

A Chiral Borane Catalyzed Asymmetric Hydrosilylation of Imines

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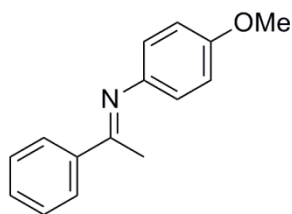
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Supporting Information

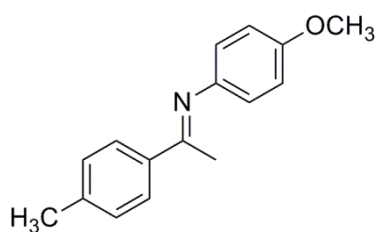
General consideration: All air-sensitive compounds were handled under an atmosphere of argon or in a nitrogen-filled glovebox. ^1H NMR and ^{13}C NMR spectra were recorded on Bruker AV 400 at ambient temperature with CDCl_3 as solvent and TMS as internal standard. Chemical shifts (δ) were given in ppm, referenced to the residual proton resonance of TMS (0), to the carbon resonance of the CDCl_3 (77.23). Coupling constants (J) were given in Hertz (Hz). IR spectrums were recorded on Perkin-Elmer-983 spectrometer. Optical rotations were measured with PerkinElmer 341 polarimeter. Flash column chromatography was performed on silica gel (200-300 mesh). All solvents were purified by conventional methods, distilled before use. Commercially available reagents were used without further purification. $\text{HB}(\text{C}_6\text{F}_5)_2$ was prepared according to reported method.¹

Representative procedure for the metal-free catalytic asymmetric hydrosilylation of imines (Table 2,

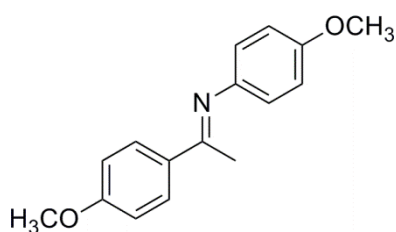
Entry 1): To a schlenk tube was added $\text{HB}(\text{C}_6\text{F}_5)_2$ (0.0034 g, 0.010 mmol), chiral diene **4g** (0.0061 g, 0.005 mmol) and dry toluene (1.0 mL) in glovebox. The resulting mixture was stirred at room temperature for 10 min followed by the addition of PhMe_2SiH (0.0749 g, 0.55 mmol) and stirring for 5 min at room temperature. To the resulting solution, imine **1a** (0.1126 g, 0.5 mmol) was added. After the schlenk was sealed, the reaction mixture was stirred at 0 °C for 8 h and the solvent was removed under reduced pressure. The crude residue was purified by flash chromatography on silica gel using petroleum ether/ethyl acetate (50/1) as the eluent to give the desired (*R*)-**3a** as a yellow oil (0.1130 g, >99% yield, 67% ee).



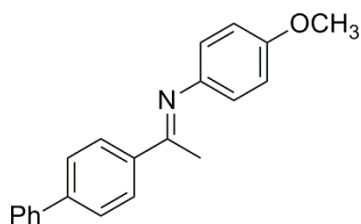
(*E*)-4-methoxy-*N*-(1-phenylethylidene)aniline (1a**)²:** ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.99-7.97 (m, 2H), 7.46-7.44 (m, 3H), 6.93 (d, J = 8.8 Hz, 2H), 6.72 (d, J = 8.8 Hz, 2H), 3.83 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 165.9, 156.1, 145.0, 139.9, 130.5, 128.5, 127.3, 120.9, 114.4, 55.6, 17.5.



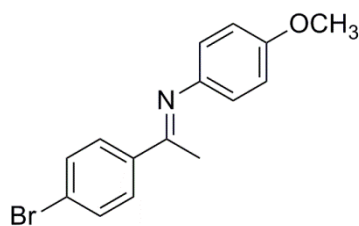
(*E*)-4-methoxy-*N*-(1-(*p*-tolyl)ethylidene)aniline (1b)²: ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.87 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 6.76 (d, *J* = 8.8 Hz, 2H), 3.82 (s, 3H), 2.41 (s, 3H), 2.24 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 165.7, 156.0, 145.1, 140.7, 137.2, 129.2, 127.3, 121.0, 114.4, 55.6, 21.6, 17.4.



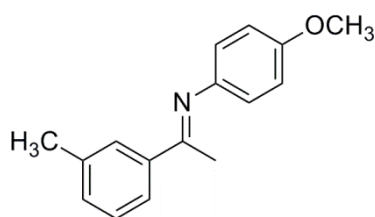
(*E*)-4-methoxy-*N*-(1-(4-methoxyphenyl)ethylidene)aniline (1c)²: ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.93 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 6.74 (d, *J* = 8.8 Hz, 2H), 3.86 (s, 3H), 3.82 (s, 3H), 2.22 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 165.0, 161.6, 156.0, 145.3, 132.7, 128.9, 121.1, 114.4, 113.8, 55.7, 55.6, 17.3.



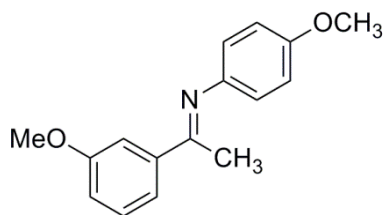
(*E*)-*N*-(1-([1,1'-biphenyl]-4-yl)ethylidene)-4-methoxyaniline (1d)³: ¹H NMR (400 MHz, CDCl₃, ppm): δ 8.06 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.50-7.46 (m, 2H), 7.39 (dd, *J* = 7.2, 7.6 Hz, 1H), 6.94 (d, *J* = 8.8 Hz, 2H), 6.79 (d, *J* = 8.8 Hz, 2H), 3.84 (s, 3H), 2.30 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 165.5, 156.2, 145.0, 143.2, 140.6, 138.8, 129.1, 127.9, 127.8, 127.3, 127.2, 121.0, 114.5, 55.7, 17.5.



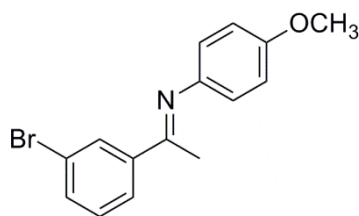
(*E*)-*N*-(1-(4-bromophenyl)ethylidene)-4-methoxyaniline (1e)⁴: ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.84 (d, *J* = 8.8 Hz, 2H), 7.56 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 6.75 (d, *J* = 8.8 Hz), 3.82 (s, 3H), 2.23 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 164.7, 156.3, 144.6, 138.8, 131.7, 128.9, 125.1, 120.9, 114.5, 55.7, 17.3.



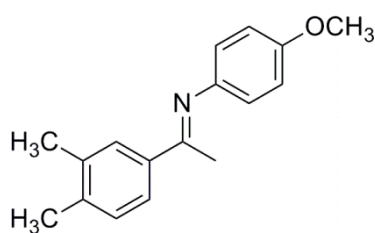
(*E*)-4-methoxy-*N*-(1-(*m*-tolyl)ethylidene)aniline (1f)⁴: ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.83 (s, 1H), 7.73 (d, *J* = 7.6 Hz, 1H), 7.34 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.28 (d, *J* = 7.6 Hz, 1H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.77 (d, *J* = 8.8 Hz, 2H), 3.82 (s, 3H), 2.43 (s, 3H), 2.25 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 166.2, 156.1, 145.0, 139.9, 138.2, 131.3, 128.4, 127.8, 124.5, 120.9, 114.4, 55.6, 21.7, 17.6.



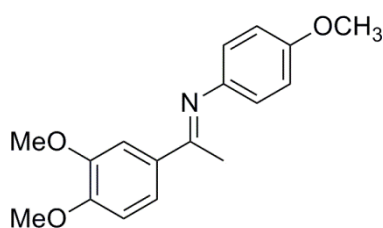
(*E*)-4-methoxy-*N*-(1-(3-methoxyphenyl)ethylidene)aniline (1g)⁶: ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.57-7.58 (m, 1H), 7.50 (d, *J* = 7.6 Hz, 1H), 7.35 (dd, *J* = 8.0, 8.0 Hz, 1H), 7.01 (dd, *J* = 2.6, 8.0 Hz, 1H), 6.91 (d, *J* = 8.4 Hz, 2H), 6.76 (d, *J* = 8.4 Hz, 2H), 3.88 (s, 3H), 3.82 (s, 3H), 2.24 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 165.8, 159.9, 156.1, 145.0, 141.4, 129.5, 121.0, 120.0, 116.9, 114.4, 112.0, 55.7, 55.6, 17.7.



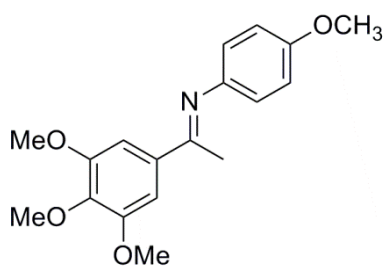
(*E*)-*N*-(1-(3-Bromophenyl)ethylidene)-4-methoxyaniline (1h)³: ¹H NMR (400 MHz, CDCl₃, ppm): δ 8.14 (m, 1H), 7.86 (m, 1H), 7.58 (d, *J* = 8.8 Hz, 1H), 7.31 (dd, *J* = 8.0, 8.0 Hz, 1H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 3.82 (s, 3H), 2.23 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 164.4, 156.3, 144.4, 141.9, 133.3, 130.4, 130.0, 125.9, 122.9, 120.9, 114.4, 55.6, 17.4.



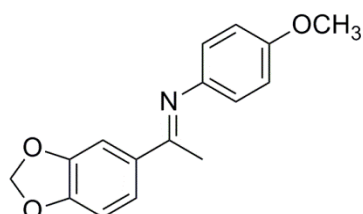
(*E*)-*N*-(1-(3,4-dimethylphenyl)ethylidene)-4-methoxyaniline (1i): light yellow solid, m.p. 70-72 °C; IR (film): 1628, 1604, 1504 cm⁻¹; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.81 (s, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 1H), 6.92 (d, *J* = 8.4 Hz, 2H), 6.77 (d, *J* = 8.4 Hz, 2H), 3.82 (s, 3H), 2.34 (s, 3H), 2.33 (s, 3H), 2.24 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 166.0, 156.0, 145.2, 139.4, 137.6, 136.7, 129.7, 128.3, 124.9, 121.0, 114.3, 55.6, 20.0, 19.9, 17.4; HRMS (ESI) calcd. for C₁₇H₂₀ON (M+H): 254.1539, Found: 254.1538.



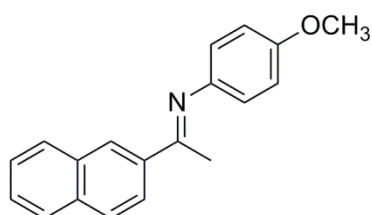
(*E*)-*N*-(1-(3,4-dimethoxyphenyl)ethylidene)-4-methoxyaniline (1j)²: ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.71 (s, 1H), 7.43 (d, *J* = 8.4 Hz, 1H), 6.91 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 1H), 6.74 (d, *J* = 8.8 Hz, 2H), 3.96 (s, 3H), 3.94 (s, 3H), 3.81 (s, 3H), 2.22 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 164.9, 156.0, 151.3, 149.1, 145.2, 132.9, 121.0, 120.9, 114.4, 110.2, 109.6, 56.2, 55.7, 17.2.



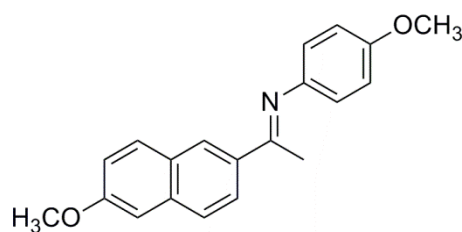
(*E*)-4-methoxy-*N*-(1-(3,4,5-trimethoxyphenyl)ethylidene)aniline (1k)¹²: ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.23 (s, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 6.74 (d, *J* = 8.8 Hz, 2H), 3.93 (s, 6H), 3.90 (s, 3H), 3.81 (s, 3H), 2.23 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 165.2, 156.1, 153.2, 144.9, 140.4, 135.4, 120.9, 114.4, 104.7, 61.1, 56.5, 55.6, 17.5.



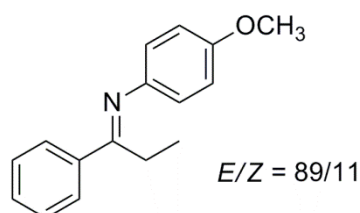
(*E*)-*N*-(1-(benzo[d][1,3]dioxol-5-yl)ethylidene)-4-methoxyaniline (1l)⁹: ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.57 (s, 1H), 7.43 (d, *J* = 8.8 Hz, 1H), 6.90 (d, *J* = 8.8 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 1H), 6.73 (d, *J* = 8.8 Hz, 2H), 6.01 (s, 2H), 3.81 (s, 3H), 2.20 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 164.7, 156.0, 149.7, 148.2, 145.0, 134.6, 122.2, 121.0, 114.4, 107.9, 107.4, 101.6, 55.7, 17.4.



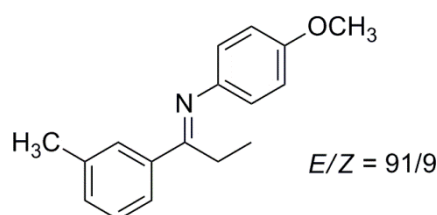
(*E*)-4-methoxy-*N*-(1-(naphthalen-2-yl)ethylidene)aniline (1m)¹⁰: ¹H NMR (400 MHz, CDCl₃, ppm): δ 8.34 (s, 1H), 8.23 (dd, *J* = 1.6, 8.6 Hz, 1H), 7.94-7.86 (m, 3H), 7.56-7.51 (m, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 6.81 (d, *J* = 8.8 Hz, 2H), 3.84 (s, 3H), 2.38 (s, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 165.7, 156.2, 145.1, 137.3, 134.6, 133.2, 129.1, 128.2, 127.9, 127.3, 126.5, 124.4, 121.0, 114.5, 55.7, 17.5.



(E)-4-methoxy-N-(1-(6-methoxynaphthalen-2-yl)ethylidene)aniline (1n): yellow solid, m.p. 210-213 °C; IR (film): 1616, 1601, 1504 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.27 (s, 1H), 8.19 (d, $J = 8.4$ Hz, 1H), 7.82 (d, $J = 8.4$ Hz, 1H), 7.78 (d, $J = 8.4$ Hz, 1H), 7.20-7.17 (m, 2H), 6.93 (d, $J = 8.8$ Hz, 2H), 6.80 (d, $J = 8.8$ Hz, 2H), 3.95 (s, 3H), 3.83 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 165.7, 158.9, 156.1, 145.2, 136.0, 135.3, 130.6, 128.5, 127.6, 127.0, 125.0, 121.1, 119.4, 114.4, 105.9, 55.7, 55.6, 17.4; HRMS (ESI) calcd. for $\text{C}_{20}\text{H}_{20}\text{O}_2\text{N}$ ($\text{M}+\text{H}$): 306.1489, Found: 306.1486.

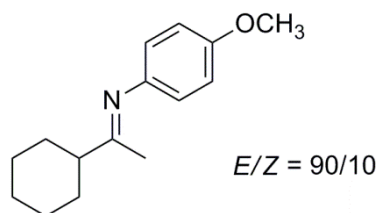


(E)-4-methoxy-N-(1-phenylpropylidene)aniline (1o)⁵: ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.94-7.89 (m, 2H), 7.47-7.43 (m, 3H), 6.91 (d, $J = 8.8$ Hz, 2H), 6.75 (d, $J = 8.8$ Hz, 2H), 3.82 (s, 3H), 2.69 (q, $J = 7.6$ Hz, 2H), 1.09 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 171.5, 156.0, 145.1, 138.5, 130.4, 128.6, 127.7, 120.4, 114.5, 55.7, 23.4, 13.0.

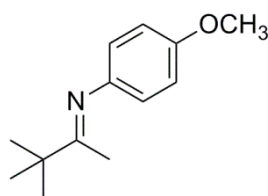


(E)-4-methoxy-N-(1-(m-tolyl)propylidene)aniline (1p): yellow solid, m.p. 62-64 °C; IR (film): 1628, 1604, 1503 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.77 (s, 1H), 7.66 (d, $J = 7.6$ Hz, 1H), 7.33 (dd, $J = 7.6$, 7.6 Hz, 1H), 7.27 (d, $J = 8.0$ Hz, 1H), 6.91 (d, $J = 8.8$ Hz, 2H), 6.75 (d, $J = 8.8$ Hz, 2H), 3.81 (s, 3H), 2.68 (q, $J = 7.6$ Hz, 2H), 2.42 (s, 3H), 1.08 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ

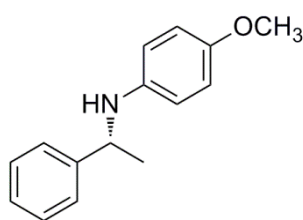
171.8, 155.9, 145.1, 138.5, 138.4, 131.2, 128.5, 128.3, 124.8, 120.5, 114.4, 55.6, 23.5, 21.7, 13.1; HRMS (ESI) calcd. for C₁₇H₂₀ON (M+H): 254.1539, Found: 254.1537.



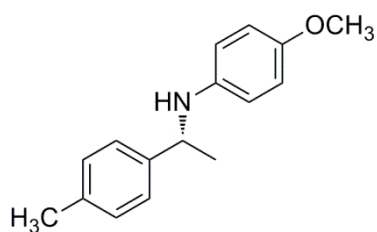
(*E*)-*N*-(1-cyclohexylethylidene)-4-methoxyaniline (1q)²: yellow oil; ¹H NMR (400 MHz, C₆D₆, ppm): δ 6.81 (d, *J* = 8.8 Hz, 2H), 6.66 (d, *J* = 8.8 Hz, 2H), 3.36 (s, 3H), 2.15-2.08 (m, 1H), 1.88 (d, *J* = 12.0 Hz, 2H), 1.76-1.69 (m, 2H), 1.62-1.59 (m, 1H), 1.54 (s, 3H), 1.49-1.41 (m, 2H), 1.27-1.10 (m, 3H); ¹³C NMR (100 MHz, C₆D₆, ppm): δ 174.1, 156.2, 146.0, 120.8, 114.6, 55.0, 49.3, 30.6, 26.6, 25.7, 17.8.



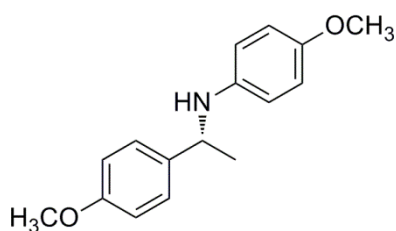
(*E*)-*N*-(3,3-dimethylbutan-2-ylidene)-4-methoxyaniline (1r)¹¹: ¹H NMR (400 MHz, CDCl₃, ppm): δ 6.83 (d, *J* = 8.8 Hz, 2H), 6.57 (d, *J* = 8.8 Hz, 2H), 3.77 (s, 3H), 1.74 (s, 3H), 1.21 (s, 9H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 178.1, 155.6, 145.6, 120.3, 114.3, 55.6, 40.4, 28.0, 15.3.



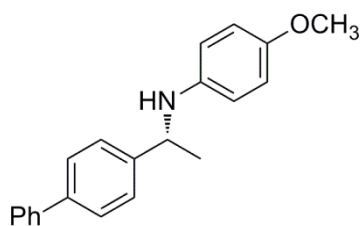
(*R*)-4-methoxy-*N*-(1-phenylethyl)aniline (3a)²: yellow oil, [α]_D²⁵ = +6.0 (*c* 1.01, CHCl₃) (67% ee) [lit.: [α]_D²⁰ = +8.5 (*c* 0.87, CHCl₃) (98% ee for *R* isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.36-7.28 (m, 4H), 7.23-7.19 (m, 1H), 7.68 (d, *J* = 8.8 Hz, 2H), 6.46 (d, *J* = 8.8 Hz, 2H), 4.40 (q, *J* = 6.8 Hz, 1H), 3.68 (s, 3H), 1.48 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 152.1, 145.7, 141.8, 128.8, 127.0, 126.1, 115.0, 114.8, 55.9, 54.4, 25.3.



(*R*)-4-methoxy-*N*-(1-(*p*-tolyl)ethyl)aniline (3b)²: yellow oil, $[\alpha]_{\text{D}}^{25} = +11.5$ (*c* 1.12, CHCl₃) (61% ee) [lit.: $[\alpha]_{\text{D}}^{25} = +7.7$ (*c* 1.05, CHCl₃) (98% ee for *R* isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.29 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.73 (d, *J* = 8.8 Hz, 2H), 6.53 (d, *J* = 8.8 Hz, 2H), 4.43 (q, *J* = 6.4 Hz, 1H), 3.90 (brs, 1H), 3.72 (s, 3H), 2.36 (s, 3H), 1.52 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 152.1, 142.5, 141.6, 136.5, 129.5, 126.0, 114.9, 114.8, 55.9, 54.2, 25.2, 21.2.

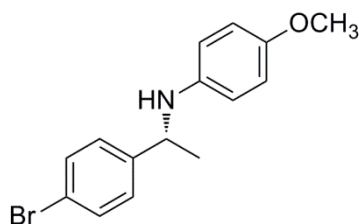


(*R*)-4-methoxy-*N*-(1-(4-methoxyphenyl)ethyl)aniline (3c)²: yellow oil, $[\alpha]_{\text{D}}^{25} = +10.1$ (*c* 1.00, CHCl₃) (65% ee) [lit.: $[\alpha]_{\text{D}}^{20} = +24.2$ (*c* 1.05, CHCl₃) (98% ee for *R* isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.28 (d, *J* = 8.4 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H), 6.70 (d, *J* = 8.8 Hz, 2H), 6.48 (d, *J* = 8.8 Hz, 2H), 4.38 (q, *J* = 6.4 Hz, 1H), 3.79 (s, 3H), 3.70 (s, 3H), 1.48 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 158.7, 152.1, 141.9, 137.7, 127.1, 115.0, 114.8, 114.2, 56.0, 55.4, 53.8, 25.3.

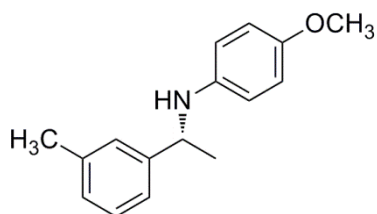


(*R*)-*N*-(1-([1,1'-biphenyl]-4-yl)ethyl)-4-methoxyaniline (3d)³: white solid, $[\alpha]_{\text{D}}^{25} = +41.0$ (*c* 0.96, CHCl₃) (61% ee) [lit.: $[\alpha]_{\text{D}}^{25} = +41.9$ (*c* 2.30, CHCl₃) (74% ee for *R* isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.62-7.58 (m, 4H), 7.46 (m, 4H), 7.36 (dd, *J* = 7.2, 7.2 Hz, 1H), 6.75 (d, *J* = 8.8 Hz, 2H), 6.55 (d, *J* = 8.8 Hz, 2H), 4.50 (q, *J* = 6.8 Hz, 1H), 3.73 (s, 3H), 1.57 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (100 MHz,

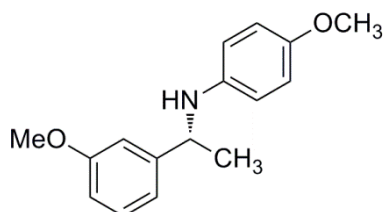
CDCl₃, ppm): δ 152.1, 144.8, 141.7, 141.1, 139.9, 128.9, 127.6, 127.3, 127.2, 126.5, 115.0, 114.8, 55.9, 54.2, 25.3.



(*R*)-*N*-(1-(4-bromophenyl)ethyl)-4-methoxyaniline (3e)^{5,6}: yellow oil, $[\alpha]_{\text{D}}^{25} = +13.5$ (*c* 1.10, CHCl₃) (59% ee) [lit.: $[\alpha]_{\text{D}}^{25} = +28.4$ (*c* 2.00, CHCl₃) (94% ee)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.42 (d, *J* = 8.8 Hz, 2H), 7.23 (d, *J* = 8.8 Hz, 2H), 6.68 (d, *J* = 8.8 Hz, 2H), 6.43 (d, *J* = 8.8 Hz, 2H), 4.35 (q, *J* = 6.8 Hz, 1H), 3.69 (s, 3H), 1.46 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 152.3, 144.8, 141.4, 131.9, 127.9, 120.6, 115.0, 114.8, 55.9, 54.0, 25.3.

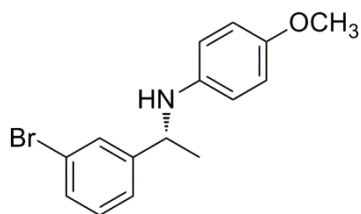


(*R*)-4-methoxy-*N*-(1-(*m*-tolyl)ethyl)aniline (3f)^{4,6}: yellow oil, $[\alpha]_{\text{D}}^{25} = +2.5$ (*c* 0.98, CHCl₃) (68% ee) [lit.: $[\alpha]_{\text{D}}^{25} = +8.1$ (*c* 2.00, CHCl₃) (89% ee)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.33-7.26 (m, 3H), 7.14 (d, *J* = 7.2 Hz, 1H), 6.81 (d, *J* = 8.8 Hz, 2H), 6.58 (d, *J* = 8.8 Hz, 2H), 4.47 (q, *J* = 6.8 Hz, 1H), 3.87 (brs, 1H), 3.78 (s, 3H), 2.44 (s, 3H), 1.58 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 151.9, 145.7, 141.8, 138.2, 128.6, 127.7, 126.7, 123.0, 114.8, 114.6, 55.8, 54.4, 25.2, 21.7.

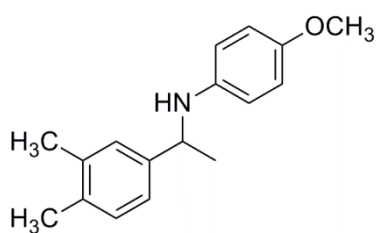


(*R*)-4-methoxy-*N*-(1-(3-methoxyphenyl)ethyl)aniline (3g)^{6,7}: yellow oil, $[\alpha]_{\text{D}}^{25} = +5.5$ (*c* 1.05, CHCl₃) (72% ee) [lit.: $[\alpha]_{\text{D}}^{25} = -6.6$ (*c* 0.45, CHCl₃) (96% ee)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.27 (m, 1H),

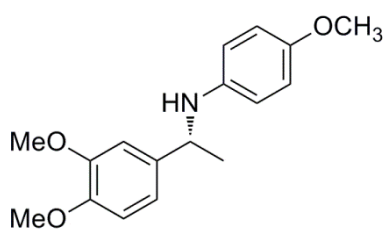
7.01-6.98 (m, 2H), 6.80 (d, $J = 8.0$ Hz, 1H), 6.73 (d, $J = 8.4$ Hz, 2H), 6.52 (d, $J = 8.4$ Hz, 2H), 4.42 (q, $J = 6.4$ Hz, 1H), 3.90 (brs, 1H), 3.81 (s, 3H), 3.72 (s, 3H), 1.52 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 160.0, 152.0, 147.6, 141.7, 129.8, 118.4, 114.9, 114.7, 112.1, 111.8, 55.8, 55.3, 54.4, 25.2.



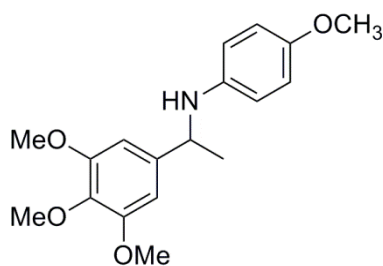
(*R*)-*N*-(1-(3-bromophenyl)ethyl)-4-methoxyaniline (3h)³: yellow oil, $[\alpha]_{\text{D}}^{25} = +11.7$ (c 1.08, CH_3OH) (68% ee) [lit.: $[\alpha]_{\text{D}}^{25} = +15.4$ (c 2.00, CH_3OH) (72% ee)]; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.55 (s, 1H), 7.38 (d, $J = 8.0$ Hz, 1H), 7.32 (d, $J = 7.6$ Hz, 1H), 7.19 (dd, $J = 7.6, 8.0$ Hz, 1H), 6.73 (d, $J = 8.8$ Hz, 2H), 6.47 (d, $J = 8.8$ Hz, 2H), 4.38 (q, $J = 6.8$ Hz, 1H), 3.72 (s, 3H), 1.49 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 152.2, 148.4, 141.4, 130.5, 130.2, 129.2, 124.7, 123.0, 115.0, 114.7, 55.9, 54.1, 25.4.



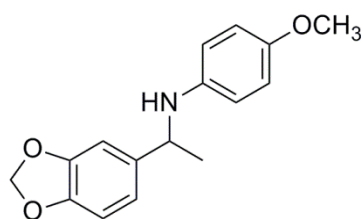
***N*-(1-(3,4-dimethylphenyl)ethyl)-4-methoxyaniline (3i)**: yellow oil, $[\alpha]_{\text{D}}^{25} = +14.7$ (c 1.07, CHCl_3) (71% ee); IR (film): 3402, 1605, 1511, 1452 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.13 (s, 1H), 7.09 (s, 2H), 6.70 (d, $J = 8.8$ Hz, 2H), 6.50 (d, $J = 8.8$ Hz, 2H), 4.36 (q, $J = 6.8$ Hz, 1H), 3.70 (s, 3H), 2.25 (s, 3H), 2.23 (s, 3H), 1.48 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 152.1, 143.1, 141.9, 136.9, 135.2, 130.0, 127.4, 123.4, 115.0, 114.8, 56.0, 54.3, 25.3, 20.1, 19.6; HRMS (ESI) calcd. for $\text{C}_{17}\text{H}_{22}\text{ON}$ ($\text{M}+\text{H}$): 256.1696, Found: 256.1695.



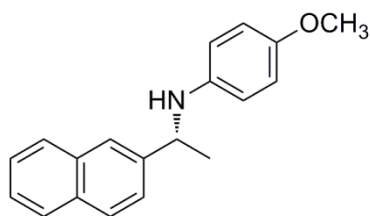
(*R*)-*N*-(1-(3,4-dimethoxyphenyl)ethyl)-4-methoxyaniline (3j)²⁸: white solid, $[\alpha]_{\text{D}}^{25} = +8.3$ (c 0.98, CHCl₃) (78% ee) [lit.: $[\alpha]_{\text{D}}^{17} = -16.0$ (c 0.72, CHCl₃) (98% ee)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 6.92-6.90 (m, 2H), 6.82 (d, $J = 8.4$ Hz, 1H), 6.70 (d, $J = 8.8$ Hz, 2H), 6.49 (d, $J = 8.8$ Hz, 2H), 4.35 (q, $J = 6.4$ Hz, 1H), 3.86 (s, 3H), 3.85 (s, 3H), 3.75 (brs, 1H), 3.70 (s, 3H), 1.49 (d, $J = 6.4$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 152.1, 149.3, 148.0, 141.9, 138.4, 117.9, 114.9, 114.8, 111.4, 109.3, 56.1, 56.0, 55.9, 54.3, 25.3.



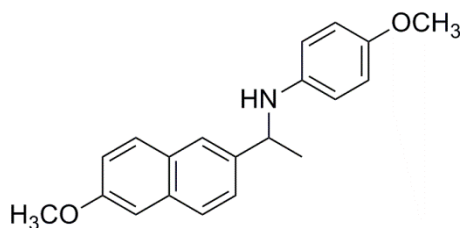
4-methoxy-*N*-(1-(3,4,5-trimethoxyphenyl)ethyl)aniline (3k): white solid, $[\alpha]_{\text{D}}^{25} = +8.1$ (c 1.00, CHCl₃) (82% ee); IR (film): 3407, 1590, 1514, 1463 cm⁻¹; ¹H NMR (400 MHz, CDCl₃, ppm): δ 6.71 (d, $J = 8.8$ Hz, 2H), 6.60 (s, 2H), 6.50 (d, $J = 8.8$ Hz, 2H), 4.31 (q, $J = 6.8$ Hz, 1H), 3.89 (brs, 1H), 3.84 (s, 3H), 3.83 (s, 3H), 3.71 (s, 3H), 1.49 (d, $J = 6.8$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 153.6, 152.2, 141.9, 141.7, 136.7, 114.9, 114.8, 102.8, 61.0, 56.3, 55.9, 55.0, 25.4; HRMS (ESI) calcd. for C₁₈H₂₄O₄N (M+H): 318.1700, Found: 318.1697.



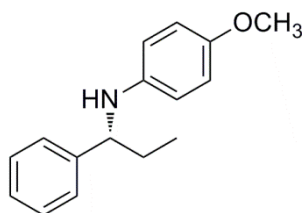
***N*-(1-(benzo[d][1,3]dioxol-5-yl)ethyl)-4-methoxyaniline (3l)**⁹: yellow oil, $[\alpha]_{\text{D}}^{25} = +11.9$ (c 0.91, CHCl₃) (59% ee) [lit.: $[\alpha]_{\text{D}}^{20} = +12.7$ (c 1.10, CHCl₃) (48% ee)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 6.95 (s, 1H), 6.90 (d, $J = 8.0$ Hz, 1H), 6.75 (d, $J = 8.0$ Hz, 1H), 6.72 (d, $J = 8.8$ Hz, 2H), 6.49 (d, $J = 8.8$ Hz, 2H), 5.92 (dd, $J = 1.2, 5.2$ Hz, 2H), 4.34 (q, $J = 6.4$ Hz, 1H), 3.71 (s, 3H), 1.46 (d, $J = 6.4$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 152.1, 148.1, 146.5, 141.7, 139.9, 119.1, 114.9, 114.7, 108.5, 106.5, 101.1, 55.9, 54.2, 25.5.



(*R*)-4-methoxy-*N*-(1-(naphthalen-2-yl)ethyl)aniline (3m)¹⁰: yellow oil, $[\alpha]_{\text{D}}^{25} = +9.0$ (*c* 1.08, EtOAc) (71% ee) [lit.: $[\alpha]_{\text{D}}^{25} = +14.3$ (*c* 0.60, EtOAc) (88% ee)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.82-7.79 (m, 3H), 7.51 (d, *J* = 8.8 Hz, 1H), 7.46-7.43 (m, 2H), 6.68 (d, *J* = 8.8 Hz, 2H), 6.51 (d, *J* = 8.8 Hz, 2H), 4.57 (q, *J* = 6.4 Hz, 1H), 3.87 (brs, 1H), 3.68 (s, 3H), 1.58 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 152.2, 143.3, 141.8, 133.8, 133.0, 128.6, 128.0, 127.9, 126.2, 125.7, 124.7, 124.5, 115.0, 114.9, 56.0, 54.7, 25.3.

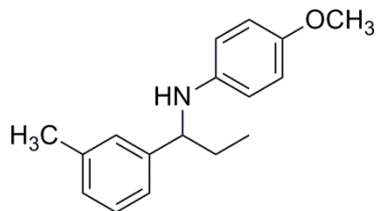


4-methoxy-*N*-(1-(6-methoxynaphthalen-2-yl)ethyl)aniline (3n): white solid, $[\alpha]_{\text{D}}^{25} = +29.9$ (*c* 0.51, CHCl₃) (74% ee); IR (film): 3403, 1607, 1514 cm⁻¹; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.74-7.68 (m, 3H), 7.47 (d, *J* = 8.4 Hz, 1H), 7.14-7.12 (m, 2H), 6.68 (d, *J* = 8.8 Hz, 2H), 6.52 (d, *J* = 8.8 Hz, 2H), 4.54 (q, *J* = 6.8 Hz, 1H), 3.91 (s, 3H), 3.86 (brs, 1H), 3.68 (s, 3H), 1.56 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 157.6, 152.1, 141.9, 140.9, 134.0, 129.5, 129.2, 127.5, 125.2, 124.4, 119.0, 115.0, 114.9, 105.9, 55.9, 55.5, 54.6, 25.3; HRMS (ESI) calcd. for C₂₀H₂₂O₂N (M+H): 308.1645, Found: 308.1644.

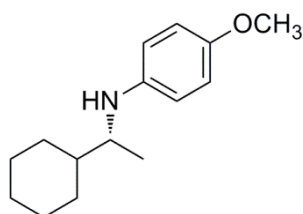


(*R*)-4-methoxy-*N*-(1-phenylpropyl)aniline (3o)^{5,6}: yellow oil, $[\alpha]_{\text{D}}^{25} = +1.3$ (*c* 0.96, EtOAc) (50% ee) [lit.: $[\alpha]_{\text{D}}^{25} = +11.9$ (*c* 0.74, EtOAc) (92% ee)]; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.32-7.28 (m, 4H),

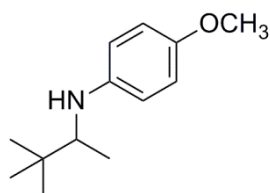
7.21-7.15 (m, 1H), 6.67 (d, $J = 8.8$ Hz, 2H), 6.45 (d, $J = 8.8$ Hz, 2H), 4.13 (t, $J = 6.4$ Hz, 1H), 3.81 (brs, 1H), 3.64 (s, 3H), 1.86-1.70 (m, 2H), 0.92 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 151.9, 144.3, 142.0, 128.6, 127.0, 126.7, 114.9, 114.6, 60.7, 55.8, 31.8, 11.0.



4-methoxy-*N*-(1-(*m*-tolyl)propyl)aniline (3p): yellow oil, $[\alpha]_{\text{D}}^{25} = +16.6$ (c 0.91, CHCl_3) (55% ee); IR (film): 3405, 1607, 1512, 1463 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.19 (dd, $J = 5.6, 14.4$ Hz, 1H), 7.13 (s, 1H), 7.11 (d, $J = 9.6$ Hz, 2H), 7.02 (d, $J = 7.2$ Hz, 1H), 6.68 (d, $J = 8.8$ Hz, 2H), 6.47 (d, $J = 8.8$ Hz, 2H), 4.09 (t, $J = 6.6$ Hz, 1H), 3.79 (s, 1H), 2.32 (s, 3H), 1.86-1.70 (m, 2H), 0.93 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 151.9, 144.4, 142.1, 138.2, 128.5, 127.8, 127.4, 123.8, 114.9, 114.6, 60.8, 55.9, 31.9, 21.7, 11.1; HRMS (ESI) calcd. for $\text{C}_{17}\text{H}_{22}\text{ON}$ ($\text{M}+\text{H}$): 256.1696, Found: 256.1694.



(*R*)-*N*-(1-cyclohexylethyl)-4-methoxyaniline (3q)²: yellow oil, $[\alpha]_{\text{D}}^{25} = -4.4$ (c 0.98, CHCl_3) (44% ee) [lit.: $[\alpha]_{\text{D}} = -19.7$ (c 0.20, CHCl_3) (98% ee for *R* isomer)]; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 6.79 (d, $J = 7.2$ Hz, 2H), 6.57 (d, $J = 7.2$ Hz, 2H), 3.76 (s, 3H), 3.27-3.22 (m, 1H), 3.24 (brs, 1H), 1.85-1.68 (m, 5H), 1.49-1.42 (m, 1H), 1.28-1.18 (m, 3H), 1.14-1.07 (m, 2H), 1.13 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 151.8, 142.4, 115.1, 114.7, 56.0, 54.3, 43.0, 30.0, 28.4, 26.8, 26.7, 26.5, 17.5.

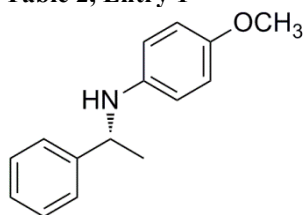


***N*-(3,3-dimethylbutan-2-yl)-4-methoxyaniline (3r)¹¹**: yellow oil, $[\alpha]_{\text{D}}^{25} = -25.7$ (c 1.12, CHCl_3) (60% ee) [lit.: $[\alpha]_{\text{D}} = -18.6$ (c 1.0, CHCl_3) (39% ee)]; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 6.79 (d, $J = 8.4$ Hz, 2H), 6.59 (d, $J = 8.4$ Hz, 2H), 3.76 (s, 3H), 3.13 (q, $J = 6.4$ Hz, 1H), 3.09 (brs, 1H), 1.88 (d, $J = 6.4$ Hz, 3H), 0.98 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 151.8, 143.1, 115.1, 114.7, 58.8, 56.0, 34.9, 26.7, 16.0.

References

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Table 2, Entry 1



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm

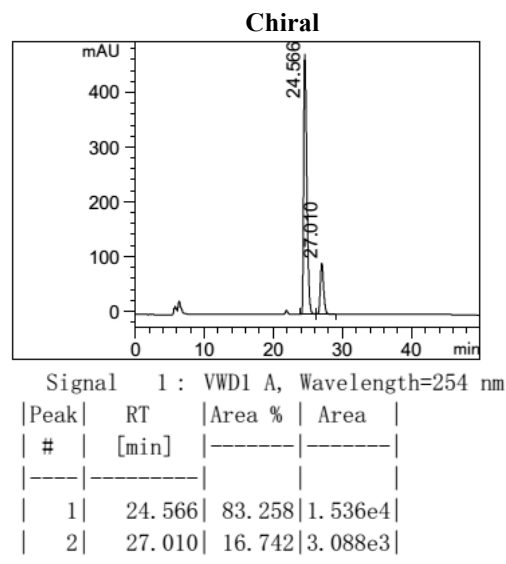
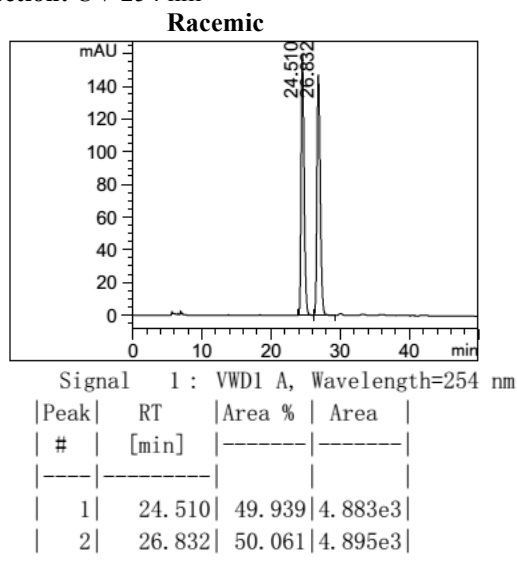
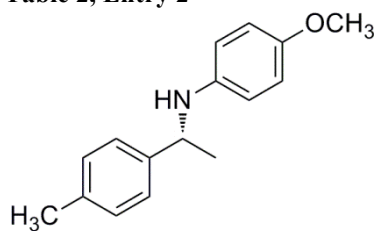


Table 2, Entry 2



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm

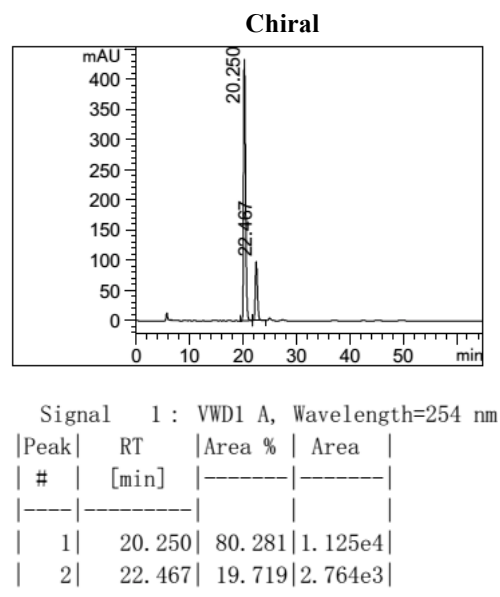
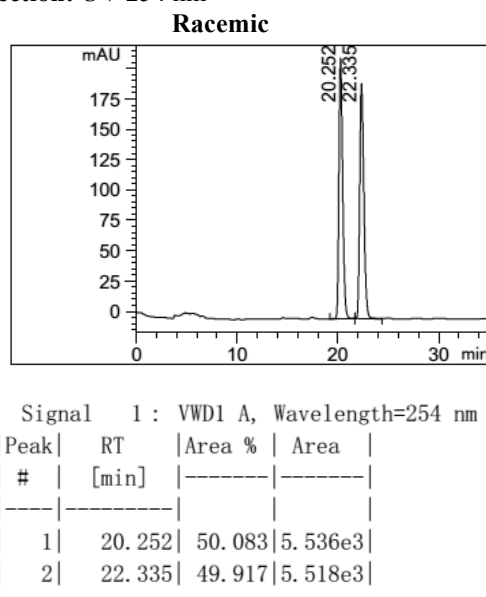
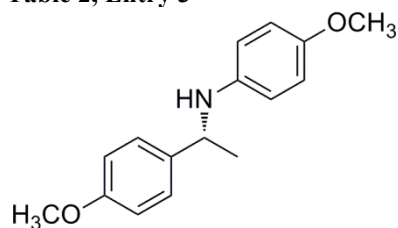
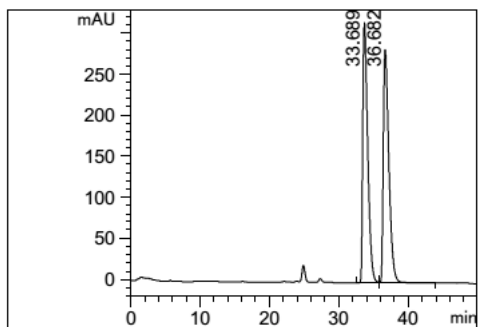


Table 2, Entry 3



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm

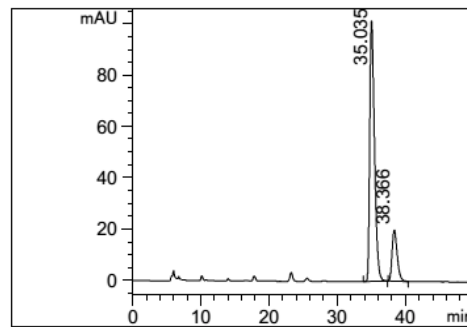
Racemic



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RT [min]	Area %	Area
1	33.689	49.891	1.582e4
2	36.682	50.109	1.589e4

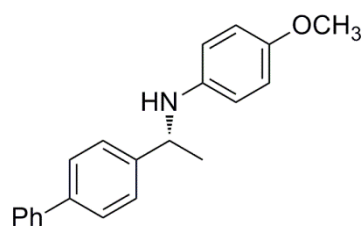
Chiral



Signal 1: VWD1 A, Wavelength=254 nm

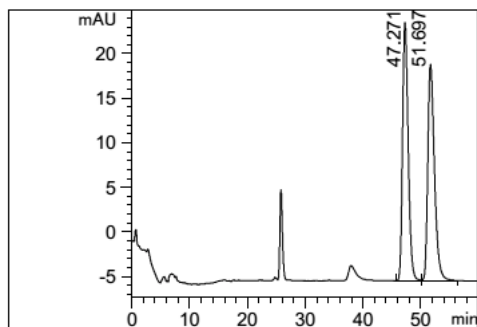
Peak #	RT [min]	Area %	Area
1	35.035	82.660	4.940e3
2	38.366	17.340	1.036e3

Table 2, Entry 4



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm

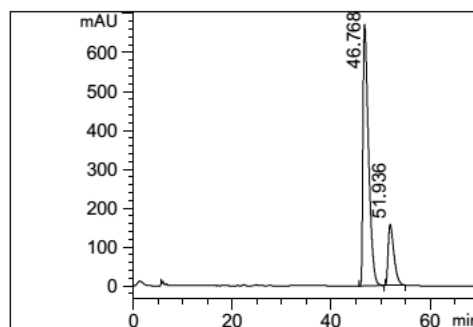
Racemic



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RT [min]	Area %	Area
1	47.271	50.222	1.966e3
2	51.697	49.778	1.948e3

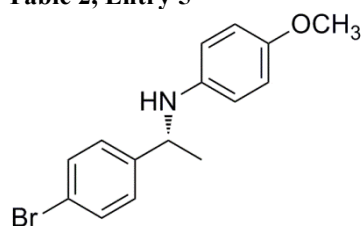
Chiral



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RT [min]	Area %	Area
1	46.768	80.515	5.279e4
2	51.936	19.485	1.278e4

Table 2, Entry 5



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 1.0 mL/min; **Detection:** UV 254 nm

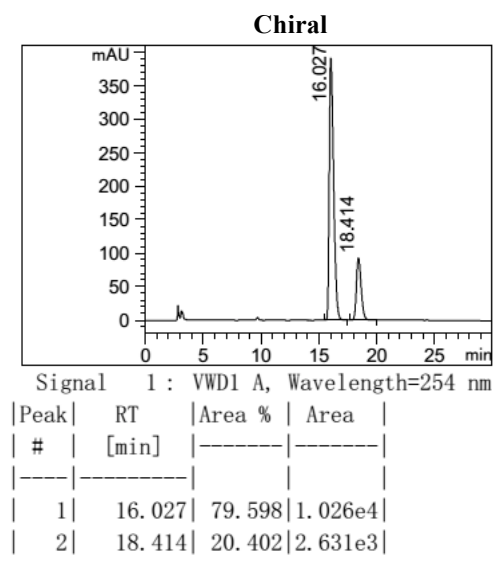
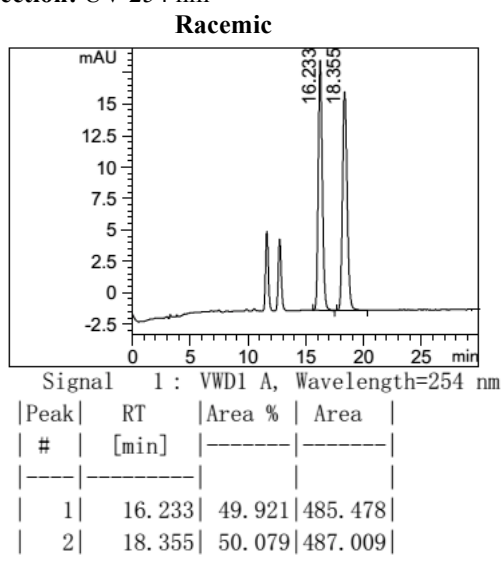
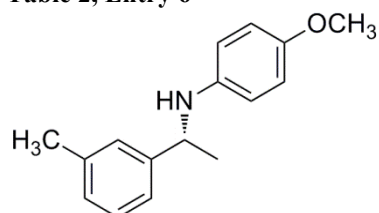


Table 2, Entry 6



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (99/1); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm

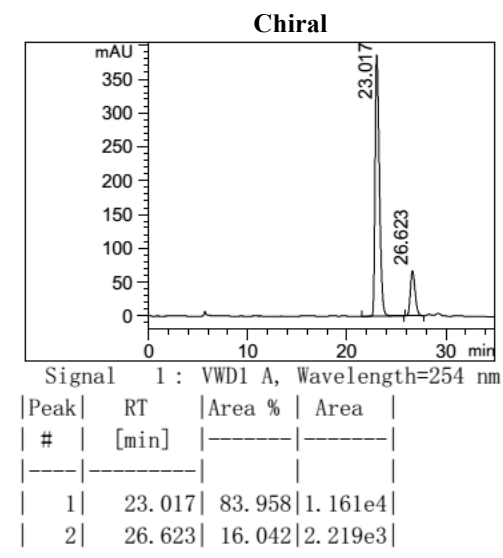
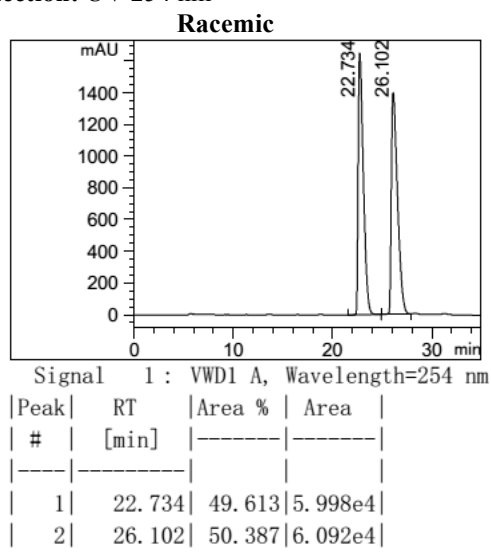
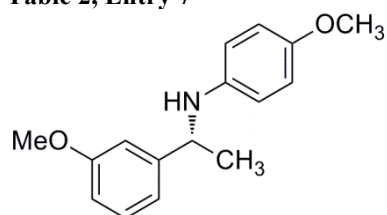


Table 2, Entry 7



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm

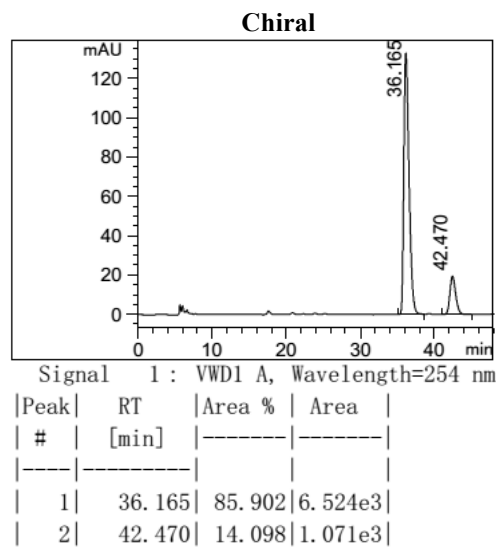
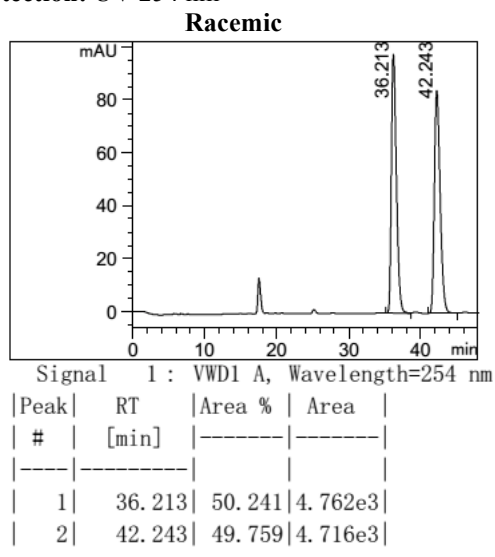
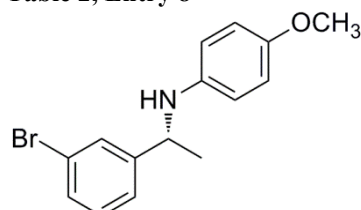


Table 2, Entry 8



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm

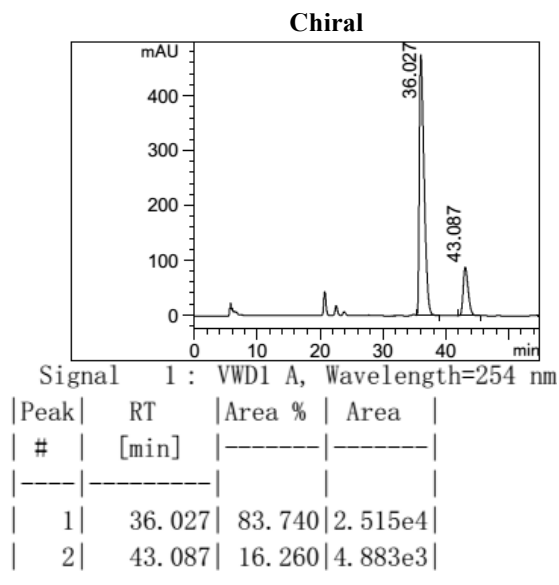
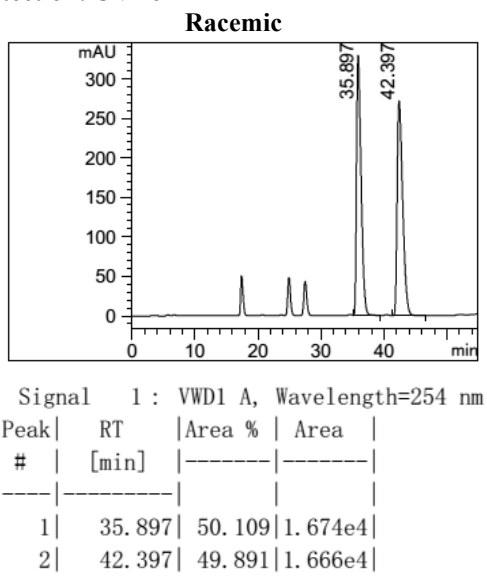
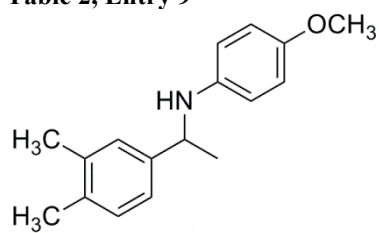
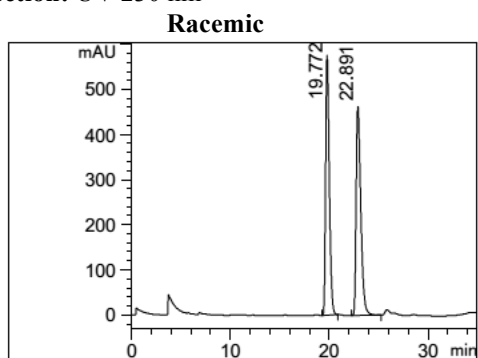


Table 2, Entry 9

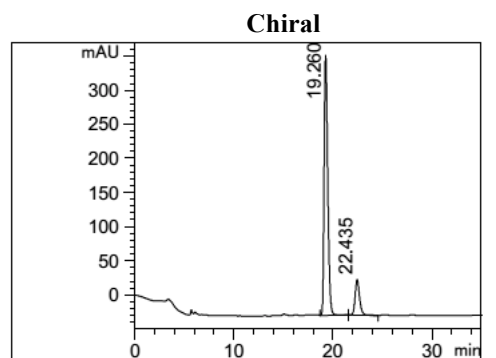


HPLC Conditions: **Column:** Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 230 nm



Signal 1: VWD1 A, Wavelength=230 nm

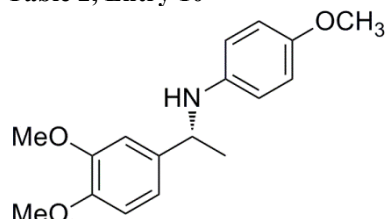
Peak #	RT [min]	Area %	Area
1	19.772	49.562	1.496e4
2	22.891	50.438	1.523e4



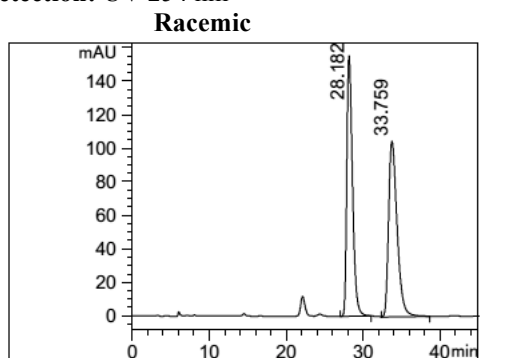
Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RT [min]	Area %	Area
1	19.260	85.431	9.647e3
2	22.435	14.569	1.645e3

Table 2, Entry 10

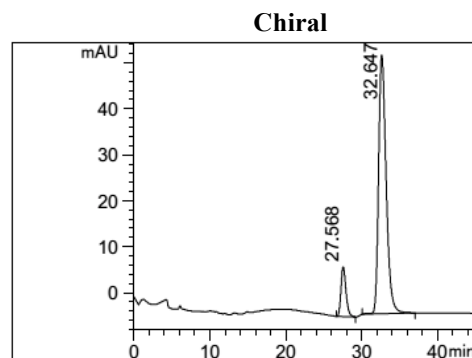


HPLC Conditions: **Column:** Chiralpak AS-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (90/10); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm



Signal 1: VWD1 A, Wavelength=254 nm

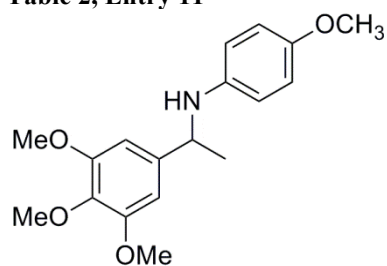
Peak #	RT [min]	Area %	Area
1	28.182	49.749	7.963e3
2	33.759	50.251	8.043e3



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RT [min]	Area %	Area
1	27.568	11.201	490.937
2	32.647	88.799	3.892e3

Table 2, Entry 11



HPLC Conditions: **Column:** Chiralpak IC-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (85/15); **Flow rate:** 0.8 mL/min; **Detection:** UV 254 nm

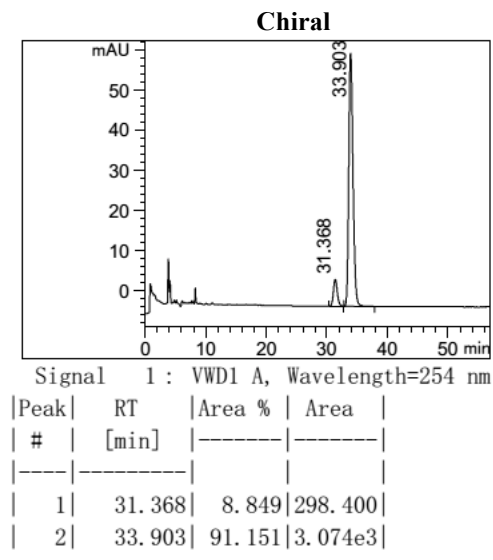
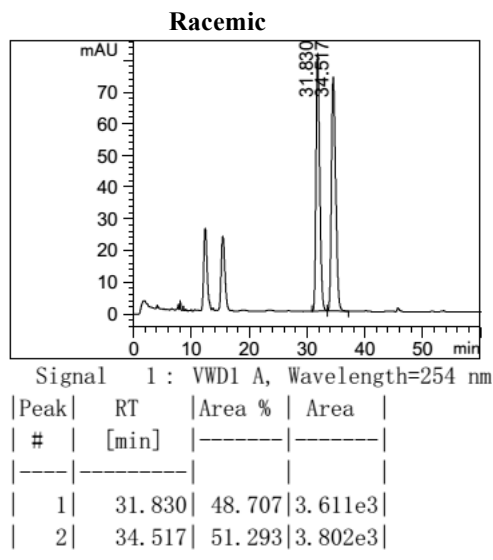
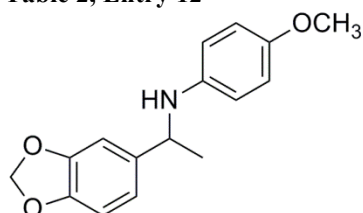


Table 2, Entry 12



HPLC Conditions: **Column:** Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (95/5); **Flow rate:** 0.5mL/min; **Detection:** UV 254 nm

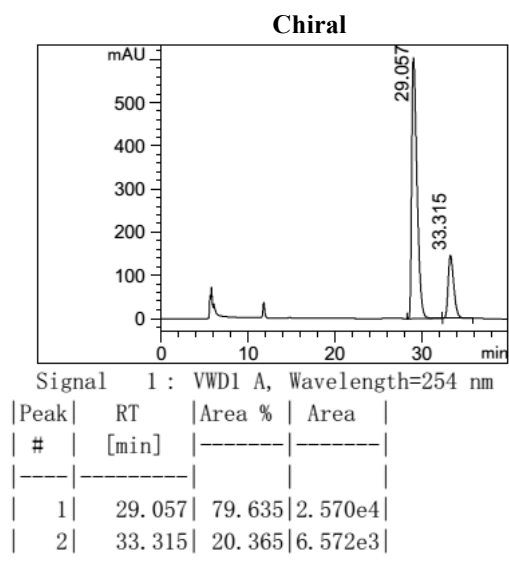
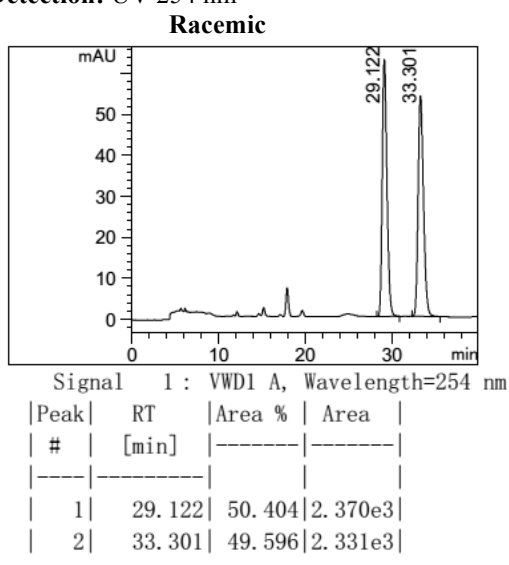
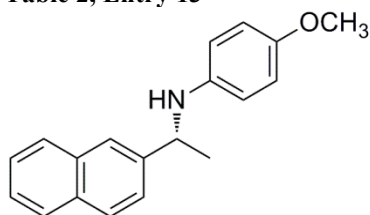
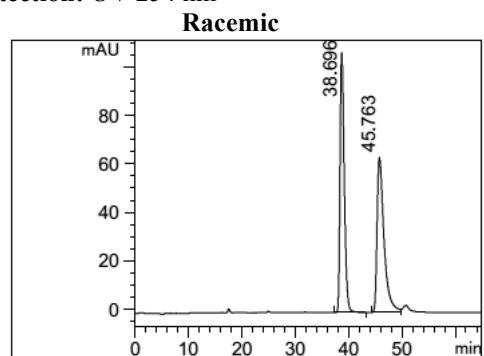


Table 2, Entry 13

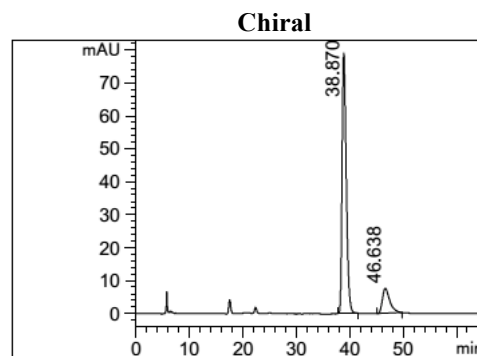


HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA (98/2); Flow rate: 0.5 mL/min; Detection: UV 254 nm



Signal 1: VWD1 A, Wavelength=254 nm

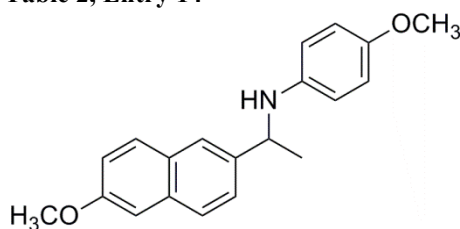
Peak #	RT [min]	Area %	Area
1	38.696	50.531	5.916e3
2	45.763	49.469	5.791e3



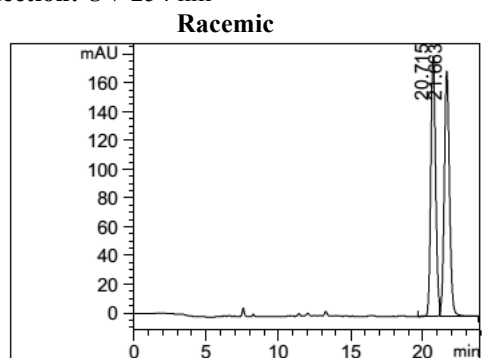
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RT [min]	Area %	Area
1	38.870	85.505	4.304e3
2	46.638	14.495	729.551

Table 2, Entry 14

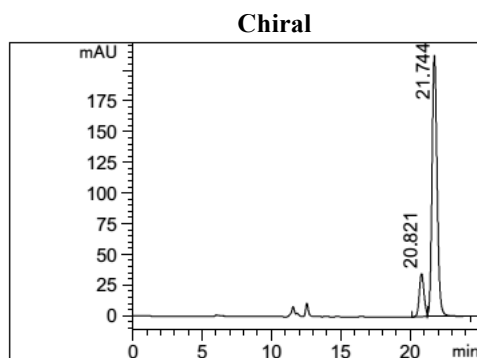


HPLC Conditions: Column: Chiralpak IC-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA (85/15); Flow rate: 0.8 mL/min; Detection: UV 254 nm



Signal 1: VWD1 A, Wavelength=254 nm

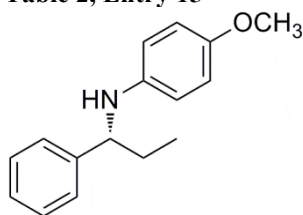
Peak #	RT [min]	Area %	Area
1	20.715	49.470	4.197e3
2	21.663	50.530	4.287e3



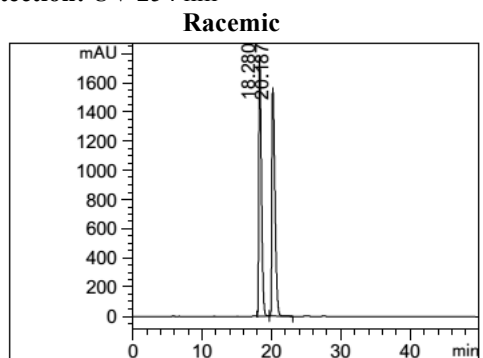
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RT [min]	Area %	Area
1	20.821	13.237	831.573
2	21.744	86.763	5.451e3

Table 2, Entry 15

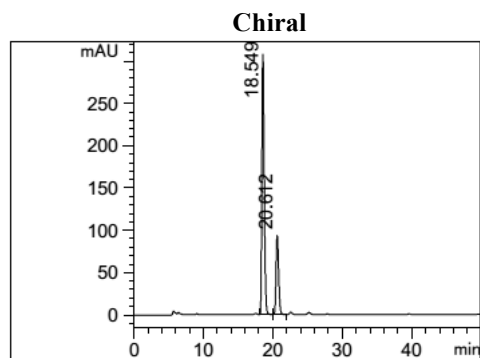


HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm



Signal 1: VWD1 A, Wavelength=254 nm

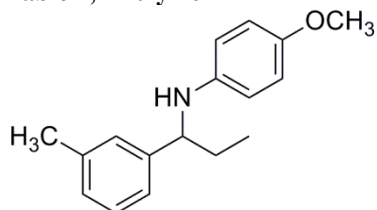
Peak #	RT [min]	Area %	Area
1	18.280	49.707	4.702e4
2	20.187	50.293	4.757e4



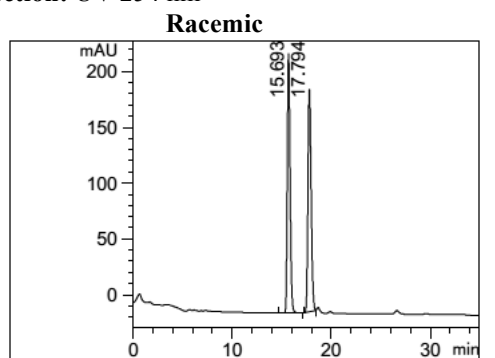
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RT [min]	Area %	Area
1	18.549	74.783	7.224e3
2	20.612	25.217	2.436e3

Table 2, Entry 16

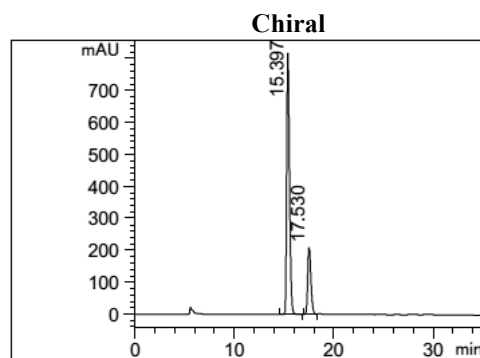


HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm



Signal 1: VWD1 A, Wavelength=254 nm

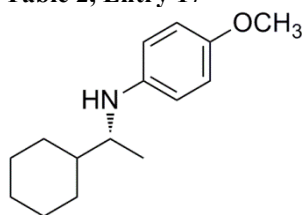
Peak #	RT [min]	Area %	Area
1	15.693	50.352	4.497e3
2	17.794	49.648	4.434e3



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RT [min]	Area %	Area
1	15.397	77.635	1.636e4
2	17.530	22.365	4.713e3

Table 2, Entry 17



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm

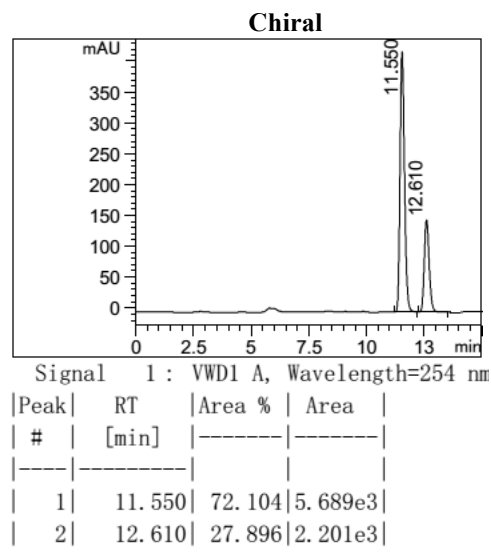
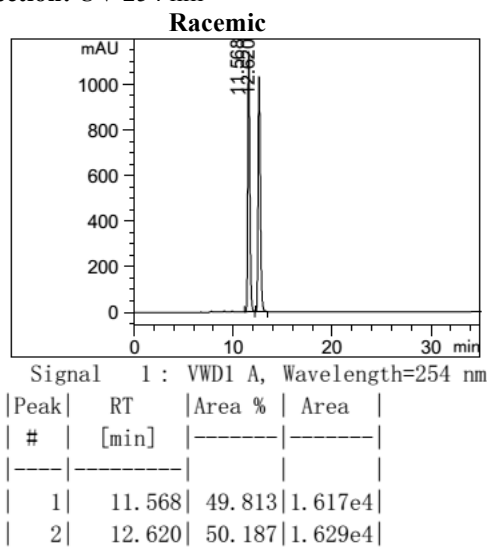
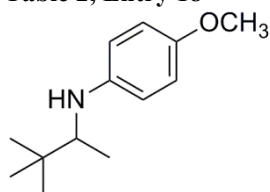
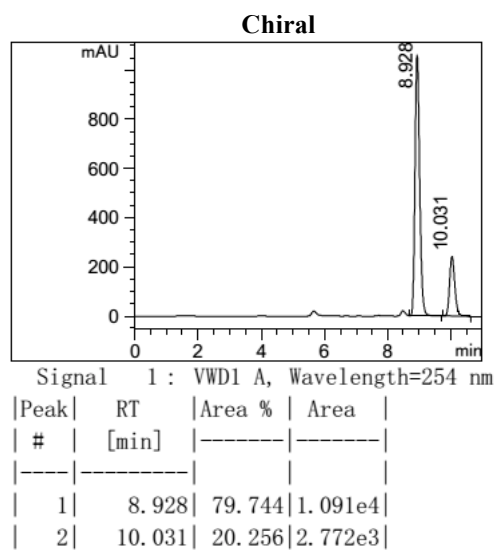
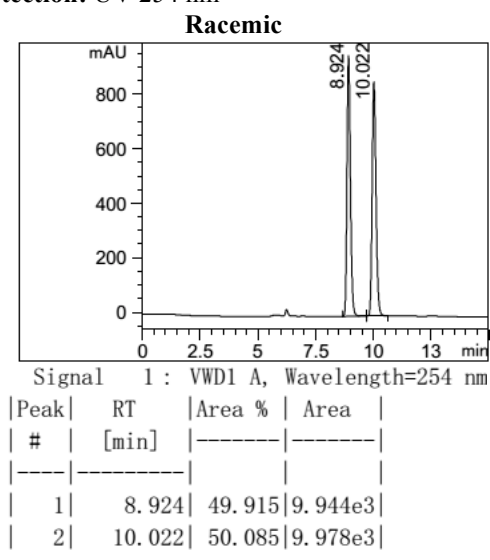
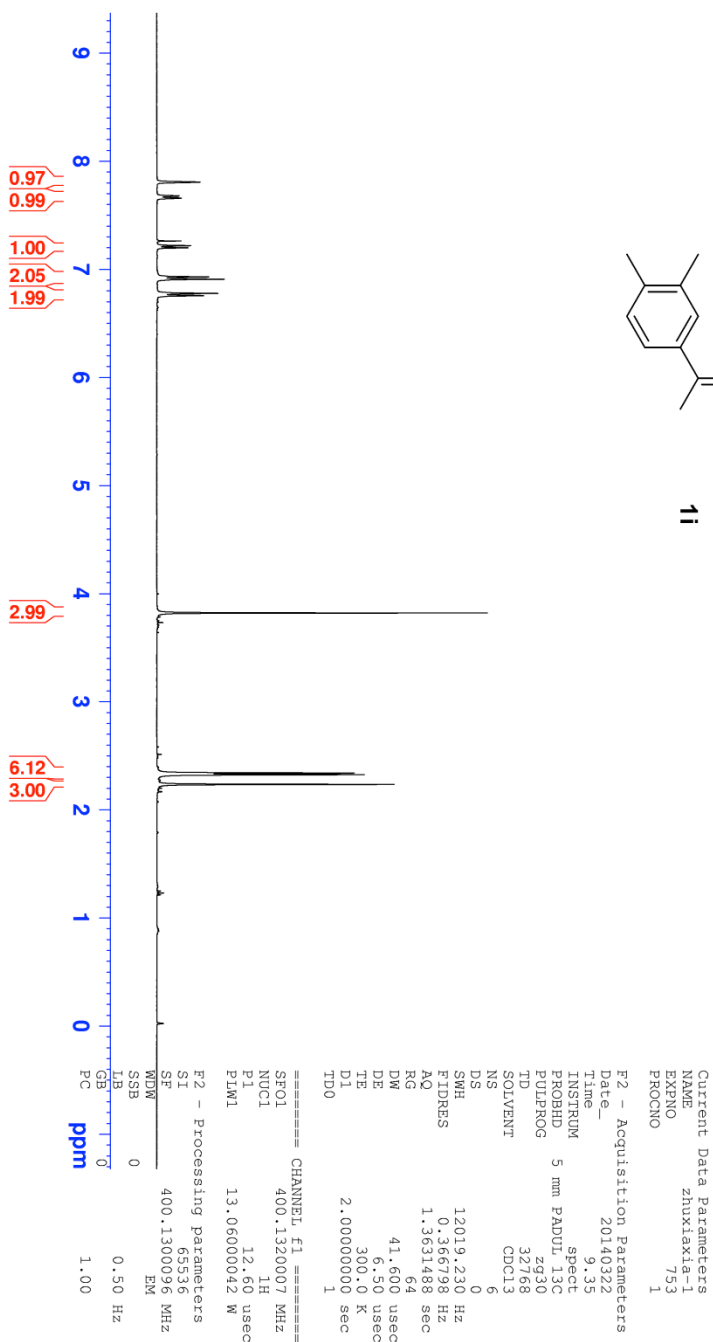
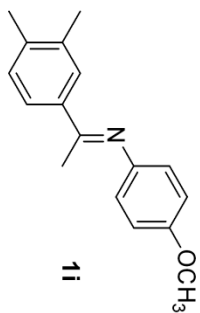


Table 2, Entry 18



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA (98/2); **Flow rate:** 0.5 mL/min; **Detection:** UV 254 nm







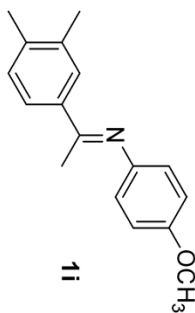
Current Data Parameters
NAME zhuxiaxia-1
EXPNO 754
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140322
Time 9.38
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 448
DS 0
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 0.6488064 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
ID0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 9.40 usec
PLW1 43.63899994 W

===== CHANNEL f2 =====
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG12 waltz16
PCPD2 90.00 usec
PLW2 13.0600042 W
PLW12 0.2535800 W
PLW13 0.2073400 W

F2 - Processing parameters
SI 32768
SF 100.6127567 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

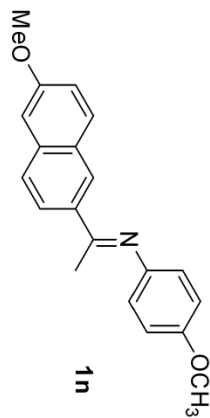


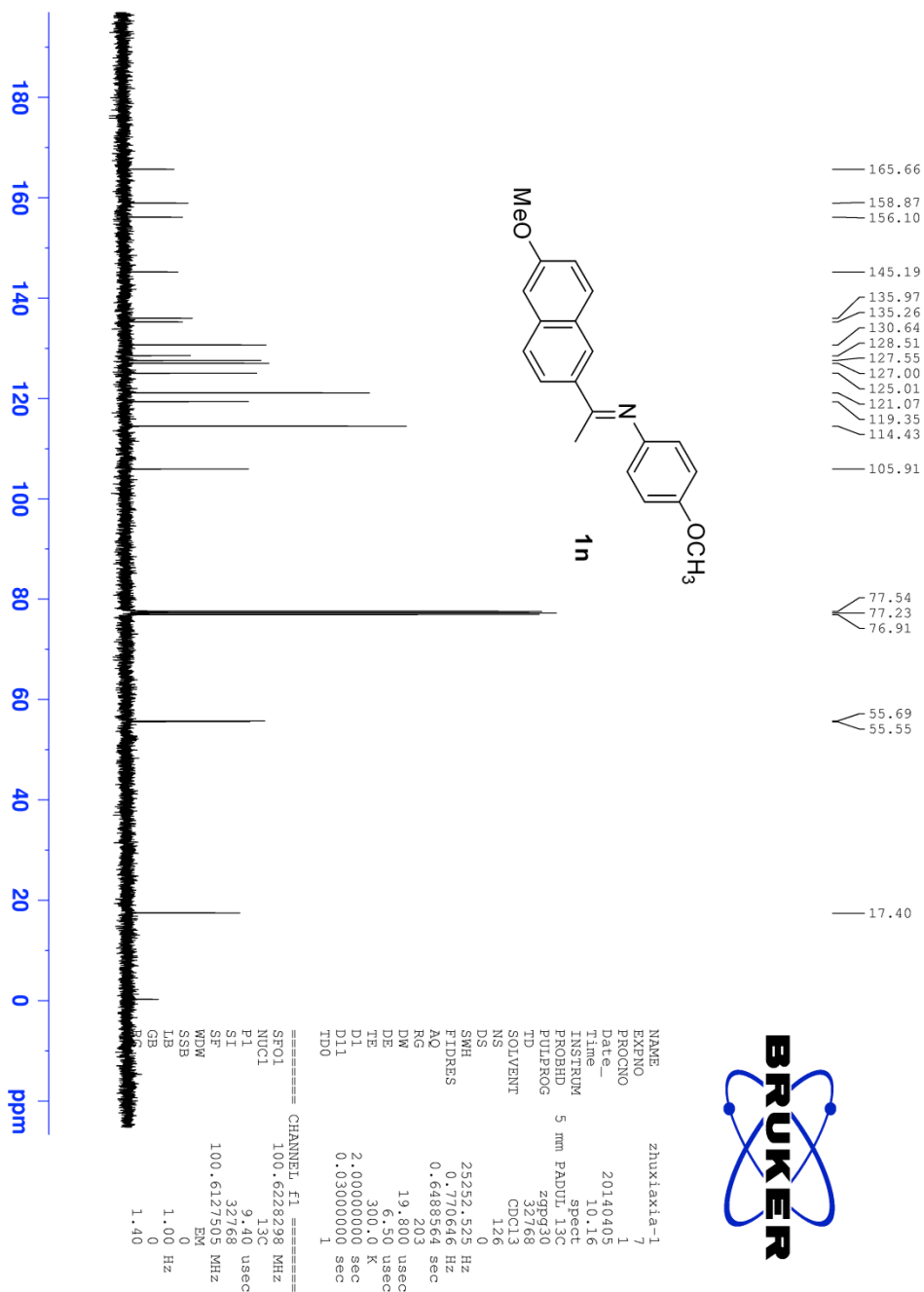
165.99
155.97
145.15
139.41
137.58
136.72
129.71
128.30
124.87
120.96
114.34

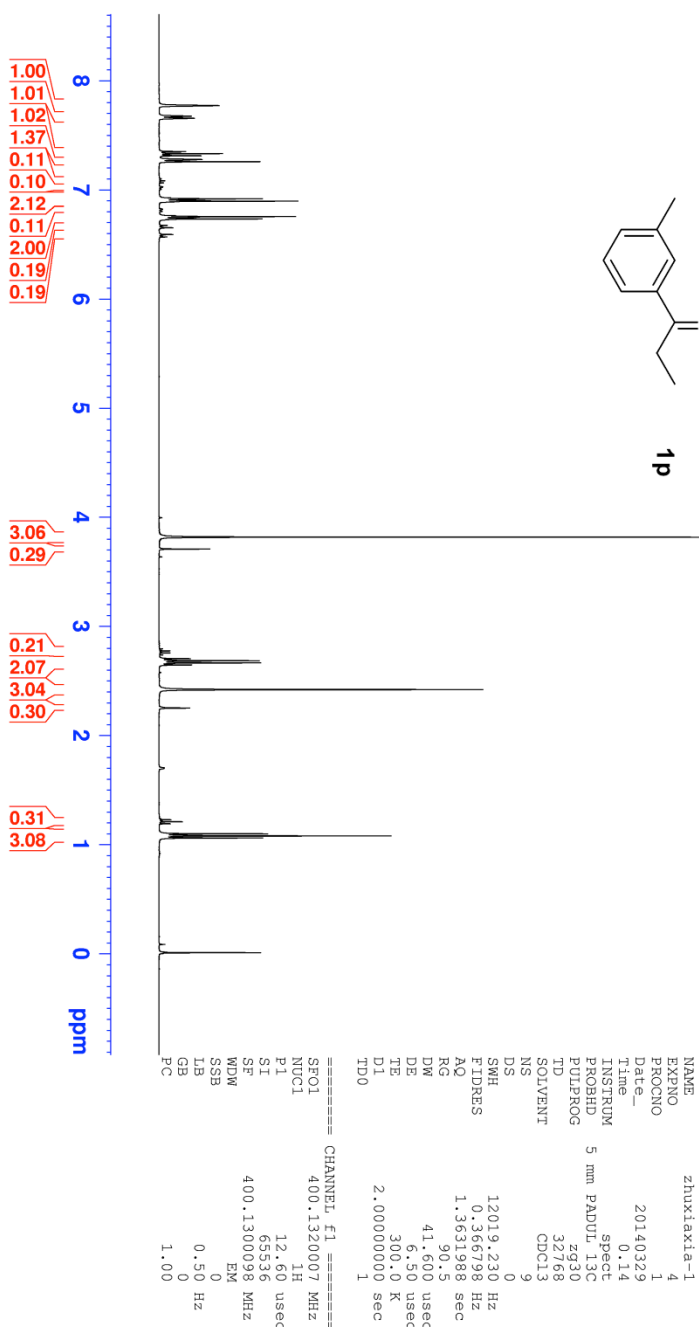
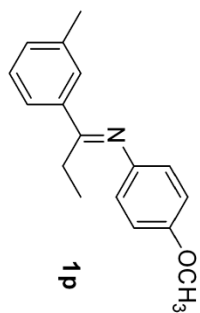
77.55
77.23
76.91

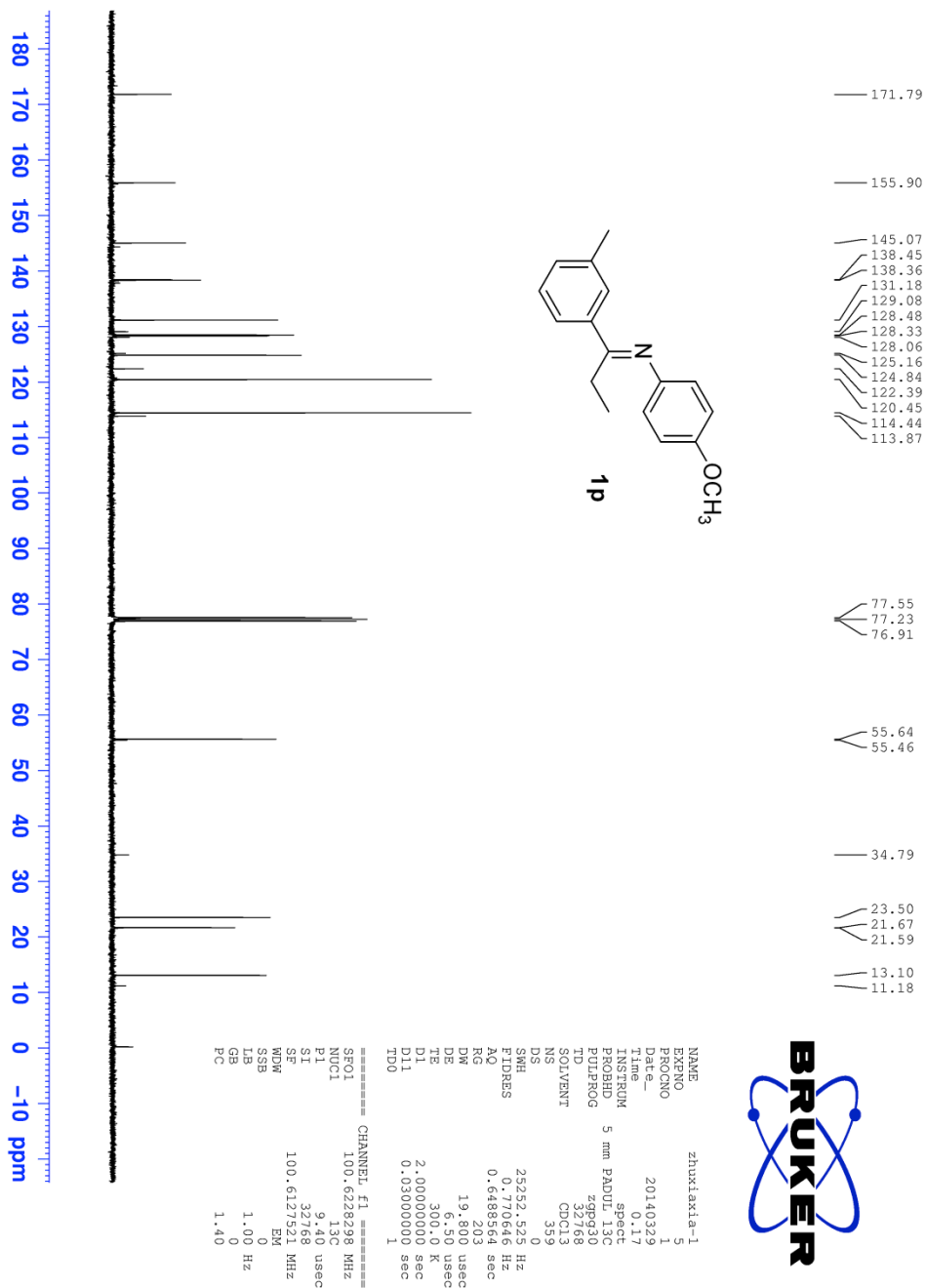
55.60

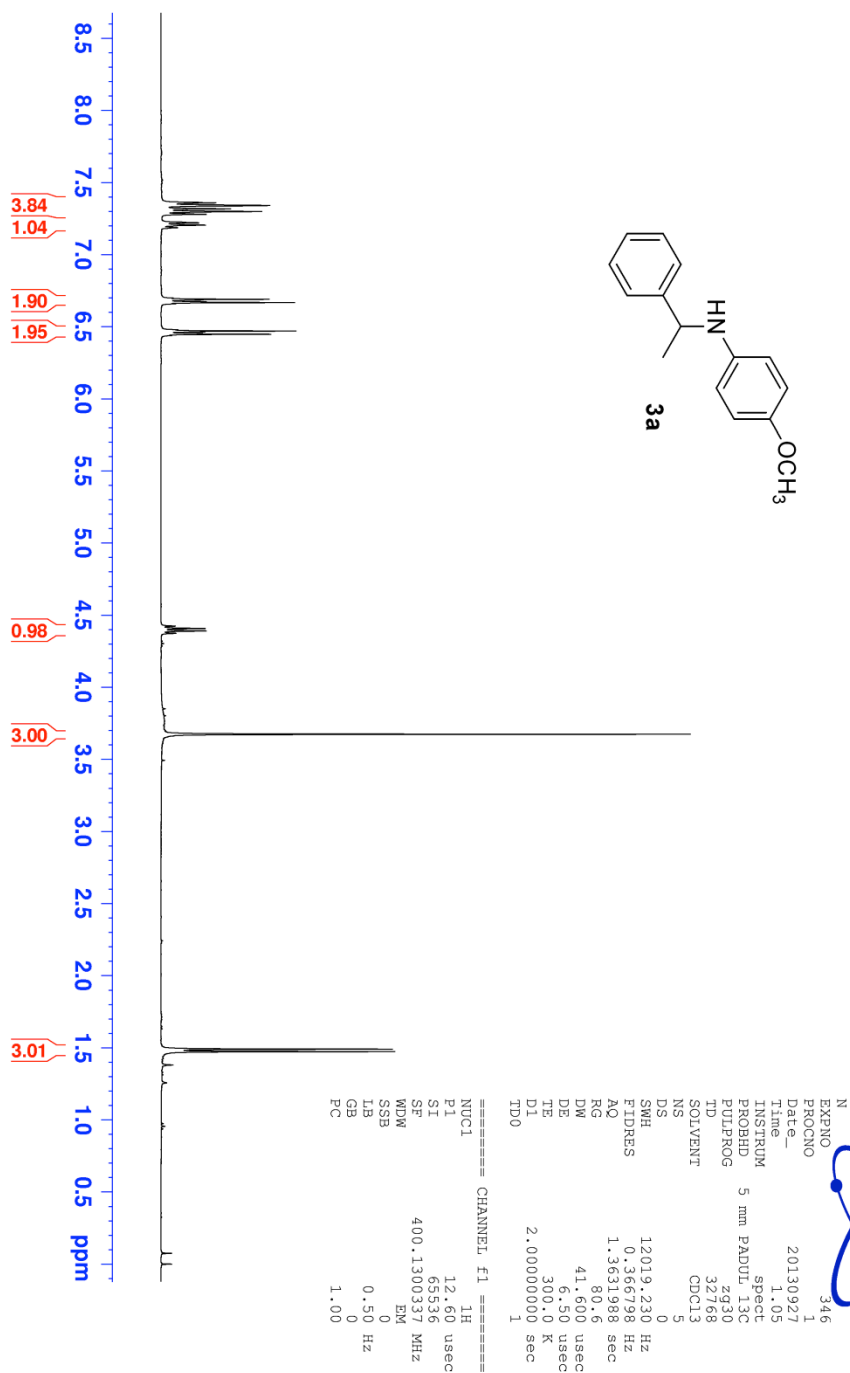
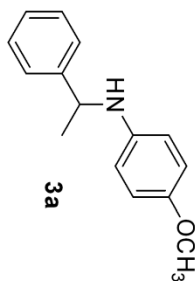
20.00
19.89
17.43

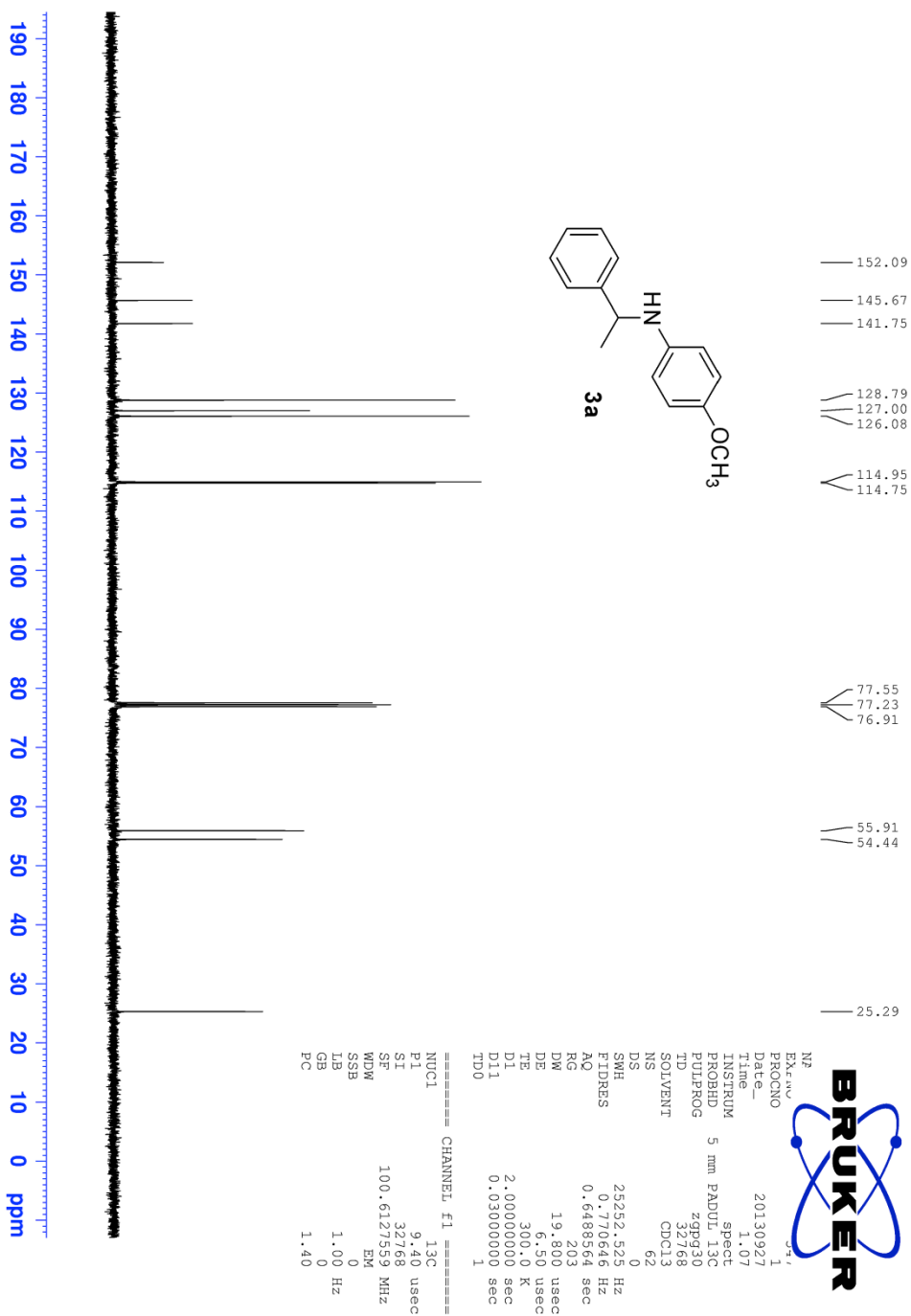


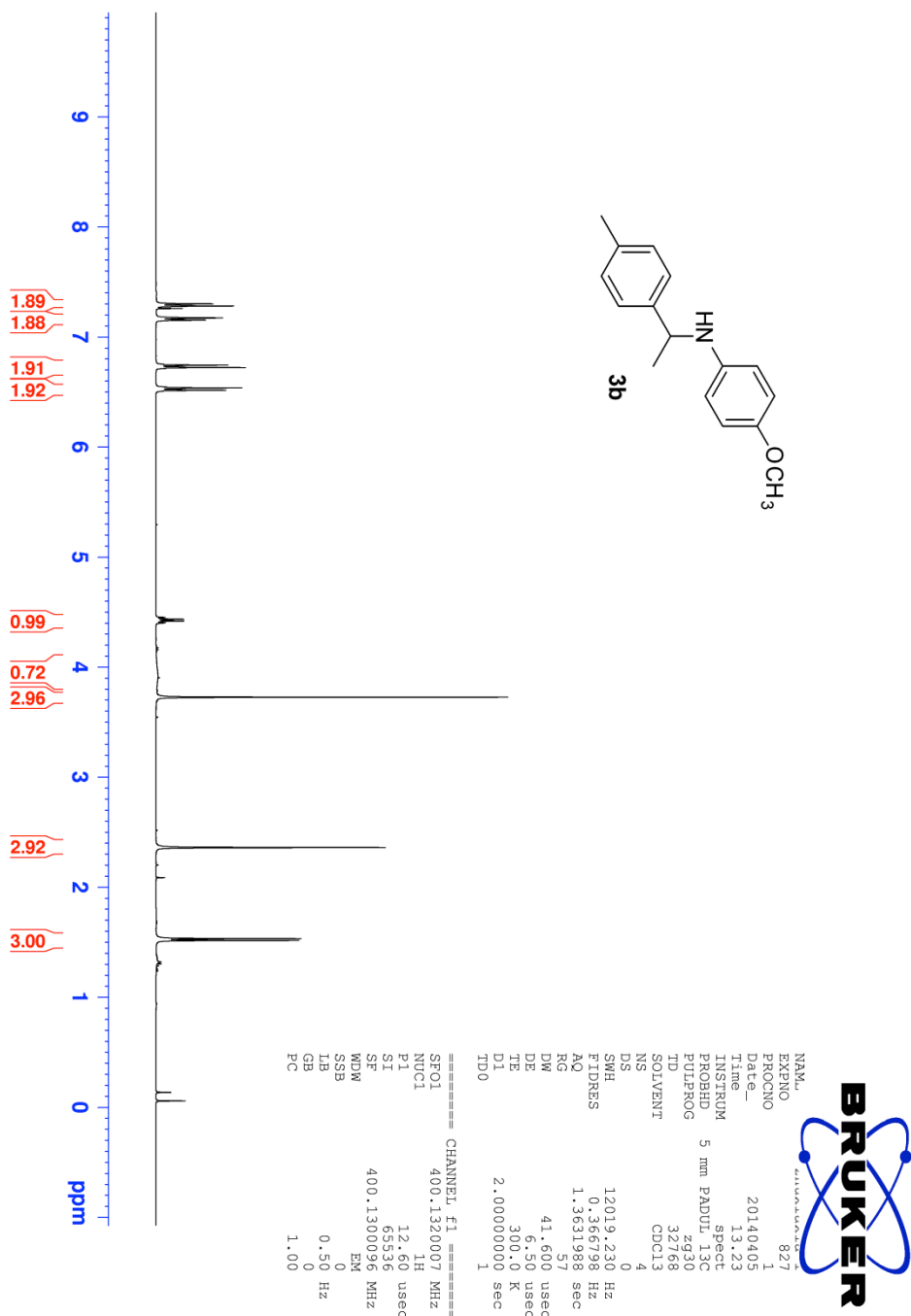
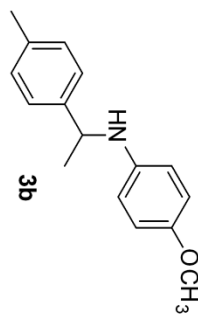


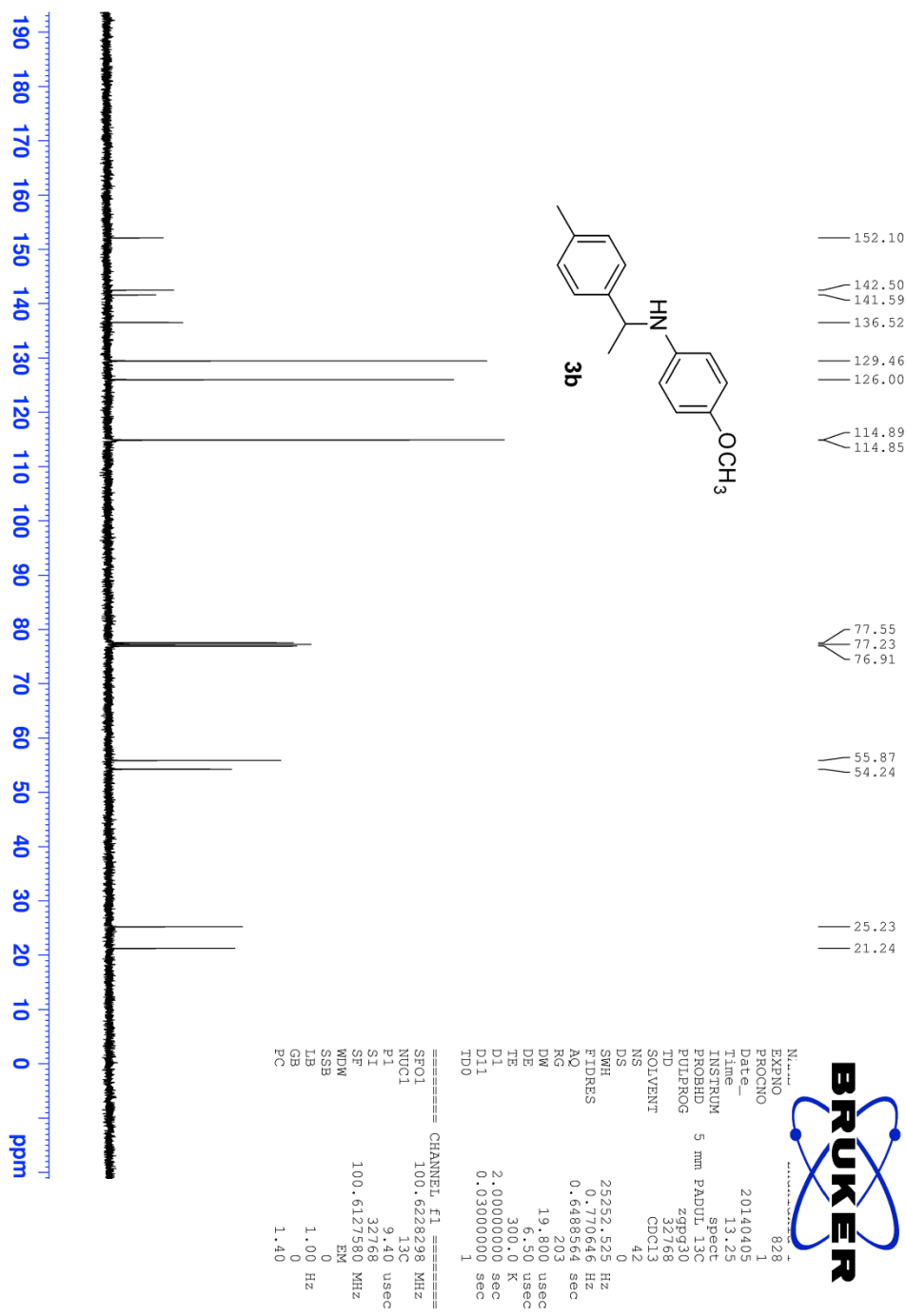








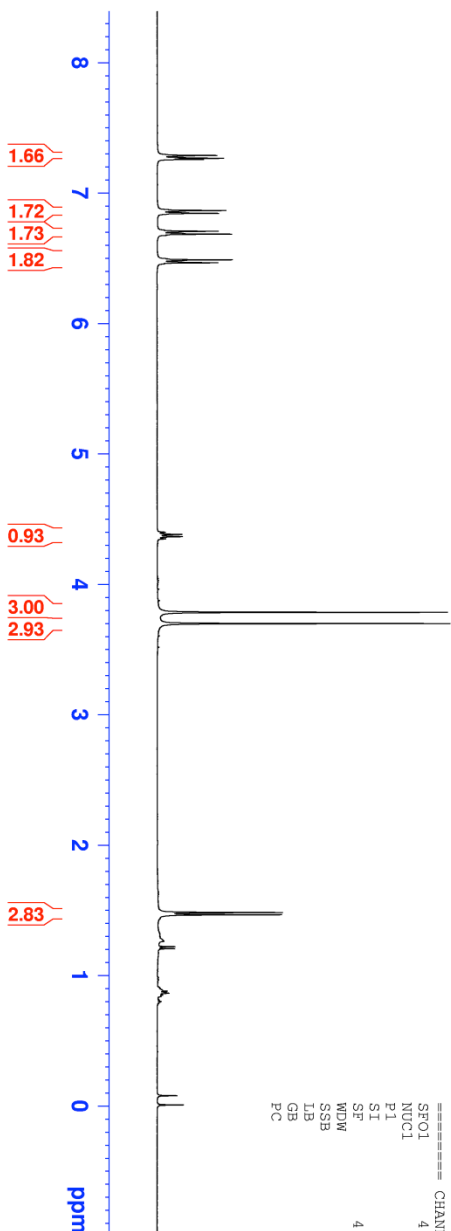
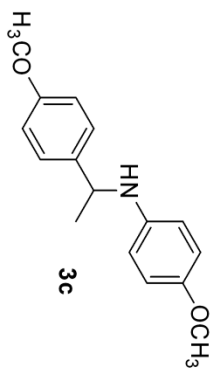


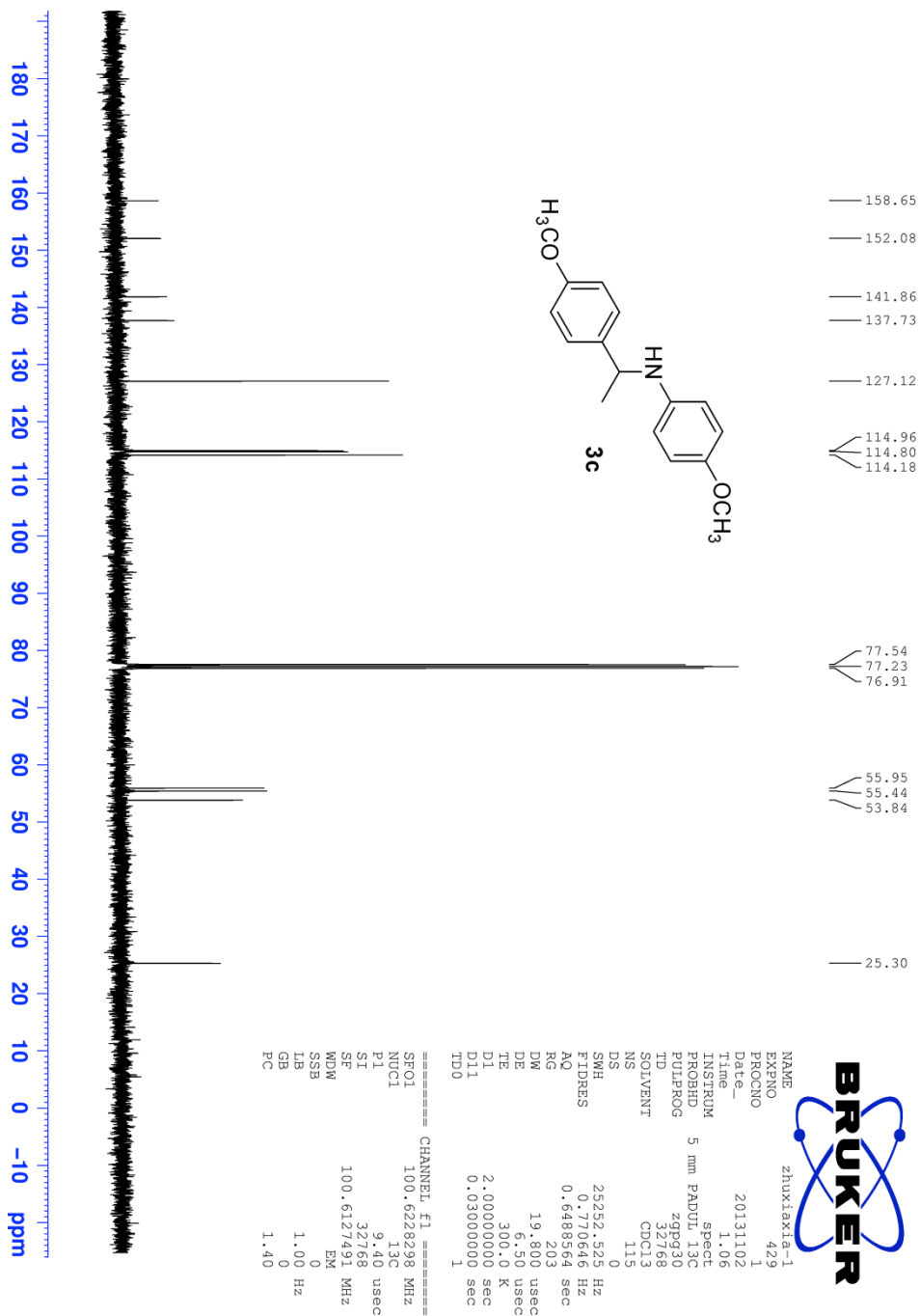


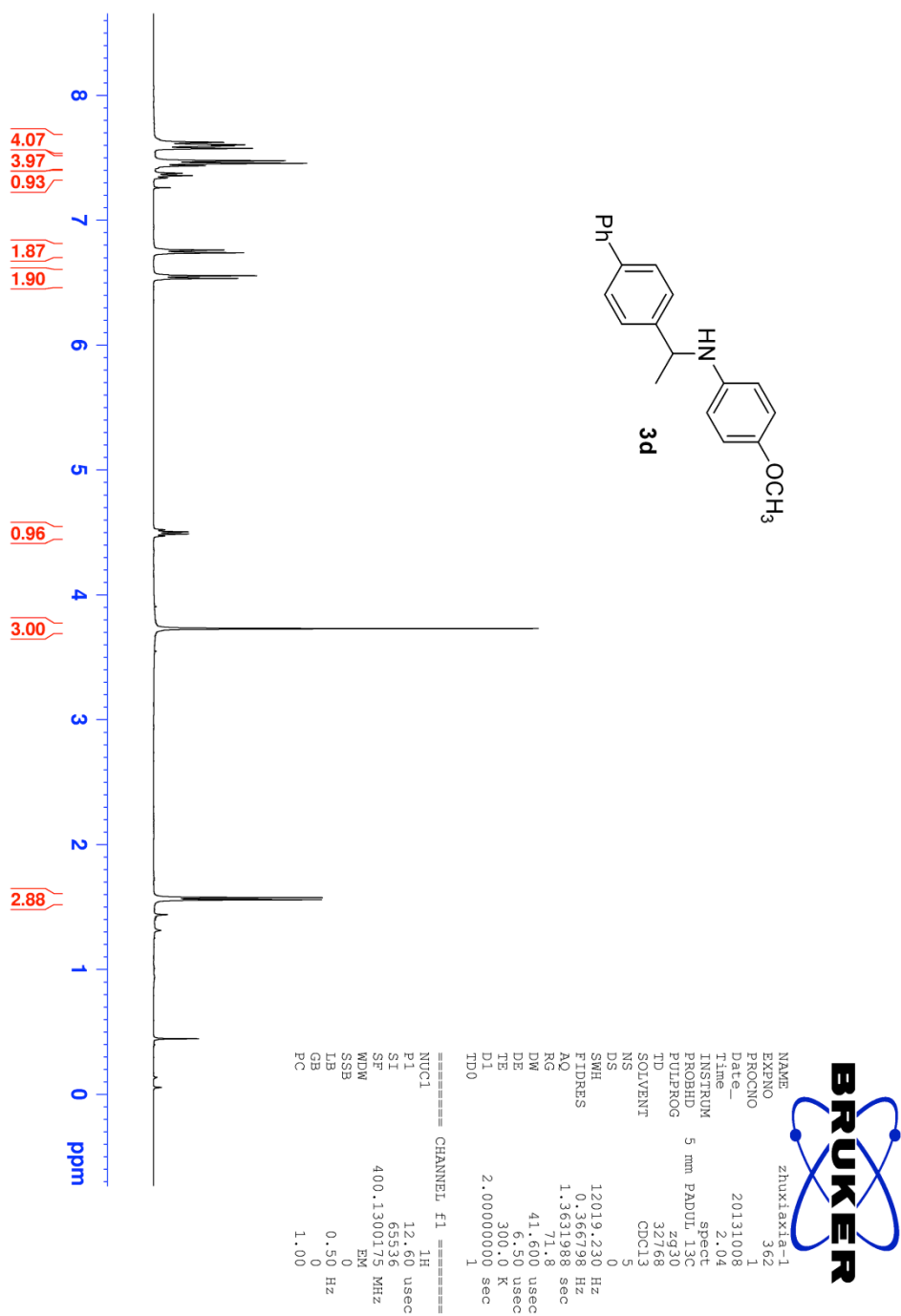


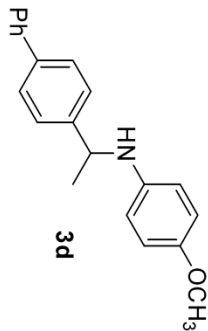
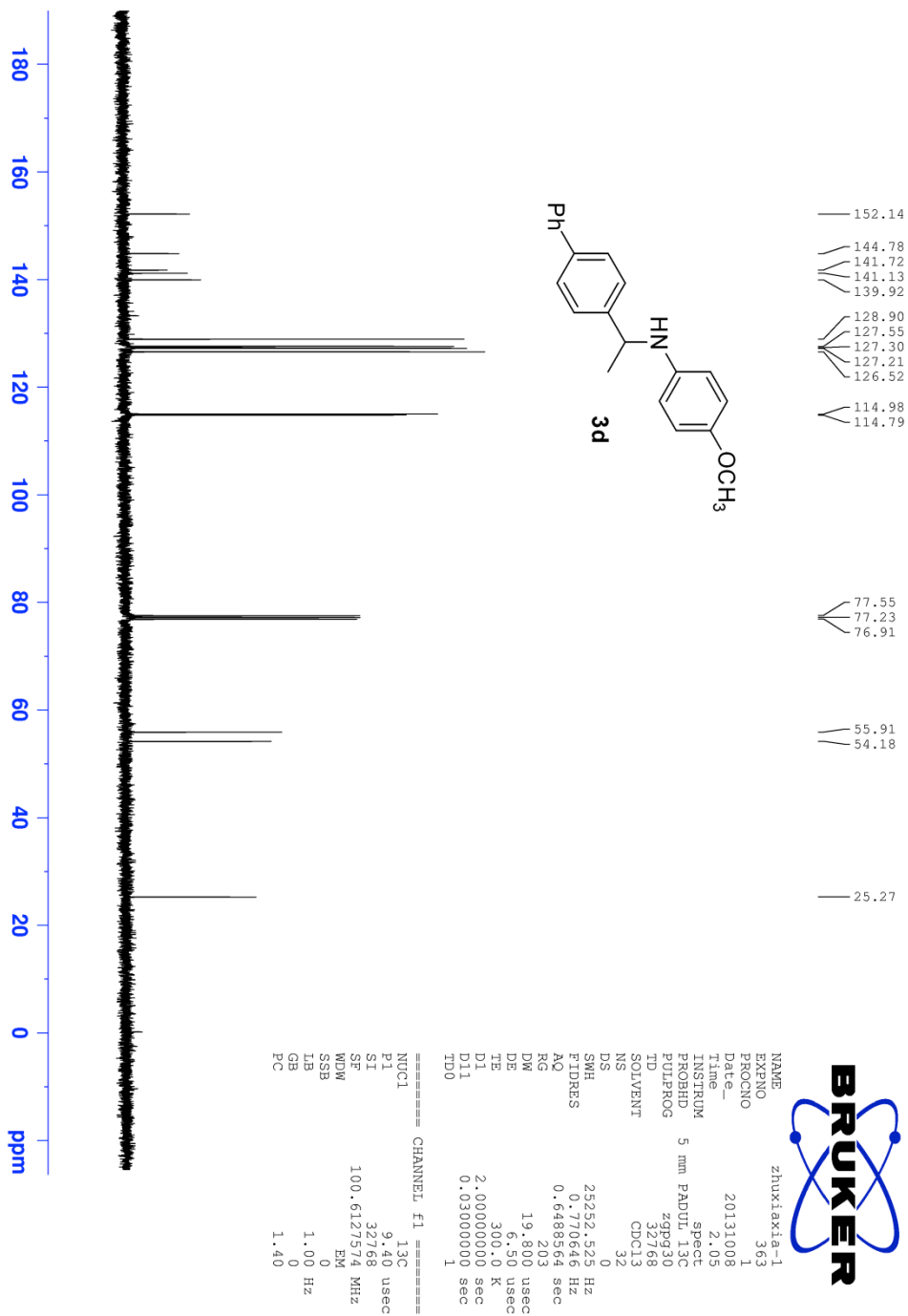
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EXPNO 428
PROCNO 1
Date_ 20131102
Time 1.04
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 6
DS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 144
RW 41.600 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

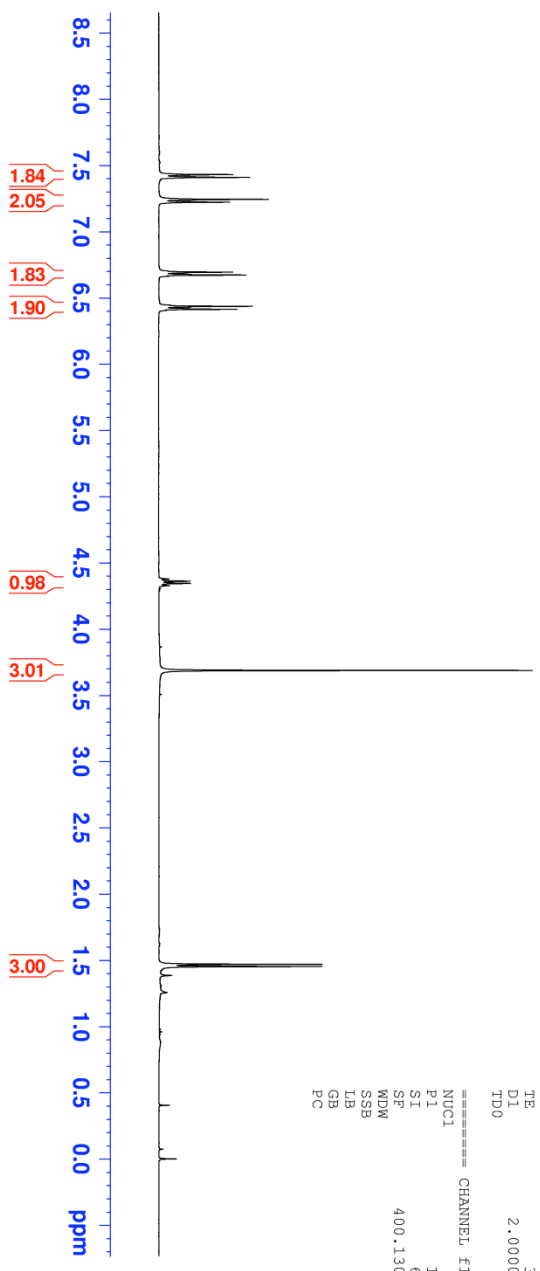
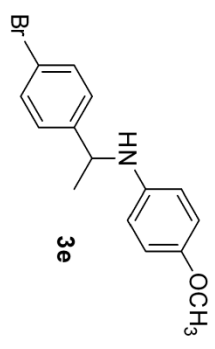
===== CHANNEL f1 =====
SFO1 400.1320007 MHz
NUC1 1H
P1 12.60 usec
SI 65336
SF 400.1300998 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00





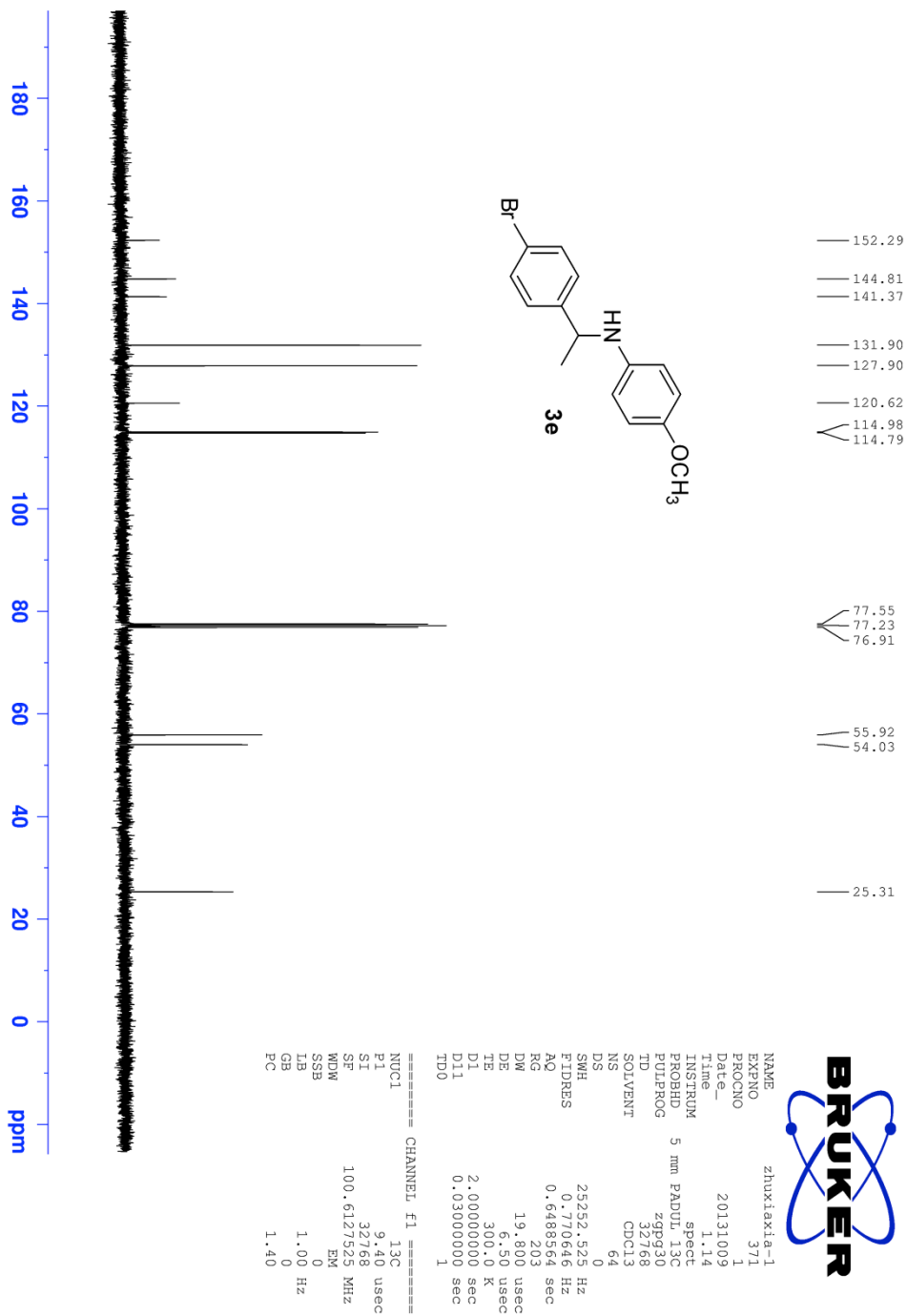


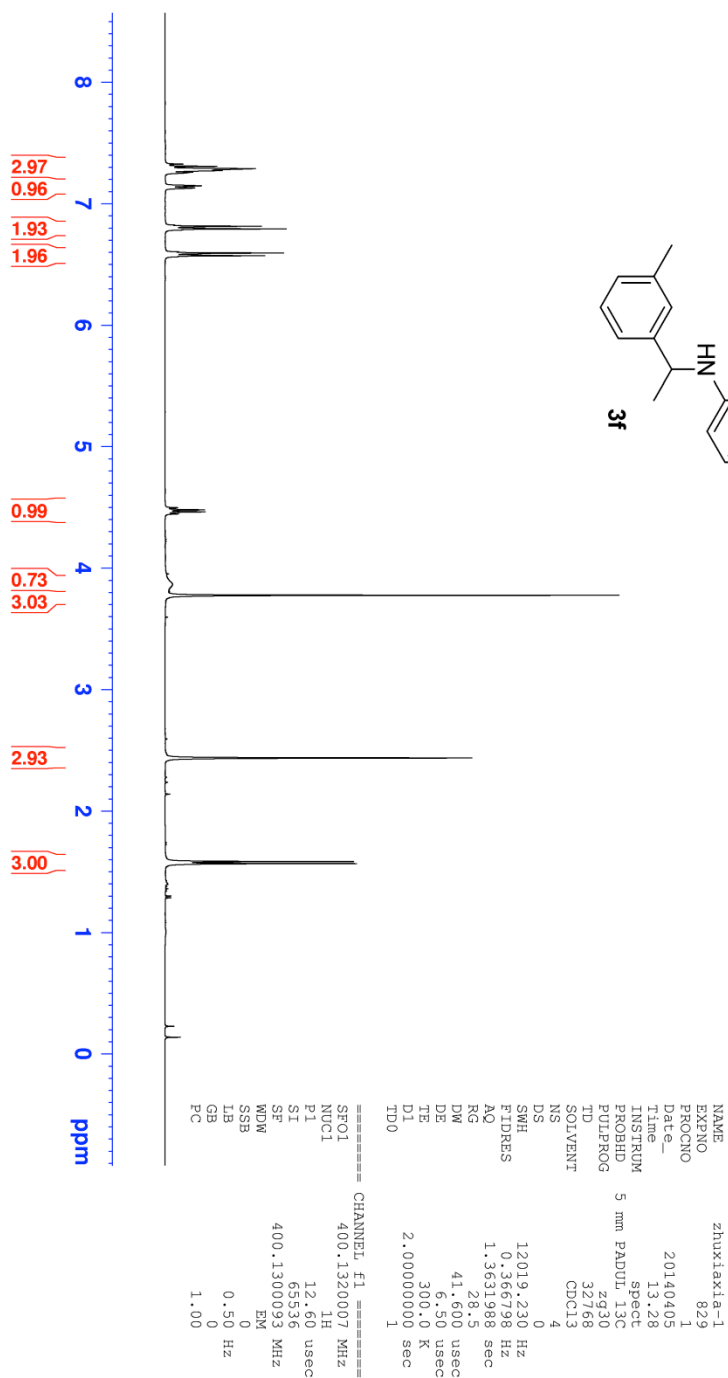
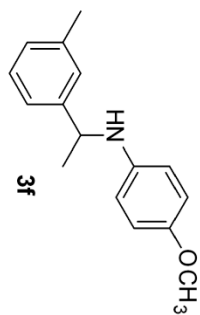


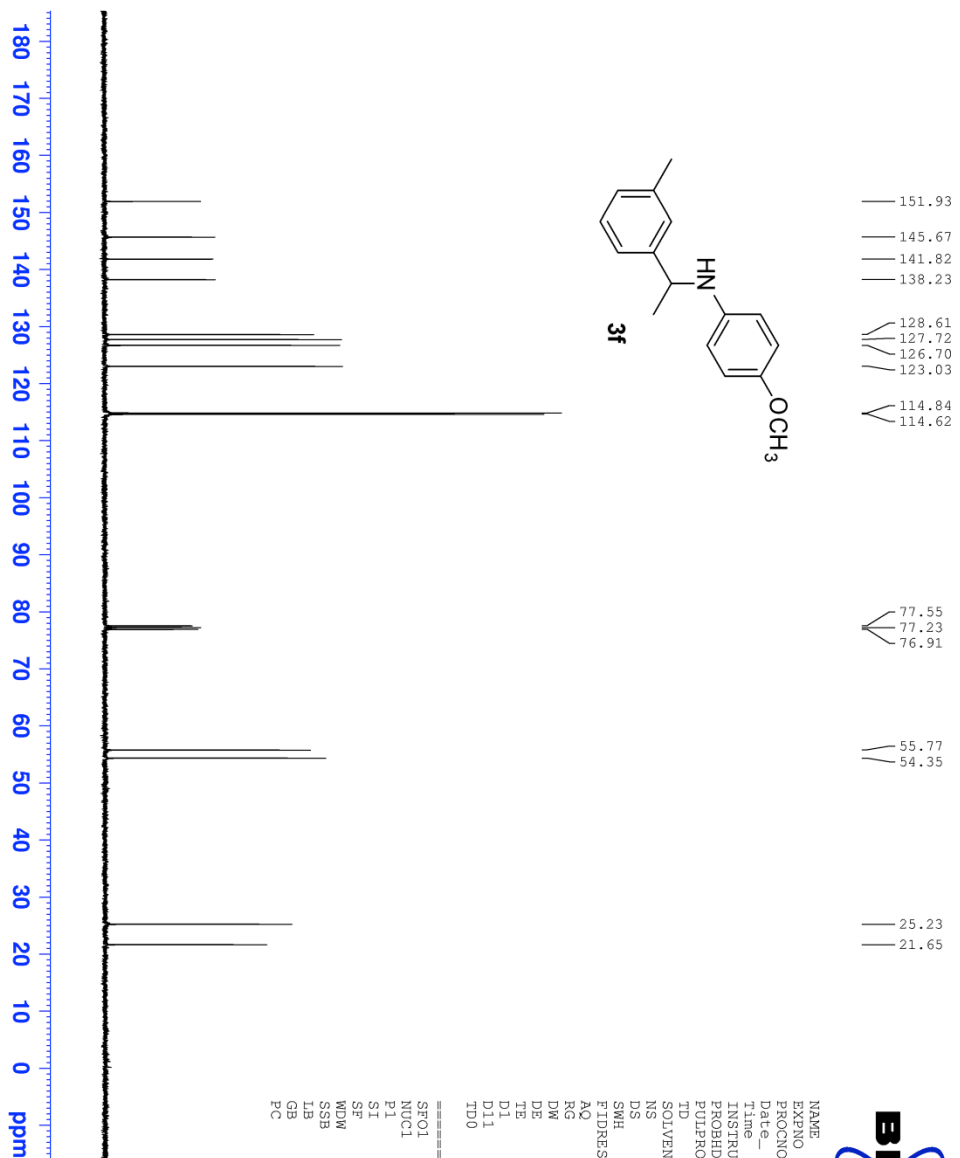


NAME: ZHUJIA14-1
EXPNO: 1
PROCNO: 1
Date_: 20131009
Time: 1.12
INSTRUM: spect
PROBHD: 5 mm PADUL 13C
PULPROG: zg30
TD: 32768
SOLVENT: CDCl3
NS: 7
DS: 0
SWH: 12019.230 Hz
FIDRES: 0.366798 Hz
AQ: 1.3631988 sec
RG: 114
DW: 41.600 usec
DE: 6.50 usec
TE: 300.0 K
D1: 2.00000000 sec
TD0: 1

===== CHANNEL f1 =====
NUC1: 1H
P1: 12.60 usec
SI: 65636
SF: 400.1300237 MHz
WDW: EM
SSB: 0
LB: 0.50 Hz
GB: 0
PC: 1.00



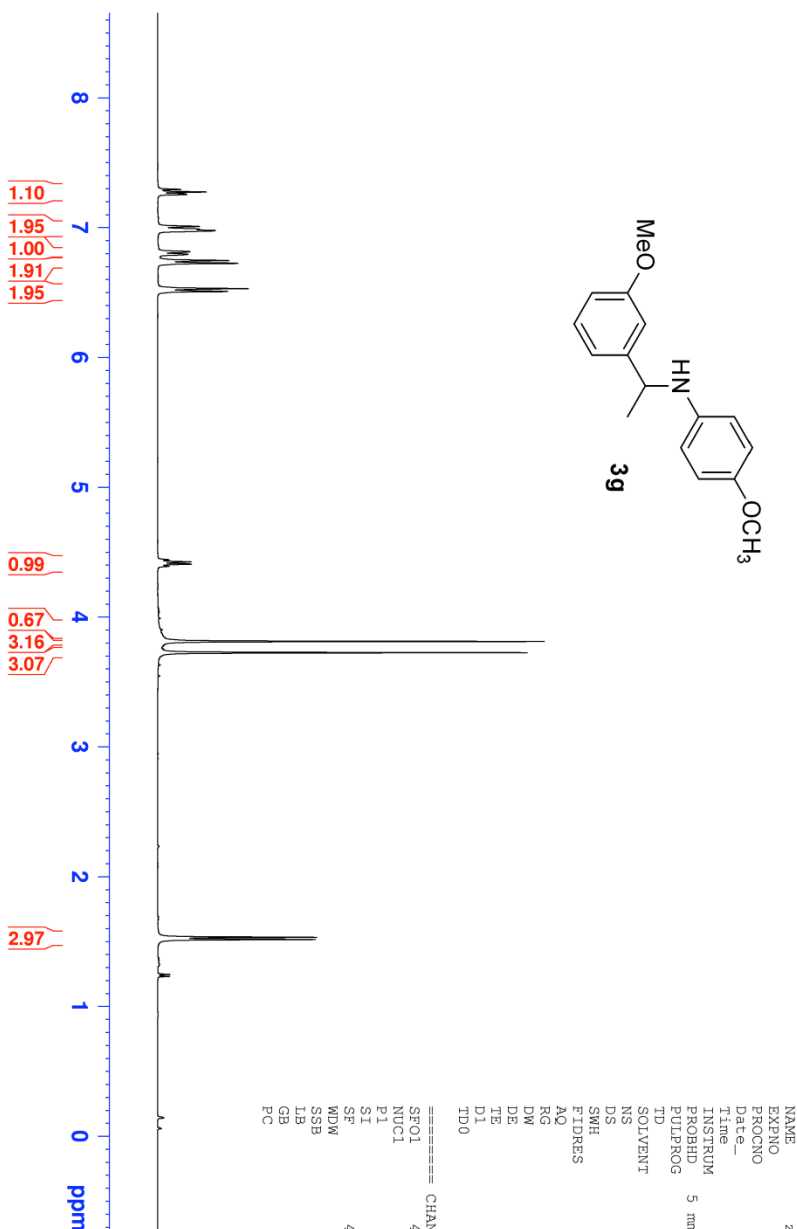
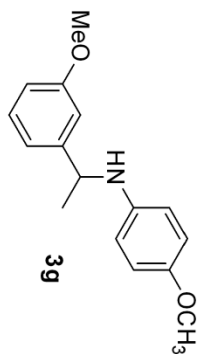




```

NAME          zhuxiaxia-1
EXPNO         830
PROCNO        1
Date_         20140405
Time          13.30
INSTRUM       spect
PROBHD        5 mm PABUL-13C
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            48
DS            1
SWH           25252.525 Hz
FIDRES        0.770646 Hz
AQ            0.6488564 sec
RG            203
RG            19.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
SFO1          100.628298 MHz
NUC1          13C
P1            9.40 usec
SI            32768
SF            100.612710 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



NAME zhuxiaxia-1
EXPNO 757
PROCNO 1
Date_ 20140323
Time 0.48
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 6
DS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 50.8
DW 41.600 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

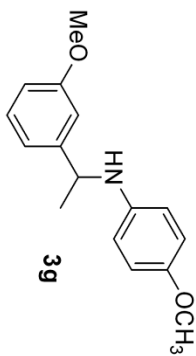
===== CHANNEL f1 =====
SFO1 400.1320007 MHz
NUC1 1H
P1 12.60 usec
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
TB 0
GB 0
PC 1.00

160.04
152.04
147.57
141.70
129.76
118.39
114.87
114.69
112.07
111.83

77.55
77.23
76.91

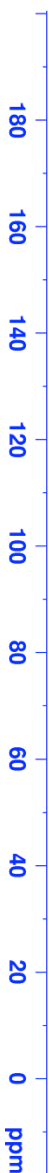
55.84
55.27
54.44

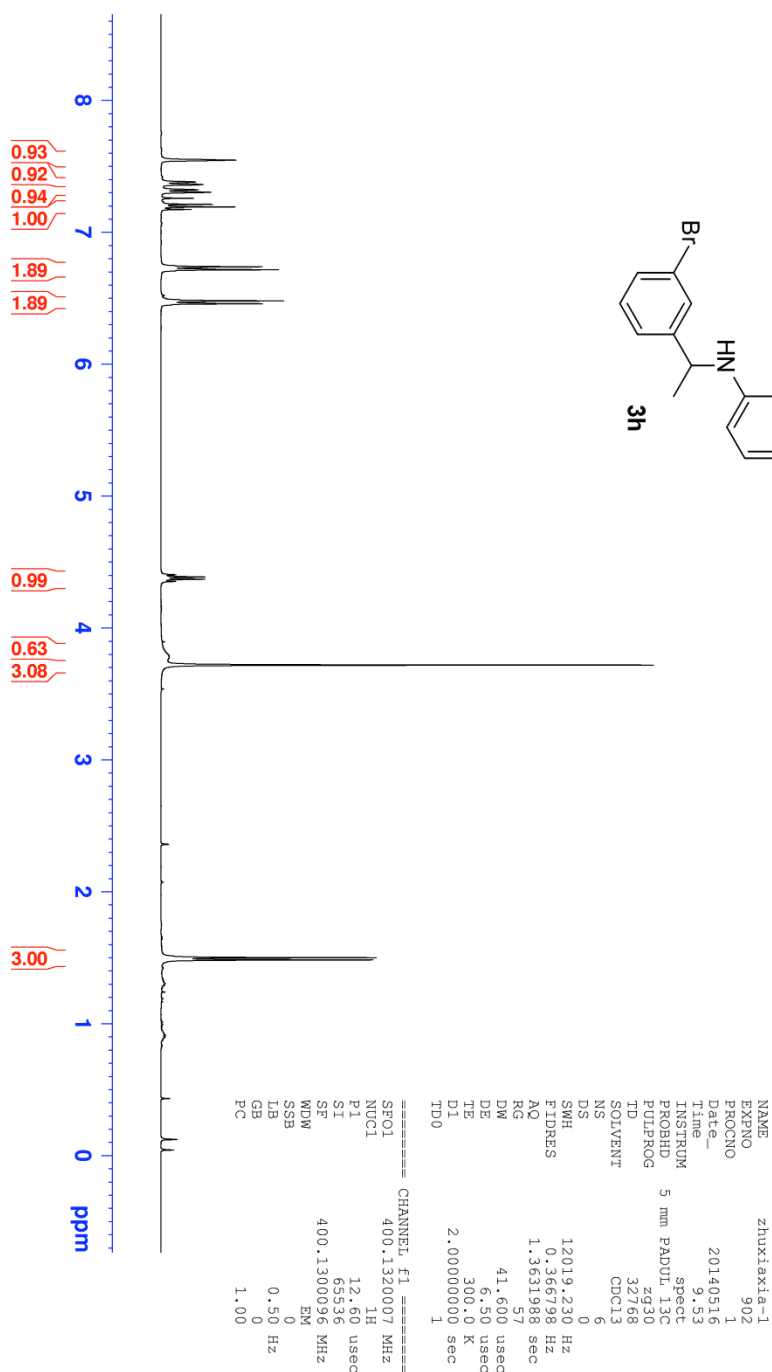
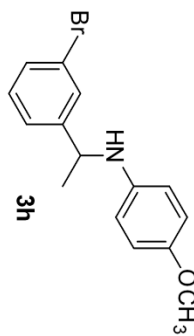
25.22

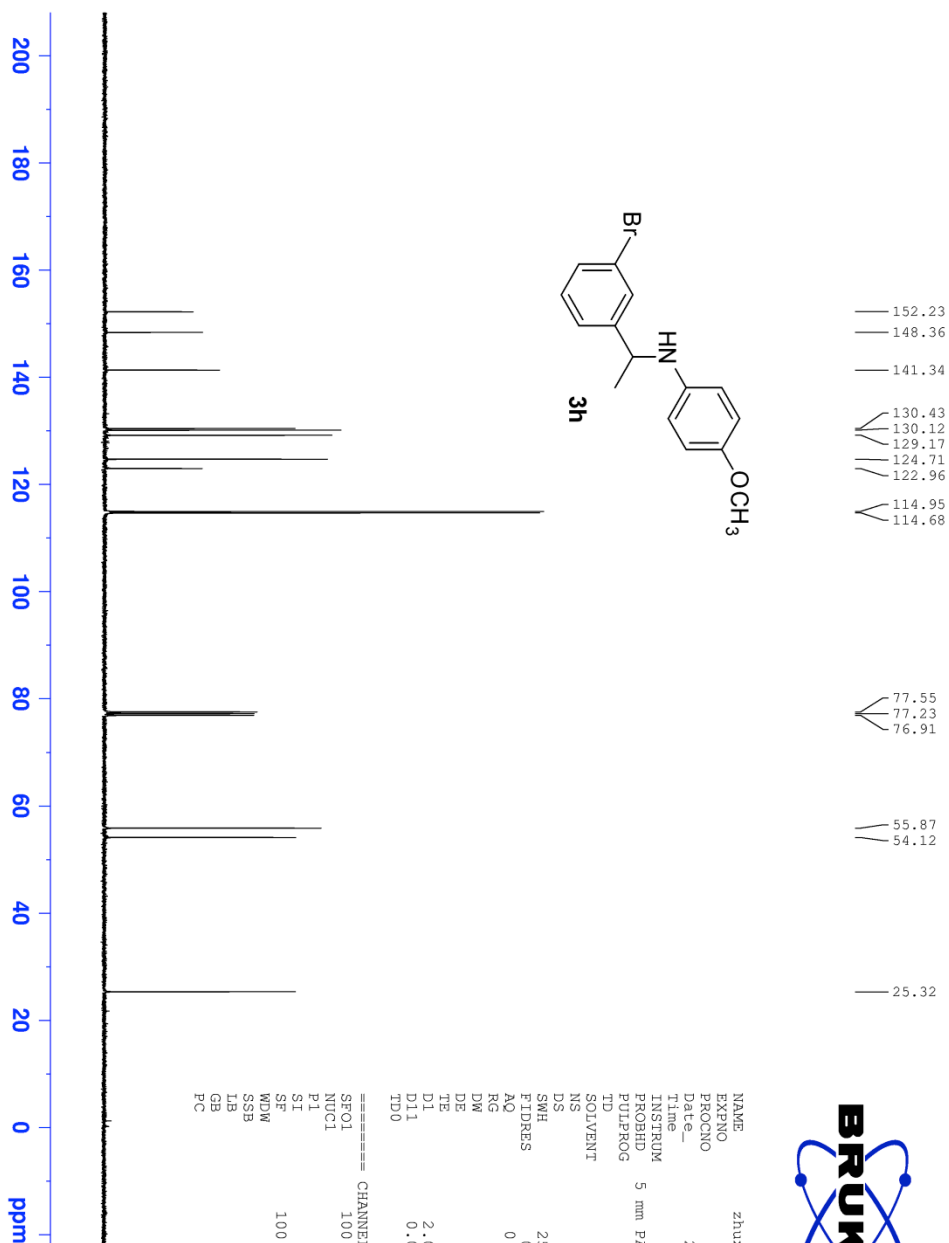


NAME zhuxiaxia-1
EXPNO 758
PROCNO 1
Date_ 20140323
Time 0.51
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 84
DS 0
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 0.6488564 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 9.40 usec
SI 32768
SF 100.6127607 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







```

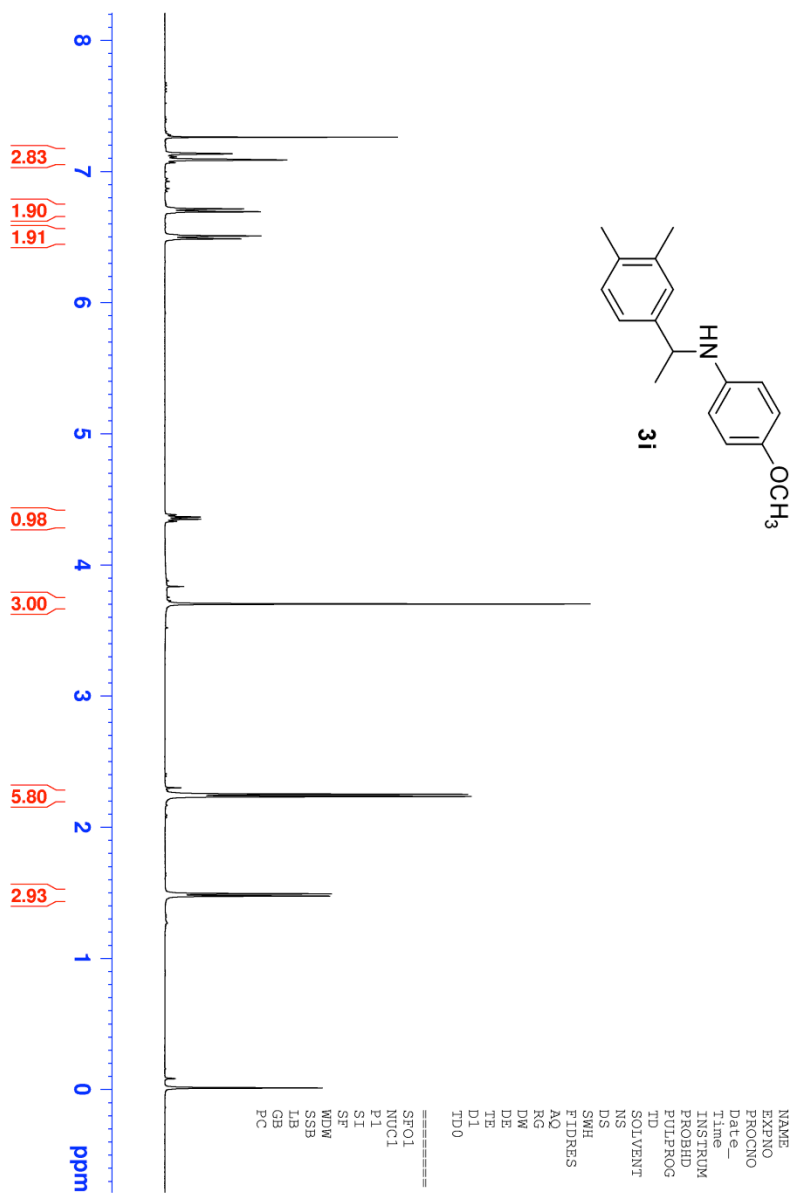
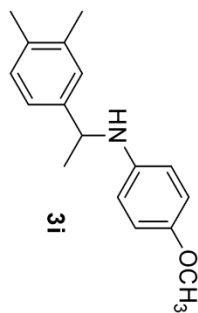
NAME zhuxiaxia-1
EXPNO 903
PROCNO 1
Date_ 20140516
Time 10.10
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 355
DS 0
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 0.6488564 sec
RG 203
DE 19.800 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

```

```

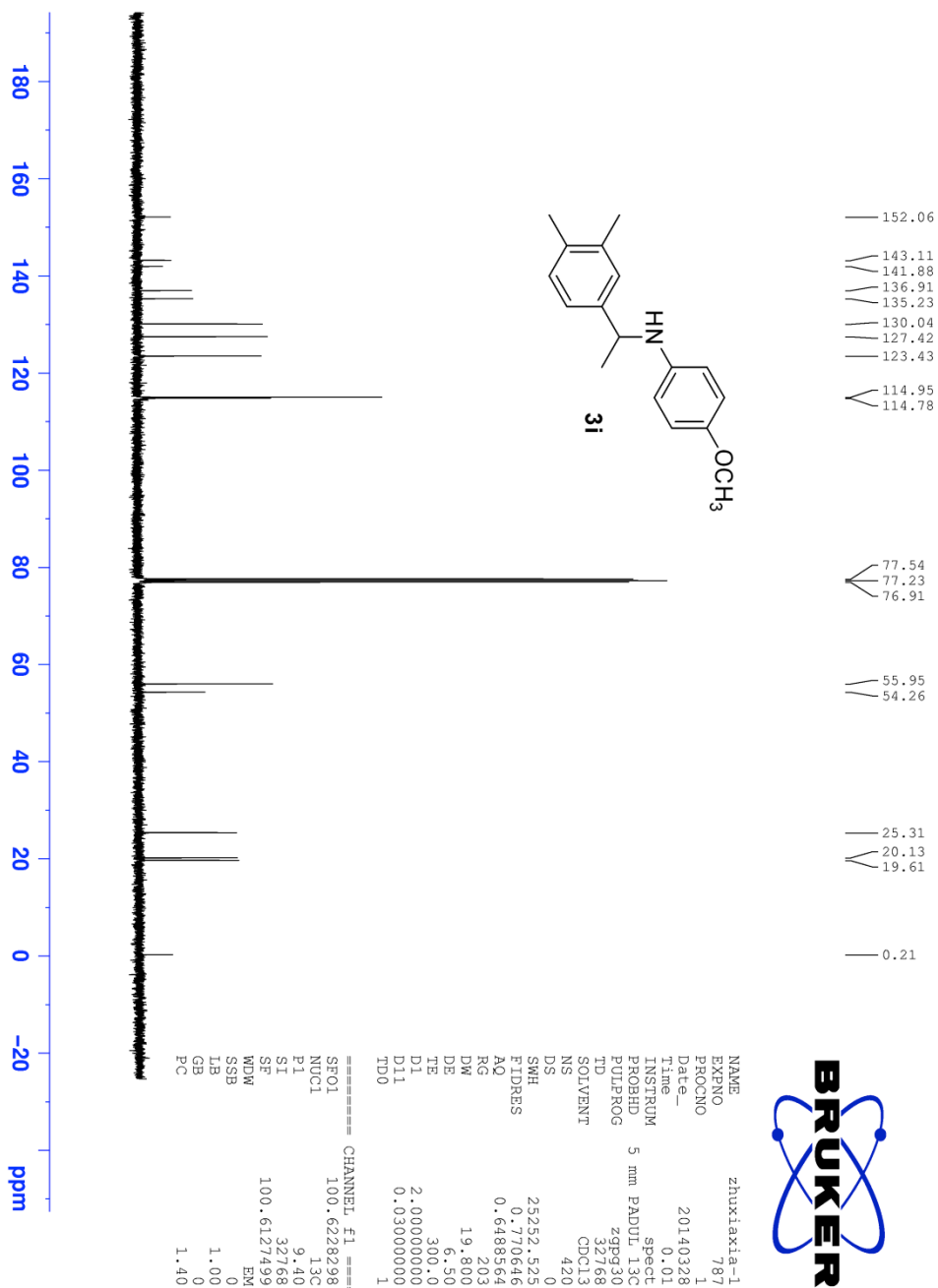
===== CHANNEL f1 =====
SFO1 100.628298 MHz
NUC1 13C
P1 9.40 usec
SI 32768
SF 100.6127576 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

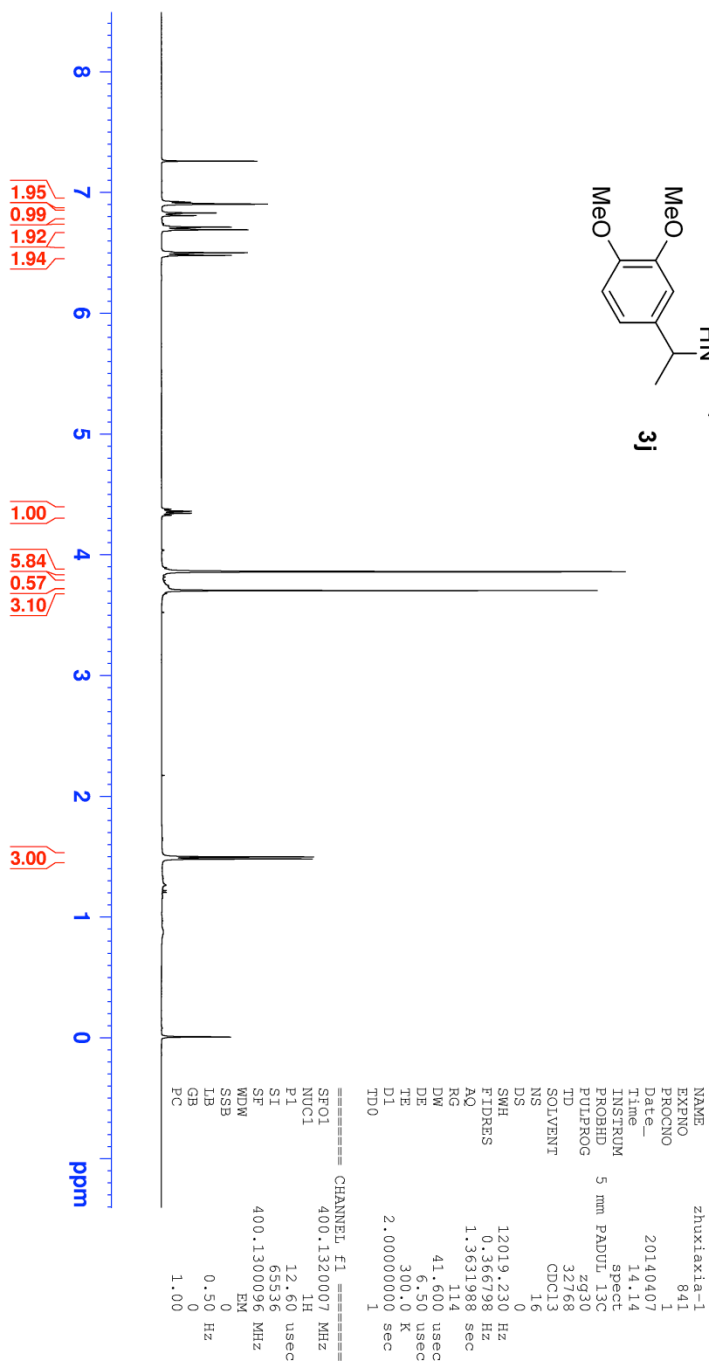
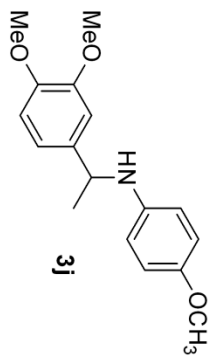
```



NAME zhuxiaixia-1
EXPNO 786
PROCNO 1
Date_ 20140327
Time 23:38
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zg30
ID 32768
SOLVENT CDCl3
DS 5
NS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 114
DW 41.600 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.1320007 MHz
NUC1 1H
P1 12.60 usec
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00





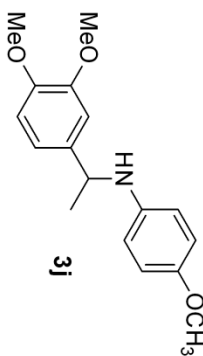
152.12
149.31
147.97
141.89
138.39

117.93
114.90
114.81
111.40
109.28

77.55
77.23
76.91

56.07
56.03
55.92
54.32

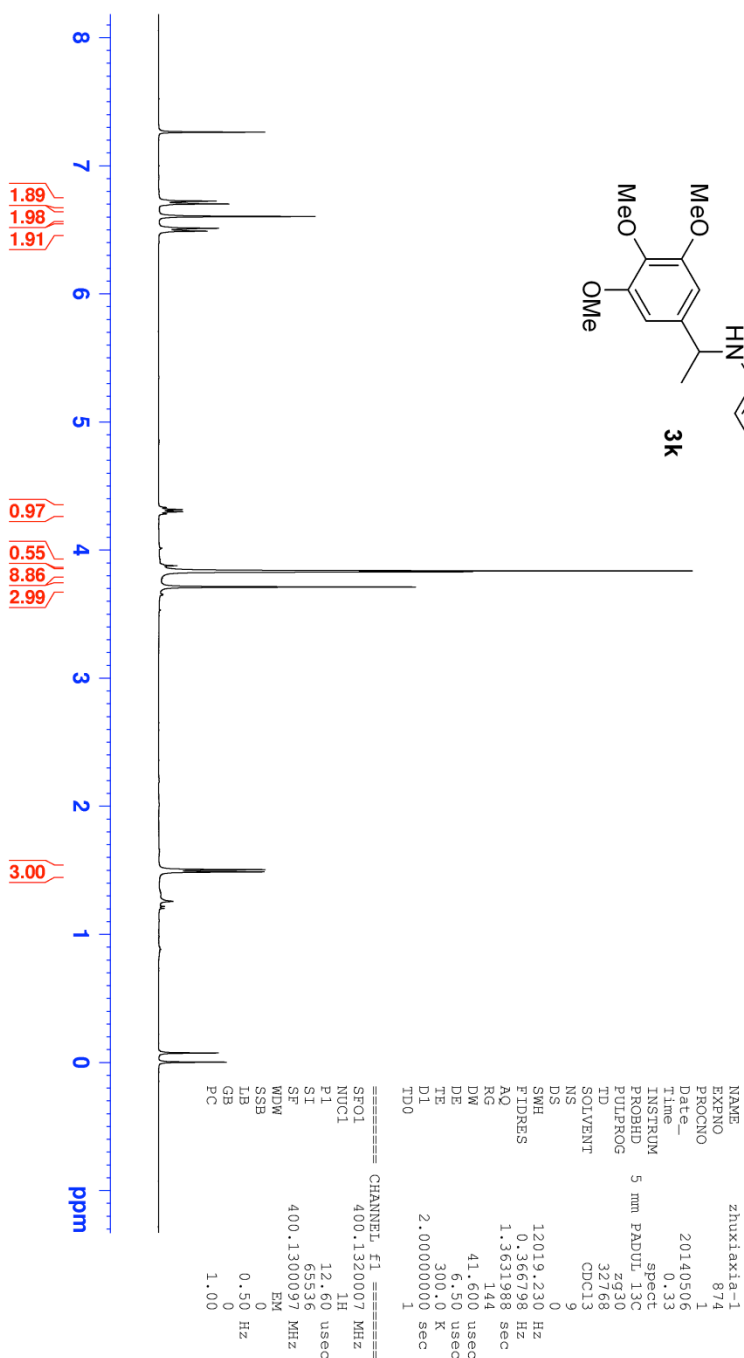
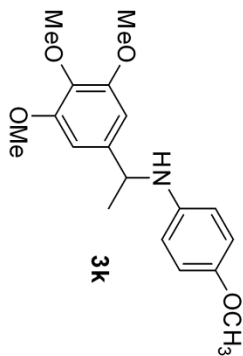
25.32

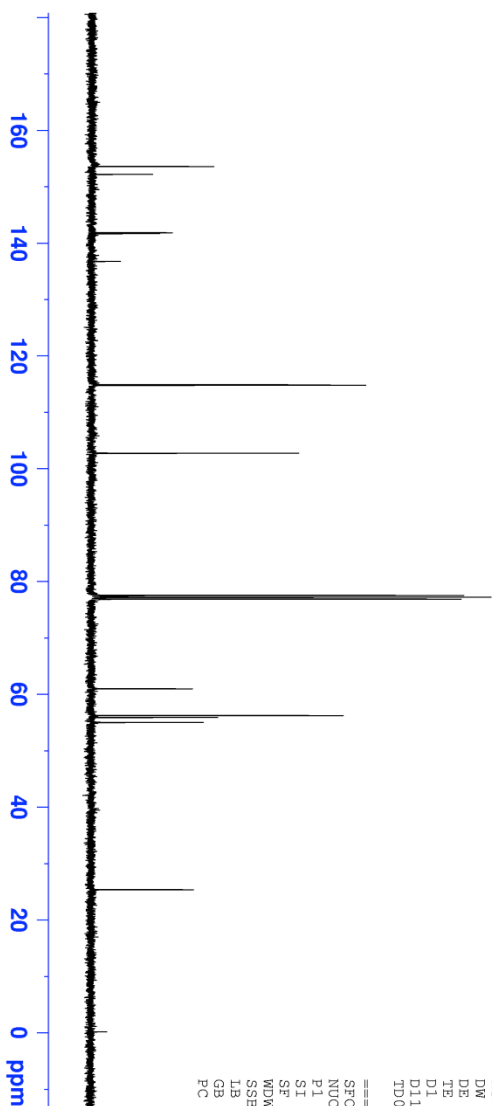
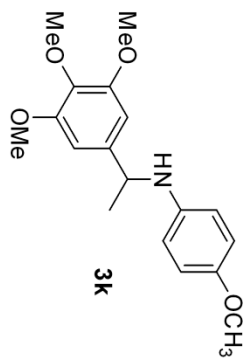


NAME zhuxiaxia-1
EXNO 842
PROCNO 1
Date_ 20140407
Time 14.18
INSTRUM spect
PROBHD 5 mm P4DUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 111
DS 0
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 0.6488564 sec
RG 203
DE 19.800 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 9.40 usec
SI 32768
SF 100.6127515 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

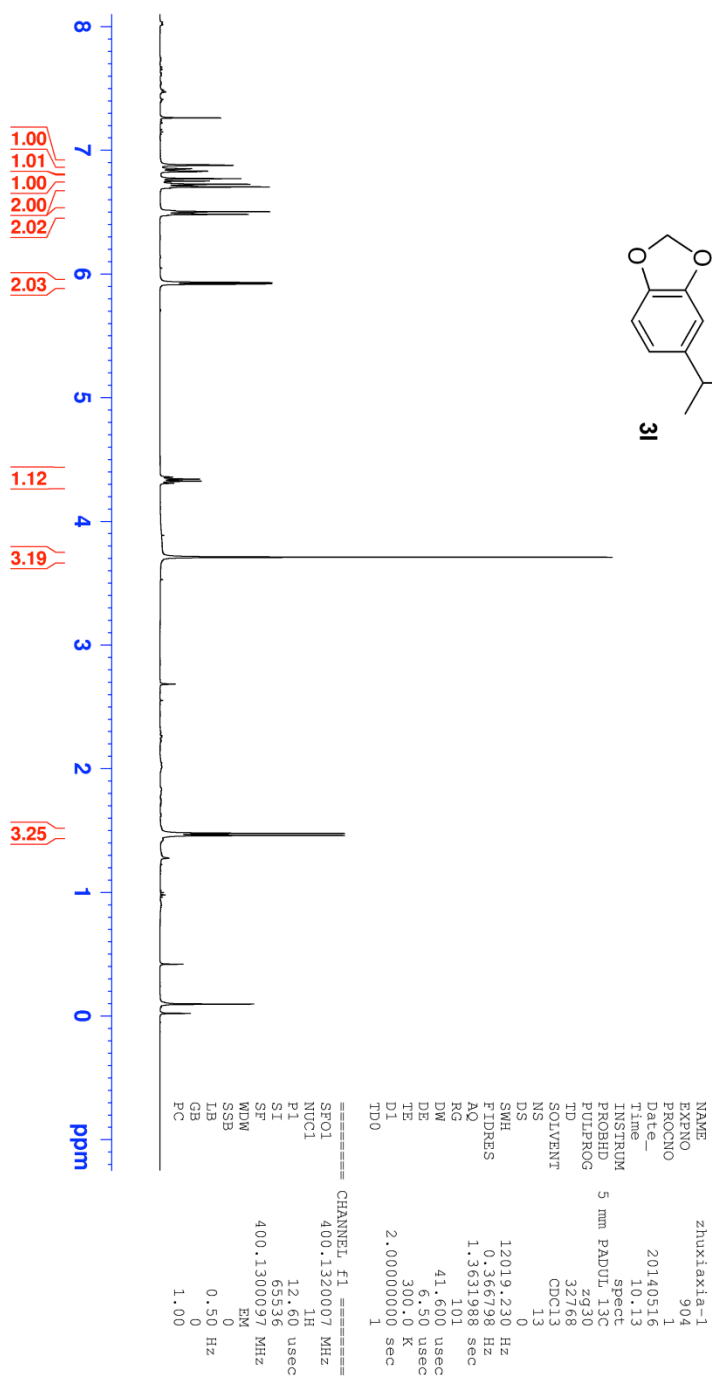
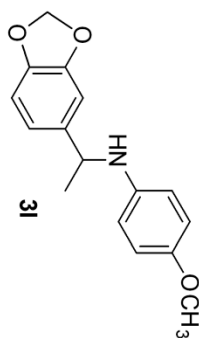


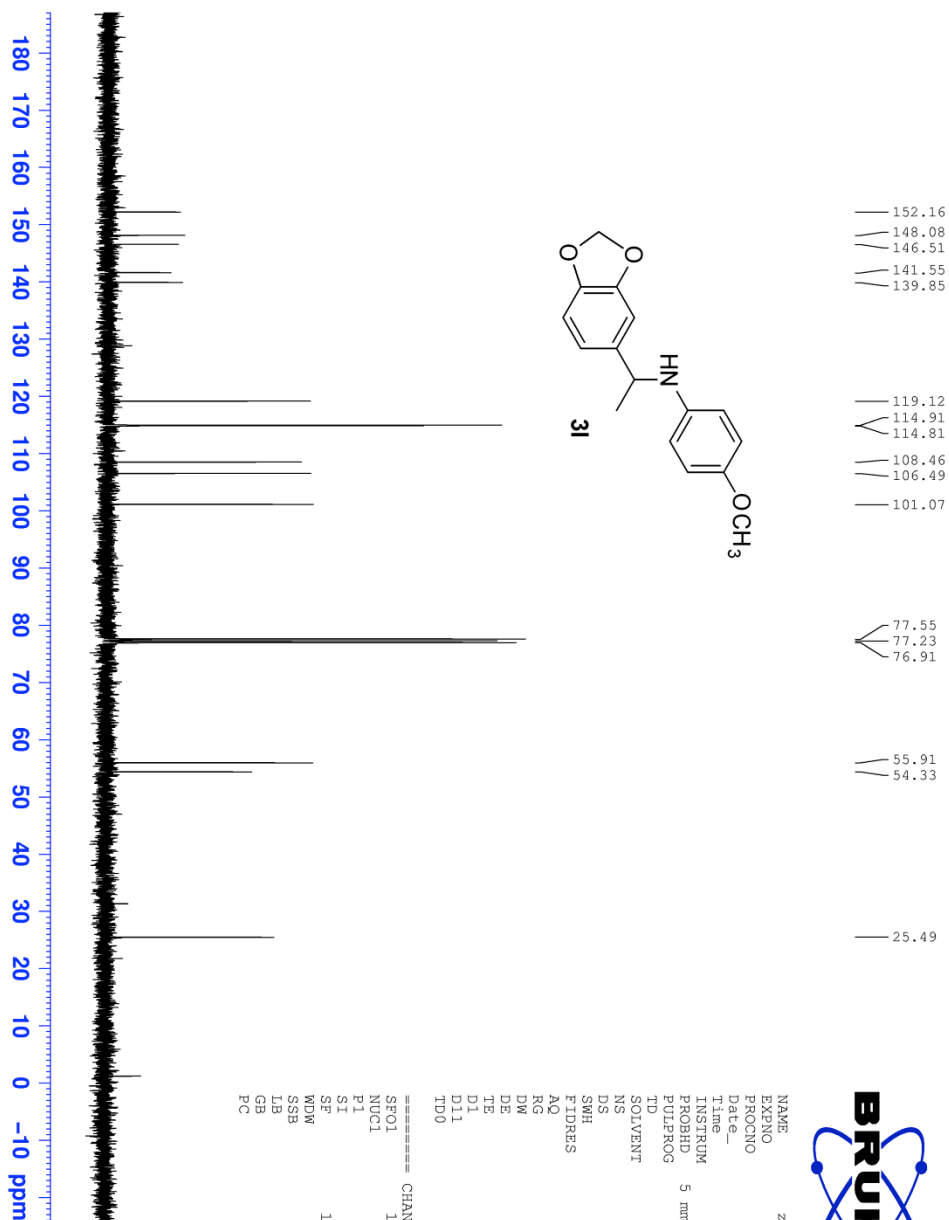




NAME zhuxiaxia-1
EXPNO 881
PROCNO 1
Date_ 20140508
Time 12.45
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 330
DS 0
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 0.648564 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 100.6228298 MHz
NUC1 13C
P1 9.40 usec
SI 32768
SF 100.6127513 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

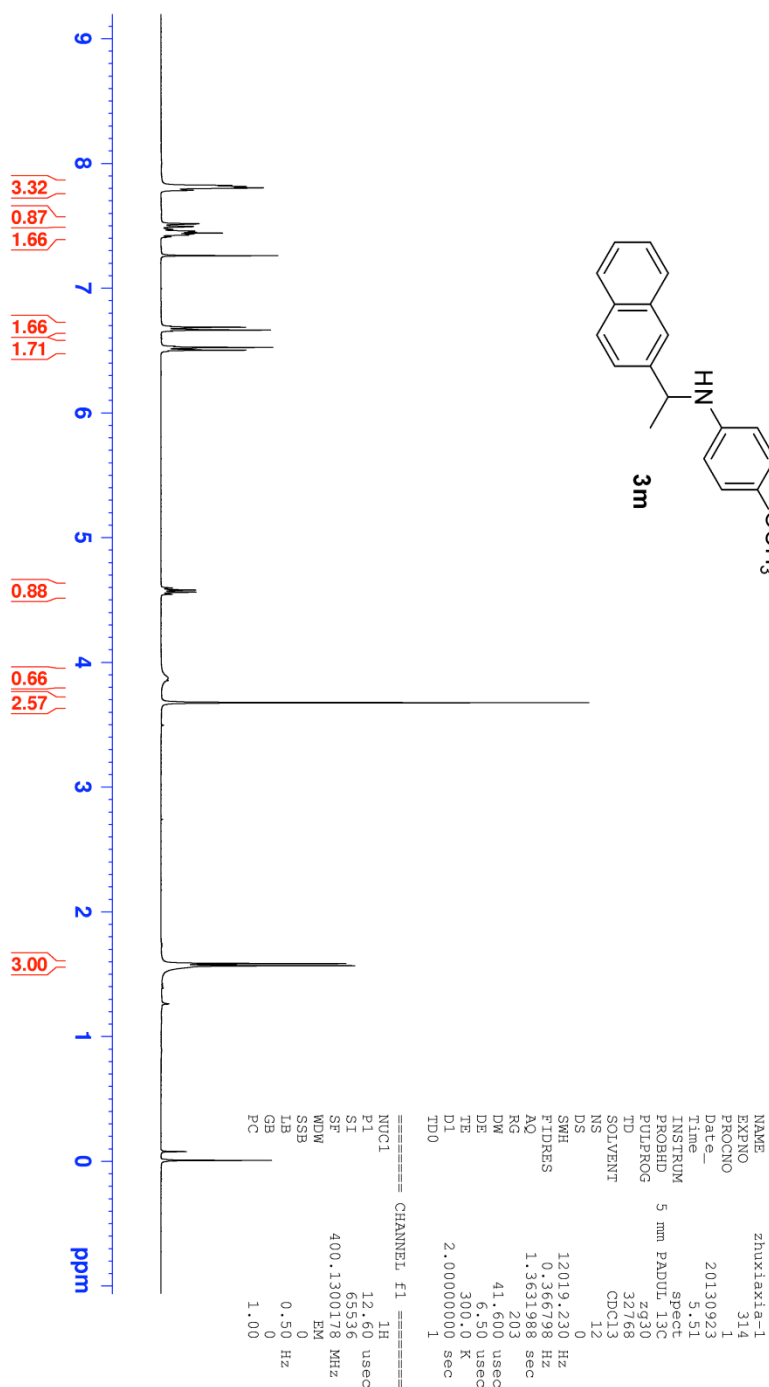
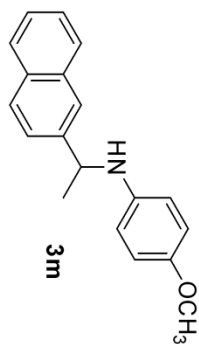


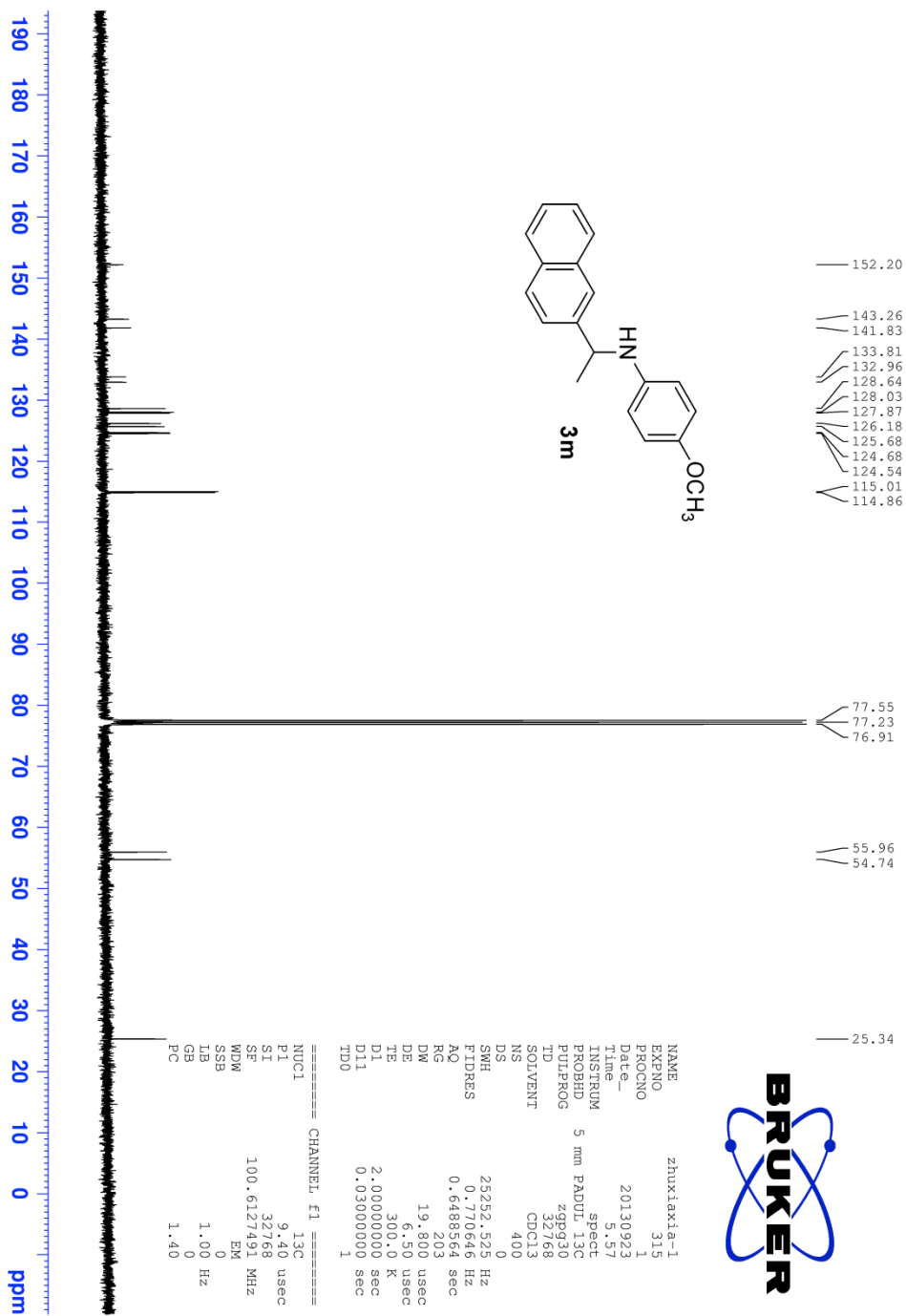


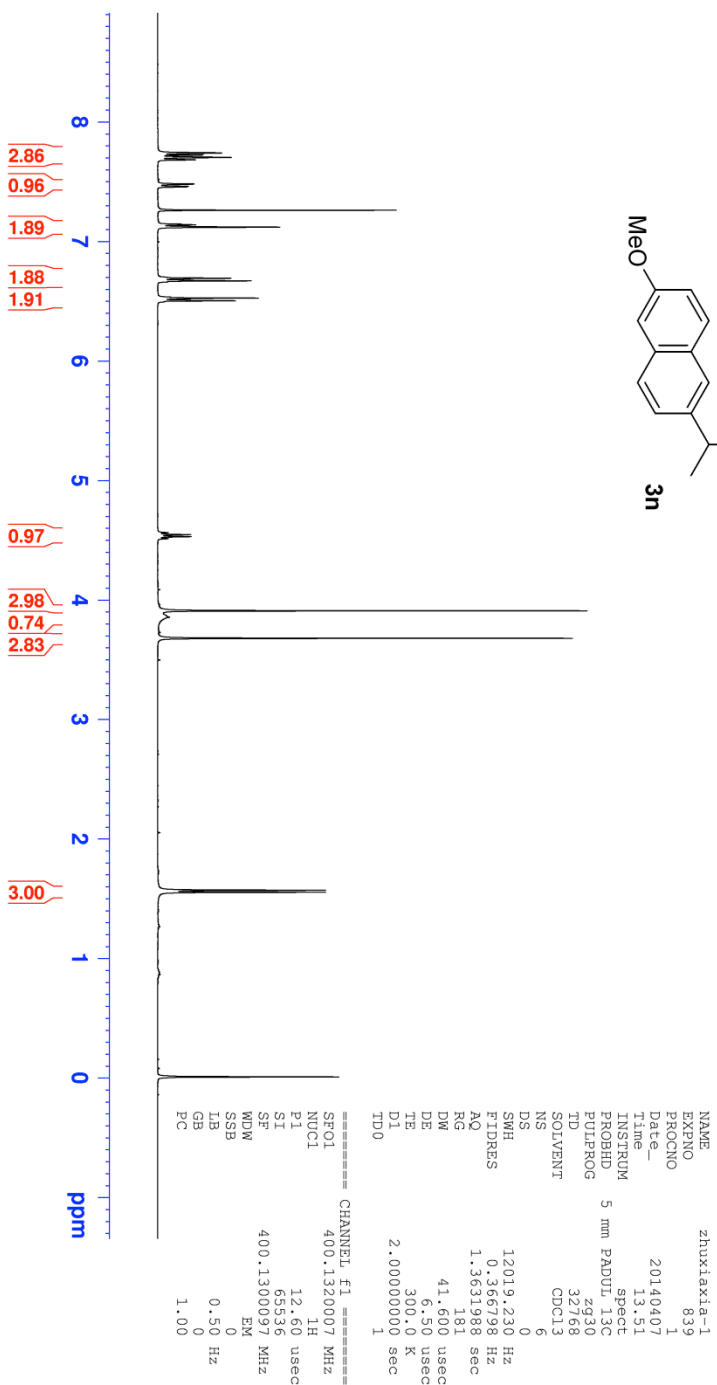
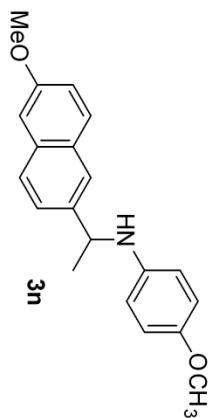
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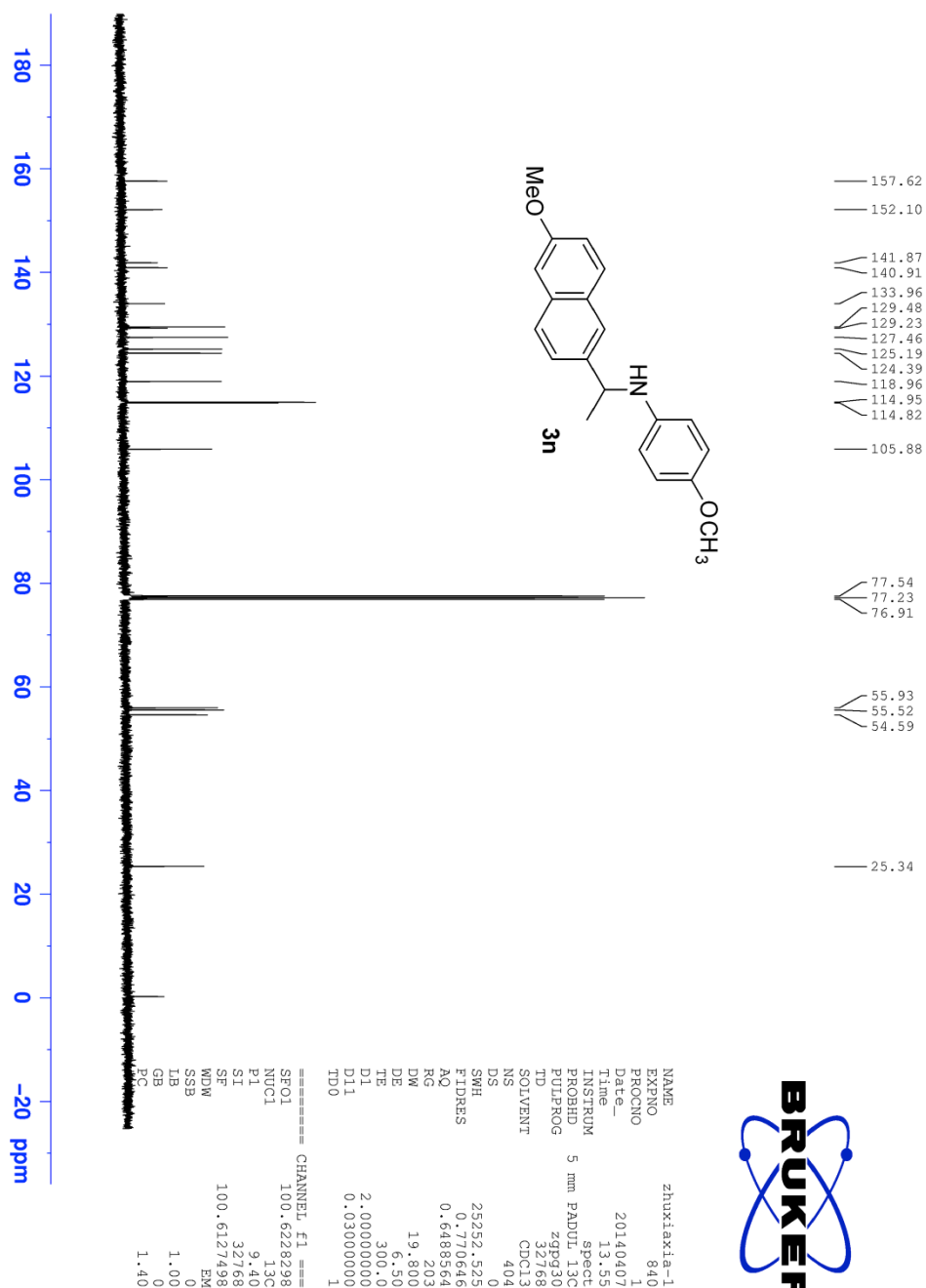
NAME          zhuxiaxia-1
EXPNO         905
PROCNO        1
Date_         20140516
Time_         10.15
INSTRUM       spect
PROBHD        5 mm PADUT, 13C
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            46
DS            0
SWH           25252.525 Hz
FIDRES        0.770646 Hz
AQ            0.6488564 sec
RG            203
RG            203
DM            19.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
SFO1          100.6228298 MHz
NUC1          13C
P1            9.40 usec
SI            32768
SF            100.6127529 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



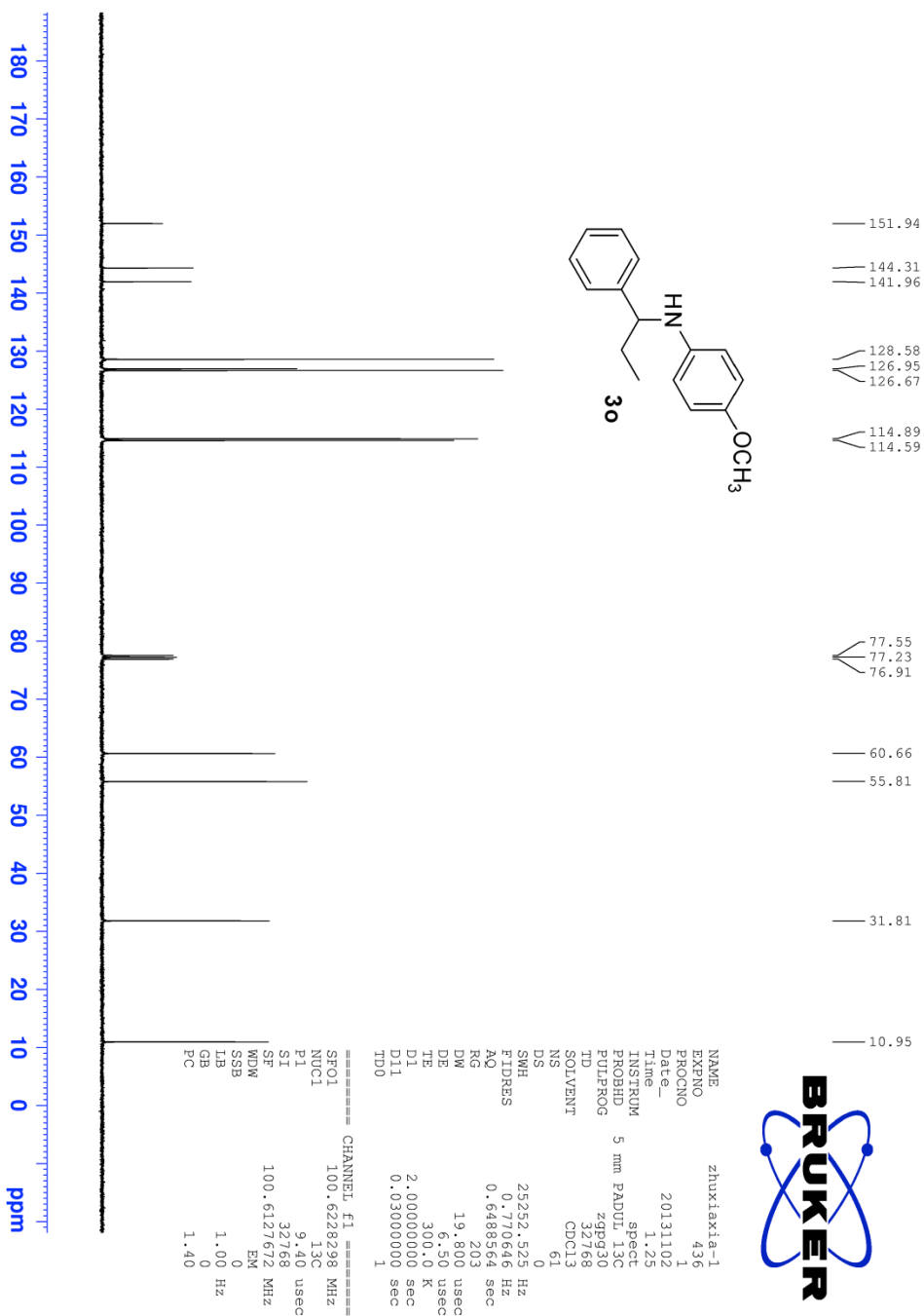


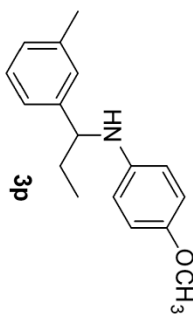


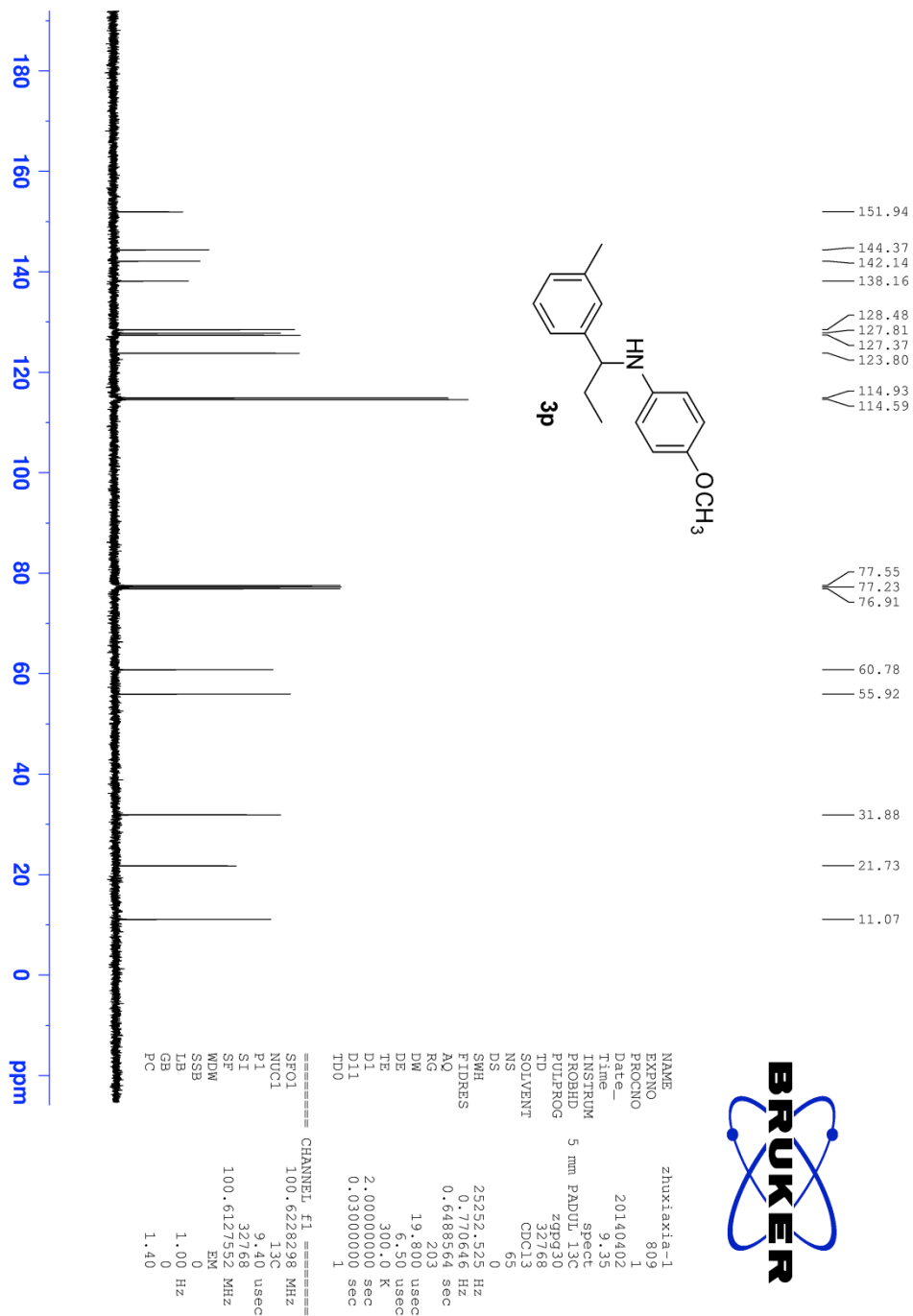


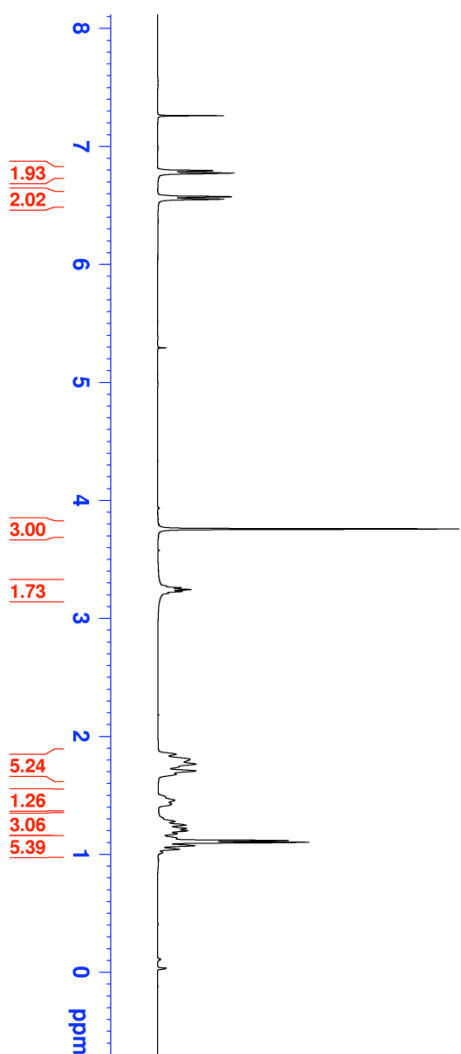
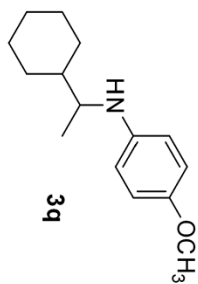


NAME	zhuxiaxia-1
EXPNO	434
PROCNO	1
Date_	20131102
Time_	1.22
INSTRUM	ADU113C
PROBHD	5 mm P
PULPROG	zgpg30
TD	32768
NS	CDCl3
SOLVENT	8
DS	0
SWH	12019.230 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	28.3
KM	41.600 used
DE	6.50 used
TE	300.0 K
TD0	2.00000000 sec
	1



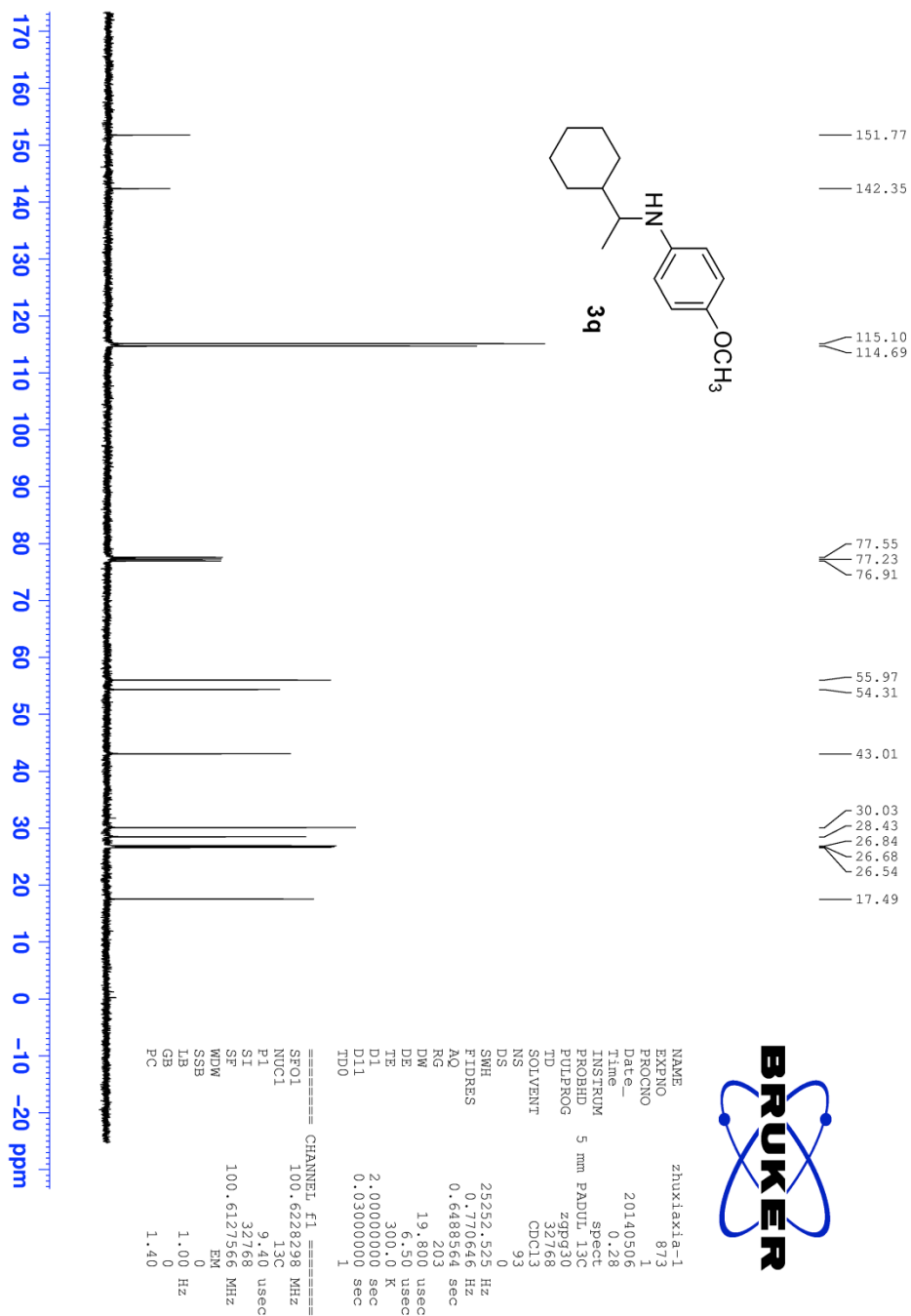


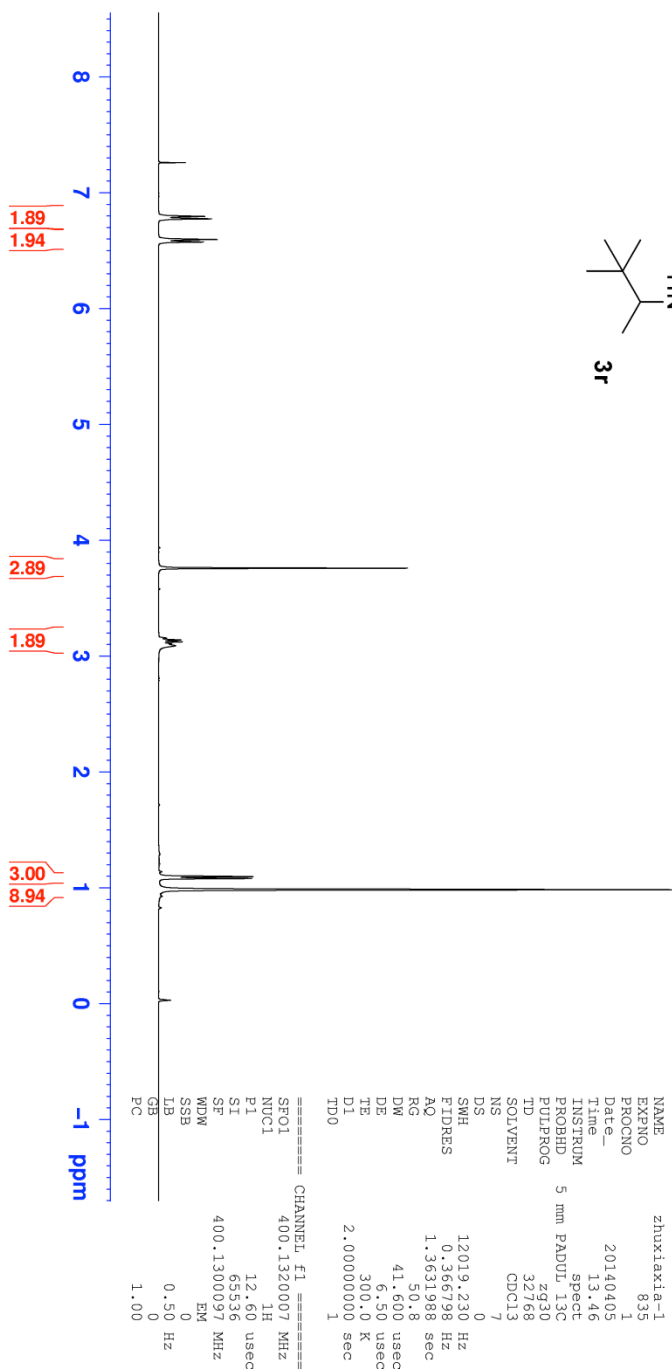
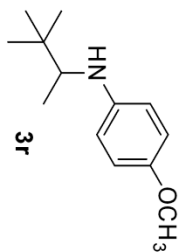




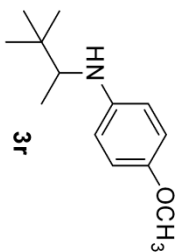
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NAME zhuxiaxia-1
EXPNO 872
PROCNO 1
Date_ 20140506
Time 0.23
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.363198 sec
RG 40.3
DW 41.600 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 400.132007 MHz
NUC1 1H
P1 12.60 usec
SI 65336
SF 400.130096 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00
```





151.76
143.09
115.11
114.70
77.55
77.23
76.91
58.79
56.02
34.89
26.74
15.95



NAME zhuxiaxia-1
EXPNO 836
PROCNO 1
Date_ 20140405
Time 13.48
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 43
DS 0
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 0.648564 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.05000000 sec
TD0 1

CHANNEL f1
SFO1 100.6228298 MHz
NUC1 13C
P1 9.40 usec
SI 32768
SF 100.6127541 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

