylsilane (TMS) with the solvent resonance as the internal standard. The chemical shifts for ^{13}C NMR were recorded in ppm downfield using the central peak of deuterochloroform (77.23 ppm) as the internal standard. Coupling constants (*J*) are reported in Hz and refer to apparent peak multiplications. TLC analysis was performed using glass-backed plates coated with 0.2 mm silica. Quantitative analysis was performed by ^{1}H NMR on Bruker DRX 400 instrument. Flash column chromatography was performed on silica gel (200-300 mesh). Enantiomeric excess was determined by HPLC analysis, using chiral column described below in detail. Optical rotations were measured with JASCO P-1010 polarimeter. The configuration was determined by comparison of rotation sign with the literature data or by analogue.

Materials: Commercially available reagents were used throughout without further purification other than those detailed below. Acetone was dried with anhydrous $CaSO_4$ and distilled over $KMnO_4$. The solvents for asymmetric hydrogenation reaction were purchased without further purification.

2. General Procedure for the Synthesis of 3-(Toluenesulfonamidoalkyl)indoles 1

3-(Toluenesulfonamidoalkyl)indoles **1a-n** were synthesized from the corresponding 2-substituted indoles and *N*-tosyl imines according to the following Method **A** or **B**.[1]

Method **A**: In a dry Schlenk tube, *N*-tosyl imines **4** (1 mmol) and $(EtO)_2POH$ (0.1 mmol) were dissolved in toluene (4 mL) under nitrogen. The solution was stirred for 10 minutes at room temperature and then for another 5 minutes at 0 °C. Subsequently, 2-substituted indoles **3** (3 mmol) were added in one portion at 0 °C. The reaction mixture was allowed to warm to room temperature naturally. After the reaction was complete (monitored by TLC), 10% $NaHCO_3$ (5 mL) was added to quench the reaction. The mixture was extracted with ethyl acetate (10 mL). The organic layer was washed by brine (10 mL), separated, and dried over anhydrous Na_2SO_4. The solvents were removed under reduced pressure and the residue was purified by flash chromatography (ethyl acetate/petroleum ether = 1/5) to afford the product.

Method **B**: In a dry Schlenk tube, 2-substituted indoles **3** (1 mmol) and I_2 (10 mol%) was dissolved in 4 mL dry CH_2Cl_2. Then the resulting mixture was stirred at 0 °C for 2 min before *N*-tosyl imines **4** (1 mmol) was added. Finally, saturated solution of sodium subsulfite was not added to quench the reaction until the starting materials were consumed as indicated by TLC (about 5 min). The mixture was extracted with CH_2Cl_2 (10 mL). The organic layer was washed by brine (10 mL), separated, and dried over anhydrous Na_2SO_4. The solvents were removed under reduced pressure and the residue was purified by flash chromatography (ethyl acetate/petroleum ether = 1/5) to afford the product.

4-Methyl-*N*-((2-methyl-1*H*-indol-3-yl)(phenyl)methyl)benzenesulfonamide (1a).[2,3] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (s, 1H), 7.46 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 7.4 Hz, 2H), 7.29-7.11 (m, 4H), 7.03 (m, 4H), 6.88 (t, *J* = 7.3 Hz, 1H), 5.82 (d, *J* = 6.8 Hz, 1H), 5.14 (d, *J* = 7.3 Hz, 1H), 2.31 (s, 3H), 2.12 (s, 3H); IR (KBr) ν 3363, 3293, 1493, 1318, 1158, 745, 698, 556 cm^{-1}.

***N*-(Cyclohexyl(2-methyl-1*H*-indol-3-yl)methyl)-4-methylbenzenesulfonamide (1b).** White solid, m.p. 94-95 ^{o}C; ^{1}H NMR (400 MHz, d^6-Acetone) δ 9.61 (s, 1H), 7.58 (d, *J* = 7.9 Hz, 1H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.08 (d, *J* = 8.0 Hz, 1H), 6.92 (t, *J* = 7.5 Hz, 1H), 6.82 (dd, *J* = 18.3, 7.6 Hz, 4H), 6.53 (d, *J* = 8.3 Hz, 1H), 4.29 (t, *J* = 9.2 Hz, 1H), 2.34-2.25 (m, 1H), 2.24 (s, 3H), 2.16 (s, 3H), 2.08-1.97 (m, 1H), 1.76 (dd, *J* = 9.2, 4.8 Hz, 1H), 1.56 (dd, *J* = 19.4, 11.5 Hz, 2H), 1.42-0.95 (m, 6H), 0.87-0.77 (m, 1H). ^{13}C NMR (100 MHz, d^6-Acetone) δ 142.03, 136.75, 133.63, 128.83, 127.24, 126.80, 120.95, 119.64, 119.17, 111.04, 110.71, 57.46, 42.54, 31.85, 30.93, 27.11, 26.81, 26.66, 21.24, 11.79. HRMS Calculated for $C_{24}H_{30}N_2O_2NaS$ $[M+Na]^+$ 419.1769, found 419.1769; IR (KBr) ν 3386, 2924, 2857, 1307, 1156, 670 cm^{-1}.

***N*-((4-Fluorophenyl)(2-methyl-1*H*-indol-3-yl)methyl)-4-methylbenzenesulfonamide (1c).** White solid, m.p. 156-157 ^{o}C; ^{1}H NMR (400 MHz, d^6-Acetone) δ 9.89 (s, 1H), 7.55 (d, *J* = 8.1 Hz, 2H), 7.44 (dd, *J* = 8.2, 5.7 Hz, 2H), 7.17 (d, *J* = 8.1 Hz, 1H), 7.09 (t, *J* = 7.2 Hz, 3H), 7.06-6.87 (m, 4H), 6.77 (t, *J* = 7.5 Hz, 1H), 5.84 (d, *J* = 7.3 Hz, 1H), 2.31 (s, 3H), 2.14 (s, 3H). ^{13}C NMR (100 MHz, d^6-Acetone) δ 163.69, 161.27, 143.18, 139.51, 138.93 (d, *J* = 3.1 Hz), 136.63, 134.06, 129.86, 129.78, 129.67, 127.58, 127.31, 121.39, 119.53 (d, *J* = 4.0 Hz), 115.37, 115.16, 111.19, 53.89, 21.32, 11.60; HRMS Calculated for $C_{23}H_{21}FN_2O_2NaS$ $[M+Na]^+$ 431.1205, found 431.1204; IR (KBr) ν 3366, 3305, 1507, 1460, 1318, 1160, 750, 668, 550 cm^{-1}.

4-Methyl-*N*-((2-methyl-1*H*-indol-3-yl)(*p*-tolyl)methyl)benzenesulfonamide (1d). White solid, m.p. 154-155 ^{o}C; ^{1}H NMR (400 MHz, d^6-Acetone) δ 9.82 (s, 1H), 7.52 (d, *J* = 7.8 Hz, 2H), 7.15 (d, *J* = 4.7 Hz, 2H), 7.15 (d, *J* = 7.5 Hz, 2H), 7.06 (t, *J* = 7.8 Hz, 4H), 6.95-6.91 (m, 2H), 6.76 (t, *J* = 7.6 Hz, 1H), 5.83 (d, *J* = 7.4 Hz, 1H), 2.29 (s, 3H), 2.25 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (100 MHz, d^6-Acetone) δ142.98, 139.81, 136.80, 136.64, 133.85, 129.56, 129.36, 127.86, 127.51, 121.27, 119.75, 119.38, 111.58, 111.11, 54.27, 21.32, 20.99, 11.68.; HRMS Calculated for $C_{24}H_{24}N_2O_2NaS$ $[M+Na]^+$ 427.1456, found 427.1447; IR (KBr) ν 3396, 1460, 1325, 1155, 747, 673, 562 cm^{-1}.

4-Methyl-*N*-((2-methyl-1*H*-indol-3-yl)(*m*-tolyl)methyl)benzenesulfonamide (1e). Pale yellow solid, m.p. 168-169 ^{o}C; ^{1}H NMR (400 MHz, d^6-Acetone) δ 9.84 (s, 1H), 7.53 (d, *J* = 8.1 Hz, 2H), 7.21-7.07 (m, 7H), 6.99-6.91 (m, 3H), 6.76 (t, *J* = 7.5 Hz, 1H), 5.85 (d, *J* = 7.5 Hz, 1H), 2.30 (s, 3H), 2.21 (s, 3H), 2.17 (s, 3H).^{13}C NMR (100 MHz, d^6-Acetone) 142.99, 142.68, 139.75, 138.04, 136.59, 133.86, 129.58, 128.63, 128.51, 128.08, 127.52, 125.04, 121.27, 119.68, 119.40, 111.59, 111.10, 110.86, 54.47, 21.48, 21.32, 11.71; HRMS Calculated for $C_{24}H_{24}N_2O_2NaS$ $[M+Na]^+$ 427.1456, found 427.1459; IR (KBr) ν 3375, 1460, 1315, 1160, 1093, 1152, 743, 668, 560 cm^{-1}..

4-Methyl-*N*-((2-methyl-1*H*-indol-3-yl)(*o*-tolyl)methyl)benzenesulfonamide (1f). White solid, m.p. 165-166 ^{o}C; ^{1}H NMR (400 MHz, d^{6}-Acetone) δ 9.88 (s, 1H), 7.78 (d, *J* = 8.3 Hz, 2H), 7.56 (d, *J* = 8.2 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.22-7.01 (m, 4H), 6.99-6.86 (m, 2H), 6.83-6.68 (m, 1H), 6.50 (s, 1H), 5.94 (d, *J* = 6.9 Hz, 1H), 2.32 (s, 3H), 2.13 (s, 3H), 2.05 (s, 3H); ^{13}C NMR (100 MHz, d^{6}-Acetone) ^{13}C NMR (101 MHz, Acetone) δ 143.24, 143.11, 142.40, 140.34, 139.70, 136.31, 134.19, 131.10, 130.17, 129.68, 128.24, 128.10, 127.64, 127.62, 127.53, 126.90, 125.99, 121.21, 119.48, 119.39, 111.07, 110.83, 109.81, 52.60, 52.50, 21.33, 21.31, 19.44, 11.83; HRMS Calculated for $C_{24}H_{24}N_2O_2NaS$ [M+Na]$^{+}$ 427.1456, found 427.1458; IR (KBr) ν 3389, 1461, 1319, 1158, 1093, 1046, 740, 672, 563 cm^{-1}.

***N*-((2-Butyl-1*H*-indol-3-yl)(phenyl)methyl)-4-methylbenzenesulfonamide (1g).** White solid, m.p. 148-149 ^{o}C; ^{1}H NMR (400 MHz, d^{6}-Acetone) δ 9.90 (s, 1H), 7.58 (d, *J* = 7.3 Hz, 2H), 7.44 (d, *J* = 7.5 Hz, 2H), 7.24 (t, *J* = 7.4 Hz, 2H), 7.21-7.07 (m, 5H), 7.05-6.87 (m, 2H), 6.75 (dd, *J* = 7.9, 7.2 Hz, 1H), 5.87 (d, *J* = 6.9 Hz, 1H), 2.56-2.49 (m, 2H), 2.32 (s, 3H), 1.54-1.46 (m, 2H), 1.30-1.24 (m, 2H), 0.85 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (100 MHz, d^{6}-Acetone) δ 143.11, 143.04, 139.66, 138.50, 136.79, 129.70, 128.67, 127.97, 127.66, 127.38, 127.30, 121.36, 120.01, 119.39, 111.25, 111.09, 54.44, 32.59, 26.37, 23.25, 21.35, 14.11; HRMS Calculated for $C_{26}H_{28}N_2O_2NaS$ [M+Na]$^{+}$ 455.1769, found 455.1768; IR (KBr) ν 3405, 2956, 1493, 1324, 1160, 742, 668, 560 cm^{-1}.

4-Methyl-*N*-((2-phenethyl-1*H*-indol-3-yl)(phenyl)methyl)benzenesulfonamide (1h). White solid, m.p. 175-176 ^{o}C; ^{1}H NMR (400 MHz, d^{6}-Acetone) δ 10.01 (s, 1H), 7.58 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 7.6 Hz, 2H), 7.14-7.27 (m, 9H), 7.09 (dd, *J* = 8.0, 2.9 Hz, 3H), 6.94 (dd, *J* = 11.0, 4.0 Hz, 2H), 6.83-6.69 (m, 1H), 5.87 (d, *J* = 7.1 Hz, 1H), 2.87-2.80 (m, 4H), 2.25 (s, 3H); ^{13}C NMR (100 MHz, d^{6}-Acetone) δ 143.20, 142.85, 142.22, 139.67, 137.50, 136.85, 129.77, 129.21, 128.64, 127.94, 127.67, 127.31, 126.86, 121.57, 120.05, 119.46, 111.81, 111.30, 54.34, 36.56, 28.92, 21.31; HRMS Calculated for $C_{30}H_{28}N_2O_2NaS$ [M+Na]$^{+}$ 503.1769, found 503.1760; IR (KBr) ν 3375, 1451, 1324, 1163, 743, 697, 669, 560 cm^{-1}.

***N*-((2,7-Dimethyl-1*H*-indol-3-yl)(phenyl)methyl)-4-methylbenzenesulfonamide (1i).** White solid, m.p. 144-145 ^{o}C; ^{1}H NMR (400 MHz, d^{6}-Acetone) δ 9.74 (s, 1H), 7.50 (d, *J* = 7.9 Hz, 2H), 7.42 (d, *J* = 7.6 Hz, 2H), 7.24 (t, *J* = 7.4 Hz, 2H), 7.17 (t, *J* = 7.3 Hz, 1H), 7.05 (d, *J* = 8.0 Hz, 2H), 6.96 (t, *J* = 9.4 Hz, 2H), 6.74 (d, *J* = 7.0 Hz, 1H), 6.67 (t, *J* = 7.5 Hz, 1H), 5.86 (d, *J* = 7.4 Hz, 1H), 2.37 (s, 3H), 2.29 (s, 3H), 2.17 (s, 3H); ^{13}C NMR (100 MHz, d^{6}-Acetone) δ 142.87, 142.82, 135.96, 133.77, 129.44, 128.70, 127.95, 127.45, 127.41, 127.04, 122.02, 120.24, 119.70, 117.46, 111.81, 54.54, 21.29, 16.82, 11.62; HRMS Calculated for $C_{24}H_{24}N_2O_2NaS$ [M+Na]$^{+}$ 427.1456, found 427.1459; IR (KBr) ν 3395, 3275, 1453, 1318, 1153, 670, 560 cm^{-1}.

***N*-(Cyclohexyl(2,7-dimethyl-1*H*-indol-3-yl)methyl)-4-methylbenzenesulfonamide (1j).** White solid, m.p. 156-157 ^{o}C; ^{1}H NMR (400 MHz, d^{6}-Acetone) δ 9.49 (s, 1H), 7.41 (d, *J* = 7.1 Hz, 1H), 7.17 (d, *J* = 7.4 Hz, 2H), 6.76-6.71 (m, 4H), 6.52 (d, *J* = 8.4 Hz, 1H), 4.27 (t, *J* = 9.1 Hz, 1H), 2.33-2.25 (m, 7H), 2.14 (s, 3H), 1.77 (d, *J* = 13.2 Hz, 1H), 1.61-1.53 (m, 2H), 1.31-1.03 (m, 5H), 0.87-0.77 (m, 2H); ^{13}C NMR (100 MHz, d^{6}-Acetone) δ 141.73, 139.87, 135.98, 133.44, 128.53, 126.63, 121.68, 120.00, 119.43, 117.42, 111.06, 57.55, 42.38, 31.87, 30.93, 27.11, 26.80, 26.64, 21.15, 16.87, 11.73; HRMS Calculated for $C_{24}H_{30}N_2O_2NaS$ [M+Na]$^{+}$ 433.1926, found 433.1938; IR (KBr) ν 3384, 2924, 2853, 1452, 1303, 1154, 667, 559 cm^{-1}.

***N*-(1-(2,7-dimethyl-1*H*-indol-3-yl)-2-methylpropyl)-4-methylbenzenesulfonamide (1k).** White solid, m.p. 166-167 ^{o}C; ^{1}H NMR (400 MHz, d^{6}-Acetone) δ 9.47 (s, 1H), 7.41 (d, *J*=7.0, 1H), 7.18 (d, *J*=8.2, 2H), 6.76-6.71 (m, 4H), 6.54 (d, *J*=8.5, 1H), 4.18 (dd, *J*=9.9, 8.7, 1H), 2.43-2.34 (m, 1H), 2.33 (s, 3H), 2.27 (s, 3H), 2.14 (s, 3H), 1.16 (d, *J*=6.5, 3H), 0.69 (d, *J*=6.7, 3H); ^{13}C NMR (100 MHz, d^6-Acetone) δ 141.76, 139.73, 135.95, 133.37, 128.53, 126.63, 121.68, 120.00, 119.42, 117.39, 111.41, 58.99, 21.14, 21.09, 20.42, 16.84, 11.70; HRMS Calculated for $C_{21}H_{26}N_2O_2NaS$ $[M+Na]^+$ 393.1613, found 393.1619; IR (KBr) ν 3421, 3352, 1460, 1158, 1098, 1023, 664, 572 cm^{-1}.

***N*-((2,7-Dimethyl-1*H*-indol-3-yl)(*p*-tolyl)methyl)-4-methylbenzenesulfonamide (1l).** yellow solid, m.p. 153-154 °C; ^1H NMR (400 MHz, d^6-Acetone) δ 9.72 (s, 1H), 7.48 (d, *J* = 7.9 Hz, 2H), 7.29 (d, *J* = 7.6 Hz, 2H), 7.11-6.95 (m, 5H), 6.90 (d, *J* = 7.4 Hz, 1H), 6.74 (d, *J* = 7.1 Hz, 1H), 6.69 (d, *J* = 7.7 Hz, 1H), 5.82 (d, *J* = 7.6 Hz, 1H), 2.37 (s, 3H), 2.28 (s, 3H), 2.26 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (100 MHz, d^6-Acetone) δ142.79, 139.76, 136.79, 135.96, 133.67, 129.38, 129.35, 127.90, 127.39, 127.05, 121.97, 120.20, 119.65, 117.55, 111.88, 54.35, 54.25, 21.30, 20.98, 16.84, 11.64; HRMS Calculated for $C_{25}H_{26}N_2O_2NaS$ $[M+Na]^+$ 441.1613, found 441.1599; IR (KBr) ν 3371, 1321, 1156, 810, 669, 558 cm^{-1}.

***N*-((2,7-Dimethyl-1*H*-indol-3-yl)(*m*-tolyl)methyl)-4-methylbenzenesulfonamide (1m).** Yellow solid, m.p. 156-158 °C; ^1H NMR (400 MHz, d^6-Acetone) δ 9.73 (s, 1H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 12.5 Hz, 2H), 7.11 (t, *J* = 7.6 Hz, 1H), 7.07-6.95 (m, 4H), 6.92 (d, *J* = 7.5 Hz, 1H), 6.74 (d, *J* = 6.9 Hz, 1H), 6.68 (t, *J* = 7.5 Hz, 1H), 5.84 (d, *J* = 7.5 Hz, 1H), 2.36 (s, 3H), 2.28 (s, 3H), 2.21 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, d^6-Acetone) δ 142.80, 142.64, 139.69, 138.02, 135.91, 133.67, 129.39, 128.61, 128.53, 128.07, 127.40, 127.07, 125.06, 121.98, 120.19, 119.67, 117.47, 111.89, 54.54, 21.48, 21.28, 16.82, 11.66; HRMS Calculated for $C_{25}H_{26}N_2O_2NaS$ $[M+Na]^+$ 411.1613, found 411.1603; IR (KBr) ν 3372, 1319, 1155, 669, 562 cm^{-1}.

***N*-((2,7-Dimethyl-1*H*-indol-3-yl)(*o*-tolyl)methyl)-4-methylbenzenesulfonamide (1n).** White solid, m.p. 159-161 °C; ^1H NMR (400 MHz, d^6-Acetone) δ 9.77 (s, 1H), 7.77 (d, *J* = 2.9 Hz, 1H), 7.53 (d, *J* = 7.9 Hz, 2H), 7.16-7.03 (m, 5H), 6.99 (d, *J* = 7.3 Hz, 1H), 6.89 (d, *J* = 6.3 Hz, 1H), 6.74 (d, *J* = 7.1 Hz, 1H), 6.68 (dd, *J* = 10.5, 4.4 Hz, 1H), 5.98-5.89 (m, 1H), 2.36 (s, 3H), 2.31 (s, 3H), 2.16 (s, 3H), 2.08 (s, 3H); ^{13}C NMR (100 MHz, d^6-Acetone) δ 142.97, 140.33, 139.69, 136.45, 135.66, 133.99, 131.13, 129.54, 128.36, 127.73, 127.56, 126.00, 121.96, 120.17, 119.79, 117.25, 110.27, 52.75, 21.32, 19.51, 16.83, 11.85; HRMS Calculated for $C_{25}H_{26}N_2O_2NaS$ $[M+Na]^+$ 441.1613, found 441.1610; IR (KBr) ν 3388, 1460, 1305, 1155, 1092, 1035, 666, 555 cm^{-1}.

3. General Procedure for Pd-Catalyzed Asymmetric Hydrogenation of 3-(Toluenesulfonamidoalkyl)indoles

(*R*)-H8-BINAP (3.8 mg, 0.006 mmol) and $Pd(OCOCF_3)_2$ (1.7 mg, 0.005 mmol) were placed in a dried schlenk tube under nitrogen atmosphere, and degassed anhydrous acetone 1 mL was added. The mixture was stirred at room temperature for 1 h, and then solvent was removed under vacuum to give the catalyst. In a glovebox, $TsOH{\cdot}H_2O$ (0.25 mmol) and substrate **1** (0.25 mmol) were stirred in 1 mL solvent (DCM and TFE were mixed in ratio of 1:1 prior to use) at room temperature for 5 min. Subsequently, the above catalyst together with 2 mL solvent was added to the reaction mixture. The hydrogenation was performed at 50 ^{o}C under H_2 (600 psi) in a stainless steel autoclave for 16-20 h. After carefully releasing the hydrogen, the resulting mixture was concentrated under vacuum and dissolved in saturated aqueous $NaHCO_3$ (5 mL). After stirring for 10 min, the mixture was extracted with CH_2Cl_2 (3×5 mL) and dried over Na_2SO_4. After purified by silica gel chromatography using petroleum ether/EtOAc (10/1) as eluent, the enantiomeric excess of the products were determined by HPLC with chiral columns (OJ-H, OD-H or AD-H).

Racemates of **2** were prepared by the hydrogenation of the 3-(toluenesulfonamidoalkyl)indoles catalyzed by $Pd(OCOCF_3)_2$/(+/-)-SynPhos in TFE.

(*2R,3R*)-(**-)-2-Methyl-3-benzylindoline (2a).**[4] [Known compound, 91% *ee*, $[\alpha]^{27}_{D}$ = -70.3 (*c* 1.0, $CHCl_3$)]; 89% yield, 87% *ee*, $[\alpha]^{27}_{D}$ = -68.0 (*c* 0.83, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.23 (d, *J* = 6.5 Hz, 3H), 2.87 (dd, *J* = 13.8, 8.9 Hz, 1H), 2.97 (dd, *J* = 13.9, 7.2 Hz, 1H), 3.53 (dd, *J* = 15.9, 7.8 Hz, 1H), 3.71 (br s, 1H), 3.96-4.03 (m, 1H), 6.54-6.65 (m, 3H), 7.00 (t, *J* = 7.4, 1H), 7.17-7.31 (m, 5H); HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 80/20, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 10.4 min, t_2 = 11.6 min (maj.).

(-)-2-Methyl-3-(cyclohexylmethyl)indoline (2b).[4] [Known compound, 94% *ee*, $[\alpha]^{29}_{D}$ = -8.6 (*c* 1.04, $CHCl_3$)]; 97% yield, 92% *ee*, $[\alpha]^{27}_{D}$ = -7.9 (*c* 0.97, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.98 (d, *J* = 11.3 Hz, 2H), 1.11 (d, *J* = 6.4 Hz, 3H), 1.20-1.89 (m, 11H), 3.23-3.27 (m, 1H), 3.71 (br s, 1H), 3.92-3.96 (m, 1H), 6.62 (d, *J* = 7.6 Hz, 1H), 6.72 (t, *J* = 7.4 Hz, 1H), 7.00-7.06 (m, 2H); HPLC (OD-H, elute: Hexanes/*i*-PrOH = 99/1, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 9.6 min, t_2 = 11.5 min (maj.).

(-)-2-Methyl-3-(4-fluorobenzyl)indoline (2c).[4] [Known compound, 88% *ee*, $[\alpha]^{28}_{D}$ = -76.3 (*c* 0.84, $CHCl_3$); 81% yield, 86% *ee*, $[\alpha]^{27}_{D}$ = -71.5 (*c* 0.83, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.24 (d, *J* = 6.1 Hz, 3H), 2.77-2.83 (m, 1H), 2.91-2.96 (m, 1H), 3.42-3.44 (m, 1H), 3.70 (br s, 1H), 3.98-4.01 (m, 1H), 6.49 (d, *J* = 6.6 Hz, 1H), 6.57-6.65 (m, 2H), 6.95-7.10 (m, 5H); HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 15.1 min, t_2 = 17.1 min (maj.).

(-)-2-Methyl-3-(4-methylbenzyl)indoline (2d).[4] [Known compound, 90% *ee*, $[\alpha]^{30}_{D}$ = -64.5 (*c* 1.0, $CHCl_3$)]; 84% yield, 84% *ee*, $[\alpha]^{27}_{D}$ = -75.9 (*c* 0.80, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.30 (d, *J* = 6.5 Hz, 3H), 2.09 (s, 3H), 2.86 (qd, *J* = 22.8, 8.0 Hz, 1H), 3.41 (d, *J* = 8.3 Hz, 1H), 3.72 (d, *J* = 7.0 Hz, 1H), 3.99-4.13 (m, 1H), 6.39 (d, *J* = 7.4 Hz, 1H), 6.54 (t, *J* = 7.3 Hz, 1H), 6.64 (d, *J* = 7.7 Hz, 1H), 7.00 (t, *J* = 7.6 Hz, 1H), δ 7.09-7.12 (m, 4H); HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 12.9 min, t_2 = 16.1 min (maj.).

(-)-2-Methyl-3-(3-methylbenzyl)indoline (2e).[4] [Known compound, 90% *ee*, $[\alpha]^{28}_{D}$ = -63.7 (*c*

0.96, $CHCl_3$); 97% yield, 87% *ee*, $[\alpha]^{28}_{D}$ = -59.8 (*c* 0.97, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.21 (d, *J* = 6.5 Hz, 3H), 2.33 (s, 3H), 2.89 (qd, *J* = 14.0, 8.0 Hz, 2H), 3.35-3.87 (m, 2H), 3.95-4.02 (m, 1H), 6.53-6.68 (m, 3H), 6.93-7.08 (m, 4H), 7.18 (t, *J* = 7.5 Hz, 1H); HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 12.7 min (maj.), t_2 = 13.6 min.

(-)-2-Methyl-3-(2-methylbenzyl)indoline (2f).[4] [Known compound, 91% *ee*, $[\alpha]^{29}_{D}$ = -79.0 (*c* 0.82, $CHCl_3$); 93% yield, 89% *ee*, $[\alpha]^{27}_{D}$ = -84.0 (*c* 0.90, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 1.23 (d, *J* = 6.5 Hz, 3H), 2.14 (s, 3H), 2.33 (s, 3H), 2.81-2.96 (m, 2H), 3.52-3.58 (m, 2H), 4.01 (p, *J* = 7.0 Hz, 1H), 6.47 (d, *J* = 7.2 Hz, 1H), 6.54 (t, *J* = 7.3 Hz, 1H), 6.86 (d, *J* = 7.3 Hz, 1H), 6.93-7.08 (m, 3H), 7.18 (t, *J* = 7.4 Hz, 1H); HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 85/15, detector: 254 nm, flow rate: 0.8 mL/min), t_1 = 11.4 min (maj.), t_2 = 15.0 min.

(-)-2-Butyl-3-benzylindoline (2g).[4] [Known compound, 94% *ee*, $[\alpha]^{28}_{D}$ = -86.3 (*c* 1.10, $CHCl_3$)]; 97% yield, 92% *ee*, $[\alpha]^{30}_{D}$ = -79.4 (*c* 1.03, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 0.93 (t, *J* = 6.9 Hz, 3H), 1.33-1.43 (m, 4H), 1.65-1.70 (m, 2H), 2.65 (dd, *J* = 13.3, 10.4 Hz, 1H), 2.99 (dd, *J* = 13.4, 5.6 Hz, 1H), 3.36-3.78 (m, 1H), 3.81 (br s, 1H), 3.81 (dd, *J* = 13.9, 7.6 Hz), 6.32 (d, *J* = 7.3 Hz, 1H), 6.50 (t, *J* = 7.4 Hz, 1H), 6.64 (d, *J* = 7.7 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 7.08 (d, *J* = 7.2 Hz, 2H), 7.18-7.28 (m, 5H); HPLC (AD-H, elute: Hexanes/*i*-PrOH = 95/5, detector: 254 nm, flow rate: 0.8 mL/min), t_1 = 7.9 min (maj.), t_2 = 10.7 min.

(-)-2-Phenethyl-3-benzylindoline (2h).[4] [Known compound, 93% *ee*, $[\alpha]^{30}_{D}$ = -74.3 (*c* 0.67, $CHCl_3$); 95% yield, 91% *ee*, $[\alpha]^{30}_{D}$ = -64.5 (*c* 1.20, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 2.01-2.07 (m, 2H), 2.62-2.80 (m, 3H), 3.01 (dd, *J* = 13.4, 5.5 Hz, 1H), 3.39-3.41 (m, 1H), 3.86-3.89 (m, 2H), 6.28 (d, *J* = 7.3 Hz, 1H), 6.51 (t, *J* = 7.4 Hz, 1H), 6.63 (d, *J* = 7.7 Hz, 1H), 6.99 (t, *J* = 7.4 Hz, 1H), 7.06 (d, *J* = 7.0 Hz, 2H), 7.21-7.32 (m, 8H); HPLC (OD-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 9.6 min, t_2 = 10.9 min (maj.).

(-)-2,7-Dimethyl-3-benzylindoline (2i).[4] (Known compound, 97% *ee*, $[\alpha]^{29}_{D}$ = -70.6 (*c* 0.88, $CHCl_3$)); 94% yield, 95% *ee*, $[\alpha]^{30}_{D}$ = -75.7 (*c* 0.93, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.25 (d, *J* = 8.5 Hz, 1H), 2.15 (s, 3H), 2.83-3.00 (m, 2H), 3.53-3.55 (m, 2H), 4.00-4.02 (m, 1H), 6.41-6.53 (m, 2H), 6.85 (m, 1H), 7.16-7.29 (m, 5H); HPLC (OD-H, elute: Hexanes/*i*-PrOH = 99/1, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 12.8 min, t_2 = 20.9 min (maj.).

(-)-2,7-Dimethyl-3-(cyclohexylmethyl)indoline (2j).[4] [Known compound, 97% *ee*, $[\alpha]^{29}_{D}$ = -17.0 (*c* 0.96, $CHCl_3$)]; 90% yield, 97% *ee*, $[\alpha]^{28}_{D}$ = -21.6 (*c* 0.80, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 0.93-0.95 (m, 2H), 1.12-1.29 (m, 6H), 1.39-1.44 (m, 2H), 1.52-1.54 (m, 1H), 1.66-1.76 (m, 4H), 1.87-1.90 (m, 1H), 2.13 (s, 3H), 3.26 (q, *J* = 7.4 Hz, 1H), 3.43 (br s, 1H), 3.93-4.00 (m, 1H), 6.67 (t, *J* = 7.4 Hz, 1H), 6.87 (d, *J* = 7.4 Hz, 1H), 6.92 (d, *J* = 7.3 Hz, 1H); HPLC (OD-H, elute: Hexanes/*i*-PrOH = 99/1, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 5.5 min, t_2 = 6.4 min (maj.).

(-)-2,7-Dimethyl-3-(2-methylpropyl)indoline (2k).[4] [Known compound, 97% *ee*, $[\alpha]^{30}_{D}$ = -3.74 (*c* 0.93, $CHCl_3$)]; 88% yield, 94% *ee*, $[\alpha]^{28}_{D}$ = -7.6 (*c* 0.47, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 0.95 (d, *J* = 6.6 Hz, 3H), 1.00 (d, *J* = 6.6 Hz, 3H), 1.14 (d, *J* = 6.5 Hz, 3H), 1.40-1.45 (m, 1H), 1.52-1.57 (m, 1H), 1.71-1.74 (m, 1H), 2.13 (s, 3H), 3.22 (q, *J* = 7.8 Hz, 1H), 3.46 (br s, 1H), 3.94-4.01 (m, 1H), 6.67 (t, *J* = 7.4 Hz, 1H), 6.87 (d, *J* = 7.4 Hz, 1H), 6.94 (d, *J* = 7.3 Hz, 1H); HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 5.1 min, t_2 = 5.7 min (maj.).

(-)-2,7-Dimethyl-3-(4-methylbenzyl)indoline (2l).[4] [Known compound, 96% *ee*, $[\alpha]^{27}_{D}$ = -69.7 (*c* 1.17, $CHCl_3$)]; 87% yield, 94% *ee*, $[\alpha]^{24}_{D}$ = -80.8 (*c* 1.17, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ = 1.23 (d, *J*=6.3, 3H), 2.14 (s, 3H), 2.34 (s, 3H), 2.71-3.02 (m, 2H), 3.46-3.66 (m, 2H), 3.84-4.20 (m, 1H), 6.41-6.67 (m, 2H), 6.86 (d, *J*=6.3, 1H), 7.07-7.09 (d, *J*=6.0, 4H); HPLC (OD-H, elute: Hexanes/*i*-PrOH

= 99/1, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 8.0 min, t_2 = 11.1 min (maj.).

(-)-2,7-Dimethyl-3-(3-methylbenzyl)indoline (2m).[4] [Known compound, 95% *ee*, $[\alpha]^{28}_{D}$ = -61.1 (*c* 1.07, $CHCl_3$)]; 97% yield, 93% *ee*, $[\alpha]^{28}_{D}$ = -75.0 (*c* 0.83, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.24 (d, *J* = 6.5 Hz, 3H), 2.15 (s, 3H), 2.34 (s, 3H), 2.84-2.96 (m, 2H), 3.52-3.58 (m, 2H), 4.00-4.03 (m, 1H), 6.47 (d, *J* = 7.3 Hz, 1H), 6.55 (t, *J* = 7.4 Hz, 1H), 6.87 (d, *J* = 7.4 Hz, 1H), 6.97-7.04 (m, 3H), 7.18 (t, *J* = 7.5 Hz, 1H); HPLC (OD-H, elute: Hexanes/*i*-PrOH = 99/1, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 8.8 min, t_2 = 12.6 min (maj.).

(-)-2,7-Dimethyl-3-(2-methylbenzyl)indoline (2n).[4] [Known compound, 94% *ee*, $[\alpha]^{29}_{D}$ = - 89.7 (*c* 0.92, $CHCl_3$)]; 97% yield, 94% *ee*, $[\alpha]^{30}_{D}$ = -87.0 (*c* 0.97, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.32 (d, *J* = 6.6 Hz, 3H), 2.09 (s, 3H), 2.15 (s, 3H), 2.80-2.95 (m, 2H), 3.44 (dd, *J* = 16.3, 7.2 Hz, 1H), 3.58 (br s, 1H), 4.05-4.08 (m, 1H), 6.27 (d, *J* = 7.3 Hz, 1H), 6.49 (t, *J* = 7.4 Hz, 1H), 6.85 (d, *J* = 7.4 Hz, 1H), 7.08-7.14 (m, 4H); HPLC (OJ-H, elute: Hexanes/*i*-PrOH = 90/10, detector: 254 nm, flow rate: 1.0 mL/min), t_1 = 6.1 min (maj.), t_2 = 7.8 min.

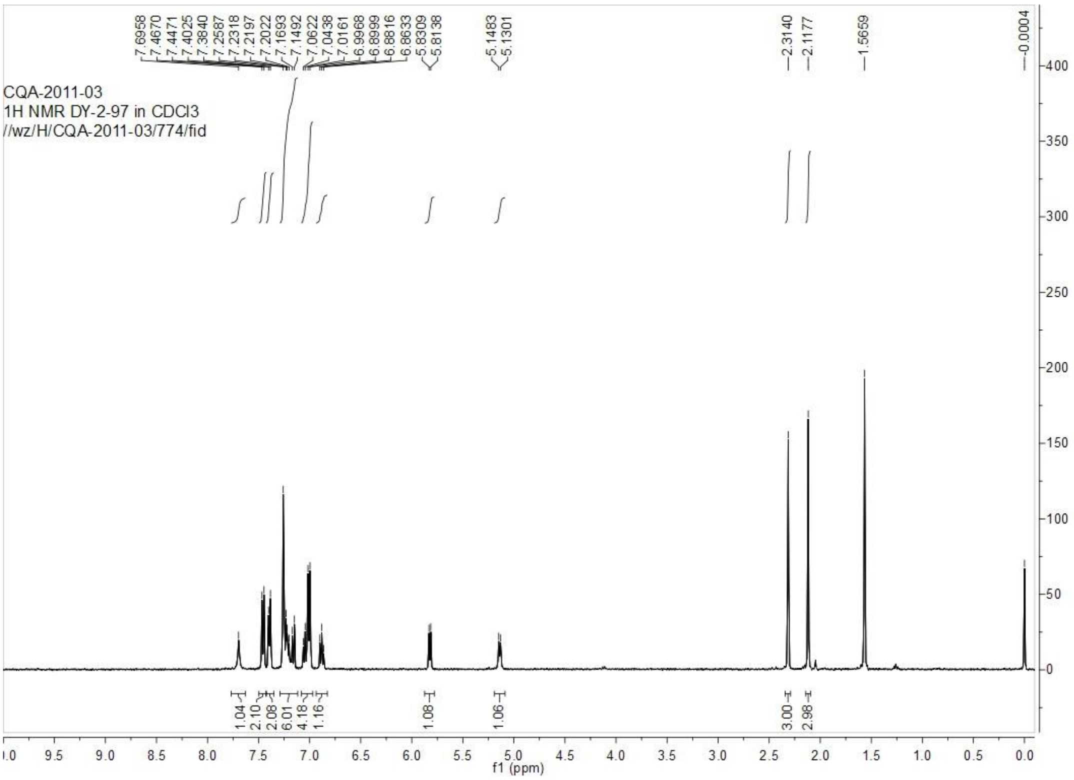
4. General Procedure for Pd-Catalyzed Tandem Reactions of 2-Substituted Indoles and *N*-Tosyl Imines [5]

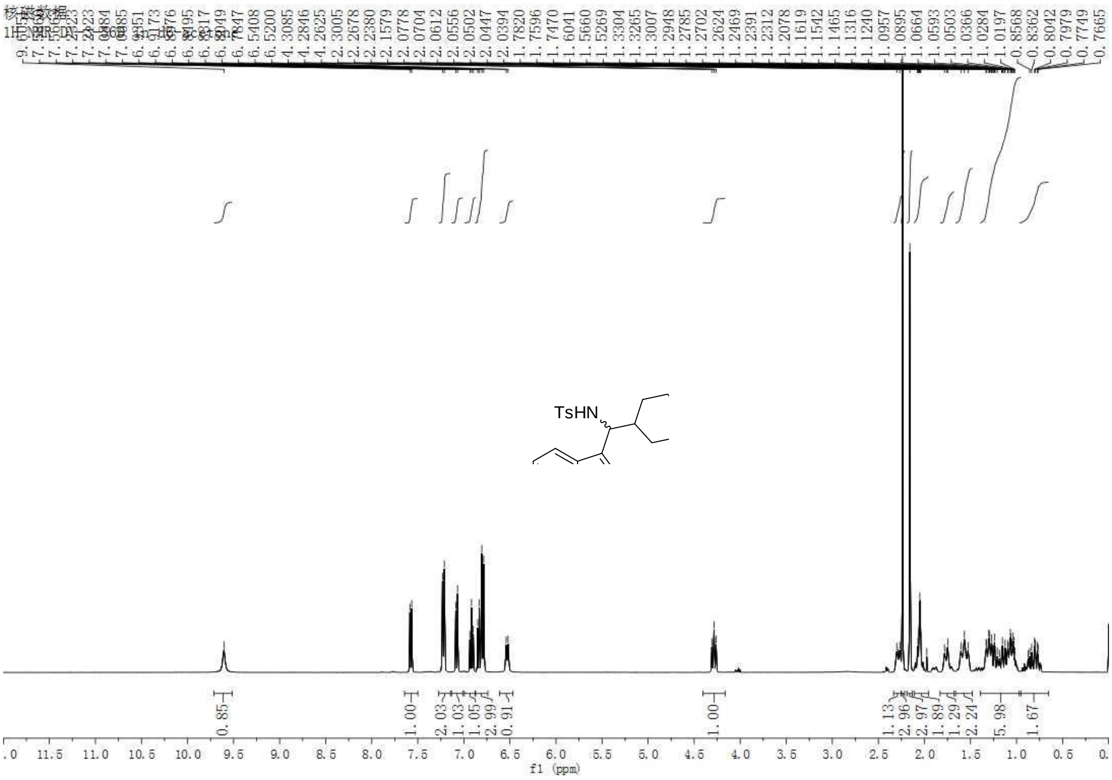
(*R*)-H8-BINAP (3.8 mg, 0.006 mmol) and $Pd(OCOCF_3)_2$ (1.7 mg, 0.005 mmol) were placed in a dried Schlenk tube under nitrogen atmosphere, and degassed anhydrous acetone was added. The mixture was stirred at rt for 1 h, then solvent was removed under vacuum to give the catalyst. In a glovebox, acid (0.25 mmol) and indole (0.25 mmol) were stirred in 1 mL DCM/TFE at room temperature for 1 min. Subsequently, *N*-tosyl imine (0.25 mmol) was added to the solution. Finally, the above catalyst together with 2 mL DCM/TFE was added to the reaction mixture. The hydrogenation was performed at 50 ^{o}C under H_2 (600 psi) in a stainless steel autoclave for 16 h. After carefully releasing the hydrogen, the resulting mixture was concentrated under vacuum and dissolved in saturated aqueous $NaHCO_3$ (5 mL). After stirring for 10 min, the mixture was extracted with CH_2Cl_2 (3×5 mL) and dried over Na_2SO_4. After purified by silica gel chromatography using petroleum ether/EtOAc (10/1) as eluent, the enantiomeric excess of the products were determined by HPLC with chiral column.

5. References

1. Q. Kang, Z.-A. Zhao, S.-L. You, *J. Am. Chem. Soc.*, 2007, **129**, 1484.
2. F. Xu, D. Huang, C. Han, W. Shen, X. Lin, Y. Wang, *J. Org. Chem.*, 2010, **75**, 8677.
3. Q.-L. He, F.-L. Sun, X.-J. Zheng, S.-L. You, *Synlett*, 2009, 1111.
4. D.-S. Wang, J. Tang, Y.-G. Zhou, M.-W. Chen, C.-B. Yu, Y. Duan, G.-F. Jiang, *Chem. Sci.*, 2011, **2**, 803.
5. Y. Duan, M.-W. Chen, Z.-S. Ye, D.-S. Wang, Q.-A. Chen, Y.-G. Zhou, *Chem. Eur. J.*, 2011, **17**, 7193.

6. Copy of NMR and HRMS Spectra

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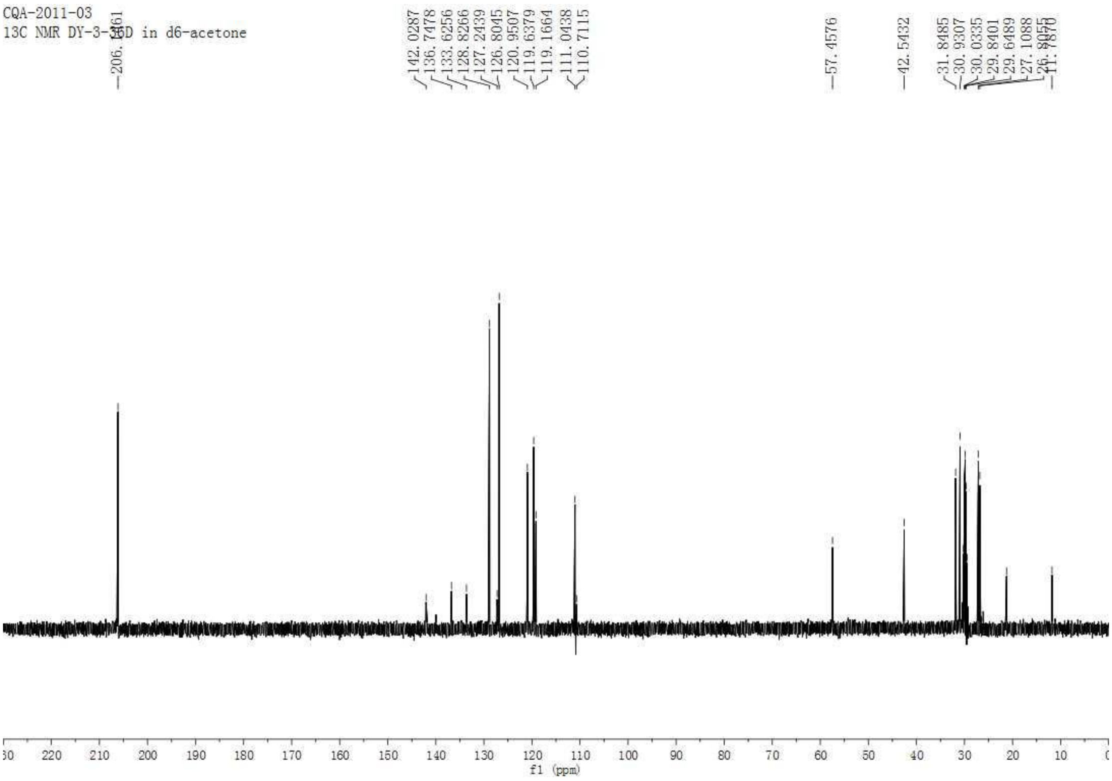
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Elemental Composition Report **Page 1**

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6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
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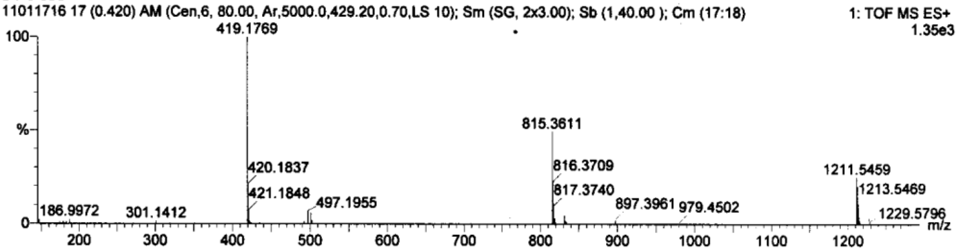

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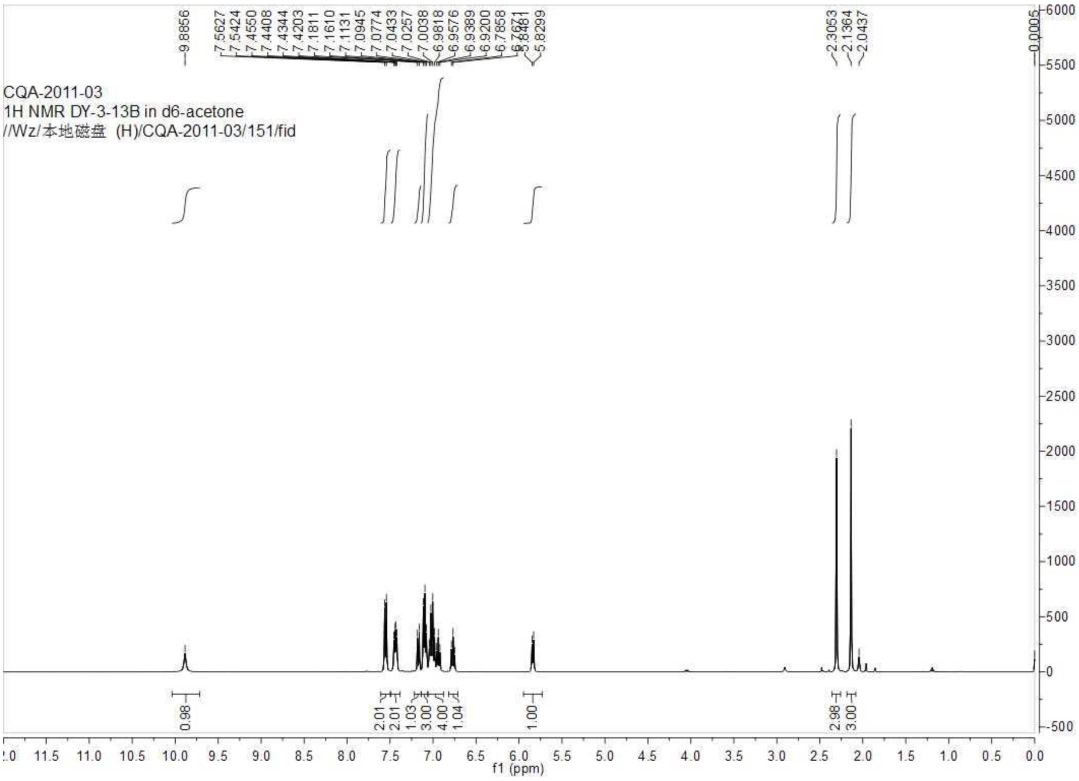
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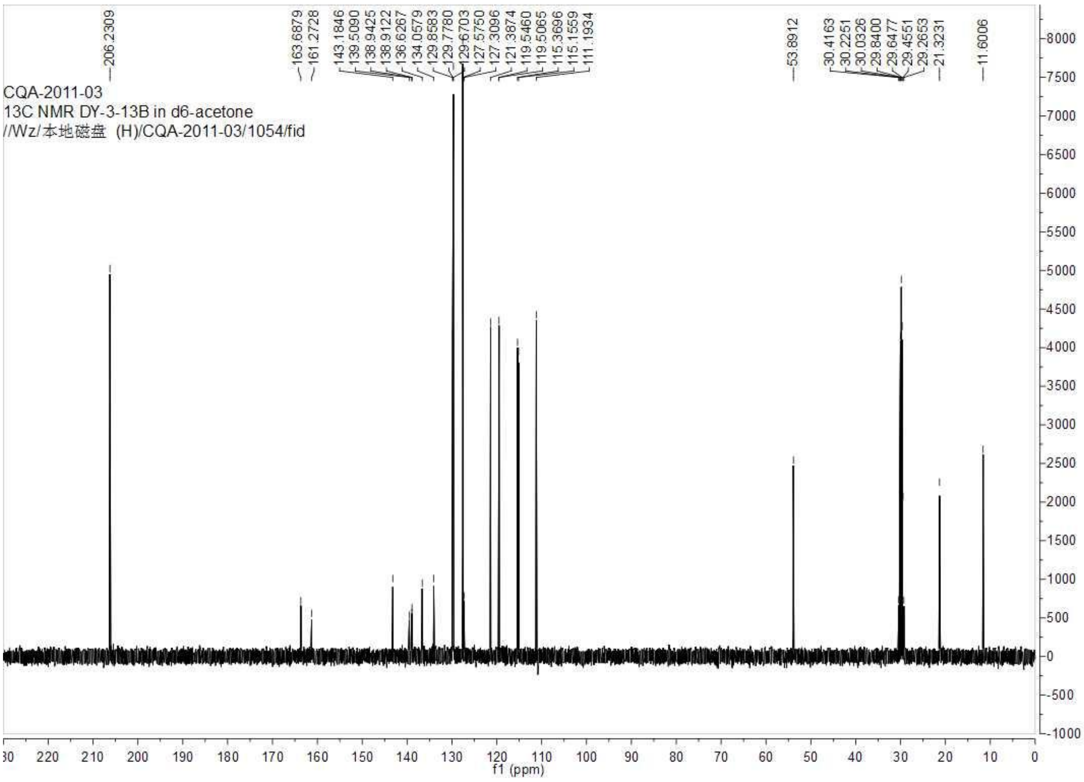
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Elemental Composition Report

Page 1

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5 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
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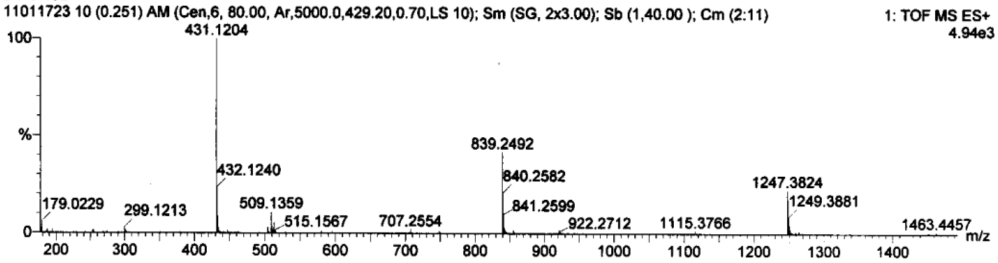

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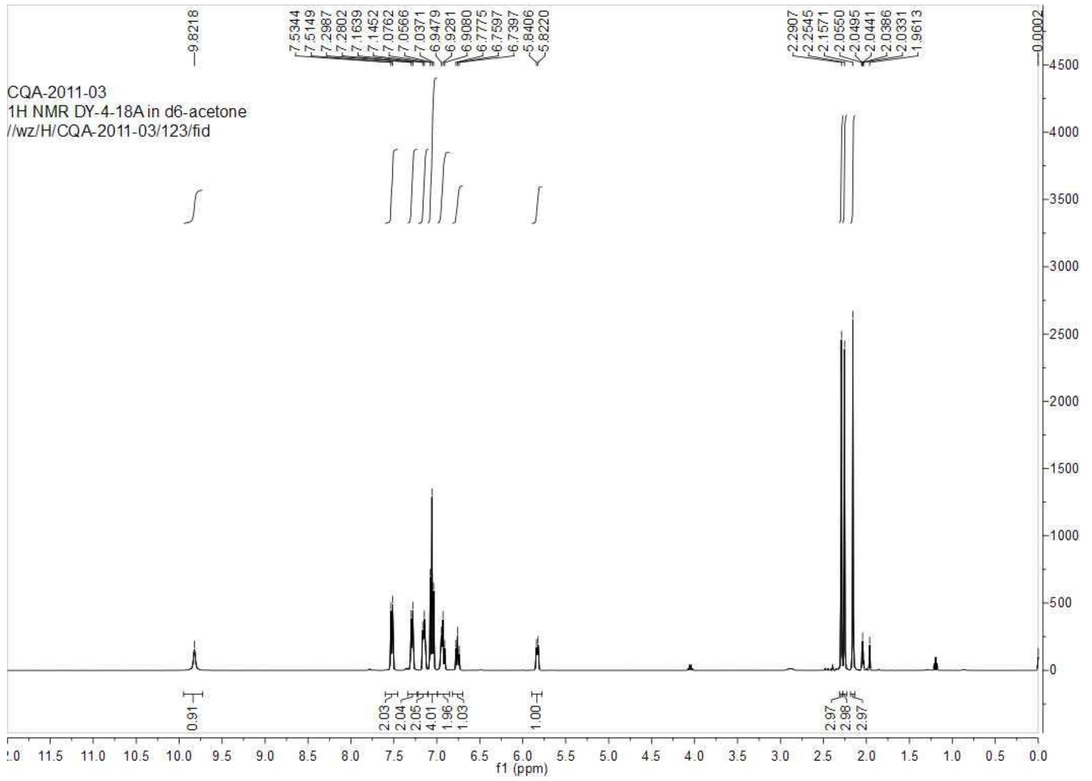
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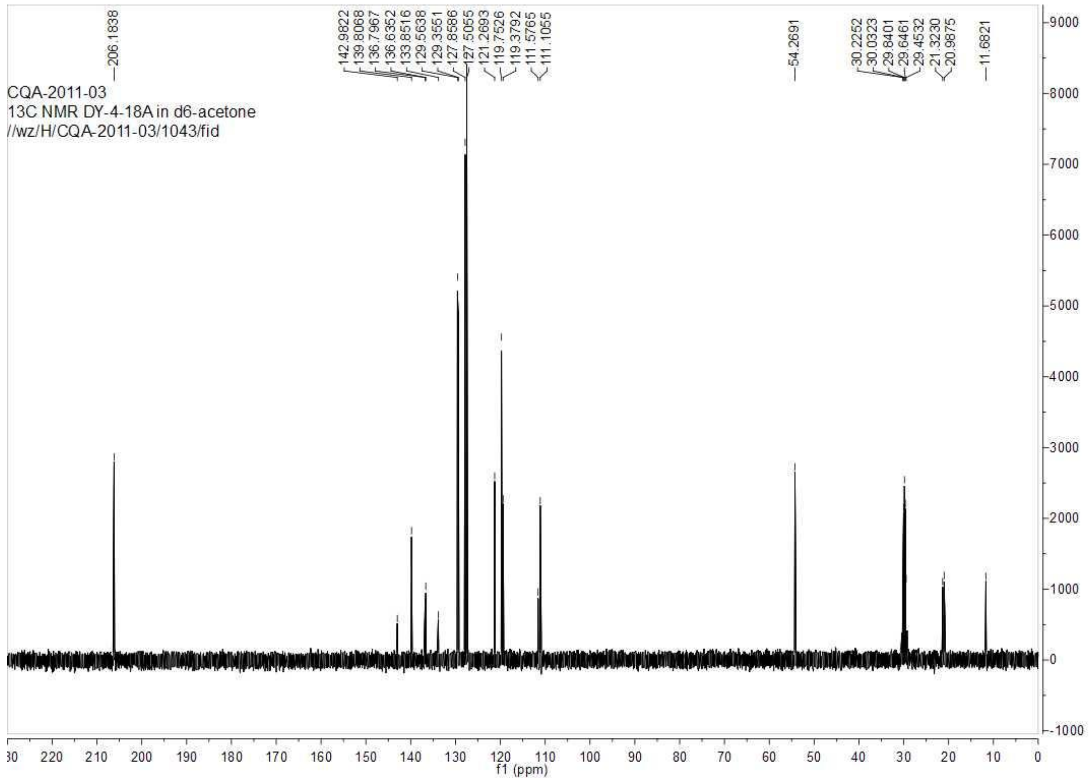
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Elemental Composition Report **Page 1**

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6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
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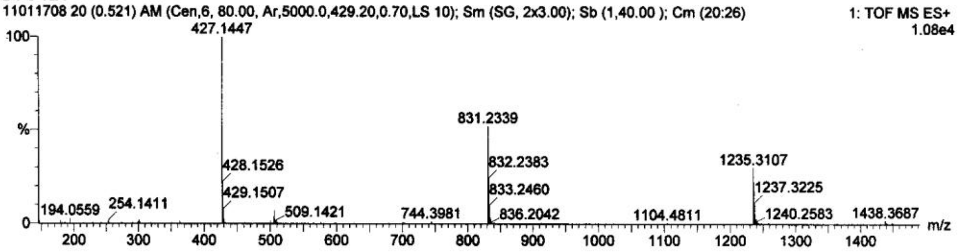

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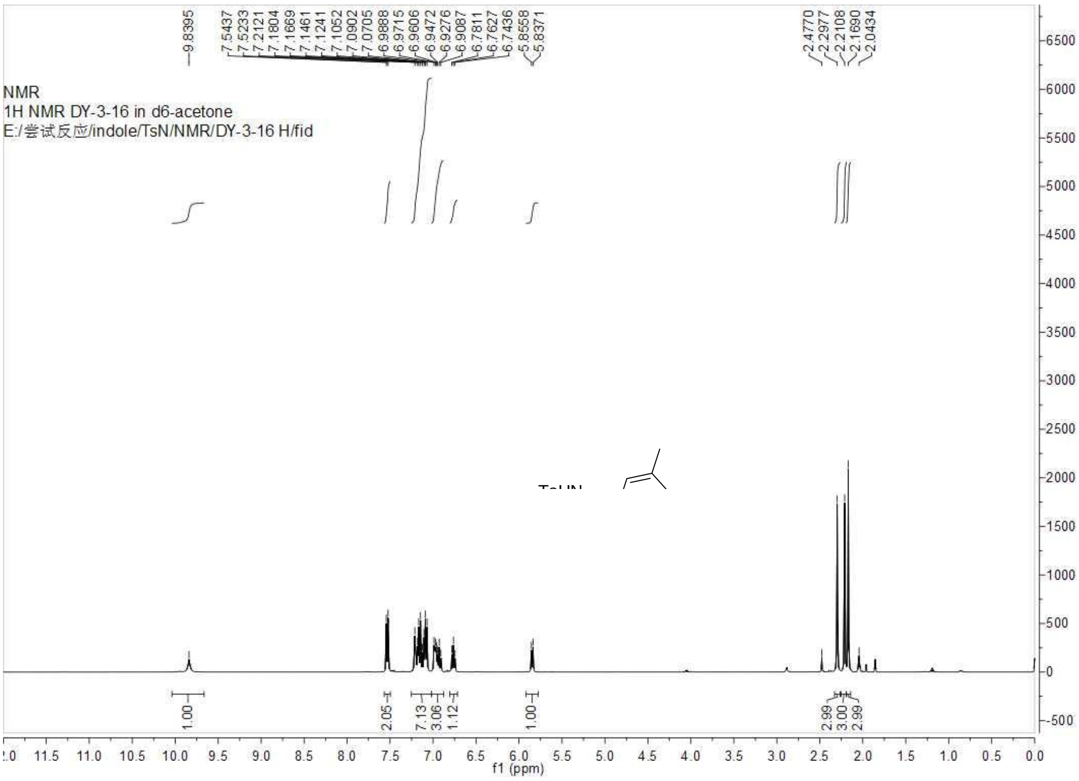
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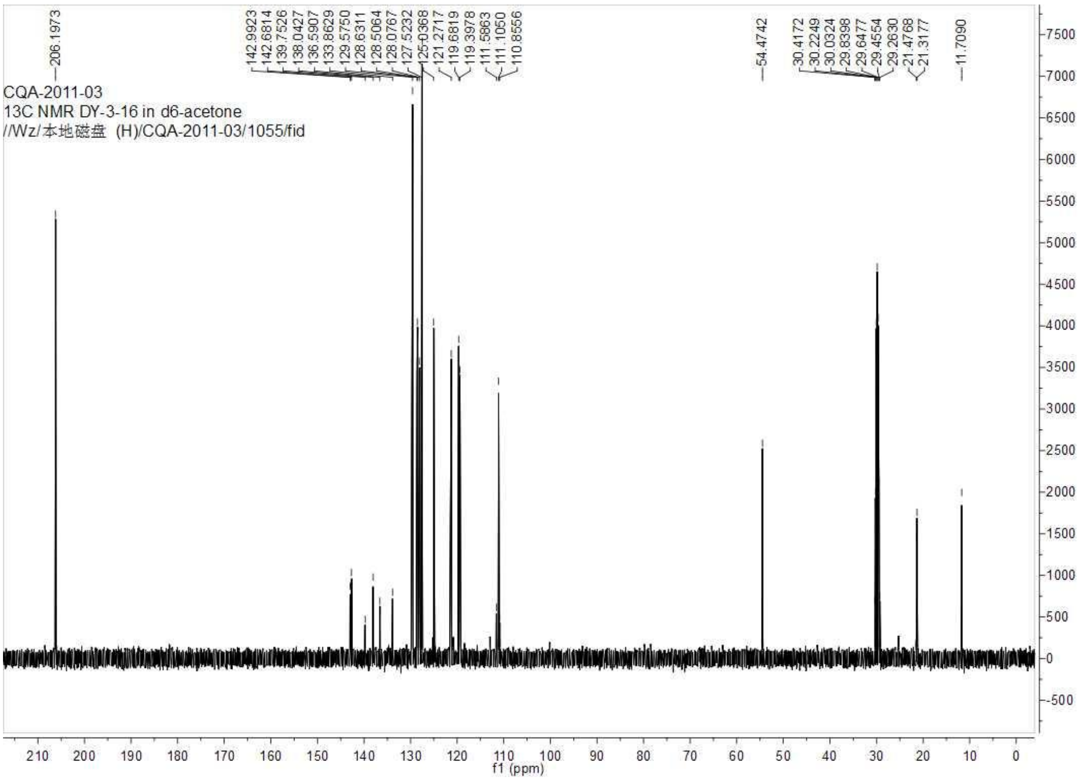
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Elemental Composition Report **Page 1**

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6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
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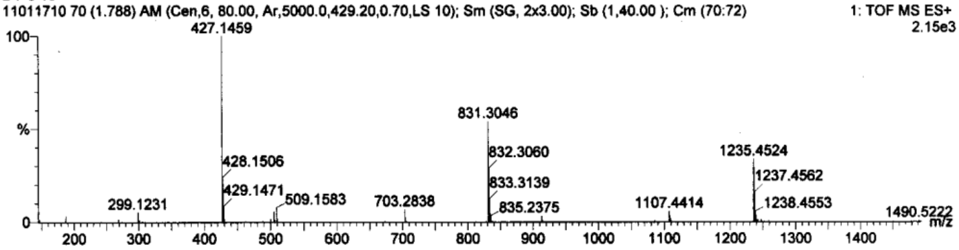

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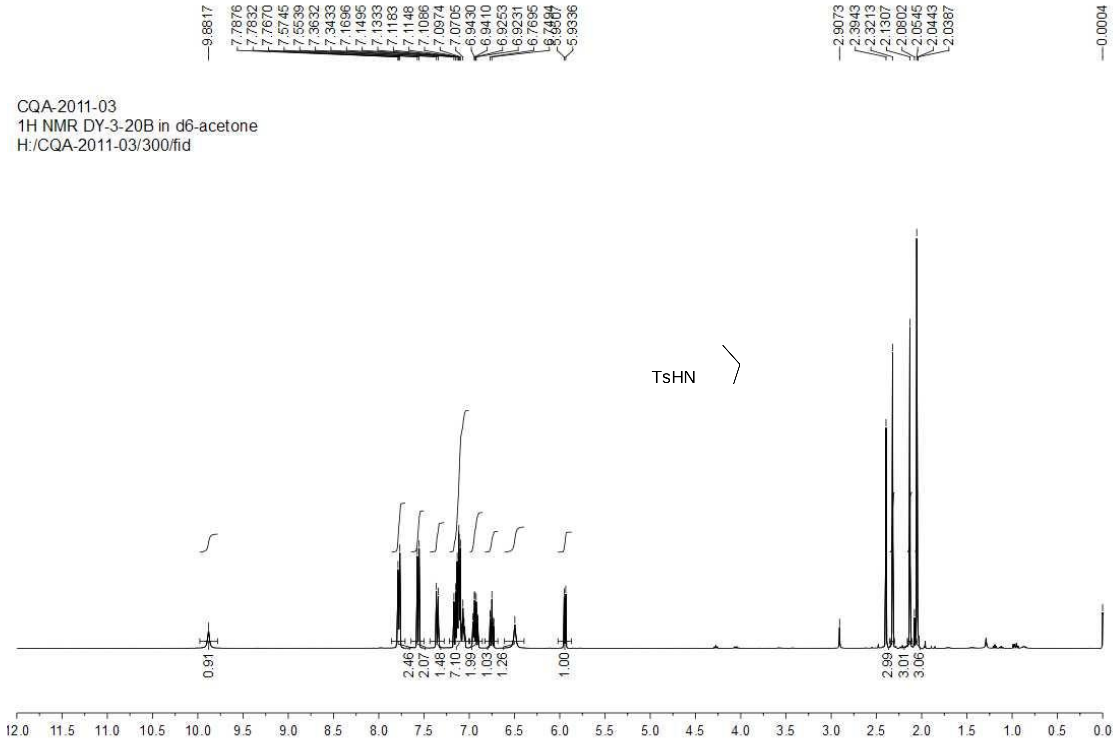
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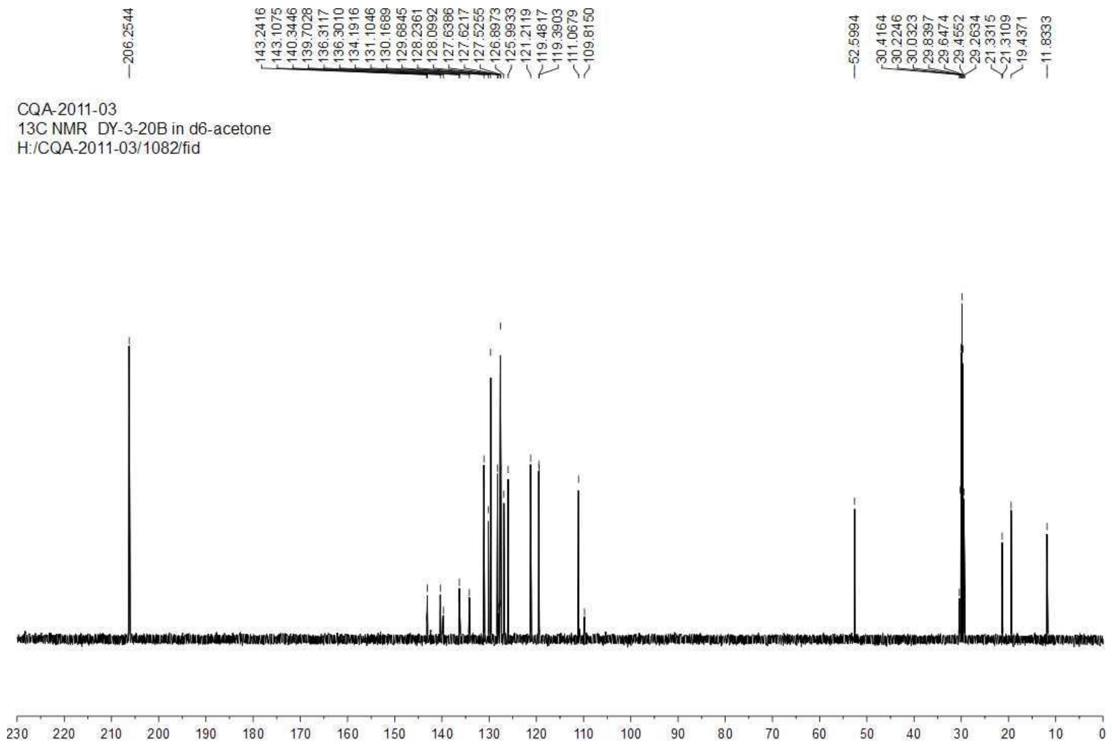
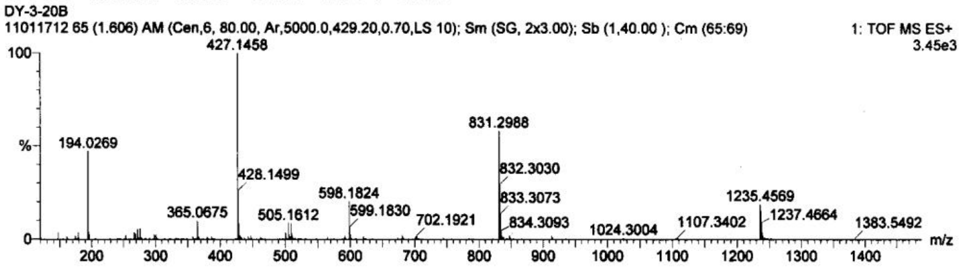

CQA-2011-03
13C NMR DY-3-20B in d6-acetone
H:/CQA-2011-03/1082/fid

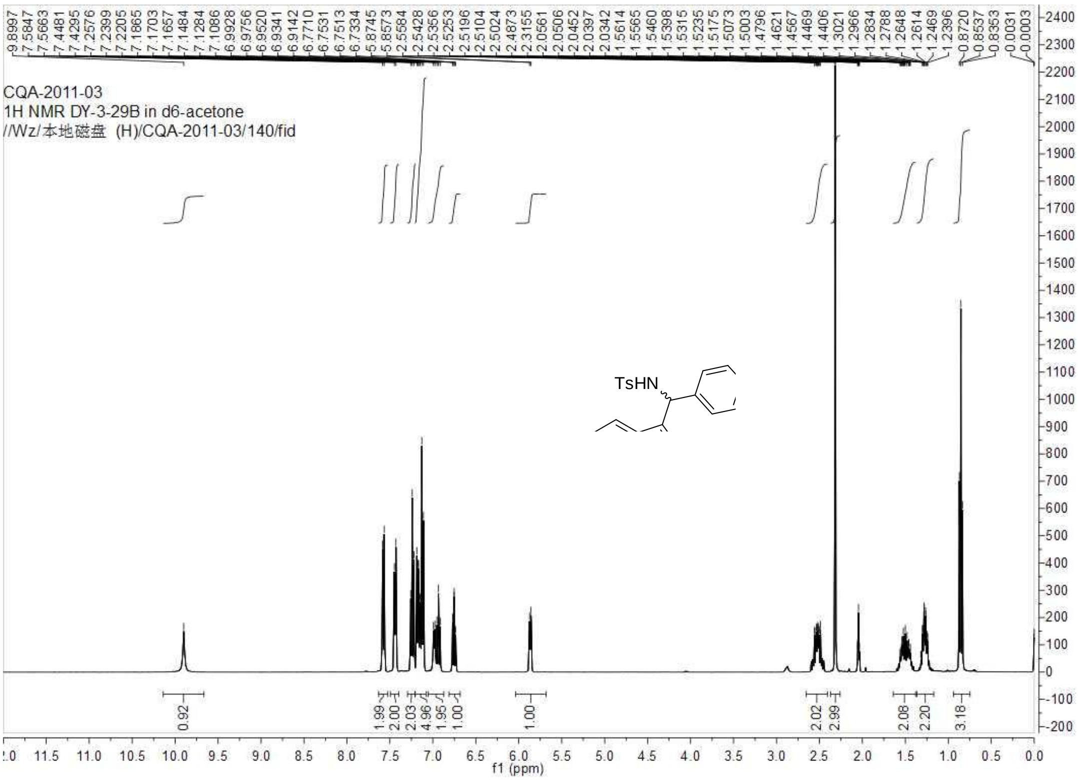
Elemental Composition Report **Page 1**

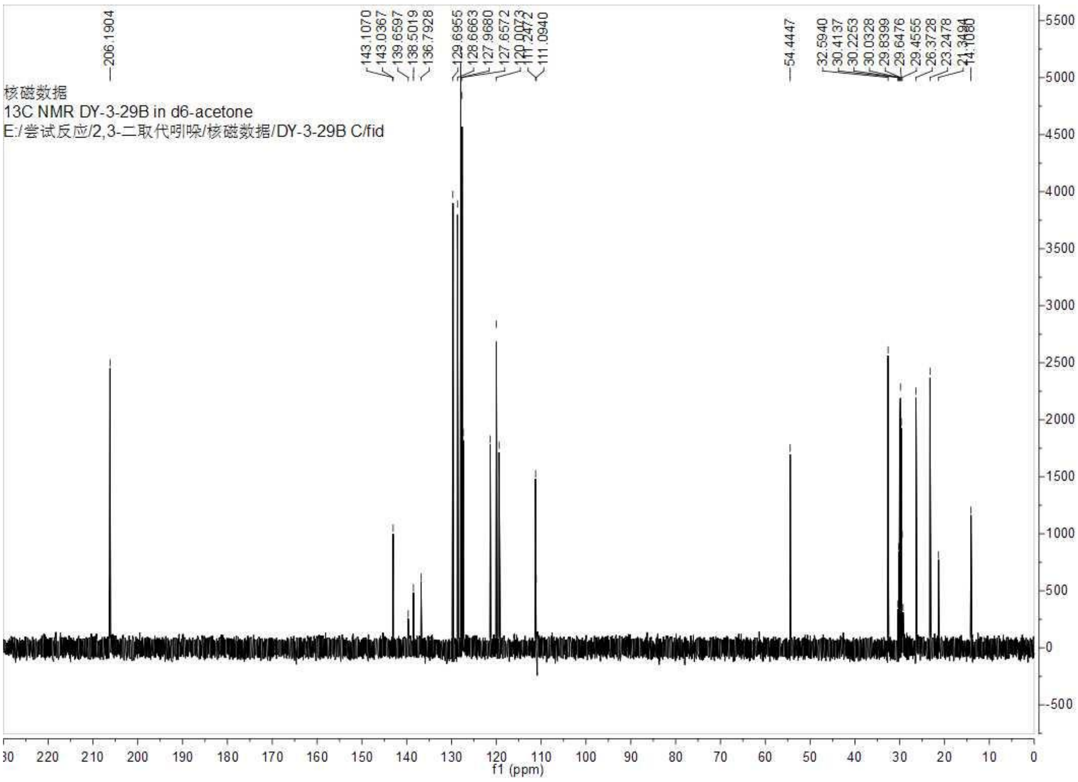
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -10.0, max = 100.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-120 N: 2-2 O: 2-2 Na: 1-1 S: 1-1



Minimum:				-10.0		
Maximum:		5.0	50.0	100.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
427.1458	427.1456	0.2	0.5	13.5	4.5	C24 H24 N2 O2 Na S

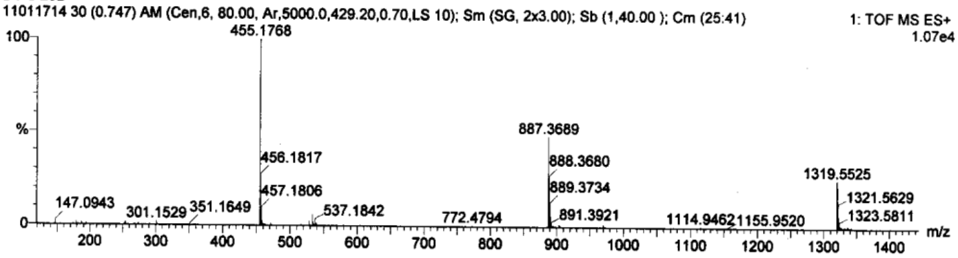

CQA-2011-03
1H NMR DY-3-29B in d6-acetone
//Wz/本地磁盘 (H)/CQA-2011-03/140/fid
TsHN
f1 (ppm)


核磁数据
13C NMR DY-3-29B in d6-acetone
f1 (ppm)

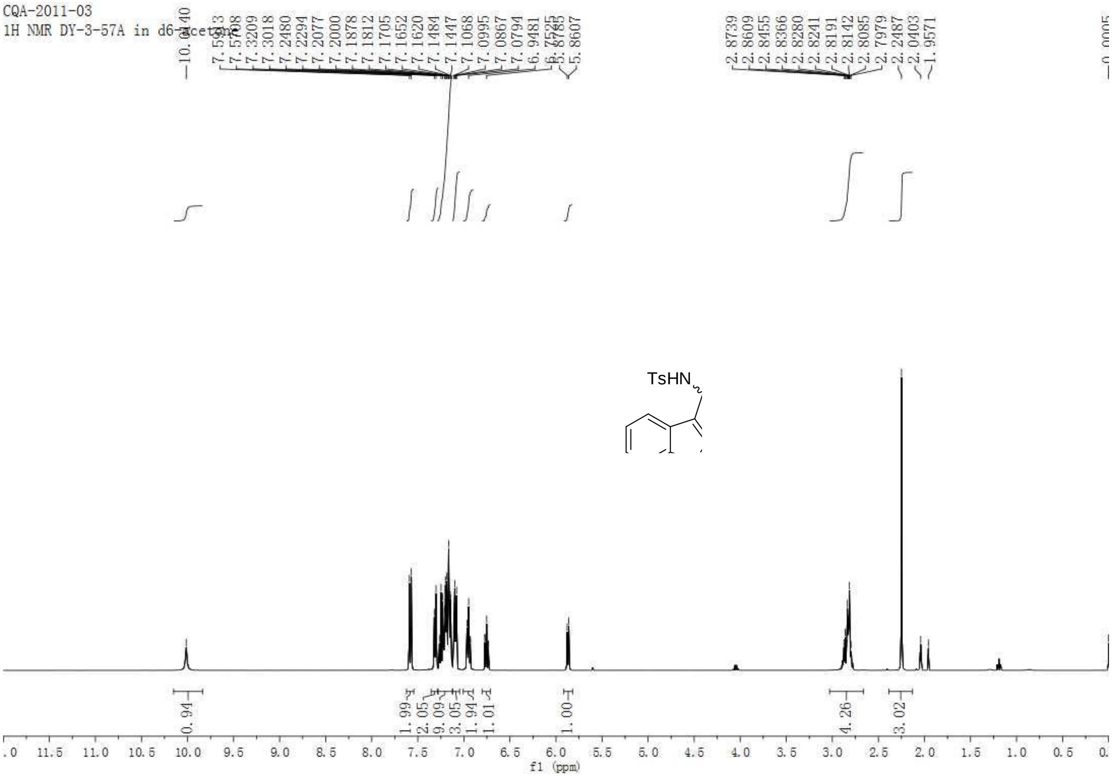
Elemental Composition Report **Page 1**

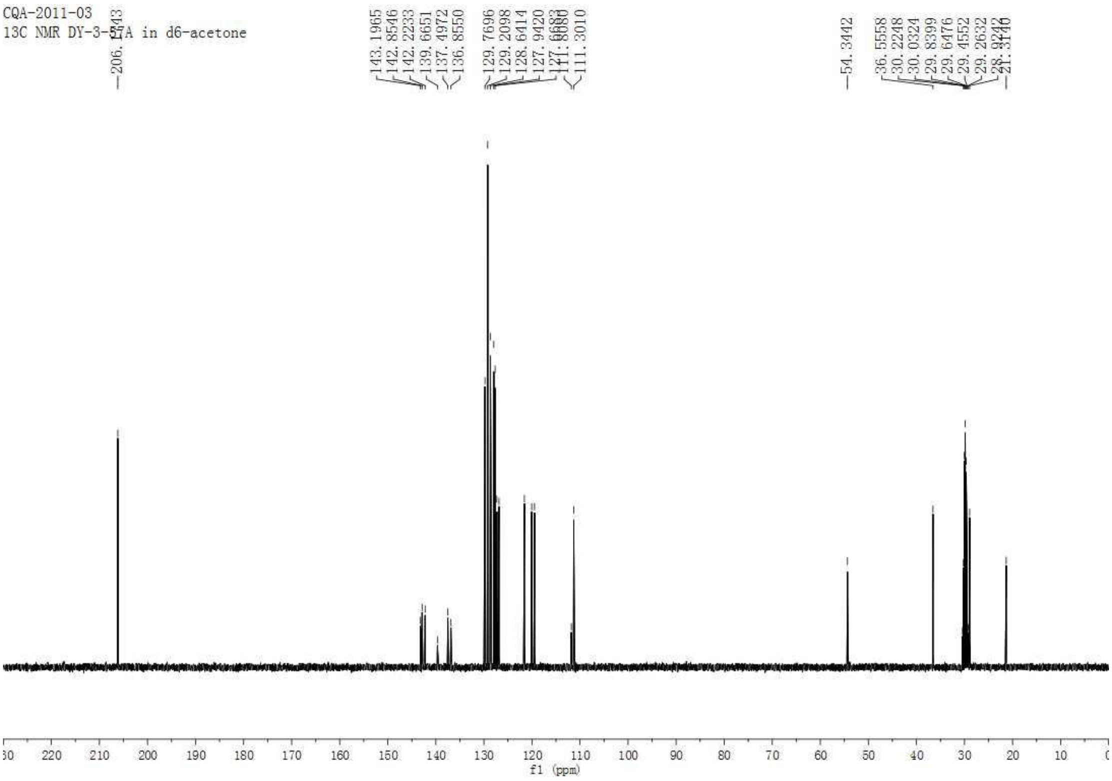
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -10.0, max = 100.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-120 N: 2-2 O: 2-2 Na: 1-1 S: 1-1
DY-3-29B
11011714 30 (0.747) AM (Cen,6, 80.00, Ar,5000.0,429.20,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (25:41)



Minimum:				-10.0		
Maximum:		5.0	50.0	100.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
455.1768	455.1769	-0.1	-0.2	13.5	20.0	C26 H28 N2 O2 Na S

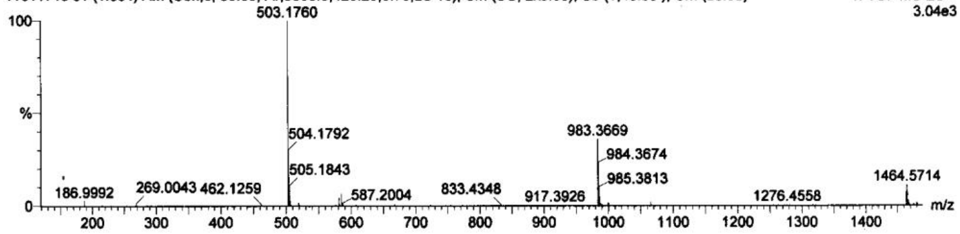

CQA-2011-03
1H NMR DY-3-57A in d6-acetone
TsHN
f1 (ppm)


CQA-2011-03
13C NMR DY-3-57A in d6-acetone
f1 (ppm)

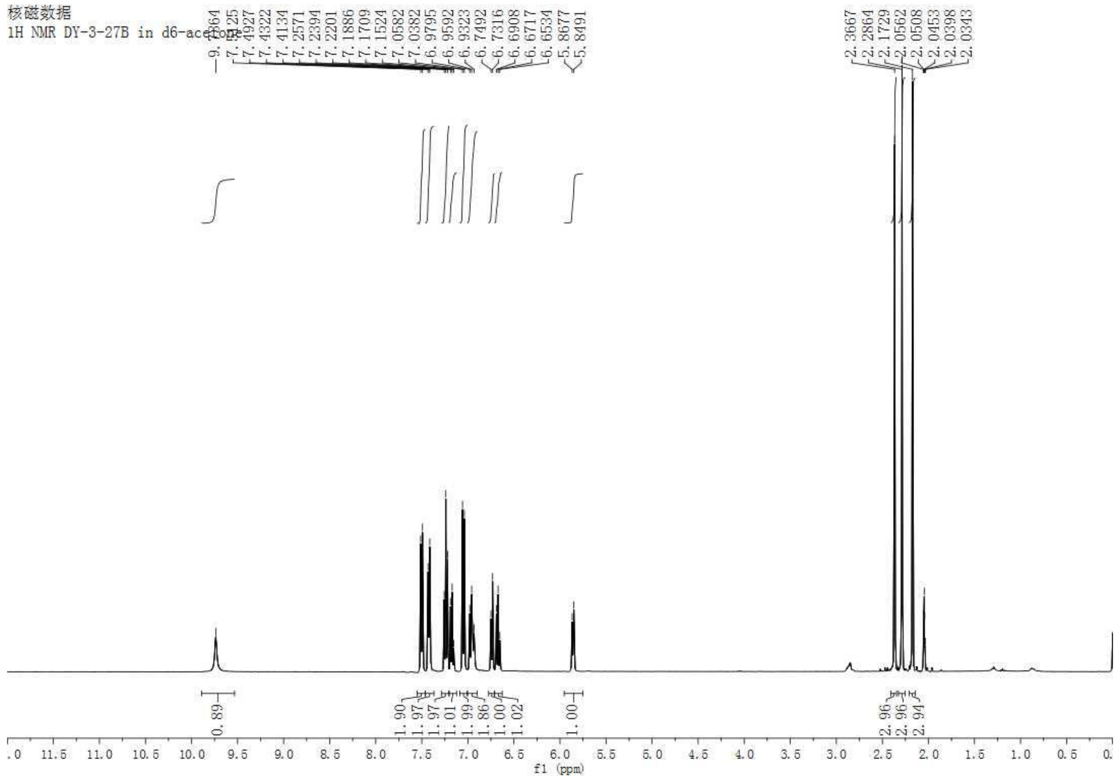
Elemental Composition Report · **Page 1**

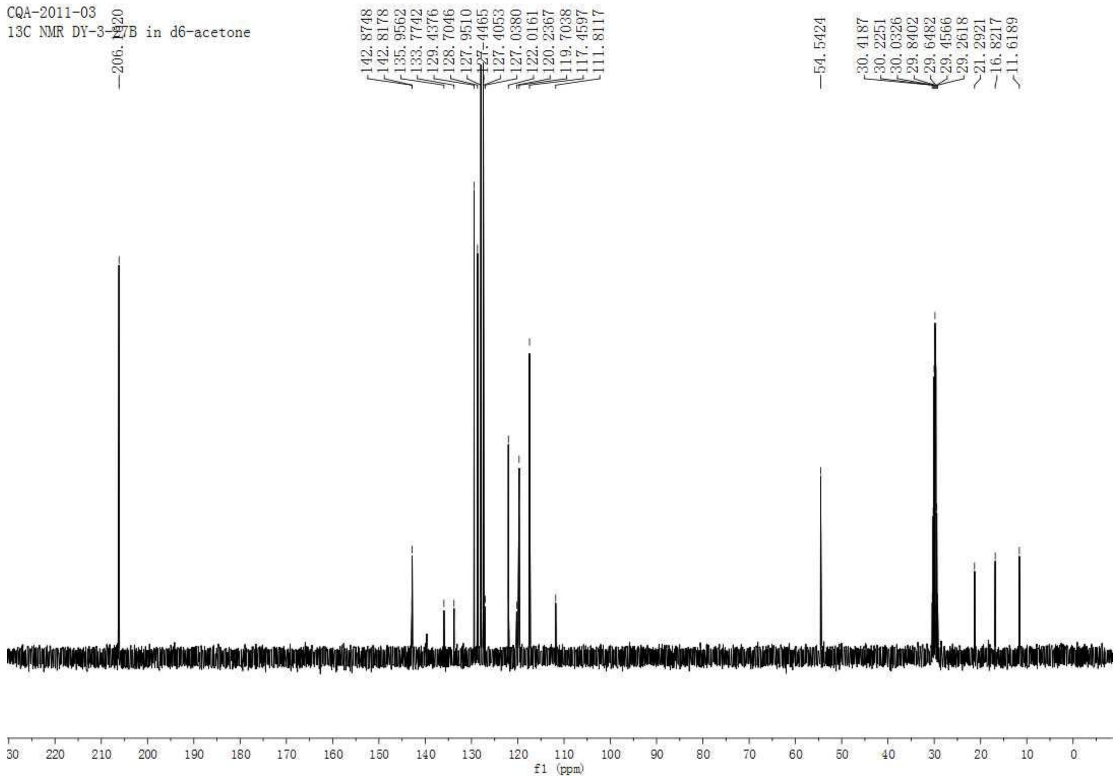
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -10.0, max = 100.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
7 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-120 N: 2-2 O: 2-2 Na: 1-1 S: 1-1
DY-3-57A
11011715 51 (1.304) AM (Cen,6, 80.00, Ar,5000.0,429.20,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (50:53) 1: TOF MS ES+ 3.04e3



Minimum:				-10.0		
Maximum:		5.0	50.0	100.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
503.1760	503.1769	-0.9	-1.8	17.5	10.4	C30 H28 N2 O2 Na S

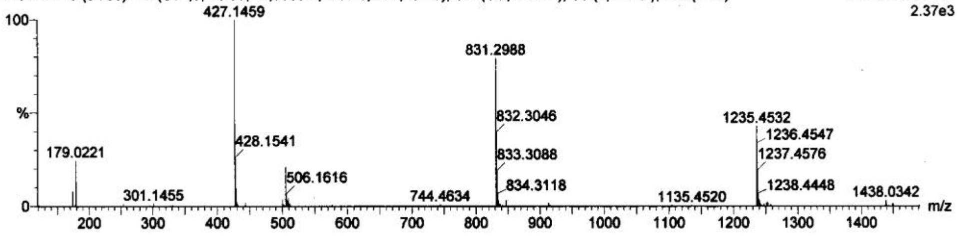

核磁数据
1H NMR DY-3-27B in d6-acetone
f1 (ppm)


CQA-2011-03
13C NMR DY-3-27B in d6-acetone
f1 (ppm)

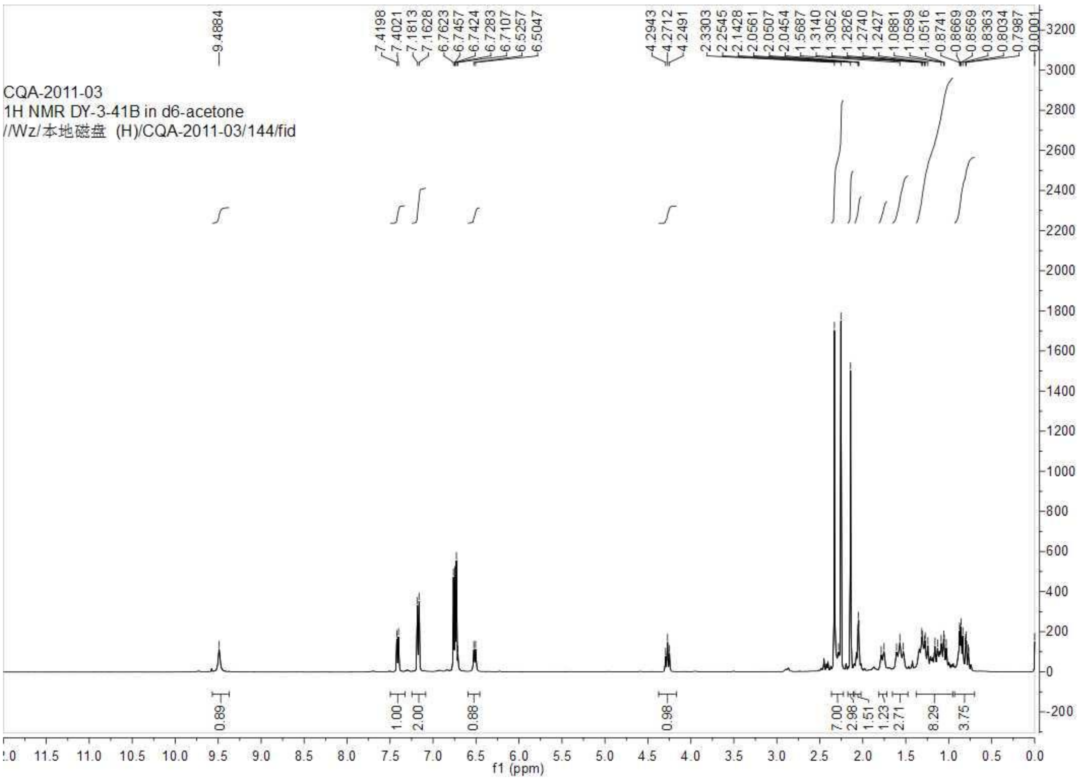
Elemental Composition Report **Page 1**

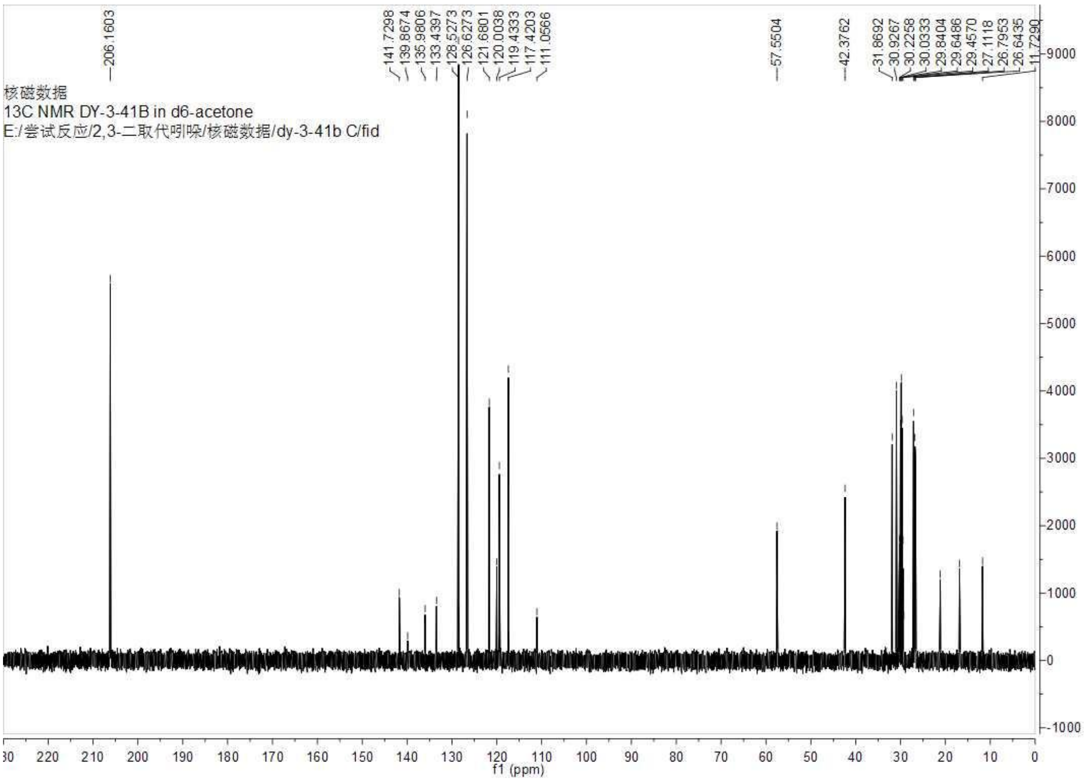
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -10.0, max = 100.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-120 N: 2-2 O: 2-2 Na: 1-1 S: 1-1
DY-3-27B
11011717 9 (0.230) AM (Cen,6, 80.00, Ar,5000.0,429.20,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (7:11) 1: TOF MS ES+ 2.37e3



Minimum:				-10.0		
Maximum:		5.0	50.0	100.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
427.1459	427.1456	0.3	0.7	13.5	3.1	C24 H24 N2 O2 Na S

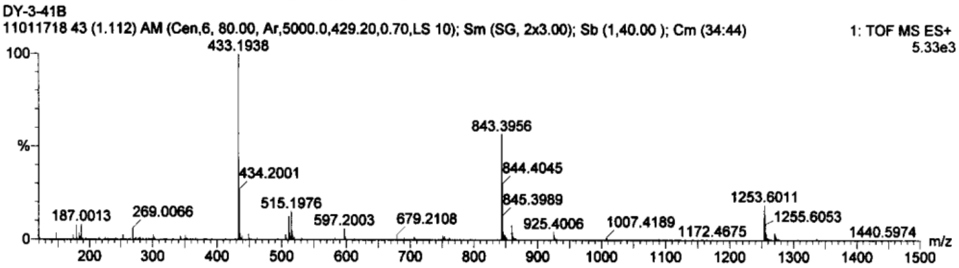

CQA-2011-03
1H NMR DY-3-41B in d6-acetone
//Wz/本地磁盘 (H)/CQA-2011-03/144/fid
f1 (ppm)


核磁数据
13C NMR DY-3-41B in d6-acetone
E:/尝试反应/2,3-二取代吲哚/核磁数据/dy-3-41b C/fid
f1 (ppm)

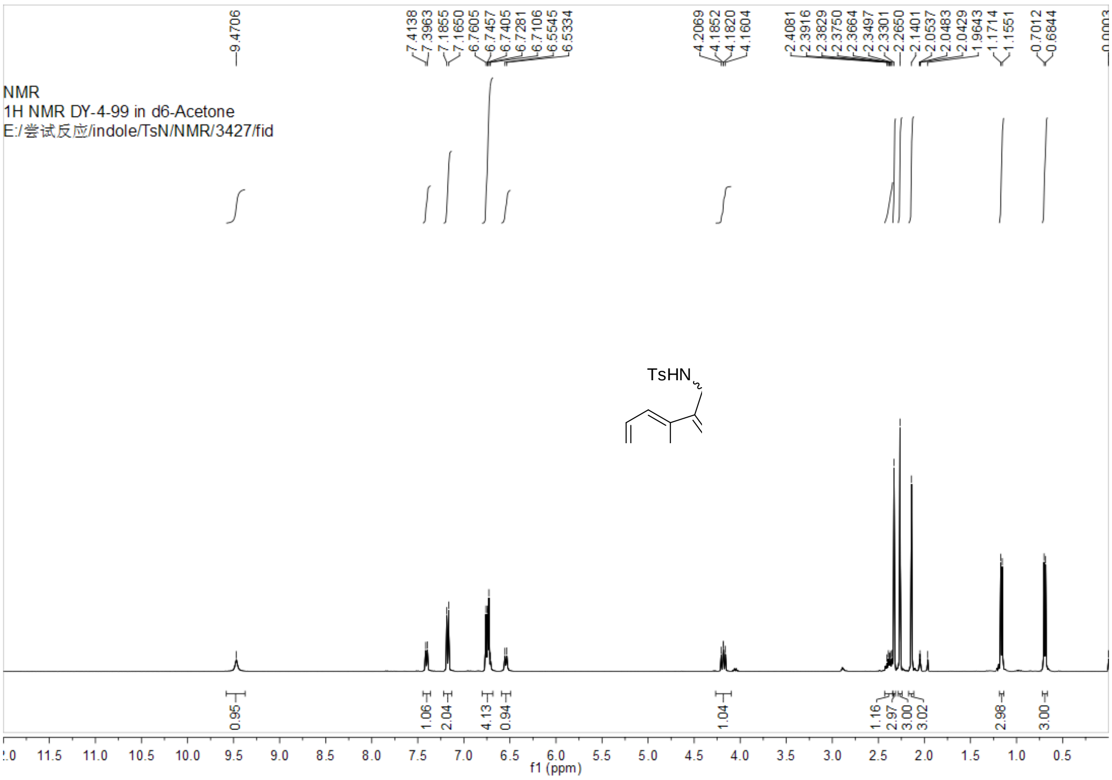
Elemental Composition Report **Page 1**

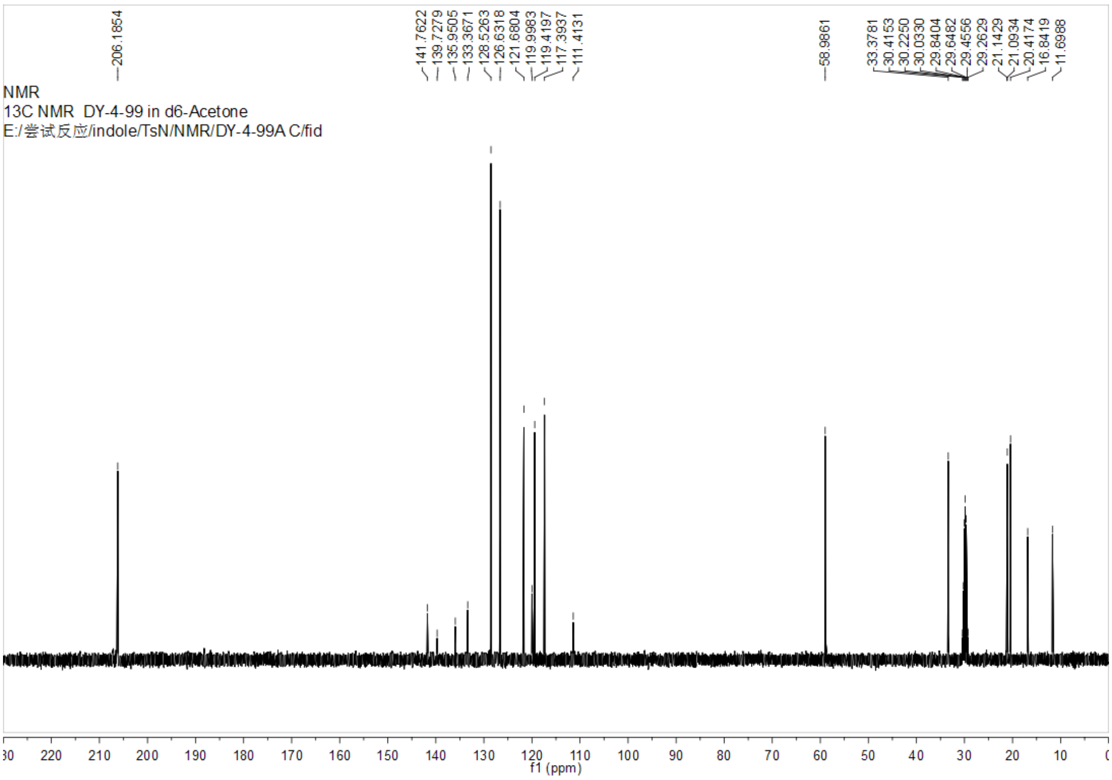
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -10.0, max = 100.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-120 N: 2-2 O: 2-2 Na: 1-1 S: 1-1



Minimum:				-10.0		
Maximum:		5.0	50.0	100.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
433.1938	433.1926	1.2	2.8	10.5	4.1	C24 H30 N2 O2 Na S

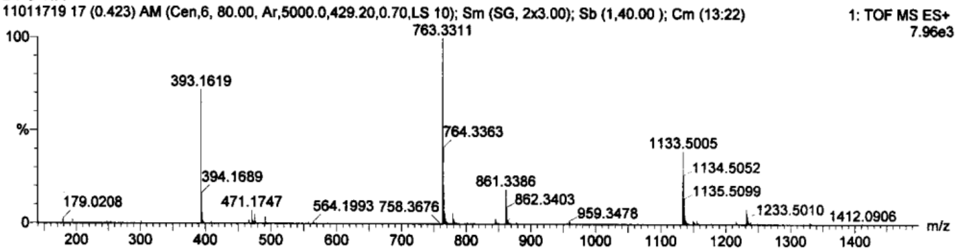

NMR
1H NMR DY-4-99 in d6-Acetone
E:/尝试反应/indole/TsN/NMR/3427/fid
TsHN
f1 (ppm)


NMR
13C NMR DY-4-99 in d6-Acetone
E:/尝试反应/indole/TsN/NMR/DY-4-99A C/fid
f1 (ppm)

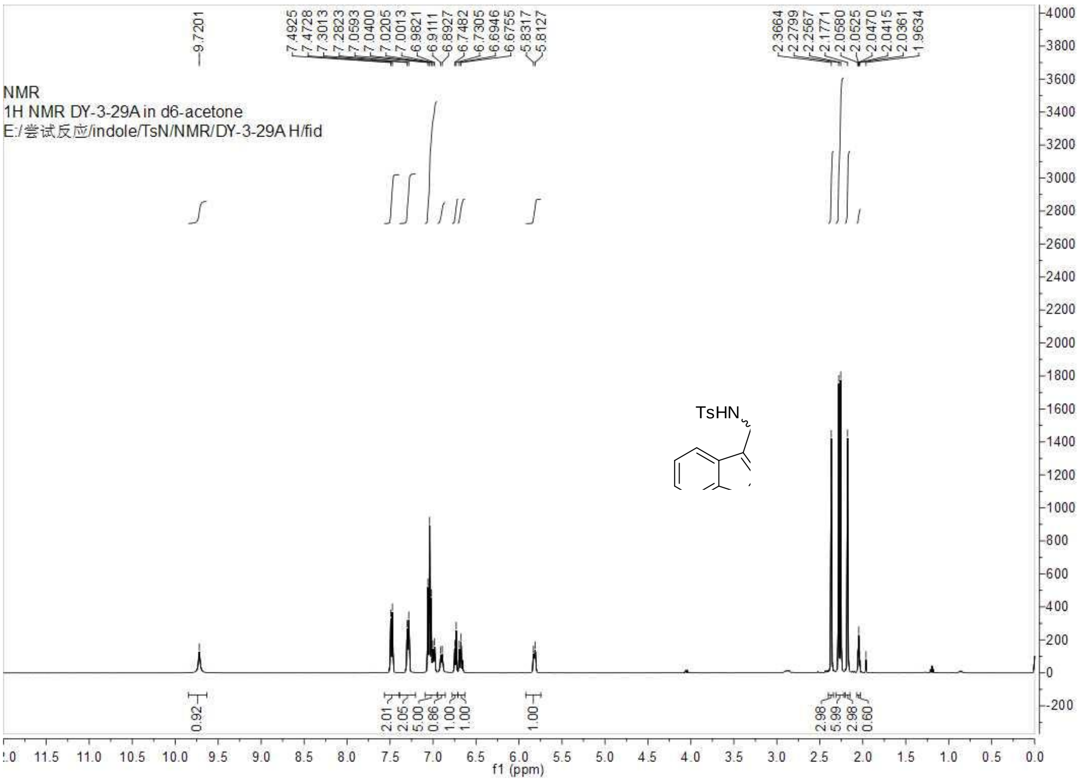
Elemental Composition Report **Page 1**

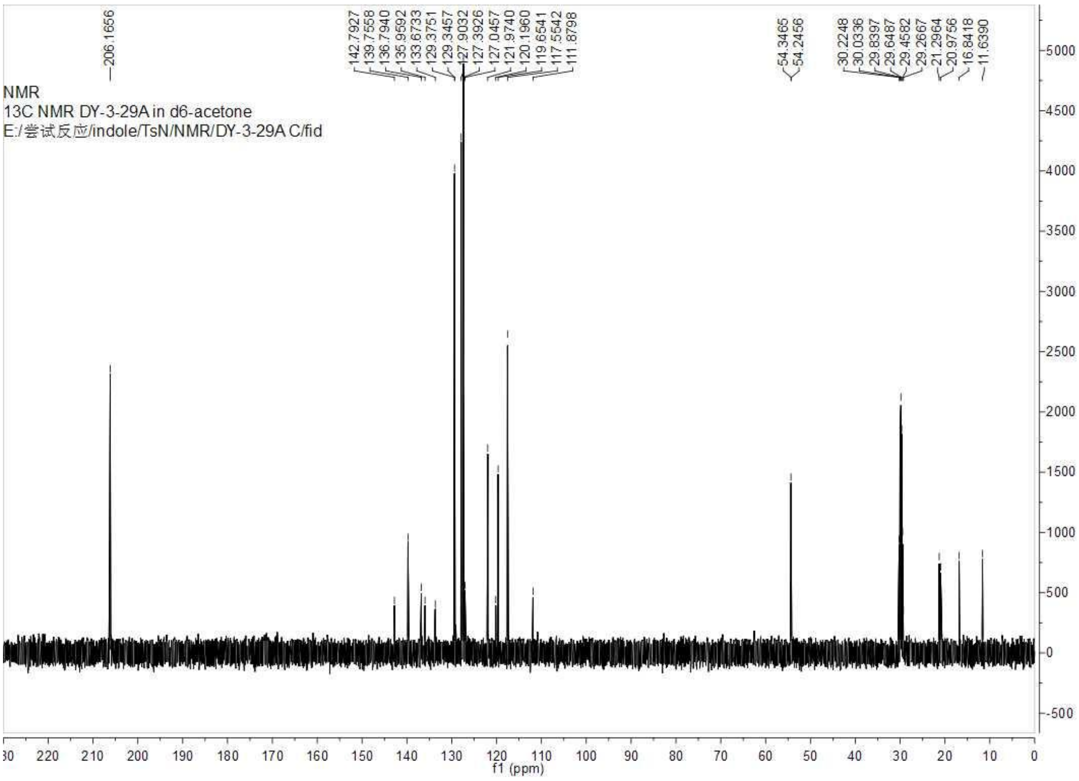
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -10.0, max = 100.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-120 N: 2-2 O: 2-2 Na: 1-1 S: 1-1
DY-3-41A
11011719 17 (0.423) AM (Cen,6, 80.00, Ar,5000.0,429.20,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (13:22)



Minimum:				-10.0		
Maximum:		5.0	50.0	100.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
393.1619	393.1613	0.6	1.5	9.5	7.9	C21 H26 N2 O2 Na S


NMR
1H NMR DY-3-29A in d6-acetone
E:/尝试反应/indole/TsN/NMR/DY-3-29A H/fid
TsHN
f1 (ppm)

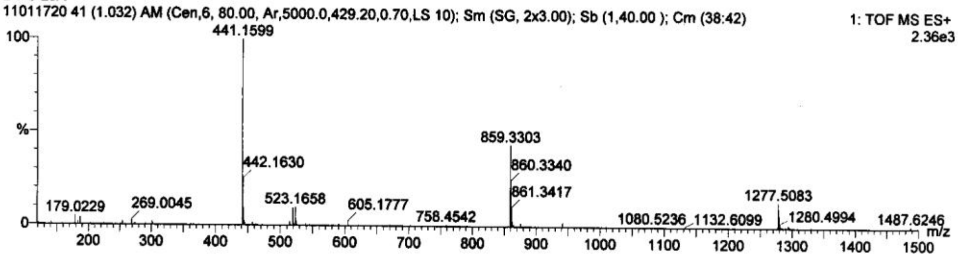

NMR
13C NMR DY-3-29A in d6-acetone
E:/尝试反应/indole/TsN/NMR/DY-3-29A C/fid
f1 (ppm)

Elemental Composition Report

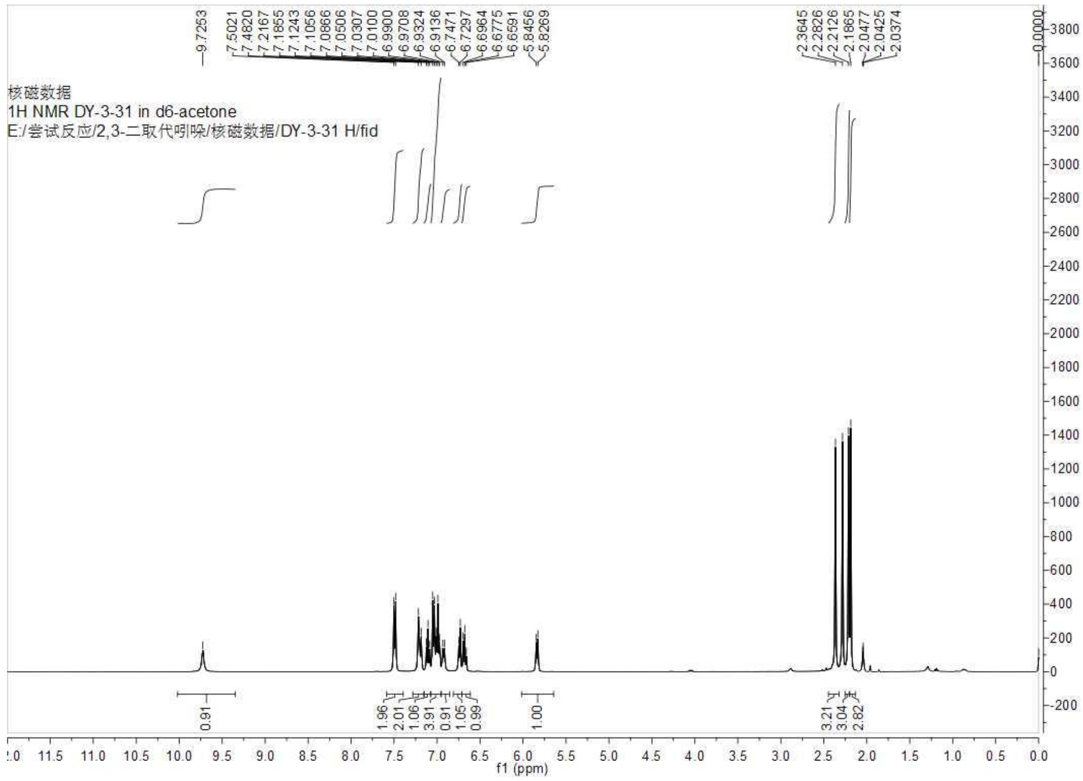
Page 1

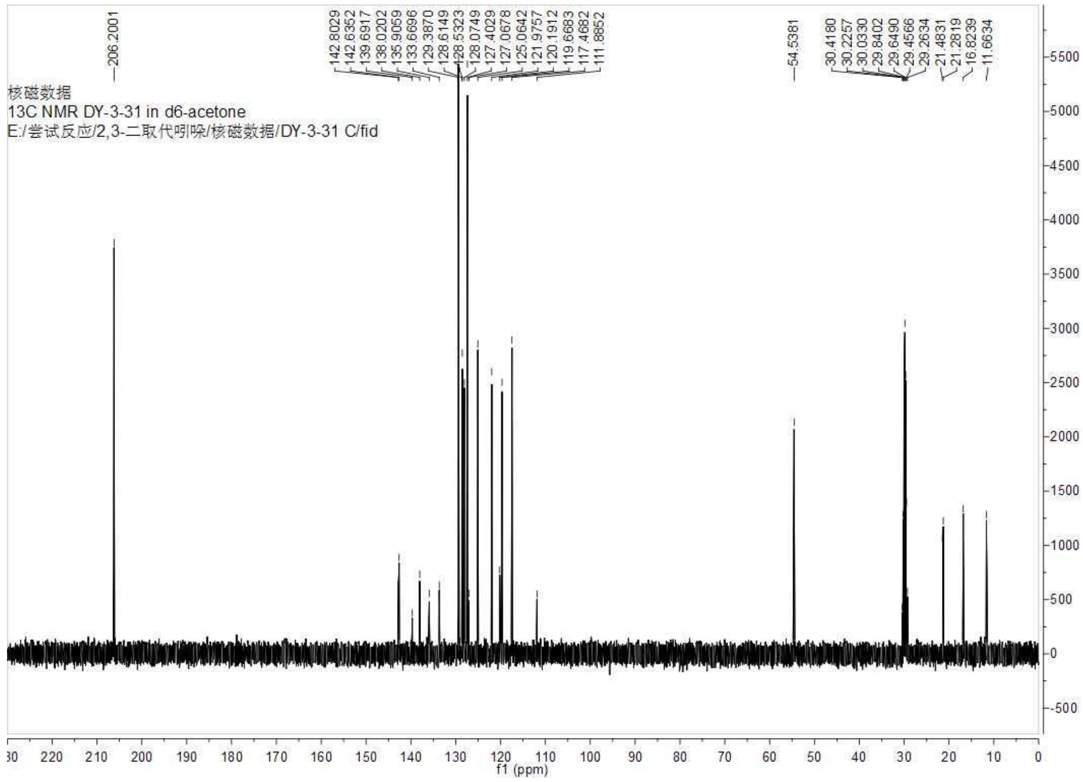
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -10.0, max = 100.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-120 N: 2-2 O: 2-2 Na: 1-1 S: 1-1
DY-3-29A
11011720 41 (1.032) AM (Cen,6, 80.00, Ar,5000.0,429.20,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (38:42)



Minimum:				-10.0		
Maximum:		5.0	50.0	100.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
441.1599	441.1613	-1.4	-3.2	13.5	8.6	C25 H26 N2 O2 Na S

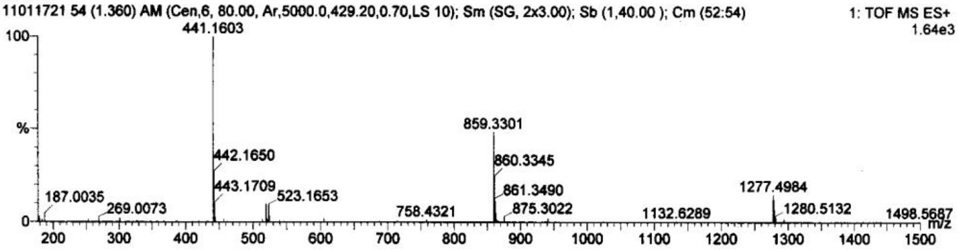

核磁数据
1H NMR DY-3-31 in d6-acetone
E:/尝试反应/2,3-二取代吲哚/核磁数据/DY-3-31 H/fid
f1 (ppm)


核磁数据
13C NMR DY-3-31 in d6-acetone
E:/尝试反应/2,3-二取代吲哚/核磁数据/DY-3-31 C/fid
f1 (ppm)

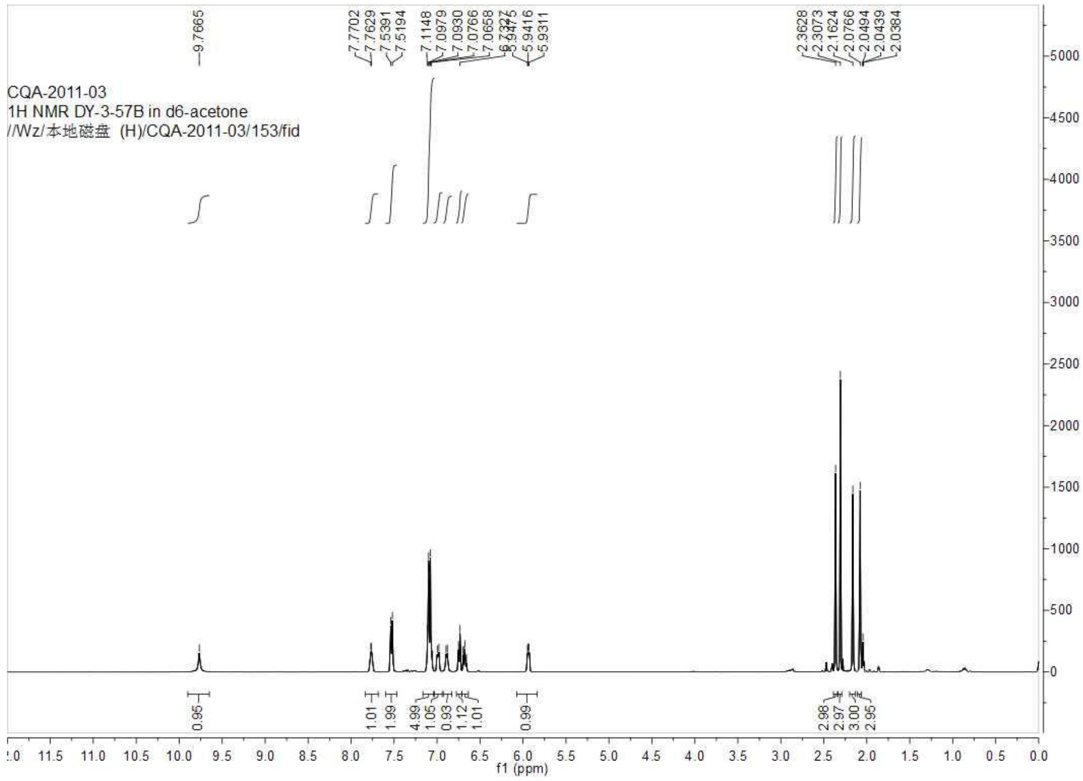
Elemental Composition Report **Page 1**

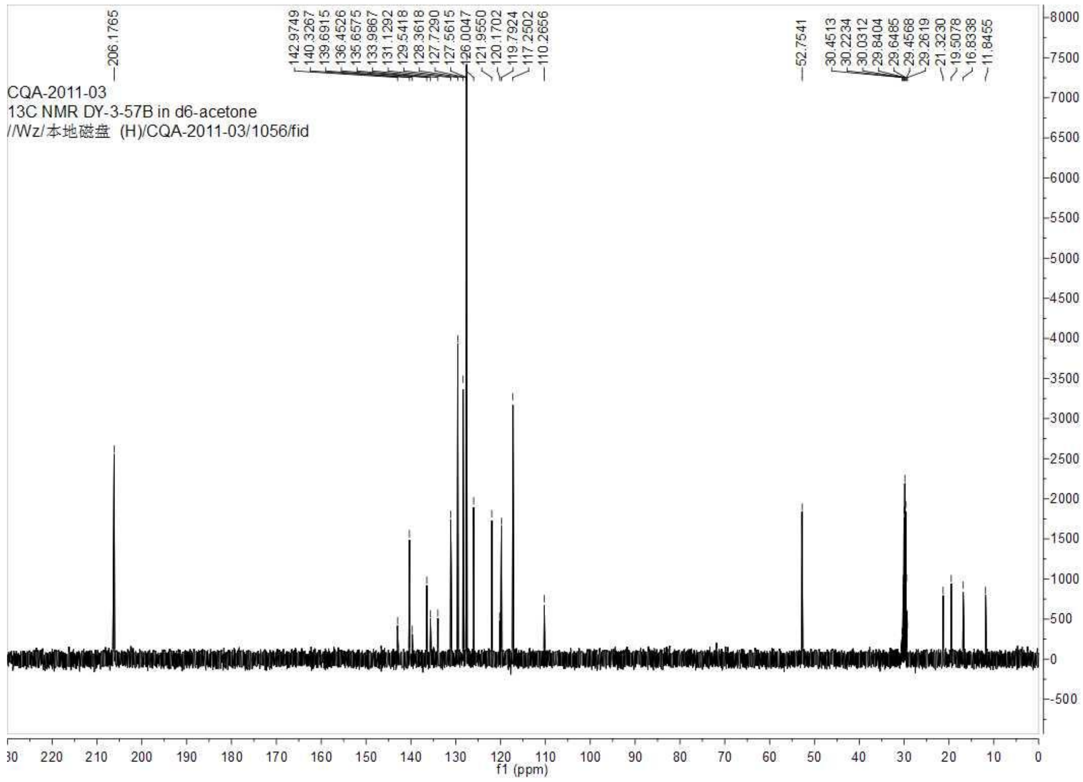
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -10.0, max = 100.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-120 N: 2-2 O: 2-2 Na: 1-1 S: 1-1
DY-3-31
11011721 54 (1.360) AM (Cen,6, 80.00, Ar,5000.0,429.20,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (52:54) 1: TOF MS ES+ 1.64e3



Minimum:				-10.0		
Maximum:		5.0	50.0	100.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
441.1603	441.1613	-1.0	-2.3	13.5	3.6	C25 H26 N2 O2 Na S

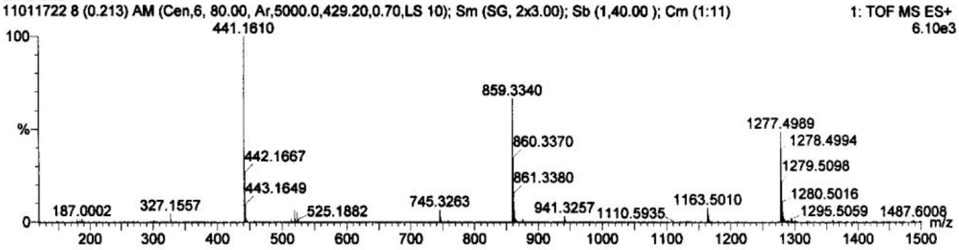

CQA-2011-03
1H NMR DY-3-57B in d6-acetone
//Wz/本地磁盘 (H)/CQA-2011-03/153/fid
f1 (ppm)


CQA-2011-03
13C NMR DY-3-57B in d6-acetone
//Wz/本地磁盘 (H)/CQA-2011-03/1056/fid
f1 (ppm)

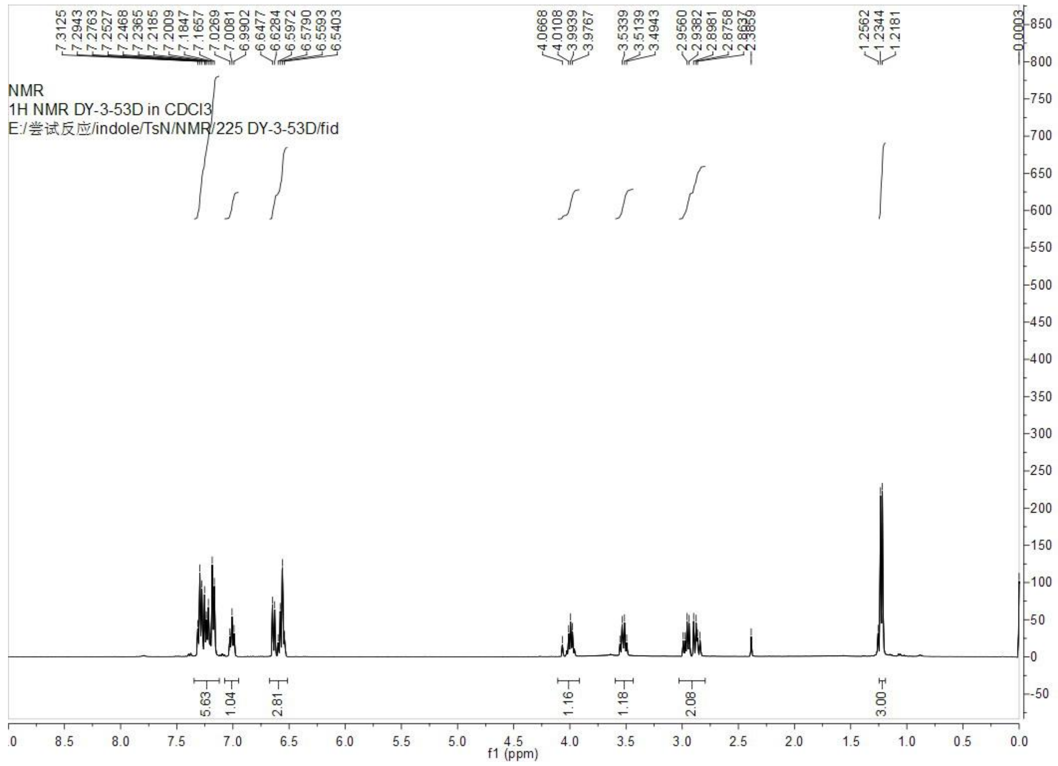
Elemental Composition Report Page 1

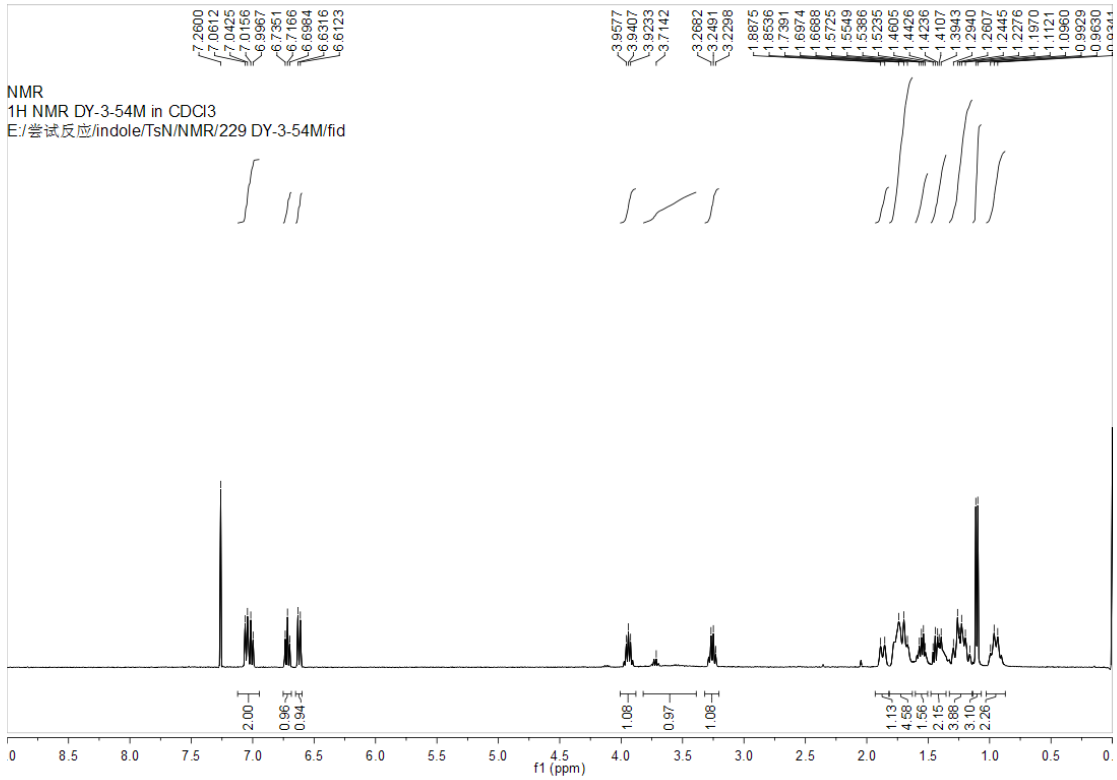
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -10.0, max = 100.0
Selected filters: None

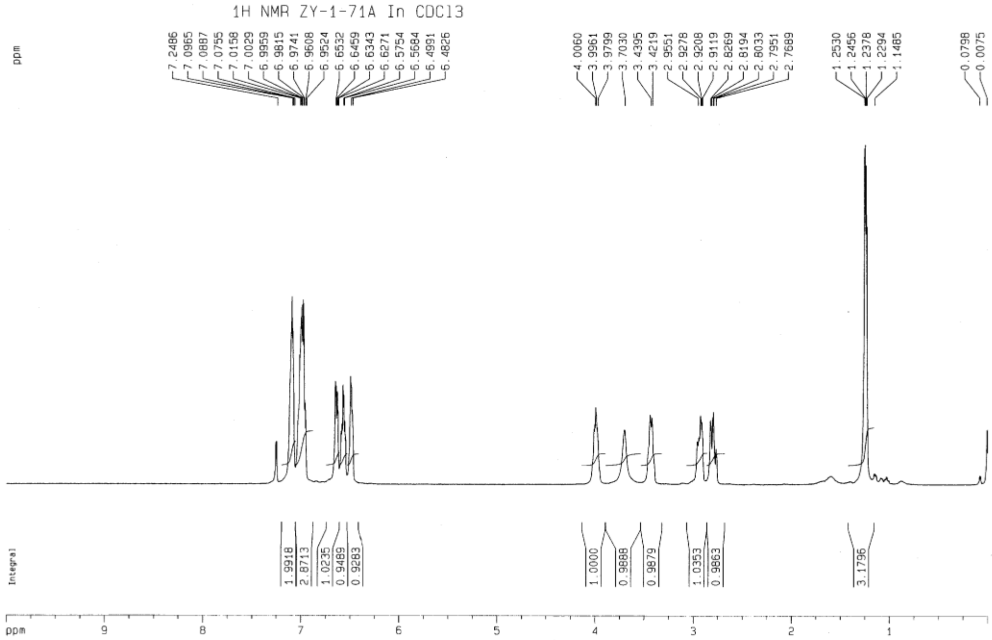
Monoisotopic Mass, Even Electron Ions
6 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-120 N: 2-2 O: 2-2 Na: 1-1 S: 1-1
DY-3-57B
11011722 8 (0.213) AM (Cen,6, 80.00, Ar,5000.0,429.20,0.70,LS 10); Sm (SG, 2x3.00); Sb (1,40.00); Cm (1:11) 1: TOF MS ES+ 6.10e3

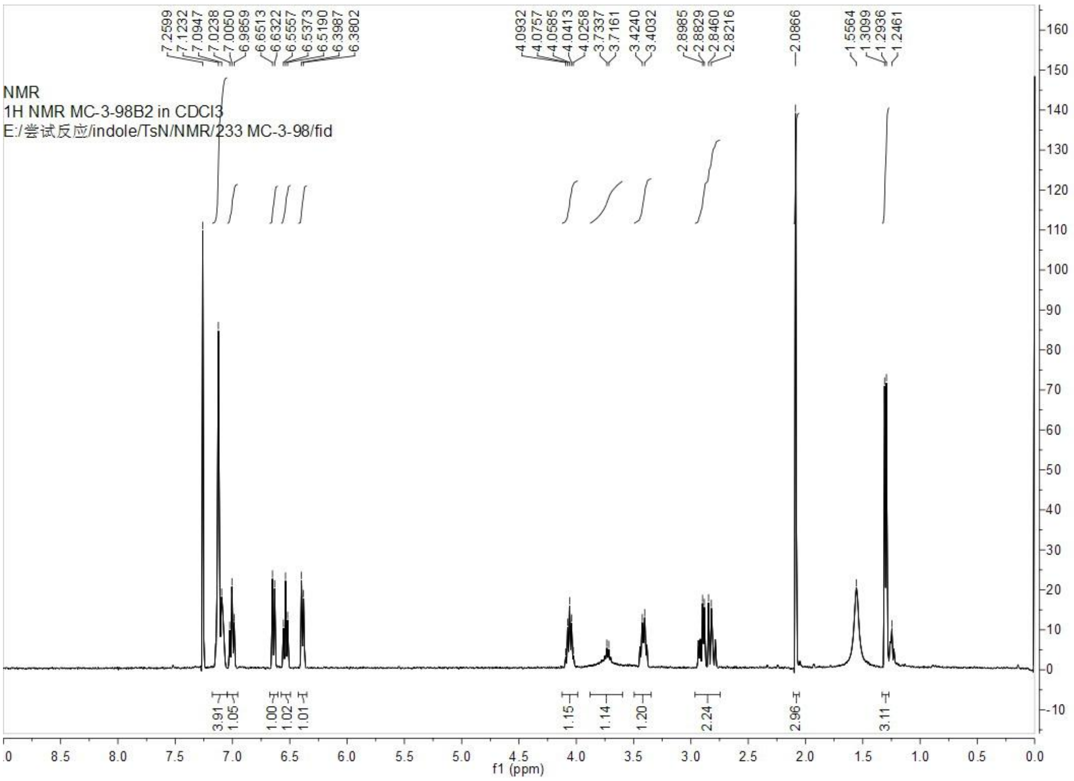


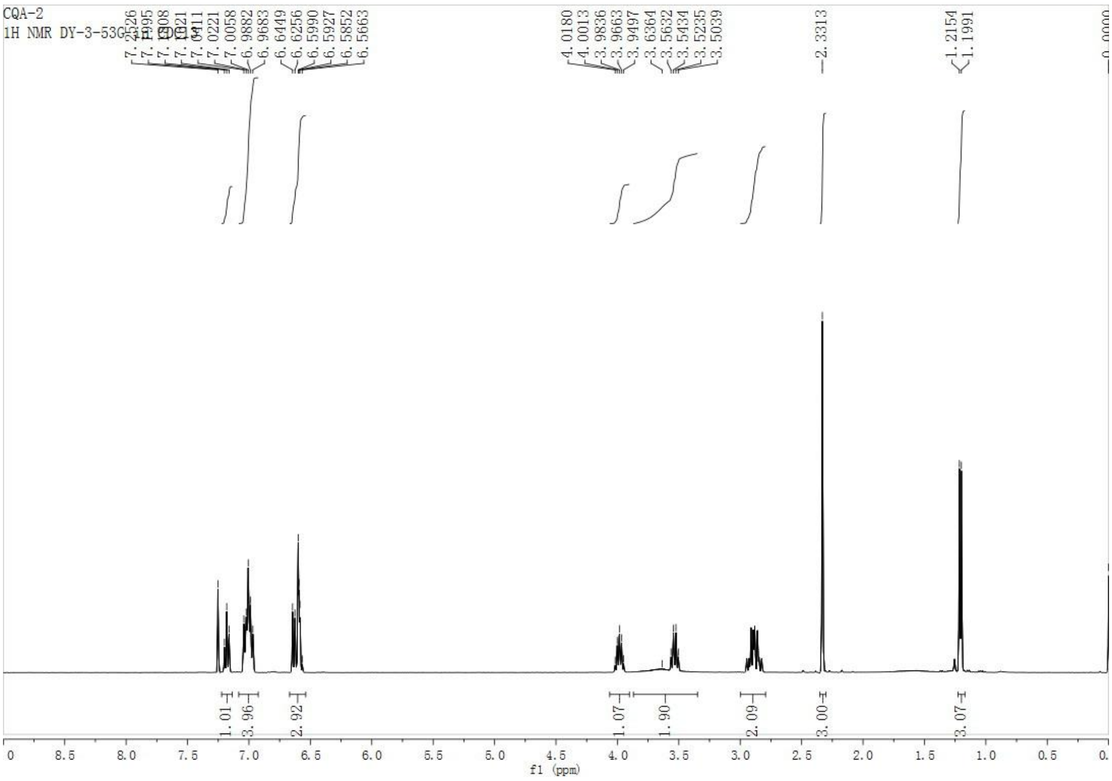
Minimum:				-10.0		
Maximum:		5.0	50.0	100.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
441.1610	441.1613	-0.3	-0.7	13.5	9.2	C25 H26 N2 O2 Na S

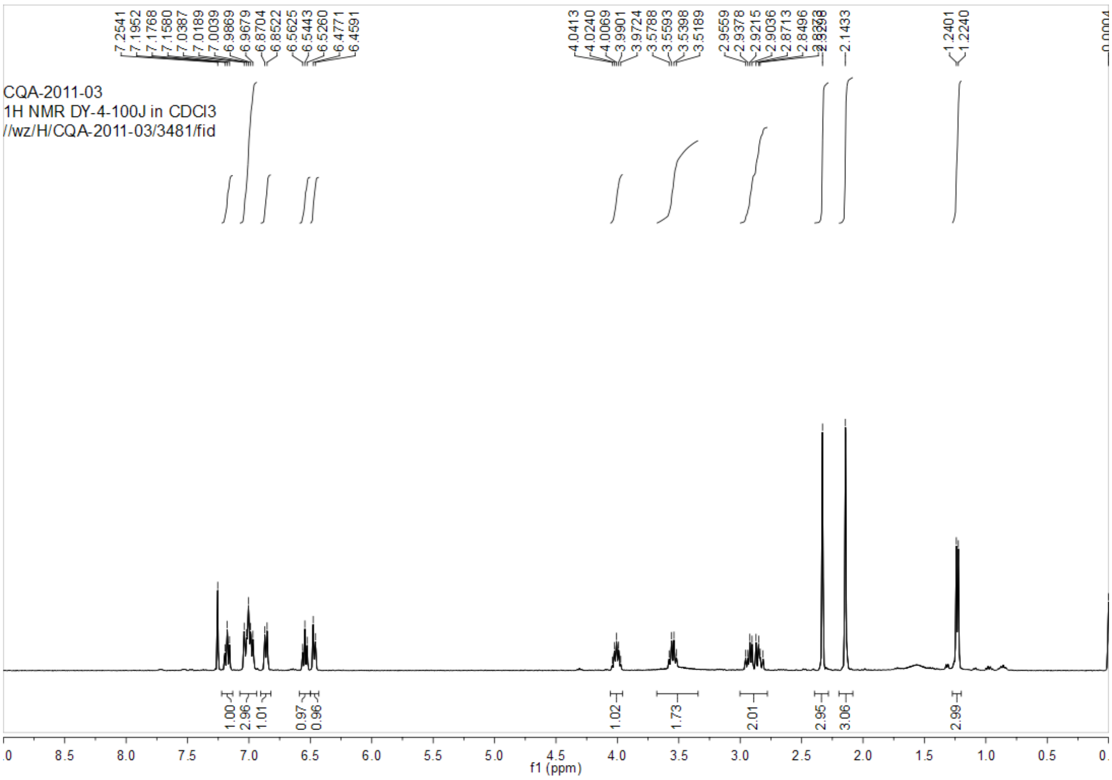

NMR
1H NMR DY-3-53D in CDCl3
E:/尝试反应/indole/TsN/NMR/225 DY-3-53D/fid
f1 (ppm)

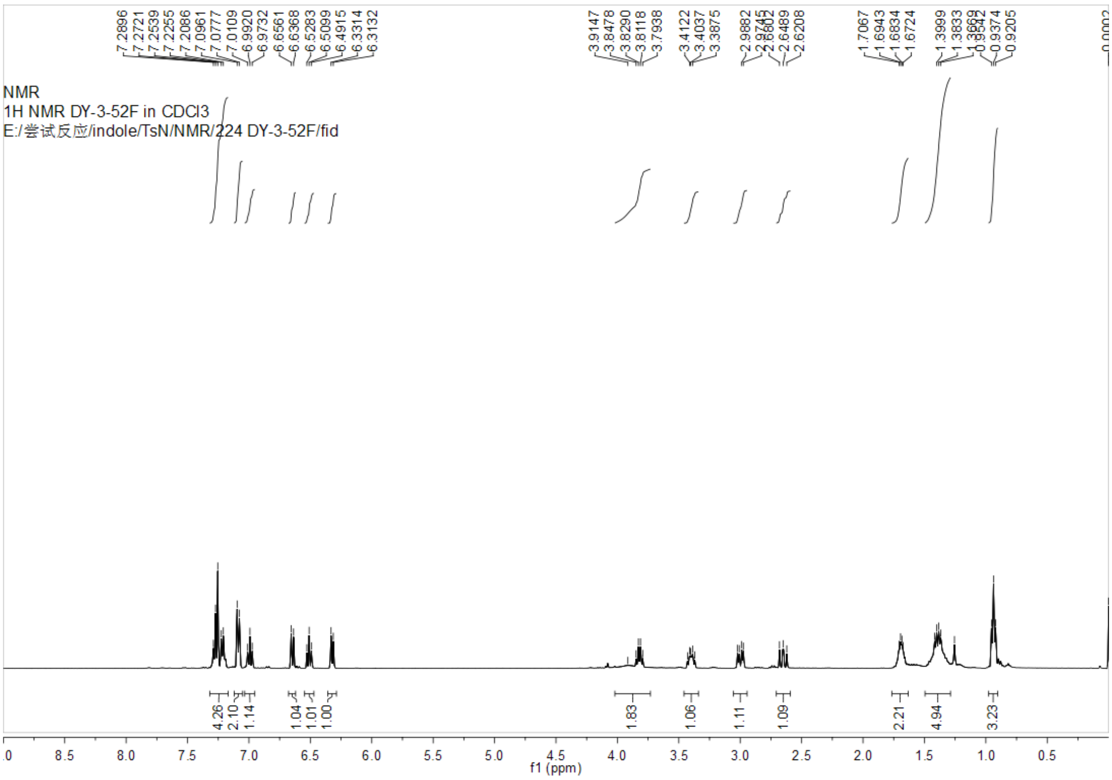

NMR
1H NMR DY-3-54M in CDCl3
E:/尝试反应/indole/TsN/NMR/229 DY-3-54M/fid
f1 (ppm)

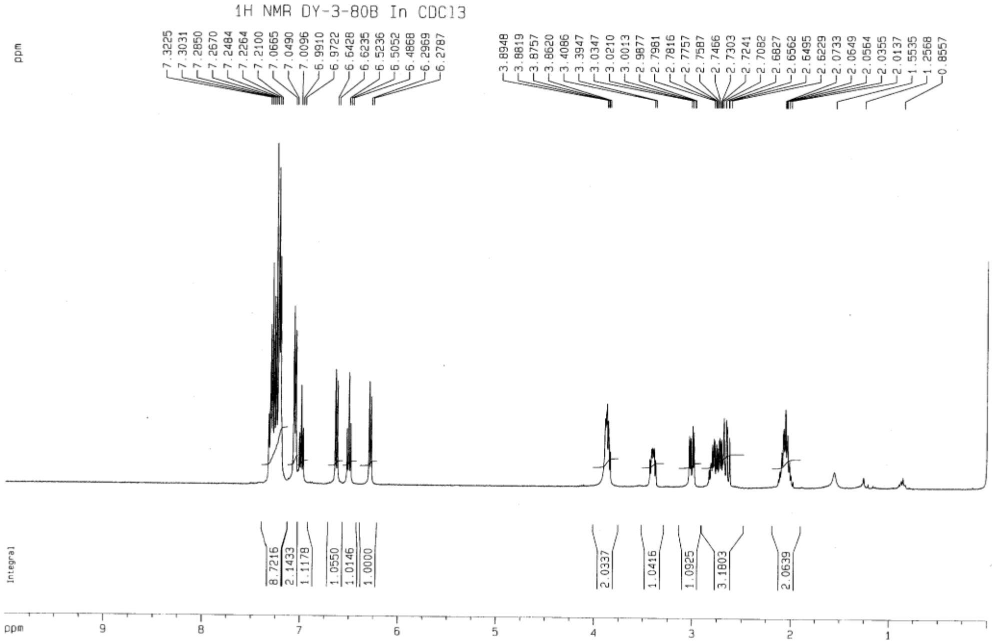

1H NMR ZY-1-71A In CDCl3

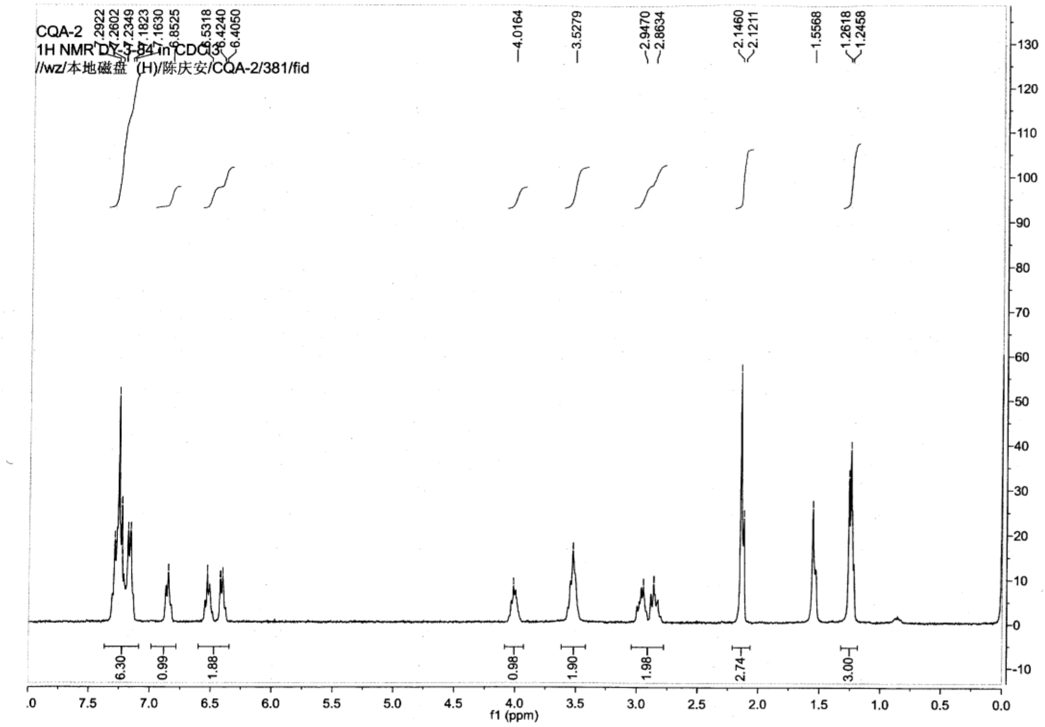

NMR
1H NMR MC-3-98B2 in CDCl3
E:/尝试反应/indole/TsN/NMR/233 MC-3-98/fid
f1 (ppm)

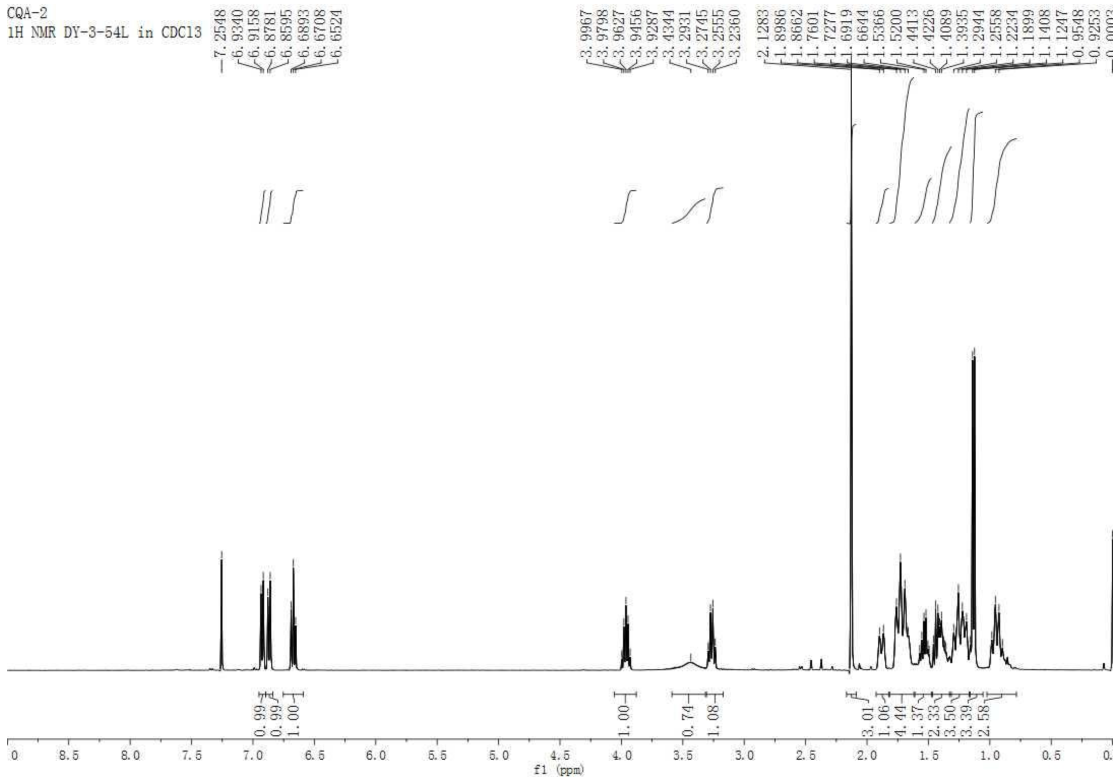

CQA-2
1H NMR DY-3-53G-1 in CDCl3
f1 (ppm)

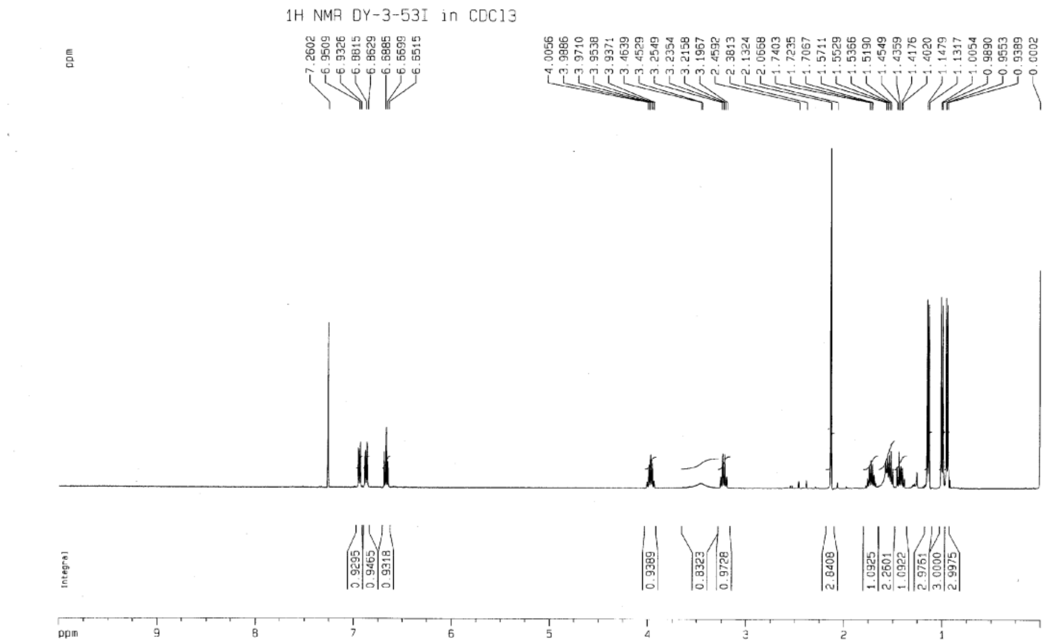

CQA-2011-03
1H NMR DY-4-100J in CDCl3
//wz/H/CQA-2011-03/3481/fid
f1 (ppm)

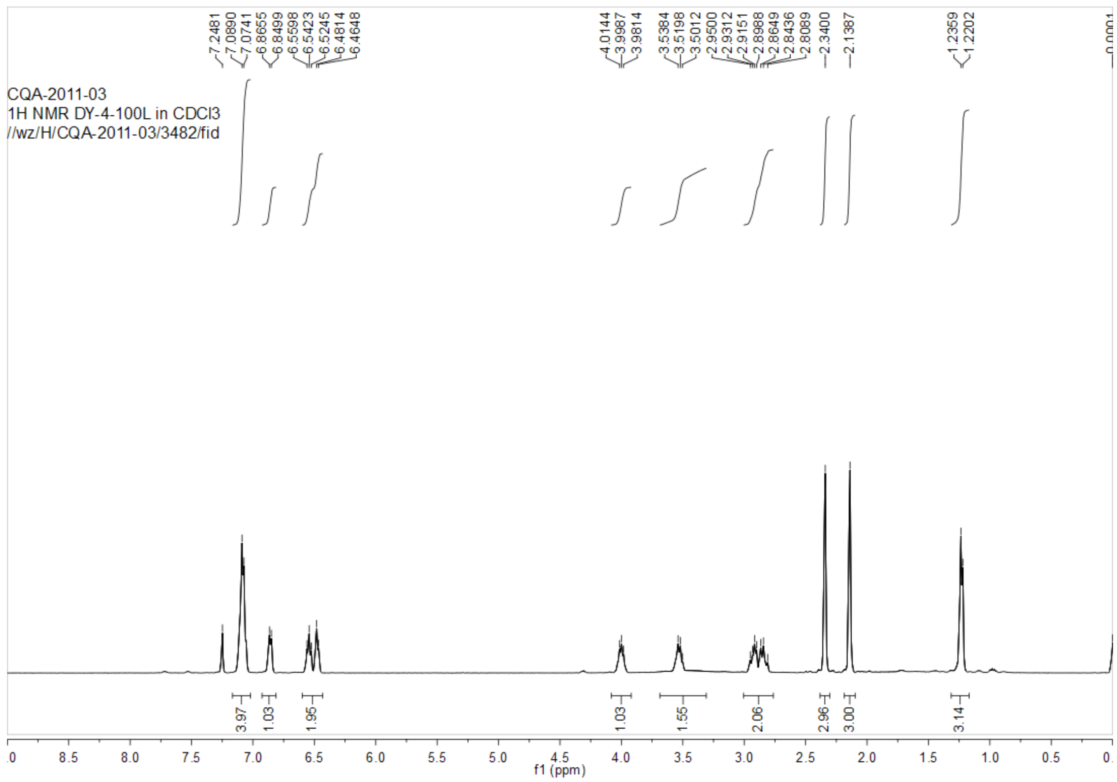

NMR
1H NMR DY-3-52F in CDCl3
E:/尝试反应/indole/TsN/NMR/224 DY-3-52F/fid
f1 (ppm)

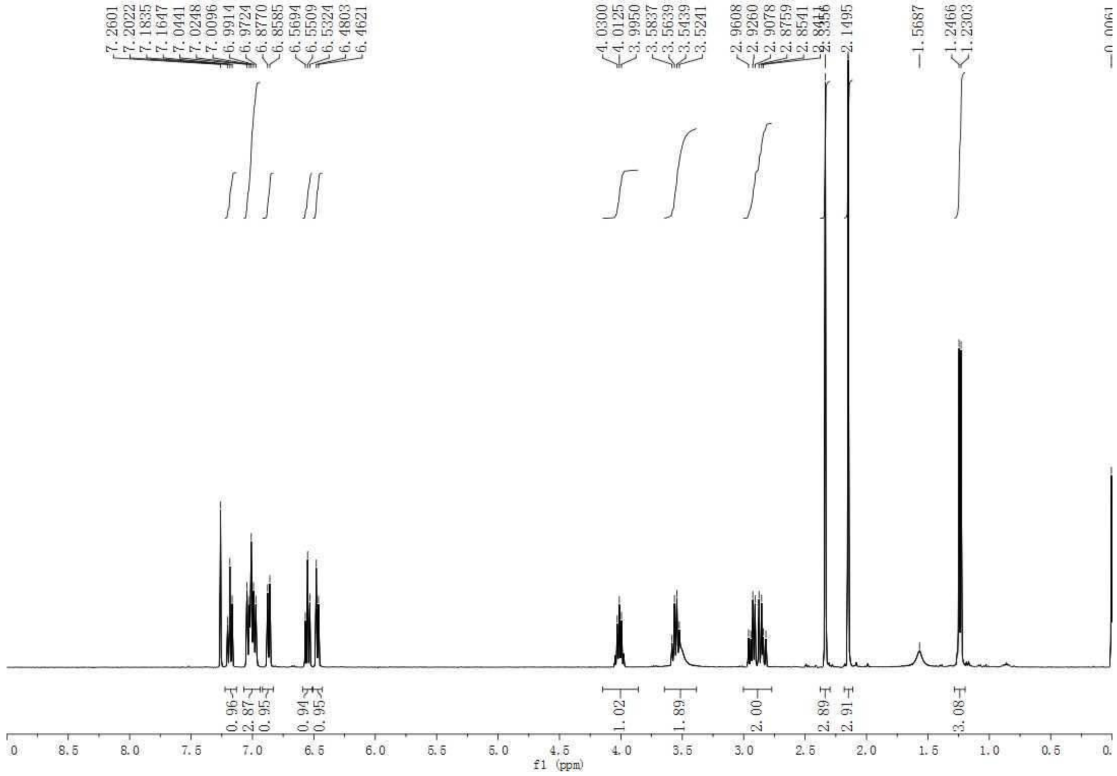

1H NMR DY-3-80B In CDCl3

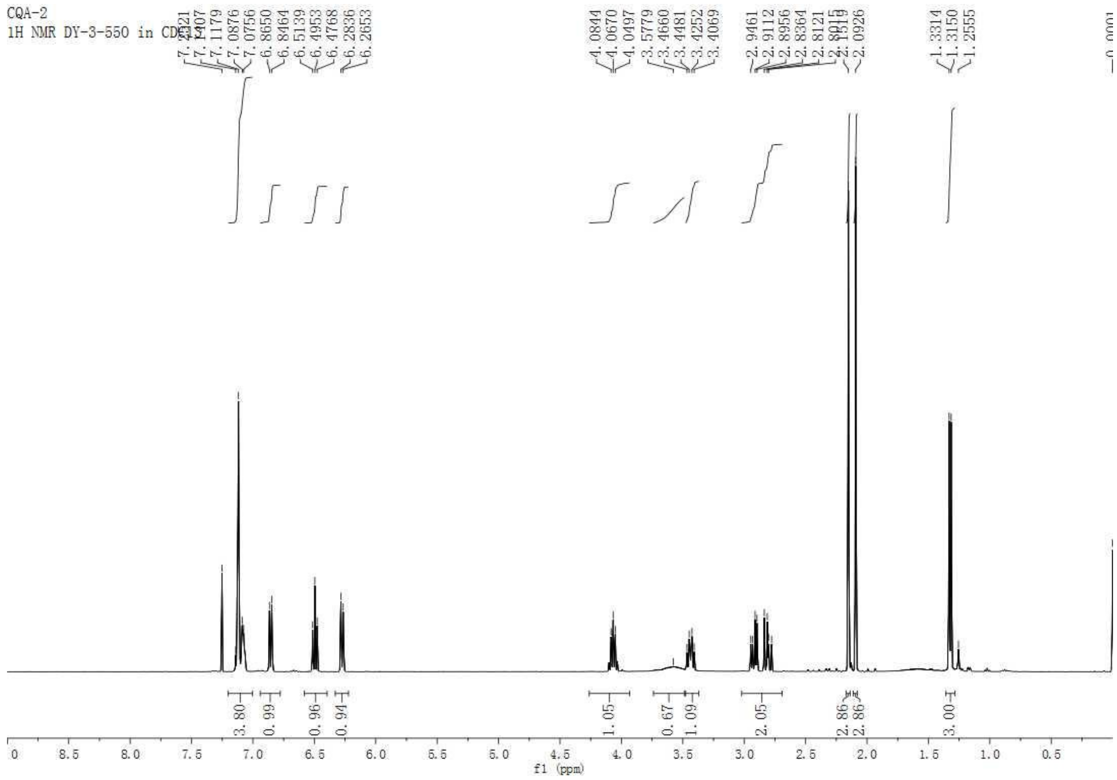

CQA-2
1H NMR DY-3-84 in CDCl3
//wz/本地磁盘 (H)/陈庆安/CQA-2/381/fid
f1 (ppm)


CQA-2
1H NMR DY-3-54L in CDCl3
f1 (ppm)


1H NMR DY-3-53I in CDCl3


CQA-2011-03
1H NMR DY-4-100L in CDCl3
//wz/H/CQA-2011-03/3482/fid
f1 (ppm)

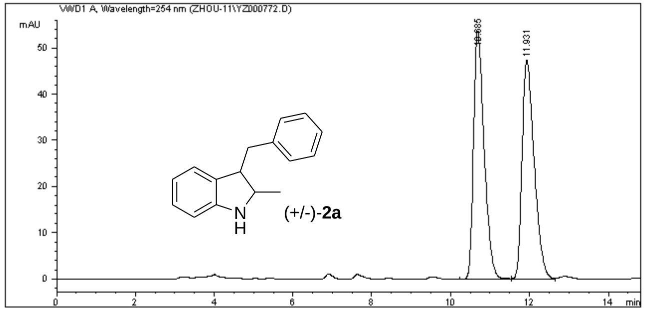



CQA-2
1H NMR DY-3-550 in CDCl3
f1 (ppm)

9. Copy of HPLC for Racemic and Chiral Compounds

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000772.D Sample Name: DY-4-93A+-
OJ-H, H/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm

Injection Date : 9/1/2011 4:21:49 PM
Sample Name : DY-4-93A+- Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/1/2011 4:17:12 PM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 4:41:56 PM by ZX
(modified after loading)



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.685	VV	0.2816	1007.65271	53.69359	49.8822
2	11.931	VV	0.3219	1012.41113	47.42553	50.1178

Totals : 2020.06384 101.11912

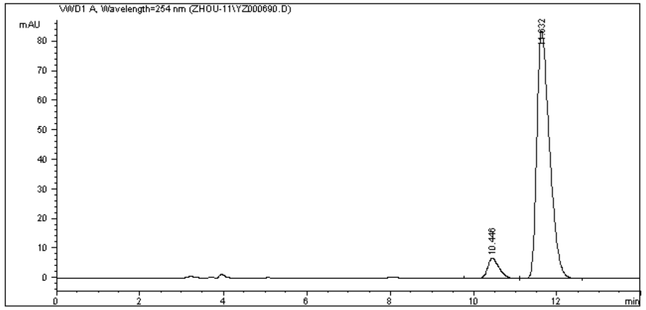
Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 9/8/2011 4:41:59 PM ZX Page 1 of 1

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000690.D Sample Name: DY-4-94A
OJ-H, H/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm

Injection Date : 8/20/2011 5:00:59 PM
Sample Name : DY-4-94A Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 8/20/2011 4:52:07 PM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 4:39:11 PM by ZX
(modified after loading)



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.446	VV	0.2845	128.01347	6.72928	6.7449
2	11.632	VV	0.3230	1769.90918	83.50908	93.2551

Totals : 1897.92265 90.23836

Results obtained with enhanced integrator!

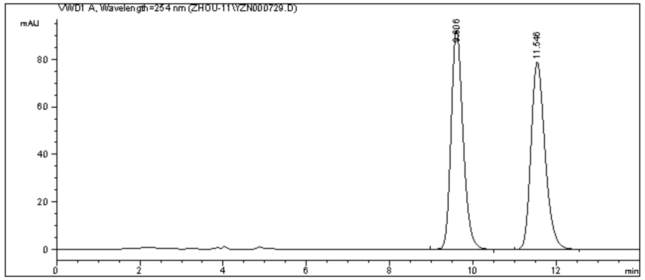
*** End of Report ***

Instrument 1 9/8/2011 4:39:35 PM ZX Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000729.D
Sample Name: DY-4-93B+-

===

Acq. Operator :
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/20/2011 4:33:50 PM
Acq. Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 8/20/2011 4:31:04 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/8/2011 4:53:55 PM
(modified after loading)
Sample Info : OD-H, H/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm



===

Area Percent Report

===

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.606	BB	0.3066	1839.55408	92.37290	49.9279
2	11.546	BB	0.3606	1844.86646	78.76810	50.0721
Totals :				3684.42053	171.14101	

===

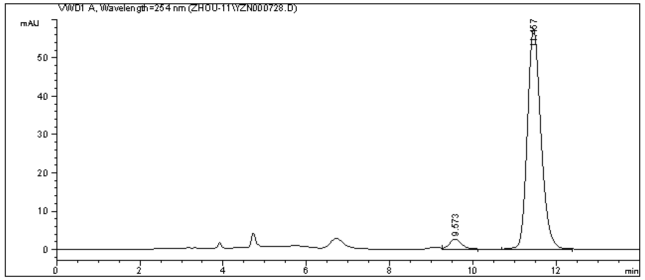
*** End of Report ***

Instrument 1 9/8/2011 4:54:08 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000728.D
Sample Name: DY-4-93B

===

Acq. Operator :
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/20/2011 4:16:25 PM
Acq. Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 8/20/2011 4:13:39 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/8/2011 4:53:55 PM
(modified after loading)
Sample Info : OD-H, H/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm



===

Area Percent Report

===

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

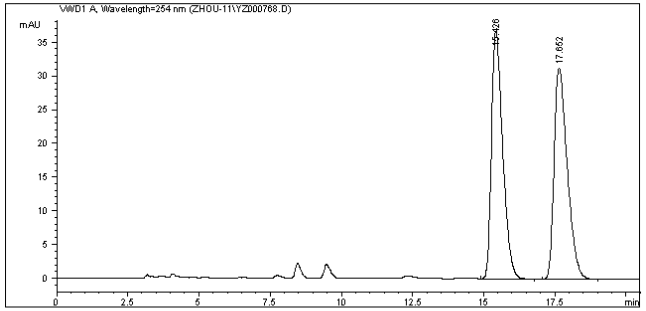
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.573	VB	0.3120	52.59864	2.58156	3.8950
2	11.457	BB	0.3491	1297.82605	57.21106	96.1050
Totals :				1350.42469	59.79262	

===

*** End of Report ***

Instrument 1 9/8/2011 4:53:57 PM Page 1 of 1

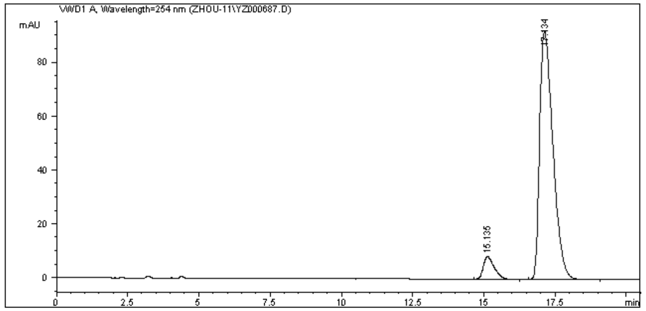
```
Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000768.D                          Sample Name: DY-4-94C+-
    OJ-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm

    =====================================================================
    Injection Date  : 9/1/2011 2:35:28 PM
    Sample Name     : DY-4-94C+-                      Location : Vial 1
    Acq. Operator   : ZX
    Acq. Method     : C:\HPCHEM\1\METHODS\SW.M
    Last changed    : 9/1/2011 1:57:34 PM by ZX
                      (modified after loading)
    Analysis Method : C:\HPCHEM\1\METHODS\SW.M
    Last changed    : 9/8/2011 4:44:46 PM by ZX
                      (modified after loading)
    =====================================================================
```




```
    =====================================================================
                         Area Percent Report
    =====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  15.426 VB    0.4141 1004.84381   36.70139  50.0372
       2  17.652 VB    0.4855 1003.35028   31.34911  49.9628

    Totals :                  2008.19409   68.05049

     Results obtained with enhanced integrator!
    =====================================================================
                          *** End of Report ***
```

Instrument 1 9/8/2011 4:44:56 PM ZX   Page 1 of 1

```
Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000687.D                          Sample Name: DY-4-94C
    OJ-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm

    =====================================================================
    Injection Date  : 8/20/2011 3:51:35 PM
    Sample Name     : DY-4-94C                        Location : Vial 1
    Acq. Operator   : ZX
    Acq. Method     : C:\HPCHEM\1\METHODS\SW.M
    Last changed    : 8/20/2011 3:39:50 PM by ZX
                      (modified after loading)
    Analysis Method : C:\HPCHEM\1\METHODS\SW.M
    Last changed    : 9/8/2011 4:44:09 PM by ZX
                      (modified after loading)
    =====================================================================
```

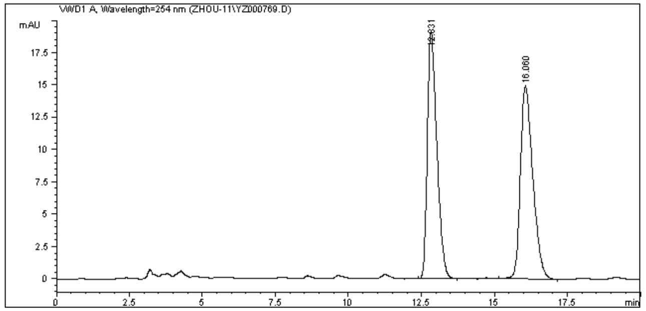



```
    =====================================================================
                         Area Percent Report
    =====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  15.135 VV    0.4039  219.80634    8.29121   6.8821
       2  17.134 VB    0.5013 2974.05859   91.90074  93.1179

    Totals :                  3193.86493  100.19195

     Results obtained with enhanced integrator!
    =====================================================================
                          *** End of Report ***
```

Instrument 1 9/8/2011 4:44:12 PM ZX   Page 1 of 1

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000769.D   Sample Name: DY-4-94D+-
OJ-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm

Injection Date : 9/1/2011 3:00:55 PM
Sample Name : DY-4-94D+-   Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/1/2011 2:57:41 PM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 4:49:15 PM by ZX
(modified after loading)



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.831	VV	0.3434	433.65973	19.11041	49.5675
2	16.060	VB	0.4512	441.22787	14.92587	50.4325

Totals : 874.88760 34.03628

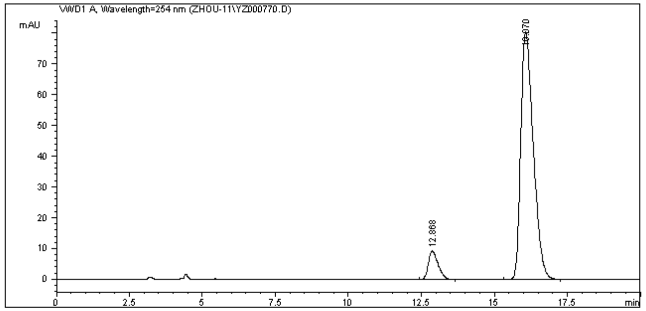
Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 9/8/2011 4:49:18 PM ZX   Page 1 of 1

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000770.D   Sample Name: DY-4-94D
OJ-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm

Injection Date : 9/1/2011 3:32:20 PM
Sample Name : DY-4-94D   Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/1/2011 3:29:13 PM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 4:48:51 PM by ZX
(modified after loading)



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.868	VV	0.3406	211.77997	9.27531	8.1506
2	16.070	VB	0.4519	2386.56934	80.55370	91.8494

Totals : 2598.34930 89.82900

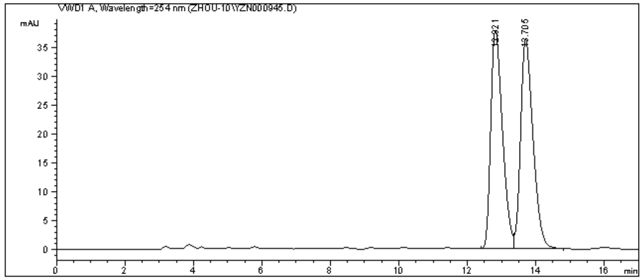
Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 9/8/2011 4:48:54 PM ZX   Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-10\YZN000945.D
Sample Name: DY-3-53G+-

```
=====================================================================
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 10/28/2010 3:23:55 PM
Acq. Method     : C:\CHEM32\1\METHODS\SW.M
Last changed    : 10/28/2010 3:14:28 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed    : 9/8/2011 7:44:34 PM
                  (modified after loading)
Sample Info     : OJ-H, H/i-PrOH =90/10, 1.0 mL/min, 30 oC, 254NM
```

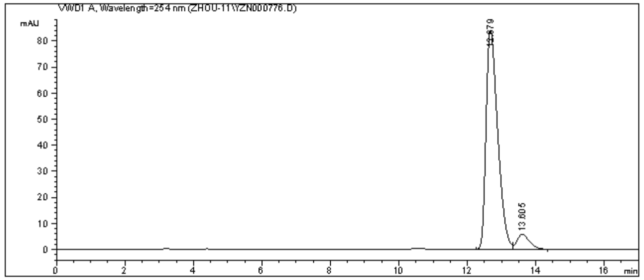



```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier:                  :      1.0000
Dilution:                    :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.821 BV    0.3638  897.20624   37.87120  49.4750
   2  13.705 VB    0.3835  916.24689   36.27848  50.5250

Totals :                  1813.45313   74.14969

=====================================================================
                          *** End of Report ***
```

Instrument 1 9/8/2011 7:44:37 PM   Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000776.D
Sample Name: DY-4-100I

```
=====================================================================
Acq. Operator   :
Acq. Instrument : Instrument 1                    Location : Vial 1
Injection Date  : 9/6/2011 2:19:43 PM
Acq. Method     : C:\CHEM32\1\METHODS\SW.M
Last changed    : 9/6/2011 1:37:07 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed    : 9/8/2011 7:50:59 PM
                  (modified after loading)
Sample Info     : OJ-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm
```

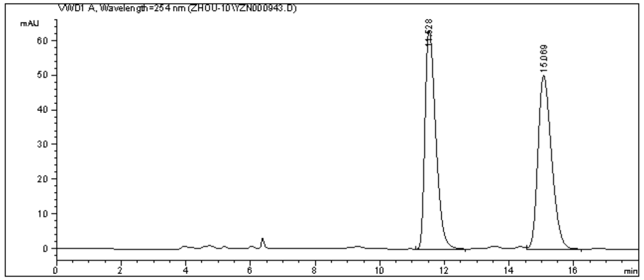



```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier:                  :      1.0000
Dilution:                    :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  12.679 BV    0.3607 1983.00977   84.18130  93.2101
   2  13.605 VB    0.3681  144.45268    5.85042   6.7899

Totals :                  2127.46245   90.03172

=====================================================================
                          *** End of Report ***
```

Instrument 1 9/8/2011 7:51:26 PM   Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-10\YZN000943.D
Sample Name: DY-3-53H+-

Acq. Operator :
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 10/28/2010 2:33:27 PM
Acq. Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 10/28/2010 2:31:22 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/8/2011 7:45:19 PM
(modified after loading)
Sample Info : OJ-H, H/i-PrOH =85/15, 0.8 mL/min, 30 oC, 254NM



Area Percent Report

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.528	VB	0.3565	1456.97620	63.16806	49.9453
2	15.069	VB	0.4517	1460.16516	49.94775	50.0547

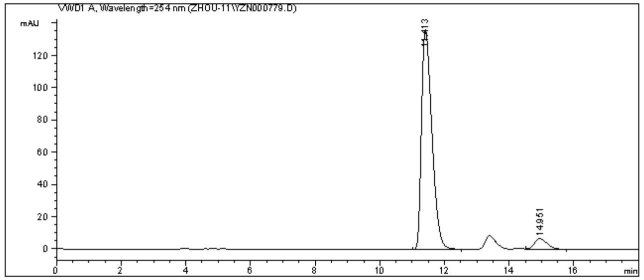
Totals : 2917.14136 113.11581

*** End of Report ***

Instrument 1 9/8/2011 7:45:22 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000779.D
Sample Name: DY-4-100J

Acq. Operator :
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 9/6/2011 3:43:58 PM
Acq. Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/6/2011 3:31:05 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/8/2011 7:52:53 PM
(modified after loading)
Sample Info : OJ-H, H/i-PrOH = 85/15, 0.8 mL/min, 30 oC, 254 nm



Area Percent Report

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.413	BB	0.3467	3042.41992	136.09575	94.2657
2	14.951	VB	0.4310	185.07315	6.58987	5.7343

Totals : 3227.49307 142.68562

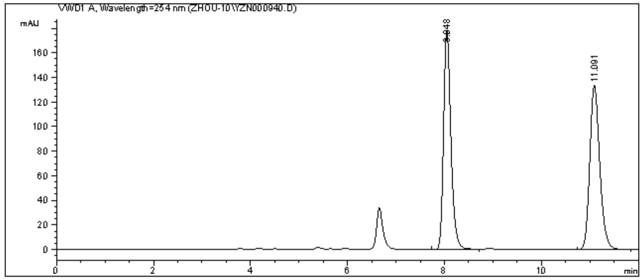
*** End of Report ***

Instrument 1 9/8/2011 7:53:00 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-10\YZN000940.D
Sample Name: DY-3-52F+-

===

Acq. Operator :
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 10/27/2010 8:19:34 PM
Acq. Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 10/27/2010 8:16:33 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/8/2011 4:58:08 PM
(modified after loading)
Sample Info : AD-H, H/i-PrOH =95/5, 0.8 mL/min, 30 oC, 254NM



===
Area Percent Report
===

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.048	BB	0.1590	1876.72498	178.64925	49.6593
2	11.091	BB	0.2172	1902.47437	133.52693	50.3407

Totals : 3779.19934 312.17618

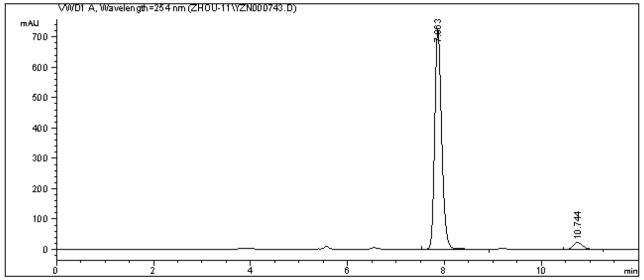
===
*** End of Report ***

Instrument 1 9/8/2011 4:58:10 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000743.D
Sample Name: DY-4-94E

===

Acq. Operator :
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/24/2011 3:23:56 PM
Acq. Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 8/24/2011 2:59:14 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/8/2011 4:56:37 PM
(modified after loading)
Sample Info : AD-H, H/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 254 nm



===
Area Percent Report
===

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.863	VB	0.1534	7247.13135	722.88898	95.9286
2	10.744	VB	0.2077	307.58405	22.68503	4.0714

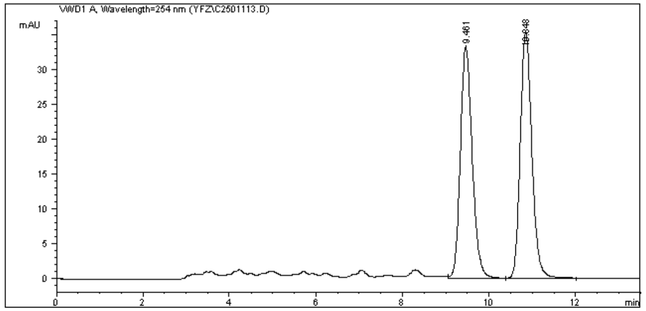
Totals : 7554.71539 745.57401

===
*** End of Report ***

Instrument 1 9/8/2011 4:56:39 PM Page 1 of 1

Data File C:\HPCHEM\1\DATA\YFZ\C2501113.D Sample Name: DY-3-80B+-
OD-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm

===
Injection Date : 12/6/2010 5:01:02 PM
Sample Name : DY-3-80B+- Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 12/6/2010 5:09:45 PM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 7:57:42 PM by ZX
(modified after loading)
===



===
Area Percent Report
===

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

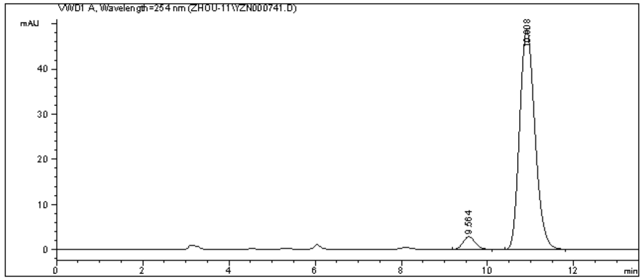
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.461	VV	0.2937	635.16913	33.33329	49.2334
2	10.848	VB	0.2857	654.94983	35.40527	50.7666

Totals : 1290.11896 68.73856

Results obtained with enhanced integrator!
===
*** End of Report ***

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000741.D
Sample Name: DY-4-95F

===
Acq. Operator :
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/24/2011 2:21:37 PM
Acq. Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 8/24/2011 2:17:33 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/8/2011 7:37:08 PM
(modified after loading)
Sample Info : OD-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm



===
Area Percent Report
===

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.564	BB	0.3081	56.31651	2.82703	4.4724
2	10.908	BB	0.3873	1202.88538	48.67994	95.5276

Totals : 1259.20189 51.50697

===
*** End of Report ***

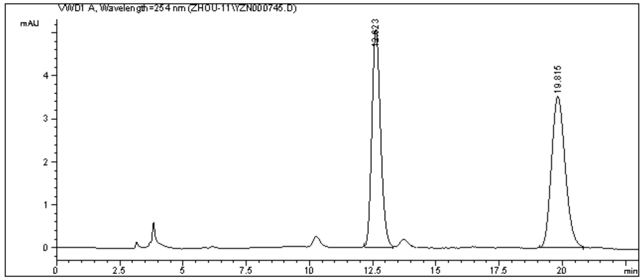
Instrument 1 9/8/2011 7:57:51 PM ZX Page 1 of 1

Instrument 1 9/8/2011 7:37:13 PM Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000745.D
Sample Name: DY-4-95G+-

===

Acq. Operator :
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/24/2011 4:39:18 PM
Acq. Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 8/24/2011 4:36:27 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/8/2011 7:39:43 PM
(modified after loading)
Sample Info : OD-H, H/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm



===
Area Percent Report
===

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.623	BB	0.3656	120.58823	5.05587	47.5335
2	19.815	BB	0.5897	133.10271	3.51989	52.4665

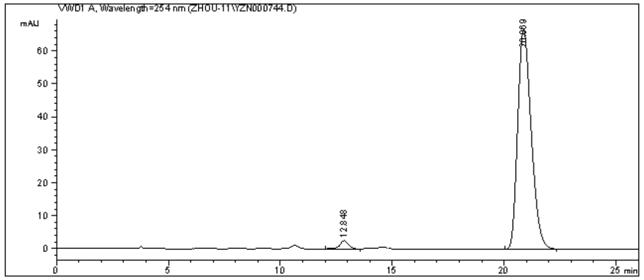
Totals : 253.69093 8.57575

===
*** End of Report ***

Data File C:\CHEM32\1\DATA\ZHOU-11\YZN000744.D
Sample Name: DY-4-95G

===

Acq. Operator :
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/24/2011 4:09:41 PM
Acq. Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 8/24/2011 3:40:54 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/8/2011 7:38:37 PM
(modified after loading)
Sample Info : OD-H, H/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm



===
Area Percent Report
===

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

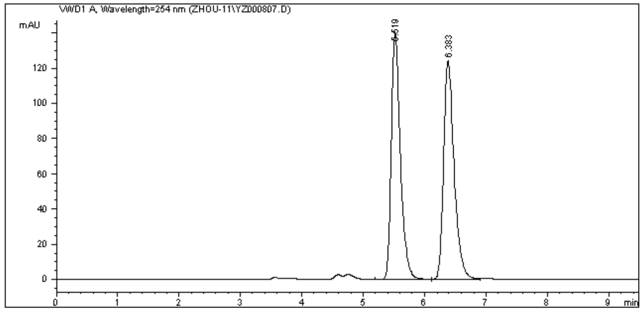
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.848	BB	0.4184	70.34047	2.52366	2.4553
2	20.869	BB	0.6486	2794.52539	66.54762	97.5447

Totals : 2864.86586 69.07127

===
*** End of Report ***

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000807.D Sample Name: DY-4-101N+-
OD-H, H/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm

Injection Date : 9/6/2011 3:31:54 PM
Sample Name : DY-4-101N+- Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/6/2011 3:39:32 PM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 4:28:02 PM by ZX
(modified after loading)



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.519	VV	0.1569	1453.65698	140.75349	49.6938
2	6.383	VV	0.1792	1471.56946	124.01060	50.3062

Totals : 2925.22644 264.76410

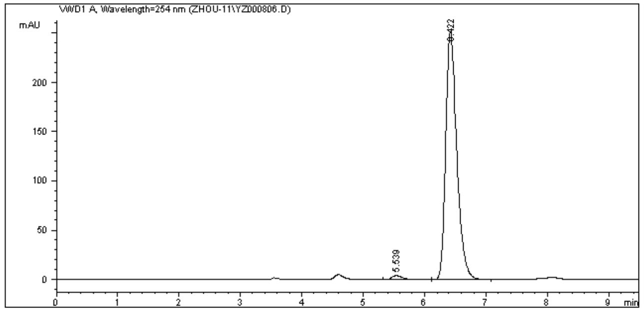
Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 9/8/2011 4:28:05 PM ZX Page 1 of 1

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000806.D Sample Name: DY-4-101N
OD-H, H/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm

Injection Date : 9/6/2011 3:18:09 PM
Sample Name : DY-4-101N Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/6/2011 3:23:06 PM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 4:28:28 PM by ZX
(modified after loading)



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.539	VB	0.1742	49.18963	4.16559	1.5626
2	6.422	BV	0.1875	3098.78125	251.33672	98.4374

Totals : 3147.97088 255.50230

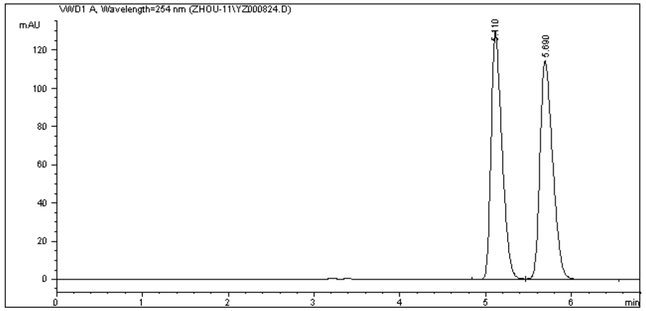
Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 9/8/2011 4:28:31 PM ZX Page 1 of 1

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000824.D Sample Name: DY-4-101M+-
OJ-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm

Injection Date : 9/8/2011 11:24:37 AM
Sample Name : DY-4-101M+- Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 11:32:09 AM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 4:24:52 PM by ZX
(modified after loading)



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.110	VV	0.1370	1166.53760	129.68788	50.0366
2	5.690	VV	0.1553	1164.82874	114.33876	49.9634

Totals : 2331.36633 244.02664

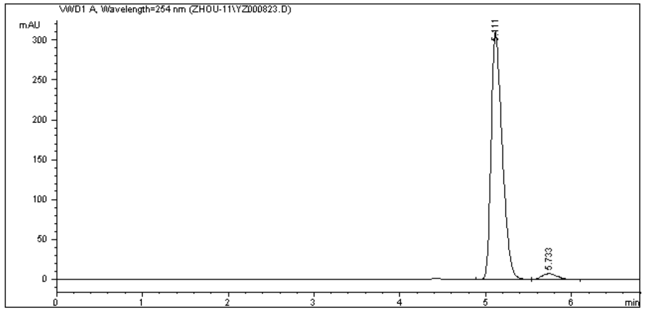
Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 9/8/2011 4:24:54 PM ZX Page 1 of 1

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000823.D Sample Name: DY-4-101M
OJ-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm

Injection Date : 9/8/2011 11:16:13 AM
Sample Name : DY-4-101M Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 11:17:11 AM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 4:24:27 PM by ZX
(modified after loading)



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.111	VV	0.1400	2834.85303	310.47525	96.9477
2	5.733	VV	0.2033	89.25226	6.83440	3.0523

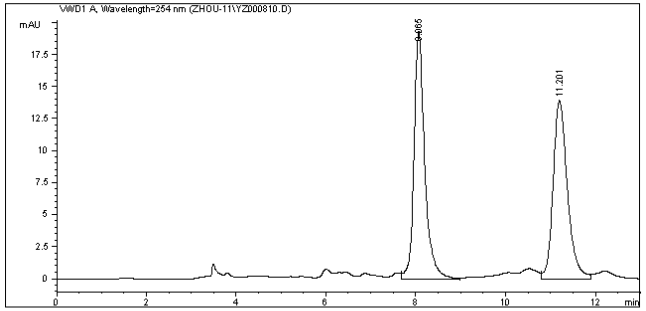
Totals : 2924.10529 317.30965

Results obtained with enhanced integrator!

*** End of Report ***

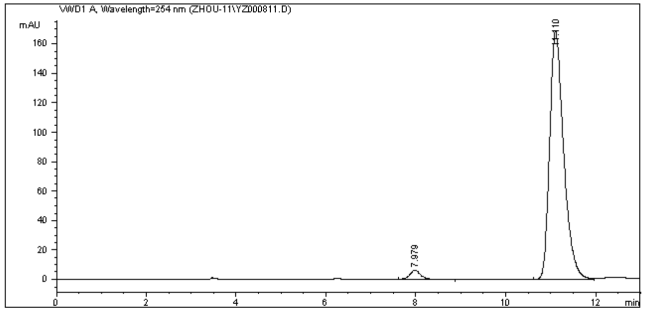
Instrument 1 9/8/2011 4:24:31 PM ZX Page 1 of 1

```
Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000810.D                    Sample Name: DY-4-101L+-
    OD-H, H/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm

    =====================================================================
    Injection Date  : 9/6/2011 4:20:58 PM
    Sample Name     : DY-4-101L+-                     Location : Vial 1
    Acq. Operator   : ZX
    Acq. Method     : C:\HPCHEM\1\METHODS\SW.M
    Last changed    : 9/6/2011 4:18:39 PM by ZX
                      (modified after loading)
    Analysis Method : C:\HPCHEM\1\METHODS\SW.M
    Last changed    : 9/8/2011 4:30:11 PM by ZX
                      (modified after loading)
    =====================================================================
```




```
    =====================================================================
                             Area Percent Report
    =====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   8.065 VB    0.2451  318.89032   19.31021  50.8424
       2  11.201 VV    0.3357  308.32336   13.91516  49.1576

    Totals :                   627.21368   33.22537

     Results obtained with enhanced integrator!
    =====================================================================
                              *** End of Report ***

Instrument 1 9/8/2011 4:30:15 PM ZX                                          Page 1 of 1
```

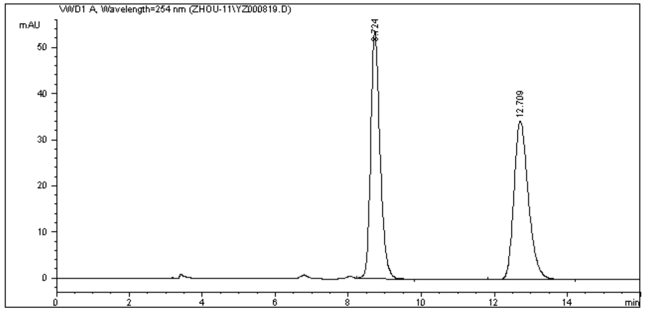
```
Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000811.D                    Sample Name: DY-4-101L
    OD-H, H/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm

    =====================================================================
    Injection Date  : 9/6/2011 4:39:55 PM
    Sample Name     : DY-4-101L                       Location : Vial 1
    Acq. Operator   : ZX
    Acq. Method     : C:\HPCHEM\1\METHODS\SW.M
    Last changed    : 9/6/2011 4:37:25 PM by ZX
                      (modified after loading)
    Analysis Method : C:\HPCHEM\1\METHODS\SW.M
    Last changed    : 9/8/2011 4:30:42 PM by ZX
                      (modified after loading)
    =====================================================================
```




```
    =====================================================================
                             Area Percent Report
    =====================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: VWD1 A, Wavelength=254 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1   7.979 VB    0.2511  104.89999    6.20470   2.7867
       2  11.110 VV    0.3323 3659.35620  168.27345  97.2133

    Totals :                  3764.25619  174.47815

     Results obtained with enhanced integrator!
    =====================================================================
                              *** End of Report ***

Instrument 1 9/8/2011 4:30:47 PM ZX                                          Page 1 of 1
```

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000819.D Sample Name: DY-4-101K+-
OD-H, H/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm

Injection Date : 9/8/2011 10:02:59 AM
Sample Name : DY-4-101K+- Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 9:37:57 AM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 4:32:13 PM by ZX
(modified after loading)



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.724	VB	0.2746	959.14655	53.51875	50.5357
2	12.709	BB	0.4233	938.81134	34.23988	49.4643

Totals : 1897.95789 87.75863

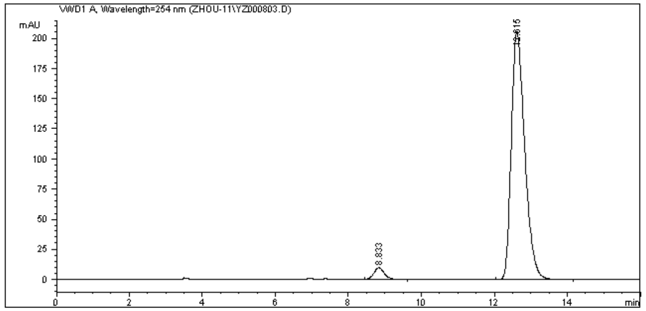
Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 9/8/2011 4:32:18 PM ZX Page 1 of 1

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000803.D Sample Name: DY-4-101K
OD-H, H/i-PrOH = 99/1, 1.0 mL/min, 30 oC, 254 nm

Injection Date : 9/6/2011 2:23:46 PM
Sample Name : DY-4-101K Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/6/2011 1:37:48 PM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 4:32:54 PM by ZX
(modified after loading)



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.833	VB	0.3050	188.91023	9.49144	3.4772
2	12.615	VB	0.3923	5243.91504	205.54976	96.5228

Totals : 5432.82527 215.04120

Results obtained with enhanced integrator!

*** End of Report ***

Instrument 1 9/8/2011 4:32:57 PM ZX Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-10\YZN000975.D
Sample Name: DY-3-550+-

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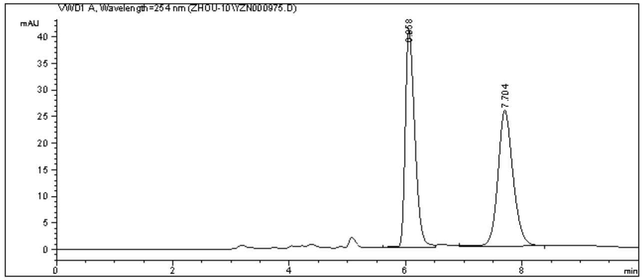
Acq. Operator :
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 11/7/2010 3:02:59 PM
Acq. Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 11/7/2010 3:01:05 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SW.M
Last changed : 9/8/2011 7:41:56 PM
(modified after loading)
Sample Info : OJ-H, H/i-PrOH =90/10, 1.0 mL/min, 30 oC, 254 NM



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Area Percent Report

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Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.058	VV	0.1746	463.79630	40.89103	50.1101
2	7.704	VB	0.2738	461.75873	25.50555	49.8899

Totals : 925.55502 66.39658

===

*** End of Report ***

Instrument 1 9/8/2011 7:41:59 PM

Page 1 of 1

Data File C:\HPCHEM\1\DATA\ZHOU-11\YZ000708.D Sample Name: DY-4-95H
OJ-H, H/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm

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Injection Date : 8/24/2011 3:35:12 PM
Sample Name : DY-4-95H Location : Vial 1
Acq. Operator : ZX
Acq. Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 8/24/2011 3:35:26 PM by ZX
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\SW.M
Last changed : 9/8/2011 5:04:08 PM by ZX
(modified after loading)

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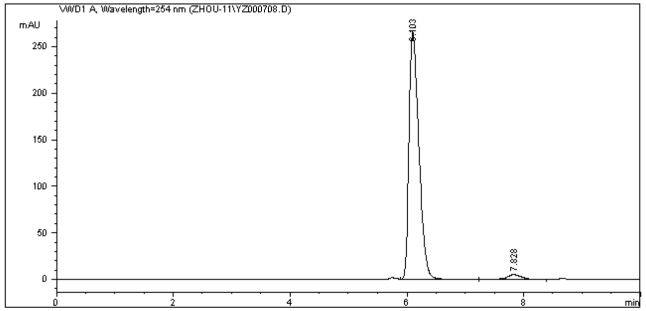

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Area Percent Report

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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.103	VV	0.1808	3128.85010	266.14951	97.1678
2	7.828	VB	0.2593	91.19790	5.21544	2.8322

Totals : 3220.04800 271.36495

Results obtained with enhanced integrator!

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*** End of Report ***

Instrument 1 9/8/2011 5:04:11 PM ZX

Page 1 of 1