Highly diastereoselective [5+1] annulation to 2,2,3-trisubstituted tetrahydroquinoxalines via intramolecular Mannich-type trapping of ammonium ylides

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Supplementary Information

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1. General Information

All moisture sensitive reactions were performed under an argon atmosphere in a well-dried reaction flask. Dichloromethane (CH_2Cl_2), 1,2-dichloroethane ($ClCH_2CH_2Cl$) and chloroform ($CHCl_3$) were freshly distilled over calcium hydride, toluene from sodium benzophenone ketyl, respectively, prior to use. ^1H NMR, ^{13}C NMR spectra were obtained utilizing a Bruker 300 MHz instrument and reported in $CDCl_3$. ^1H and ^{13}CNMR chemical shifts are reported in ppm relative to either TMS (^1H) (δ = 0 ppm) as an internal standard or the residual solvent peak (^{13}C) (δ = 77.00 ppm). Chemical shifts are reported in parts per million as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br =broad). HRMS (ESI) Mass spectra were recorded on IonSpec FT-ICR mass spectrometer. All commercially available reagents were directly used as received from vendors, unless otherwise stated. Diazo compounds **1**[1], **2aa, 2ab**, **2a**-**2m**[2] were prepared according to the literature.

2. General procedure for $Rh_2(OAc)_4$-catalyzed reaction of diazo compounds 1 and *ortho*-Aminophenyl imines 2:

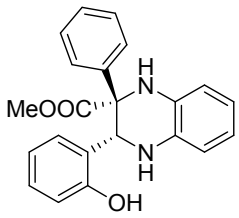
To the mixture of *ortho*-Aminophenyl imines **2** (0.1 mmol) and $Rh_2(OAc)_4$ (1 mol%) in 2 mL of dichloromethane, diazo compound **1** (0.15 mmol) in 2 mL of dichloromethane was added over 1 h via a syringe pump under argon at 40℃. After completion of the addition, the reaction mixture was further stirred at 40℃ until completion determined by TLC. Then, solvent was removed, and a portion of crude product was subjected to ^1H NMR analysis for determination of the product ratio. The crude product was purified by flash chromatography on silica gel using petroleum ether/ethyl acetate as eluent to give the corresponding products **3**.

3. Notes and References

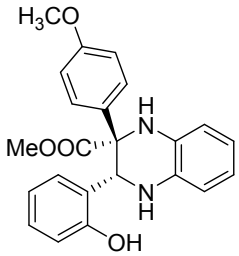
1) Doyle, M. P., Mckervey, M. A. and Ye, T. *Modern Catalytic Methods for Organic Synthesis with Diazo Compounds*; Wiley: New York, (1998).

2) D. M. Boghaei and S. Mohebi, *Tetrahedron*, 2002, **58**, 5357-5366.

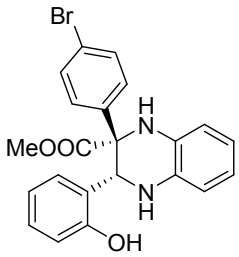
4. Characterization data of products



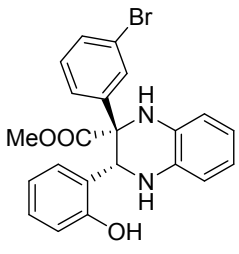
***rel*-(2*R*,3*R*)-methyl3-(2-hydroxyphenyl)-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3a).** Light yellow solid (27 mg, 75% yield), m.p. 175-176 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 8.60 (s, 1H), 7.57-7.54 (m, 2H), 7.34-7.31 (m, 3H), 6.92-6.57 (m, 8H), 5.30(s, 1H), 5,19 (s, 1H), 4.07 (s, 1H), 3.63 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 172.1, 155.3, 139.9, 131.7, 130.0, 129.2, 129.0, 128.8, 128.4, 126.3, 125.1, 120.9, 119.9, 119.7, 117.7, 117.3, 115.1, 65.1, 57.8, 53.0; HRMS: calcd for $C_{22}H_{20}N_2NaO_3$: 383.1372; found: 383.1366 $[M+Na]^+$



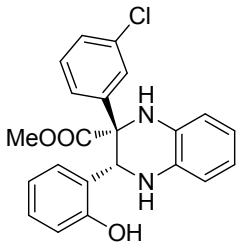
***rel*-(2*R*,3*R*)-methyl3-(2-hydroxyphenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3b).** Yellow solid (26 mg, 67% yield), m.p. 166-167 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 7.47-7.44 (m, 2H), 7.11-6.57 (m, 10H), 5.25 (s, 1H), 5.14 (s, 1H), 4.11 (s, 1H), 3.77 (s, 3H), 3.62 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 172.2, 159.4. 155.3, 131.8, 130.0, 129.2, 129.0, 127.5, 125.1, 120.9, 119.6, 117.6, 117.3, 115.1, 114.1, 64.6, 57.9, 55.2, 52.9; HRMS: calcd for $C_{23}H_{22}N_2NaO_4$: 413.1477; found: 413.1472 $[M+Na]^+$



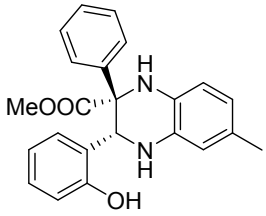
***rel*-(2*R*,3*R*)-methyl2-(4-bromophenyl)-3-(2-hydroxyphenyl)-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3c).** Yellow solid (27 mg, 63% yield), m.p. 173-174 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 7.45 (s, 4H), 7.11 (s, 1H), 6.96-6.53 (m, 7H), 5.29 (s, 1H), 5.13 (s, 1H), 4.11 (s, 1H), 3.62 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 171.6, 154.9, 139.2, 131.9, 131.0, 130.3, 129.2, 129.1, 128.2, 125.3, 122.5, 120.7, 120.2, 120.0, 117.6, 116.8, 115.2, 65.0, 57.0, 53.1; HRMS: calcd for $C_{22}H_{19}BrN_2NaO_3$: 461.0477; found: 461.0471 $[M+Na]^+$



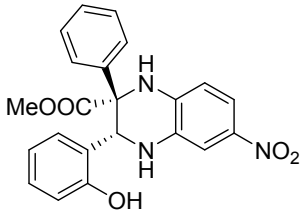
***rel*-(2*R*,3*R*)-methyl2-(3-bromophenyl)-3-(2-hydroxyphenyl)-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3d).** Yellow solid (30 mg, 70% yield), m.p. 158-159 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 8.03 (s, 1H), 7.75 (s, 1H), 7.46-7.42 (m, 2H), 7.19-7.12 (m, 2H), 6.94-6.54 (m, 7H), 5.29 (s, 1H), 5.14 (s, 1H), 4.13 (s, 1H), 3.63 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 171.6, 158.9, 154.9, 142.5, 131.5, 131.0, 130.2, 130.1, 129.6, 129.2, 125.2, 125.1, 122.9, 120.8, 120.2, 120.0, 117.6, 116.9, 115.2, 65.0, 57.0, 53.2; HRMS: calcd for $C_{22}H_{19}BrN_2NaO_3$: 461.0477; found:461.0472 $[M+Na]^+$



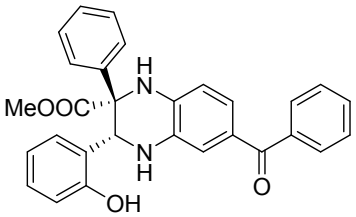
***rel*-(2*R*,3*R*)-methyl2-(3-chlorophenyl)-3-(2-hydroxyphenyl)-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3e).** Yellow solid (29 mg, 75% yield), m.p. 150-151 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 8.86 (s, 1H), 7.52-7.50 (m, 2H), 7.36-7.31 (m, 3H), 7.07-7.05 (m, 1H), 6.85-6.57 (m, 6H), 5.20 (s, 1H), 5.18 (s, 1H), 4.05 (s, 1H), 3.66 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 171.7, 156.3, 154.3, 139.4, 131.7, 129.5, 129.1, 129.0, 128.6, 126.2, 124.4, 122.9, 121.4, 119.9, 119.1, 117.7, 115.4, 112.3, 65.0, 58.3, 53.1; HRMS: calcd for $C_{22}H_{19}ClN_2NaO_3$: 417.0982; found: 417.0976 $[M+Na]^+$



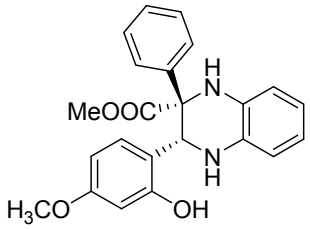
***rel*-(2*R*,3*R*)-methyl3-(2-hydroxyphenyl)-6-methyl-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3f).** Yellow solid (23 mg, 63% yield), m.p. 182-183 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 8.79 (s, 1H), 7.56-7.53 (m, 2H), 7.33-7.30 (m, 3H), 6.91-6.67 (m, 6H), 6.37 (s, 1H), 5.28(s, 1H), 5,07 (s, 1H), 4.03 (s, 1H), 3.61 (s, 3H), 2.14 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 172.1, 155.4, 139.9, 130.1, 129.4, 129.3， 129.1, 128.9, 128.8, 128.3, 126.3, 125.1, 121.4, 119.8, 117.8, 117.7, 115.2, 65.2, 58.2, 52.9, 20.6; HRMS: calcd for $C_{23}H_{22}N_2NaO_3$: 397.1528; found: 397.1523 $[M+Na]^+$



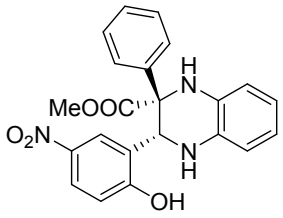
***rel*-(2*R*,3*R*)-methyl3-(2-hydroxyphenyl)-6-nitro-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3g).** Yellow solid (34 mg, 85% yield), m.p. 194-195 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 7.71-7.68 (m, 1H), 7.67-7.56 (m, 2H), 7.36-7.31 (m, 4H), 7.12-7.10 (m, 2H), 6.84-6.79 (m, 3H), 6.57 (s, 1H), 6.04 (s, 1H), 5.57 (s, 1H), 4.46 (s, 1H), 3.62 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 171.1, 153.7, 140.2, 139.6, 137.7, 129.6, 128.9, 128.5, 127.5, 126.3, 125.7, 120.9, 117.2, 116.7, 112.4, 111.0, 65.1, 53.2, 52.9; HRMS: calcd for $C_{22}H_{19}N_3NaO_5$: 428.1222; found: 428.1217 $[M+Na]^+$



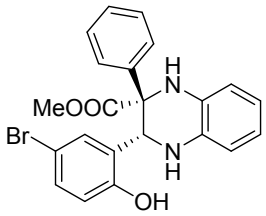
***rel*-(2*R*,3*R*)-methyl6-benzoyl-3-(2-hydroxyphenyl)-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3h).** Yellow solid (38 mg, 83% yield), m.p. 199-200 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 7.69-7.66 (m, 2H), 7.58 (m, 2H), 7.48 (m, 2H), 7.43-7.34 (m, 6H), 7.09 (m, 3H), 6.82 (m, 3H), 5.79 (s, 1H), 5.45 (s, 1H), 4.24 (s, 1H), 3.63 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 195.5, 171.5, 154.5, 140.3, 138.7, 136.8, 131.3, 129.5, 129.2, 128.98, 128.94, 128.5, 128.1, 127.96, 127.92, 126.0, 125.7, 125.2, 120.4, 118.9, 117.0, 113.0, 65.1, 54.6, 53.1; HRMS: calcd for $C_{29}H_{24}N_2NaO_4$: 487.1634; found: 487.1628 $[M+Na]^+$



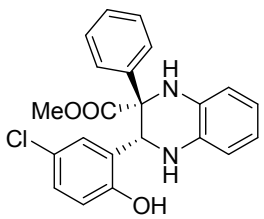
***rel*-(2*R*,3*R*)-methyl3-(2-hydroxy-4-methoxyphenyl)-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3i).** Yellow solid (30 mg, 77% yield), m.p. 145-146 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 7.54-7.51 (m, 2H), 7.36-7.26 (m, 3), 6.76-6.22 (m, 7H), 5.20 (m, 2H), 3,71 (s, 3H), 3.64 (s. 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 172.17, 160.35, 156.7, 139.7, 131.7, 130.2, 128.8, 128.3, 126.3, 120.9, 119.8, 117.2, 115.2, 106.0, 102.8, 65.3, 58.2, 55.1, 53.0; HRMS: calcd for $C_{23}H_{22}N_2NaO_4$: 413.1477; found: 413.1472 $[M+Na]^+$



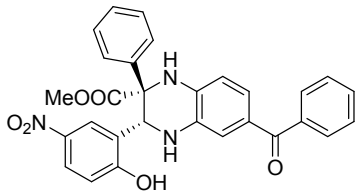
***rel*-(2*R*,3*R*)-methyl3-(2-hydroxy-5-nitrophenyl)-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3j).** Yellow solid (38 mg, 95% yield), m.p. 190-191 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 10.86 (s, 1H), 8.00 (m, 1H), 7.84 (m, 1H), 7.51-7.48 (m, 2H), 7.37-7.34 (m, 3H), 6.90-6.83 (m, 3H), 6.67- 6.68 (m, 2H), 5.32 (s, 1H), 5.23 (s, 1H), 4.01 (s, 1H), 3.73 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 171.1, 162.9, 140.1, 138.5, 132.4, 129.2, 129.0, 127.9, 126.1, 125.7, 125.4, 124.7, 123.3, 123.0, 120.1, 119.4, 118.1, 115.8, 64.6, 59.6, 53.3; HRMS: calcd for $C_{22}H_{19}N_3NaO_5$: 428.1222; found: 428.1217 $[M+Na]^+$



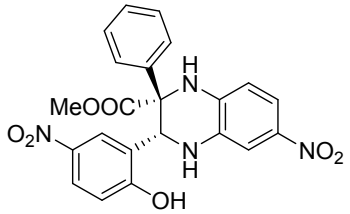
***rel*-(2*R*,3*R*)-methyl3-(5-bromo-2-hydroxyphenyl)-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3k).** Yellow solid (35 mg, 81% yield), m.p. 168-169 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 8.99 (s, 1H), 7.51-7.48 (m, 2H), 7.37-7.33 (m, 3H), 7.22-7.18 (m, 1H), 6.94-6.93 (m, 1H), 6.80-6.60 (m, 5H), 5.16 (s, 2H), 4.04 (s, 1H), 3.67 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 171.7, 154.9, 139.2, 132.1, 131.9, 131.7, 129.5, 128.9, 128.6, 126.4, 126.2, 121.5, 120.0, 119.6, 117.7, 115.4, 111.5, 65.0, 58.8, 53.1 ; HRMS: calcd for $C_{22}H_{19}BrN_2NaO_3$: 461.0477; found: 461.0471 $[M+Na]^+$



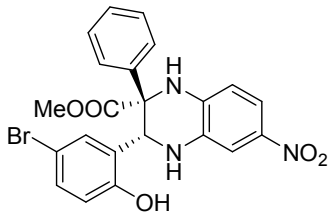
***rel*-(2*R*,3*R*)-methyl3-(5-chloro-2-hydroxyphenyl)-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3l).** Yellow solid (31 mg, 80% yield), m.p. 159-160 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 8.04 (s, 1H), 7.59 (s, 1H), 7.42-7.42 (m, 1H), 7.27-7.24 (m, 2H), 7.12 (m, 1H), 6.95-54 (m, 7H), 5,30 (s, 1H), 5.14 (s, 1H), 4.11 (s, 1H), 3.63 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 171.6, 154.9, 142.2, 134.8, 131.0, 130.1, 130.0, 129.2, 128.6, 126.7, 124.7, 120.8, 120.2, 120.0, 117.6, 116.9, 115.2, 65.1, 57.0, 53.2; HRMS: calcd for $C_{22}H_{19}ClN_2NaO_3$: 417.0982; found: 417.0976 $[M+Na]^+$



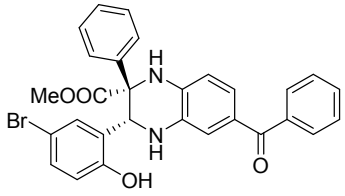
***rel*-(2R,3R)-methyl6-benzoyl-3-(2-hydroxy-5-nitrophenyl)-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3m).** Yellow solid (38 mg, 75% yield), m.p. 216-217 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 10.63 (s, 1H), 8.15-8.14 (m, 1H), 7.72 (m, 1H), 7.46-7.41 (m, 4H), 7.28-7.10 (m, 6H), 6.94-6.75 (m, 4H), 6.50 (s, 1H), 5.43 (s, 1H), 4.62 (s, 1H), 3.38 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 194.7, 170.1, 161.0, 140.8, 139.6, 138.5, 135.5, 130.5, 129.1, 128.8, 128.1, 127.6, 127.5, 127.3, 126.9, 125.3, 124.0, 123.7, 123.0, 115.6, 114.8, 112.2, 64.0. 52.3, 50.2; HRMS: calcd for $C_{29}H_{23}N_3NaO_6$: 532.1485; found: 532.1480 $[M+Na]^+$



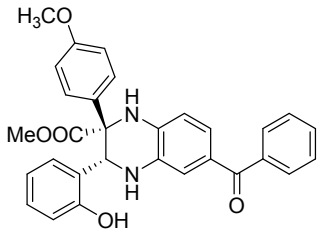
***rel*-(2*R*,3*R*)-methyl3-(2-hydroxy-5-nitrophenyl)-6-nitro-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3n).** Yellow solid (41 mg, 91% yield), m.p. 225-226 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 8.20 (s, 1H), 7.96-7.93 (m, 1H), 7.63-7.62 (m, 1H), 7.58-7.55 (m, 2H), 7.34-7.23 (m, 4), 6.97-6.89 (m, 2H), 6.32 (s, 1H), 5.58 (s, 1H) 3.61 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 170.1, 161.2, 140.5, 140.3, 139.5, 137.1, 129.2, 128.8, 128.4, 127.1, 125.4, 124.8, 123.0, 117.0, 115.5, 112.5, 110.1, 64.6, 53.1, 51.3; HRMS: calcd for $C_{22}H_{18}N_4NaO_7$: 473.1073; found: 437.1068 $[M+Na]^+$



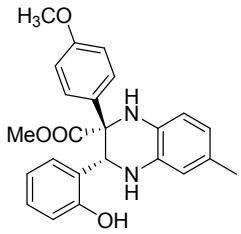
***rel*-(2*R*,3*R*)-methyl3-(5-bromo-2-hydroxyphenyl)-6-nitro-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3o).** Yellow solid (32 mg, 67% yield), m.p. 203-204 °C. ^{1}H NMR (300 MHz, $CDCl_3$+DMSO) □δ 9.32 (s, 1H), 7.37-7.31 (m, 3H), 7.11-6.97 (m, 6H), 6.86-6.76 (m, 2H), 6.56-6.53 (m, 1), 5.41 (s, 1H) 3.31 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$+DMSO) δ 169.9, 153.7, 140.6, 138.3, 137.2, 130.2, 129.5, 128.9, 128.0, 127.4, 125.3, 116.6, 115.5, 111.8, 110.5, 108.4, 63.9, 52.1, 49.7; HRMS: calcd for $C_{22}H_{18}BrN_3NaO_5$: 506.0328; found: 506.0322 $[M+Na]^+$



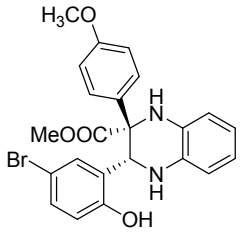
***rel*-(2*R*,3*R*)-methyl6-benzoyl-3-(5-bromo-2-hydroxyphenyl)-2-phenyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3p).** Yellow solid (33 mg, 61% yield), m.p. 210-211 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 9.23(s, 1H), 7.60-7.55 (m, 4H), 7.36-7.23 (m, 8H), 6.84-6.68 (m, 4H), 6.06 (s, 1H), 5.50(s, 1H), 3.52 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 195.2, 170. 7, 154.1, 141.1, 138.8, 135.7, 130.8, 130.6, 129.7, 129.5, 129.2, 129.1, 128.4, 127.8, 127.7, 127.6, 127.4, 125.6, 124.0, 117.1, 116.3, 111.3, 64.4, 52.6, 51.3; HRMS: calcd for $C_{29}H_{23}BrN_2NaO_4$: 565.0739; found: 565.0734 $[M+Na]^+$



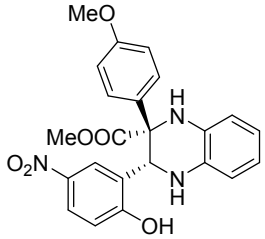
***rel*-(2*R*,3*R*)-methyl6-benzoyl-3-(2-hydroxyphenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3q).** Yellow solid (34 mg, 70% yield), m.p. 208-209 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 7.78 (s, 1H), 7.69-7.66 (m, 2H), 7.66-7.26(m, 6H), 7.09-7.07 (m, 3H), 6.87-6.69 (m, 5H), 5.83 (s, 1H), 5.44(s, 1H), 4.31 (s, 1H), 3.77 (s, 3H), 3.60 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 195.7, 171.7, 159.5, 154.5, 138.7, 136.8, 132.4, 131.3, 129.5, 129.4, 128.7, 127.9, 127.8, 127.7, 127.2, 126.0, 125.1, 120.2, 118.5, 116.7, 114.2, 112.8, 64.5, 55.2, 54.1, 53.0; HRMS: calcd for $C_{30}H_{26}N_2NaO_5$: 517.1739; found:517.1734 $[M+Na]^+$



***rel*-(2*R*,3*R*)-methyl3-(2-hydroxyphenyl)-2-(4-methoxyphenyl)-6-methyl-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3r).** Yellow solid (32 mg, 80% yield), m.p. 169-170 °C. ^{1}H NMR (300 MHz, $CDCl_3$) □δ 8.79 (s, 1H), 7.46-7.43 (m, 2H), 7.11-6.37(m, 9H), 5.16 (s, 1H), 4.95 (s, 1H), 4.00 (s, 1H), 3.77 (s, 3H), 3.61 (s, 3H), 2.14 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 172.3, 159.4, 155.4, 131.7, 130.1, 129.5, 129.3, 129.0, 127.6, 125.2, 121.4, 119.8, 117.8, 117.7, 115.2, 114.1, 64.7, 58.4, 55.2, 52.9, 20.6;HRMS: calcd for $C_{24}H_{24}N_2NaO_4$: 427.1634; found: 427.1628 $[M+Na]^+$

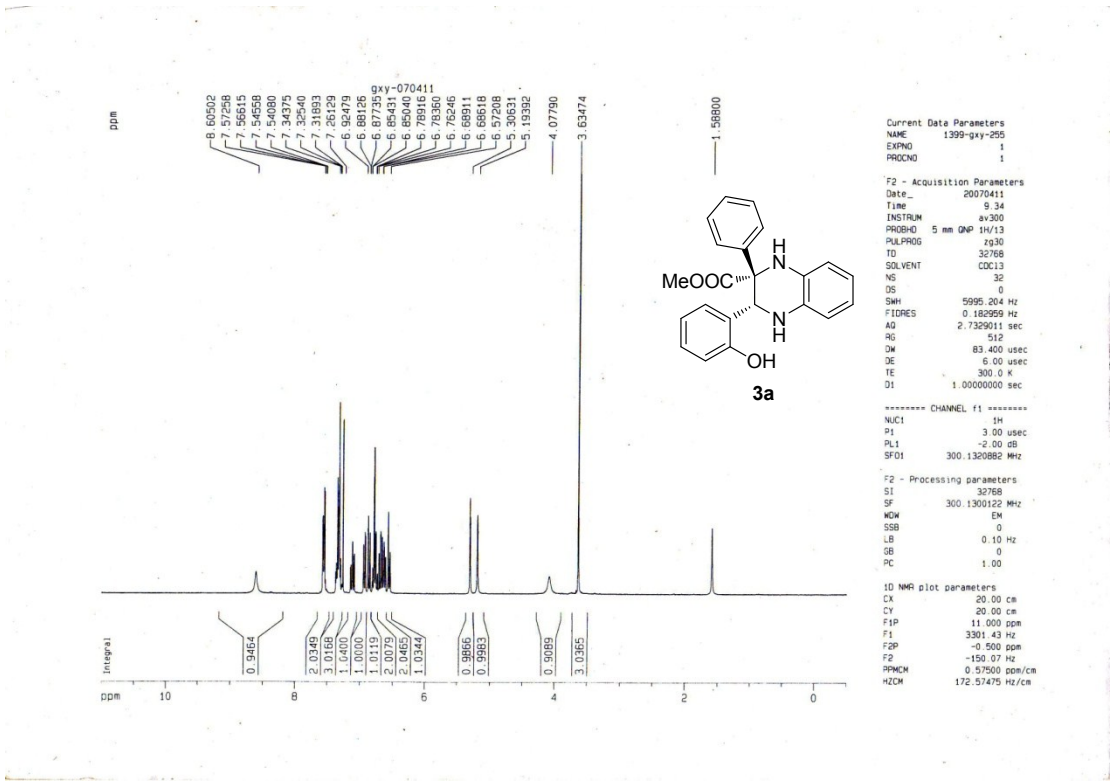


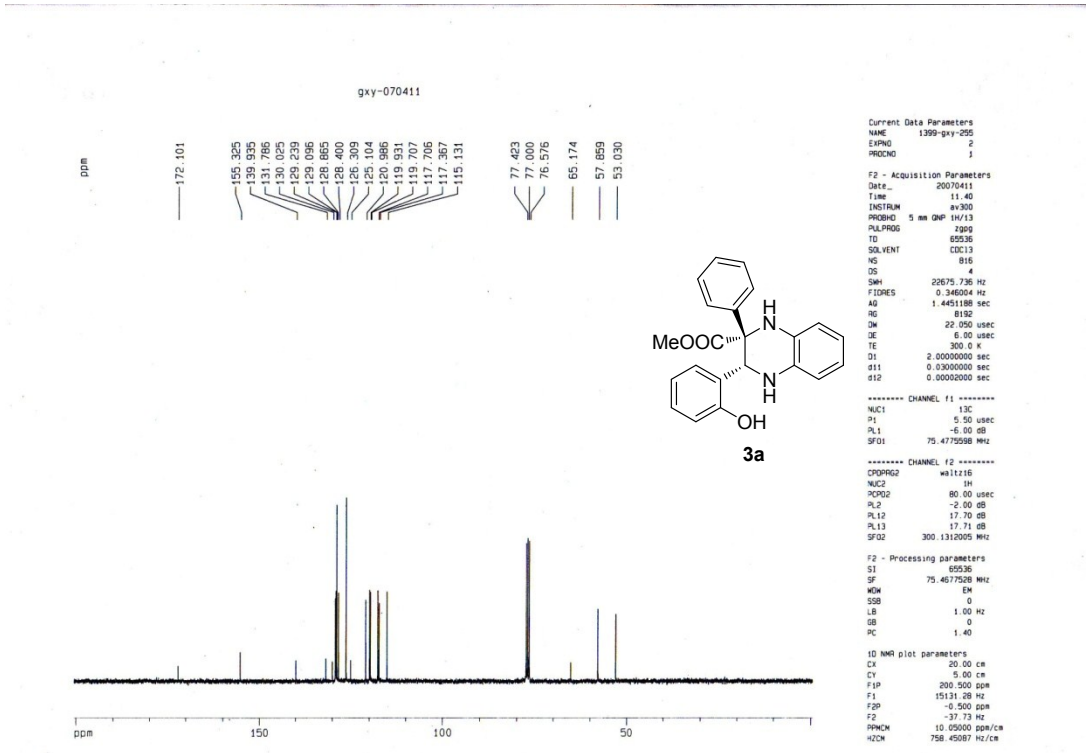
***rel*-(2*R*,3*R*)-methyl3-(5-bromo-2-hydroxyphenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3s).** Yellow solid (30 mg, 65% yield), m.p. 182-183 °C. ^{1}H NMR (300 MHz, $CDCl_3$+ DMSO) □δ 9.24 (s, 1H), 7.43-7.33 (m, 3H), 6.99-6.96(m, 1H), 6.73-6.23(m, 7H), 5.34 (s, 1H), 5.30 (s, 1H), 4.37 (s, 1H), 3.63 (s, 3H), 3.45(s, 3H); ^{13}C NMR (75MHz, $CDCl_3$) δ 171.26, 158.8, 154.1, 133.0, 130.3, 129.4, 127.0, 118.8, 118.6, 117.1, 114.8, 114.2, 113.6, 111.0, 63.6, 54.8, 52.4, 52.3; HRMS: calcd for $C_{23}H_{21}BrN_2NaO_4$: 491.0582; found: 491.0577 $[M+Na]^+$

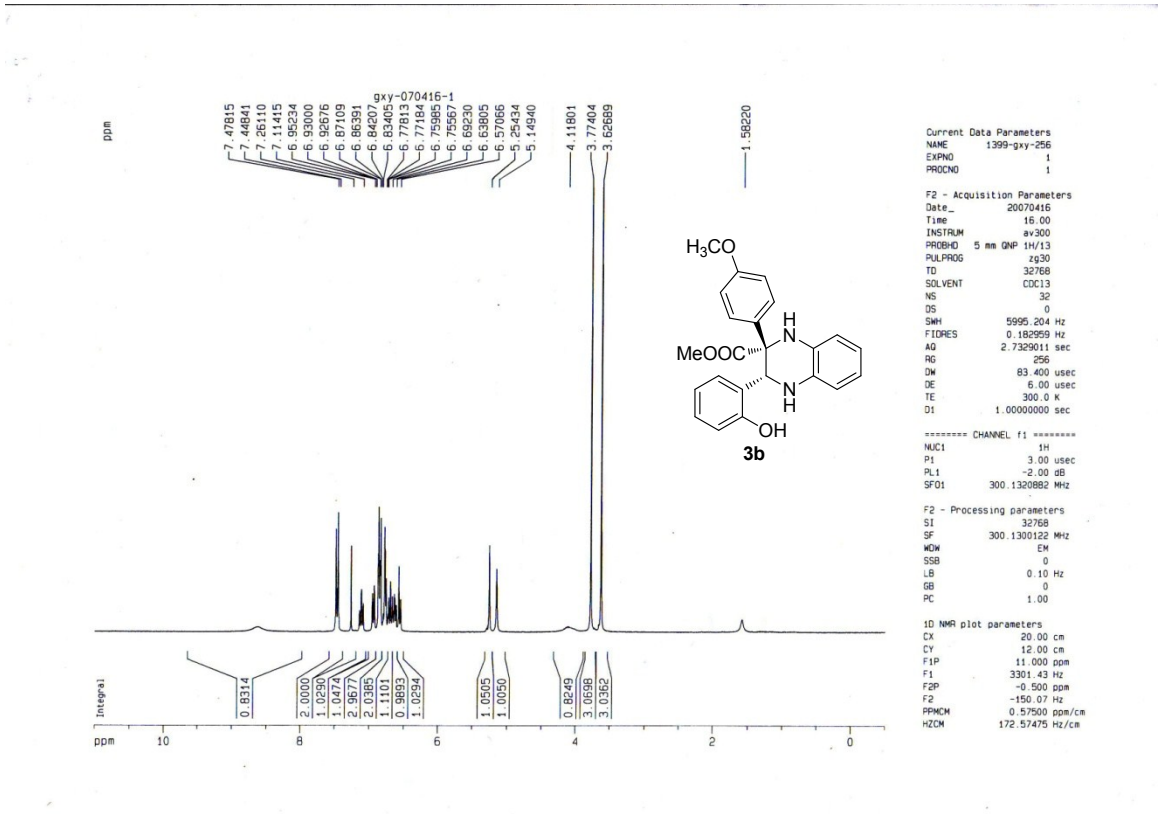


***rel*-(2*R*,3*R*)-methyl3-(2-hydroxy-5-nitrophenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroquinoxaline-2-carboxylate (3t).** Yellow solid (37 mg, 86% yield), m.p. 197-198 °C. ^{1}H NMR (300 MHz, $CDCl_3$+ DMSO) □δ 10.58 (s, 1H), 8.16 (s, 1H), 7.69-7.67 (m, 1H), 7.32-7.30 (m, 2H), 6.70-6.09 (m, 7H), 5.50 (s, 1H), 5.32 (s, 1H), 4.38 (s, 1H), 3,51 (s, 3H), 3.31 (s, 3H); ^{13}C NMR (75MHz, $CDCl_3$+DMSO) δ 170.6, 161.0, 158.5, 139.6, 132.8, 129.8, 129.7, 128.0, 126.7, 123.8, 123.7, 118.5, 118.3, 114.7, 114.2, 114.0, 113.3, 63.1, 54.6, 51.9, 50.8; HRMS: calcd for $C_{23}H_{21}N_3NaO_6$: 458.1328; found: 458.1323 $[M+Na]^+$

5. NMR spectra of products



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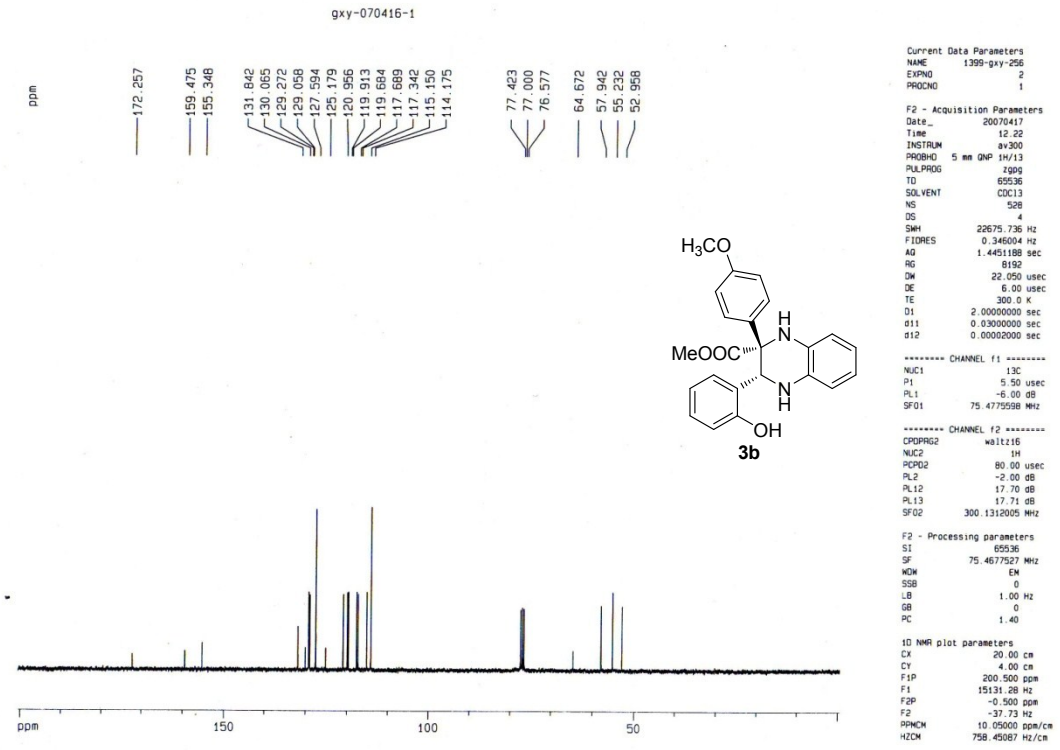
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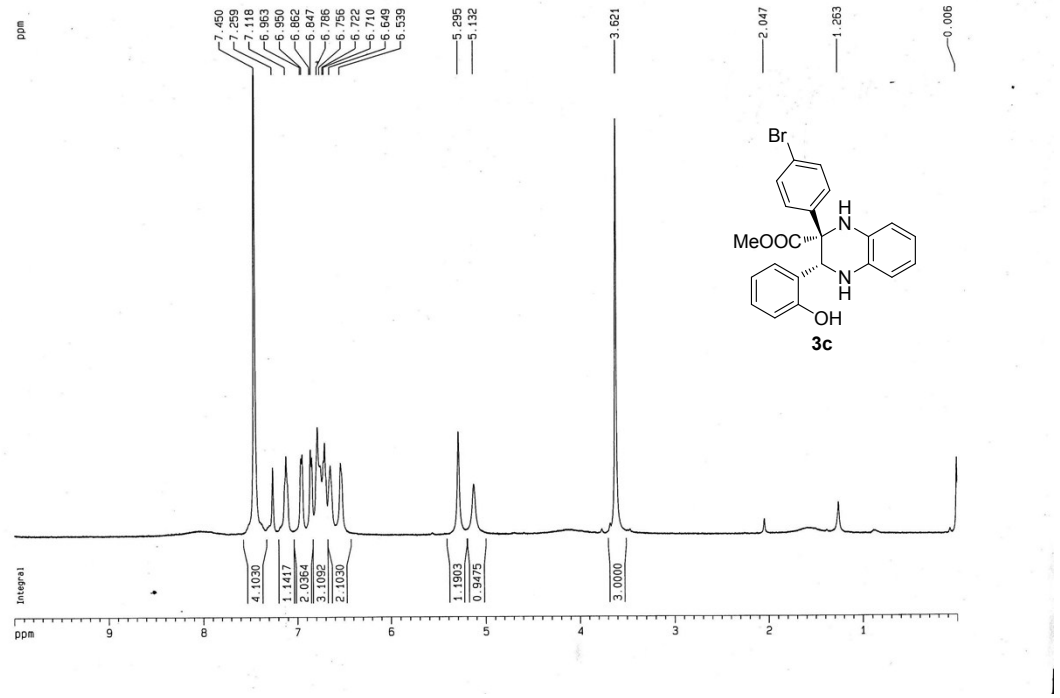
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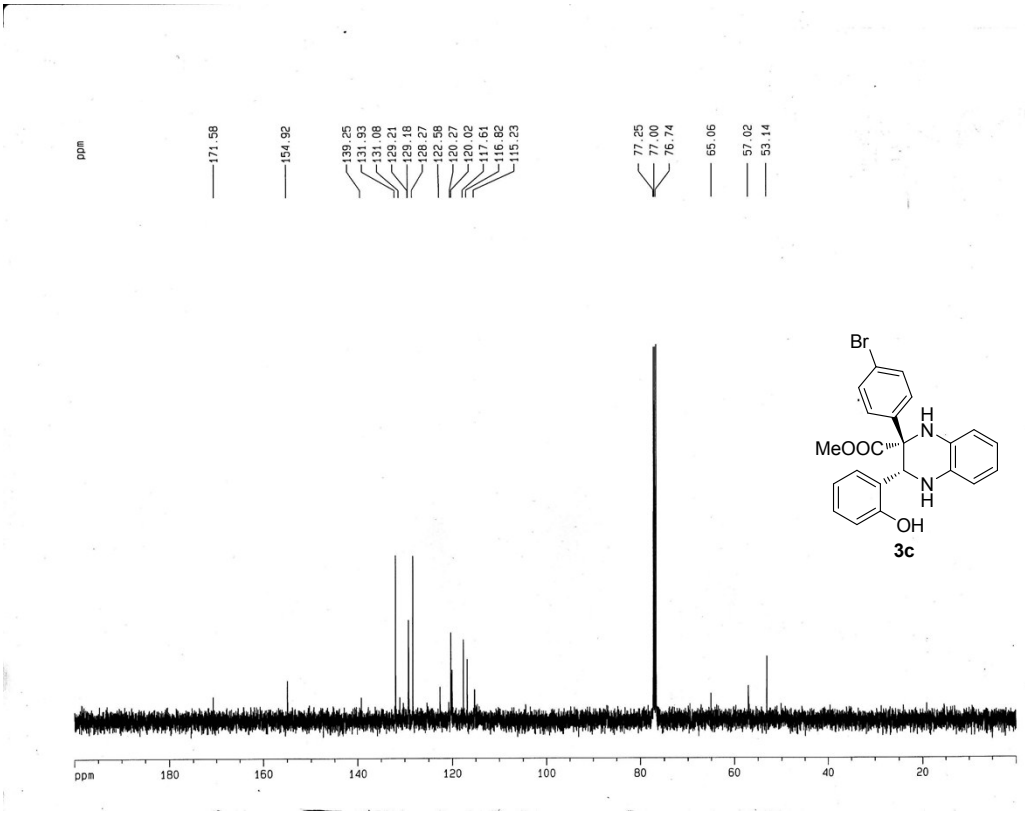
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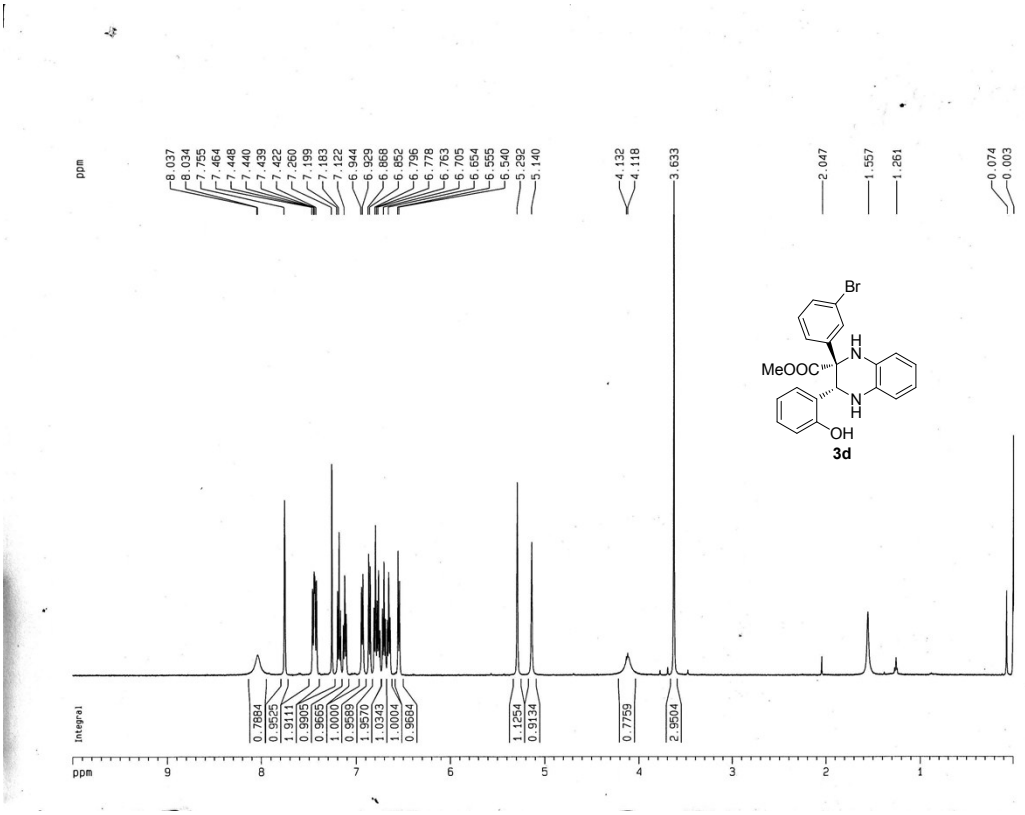
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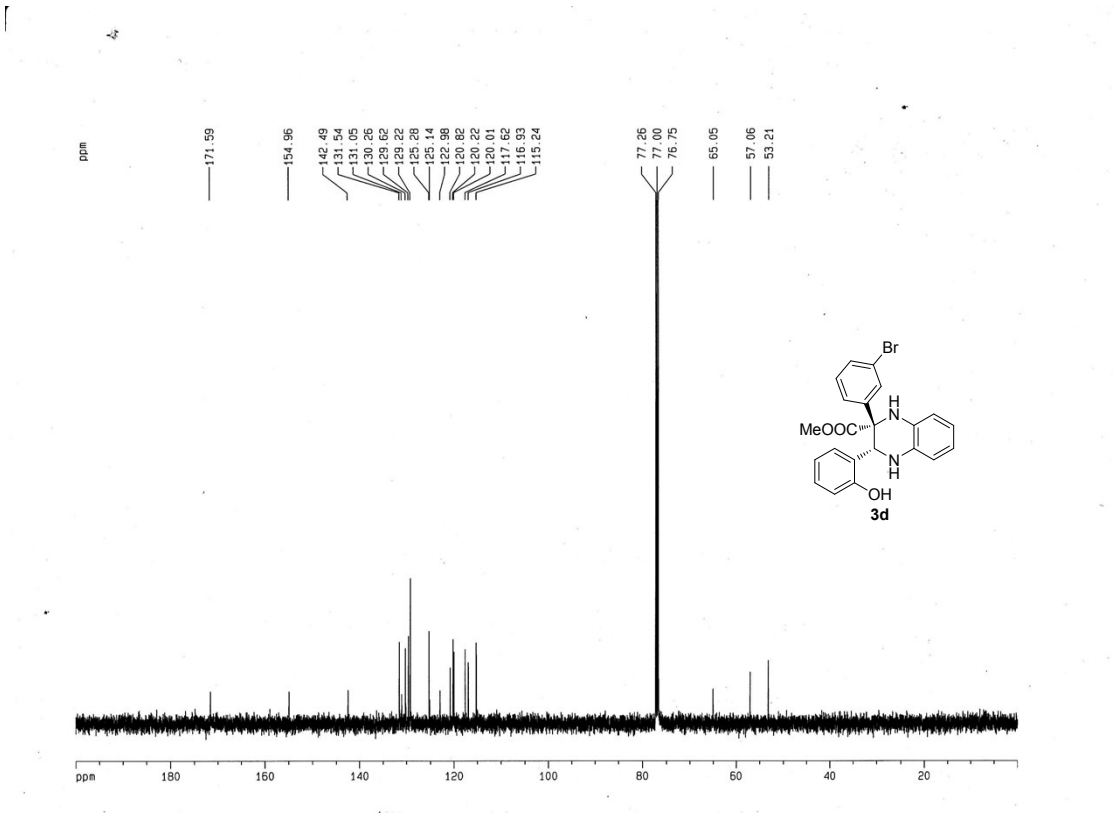
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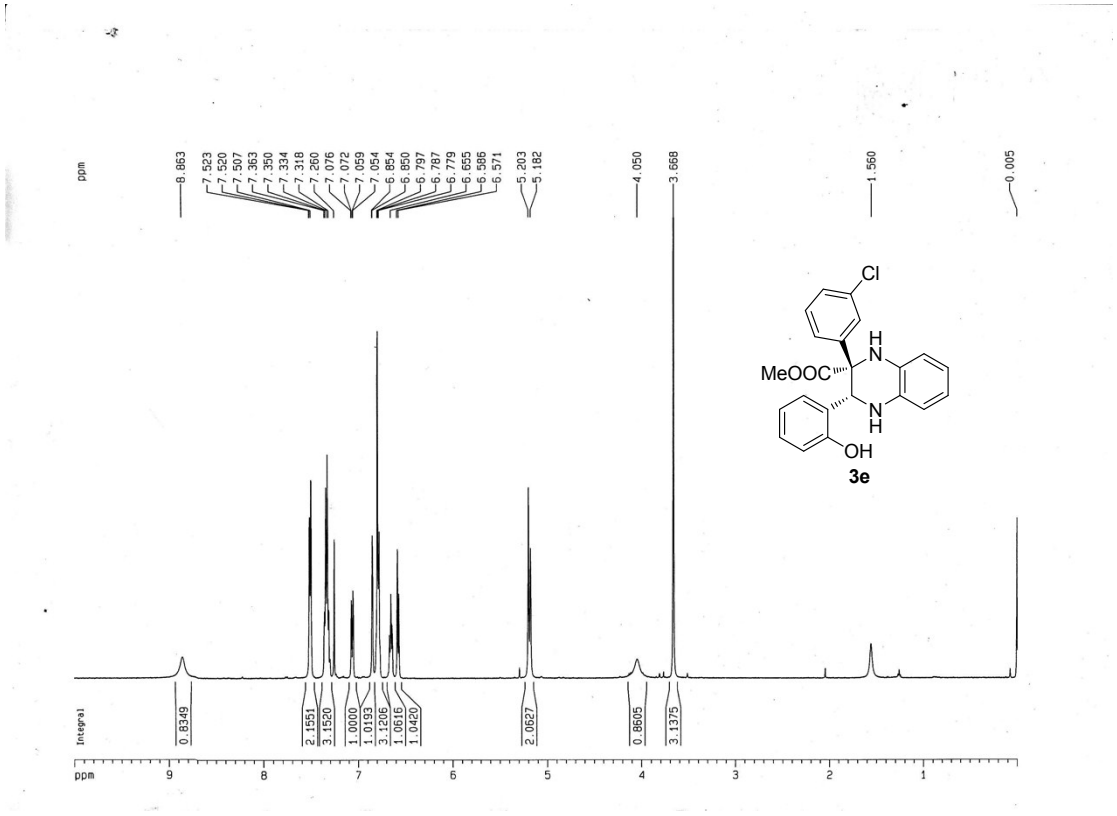
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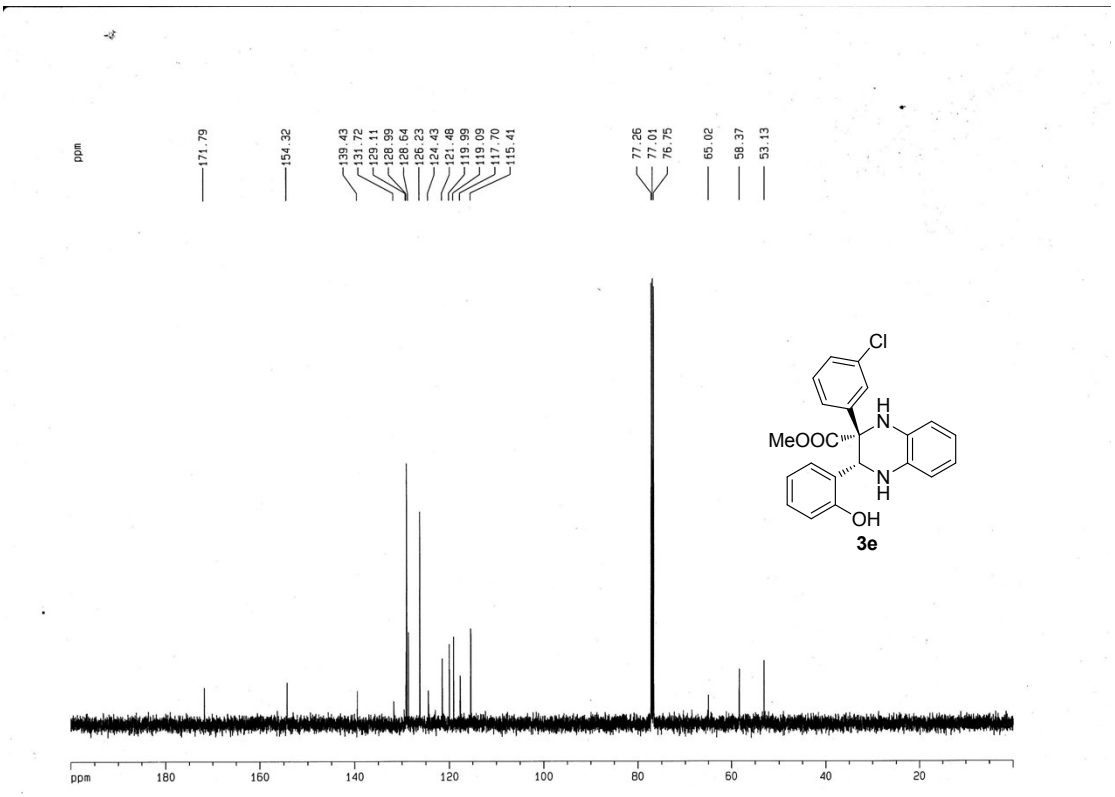
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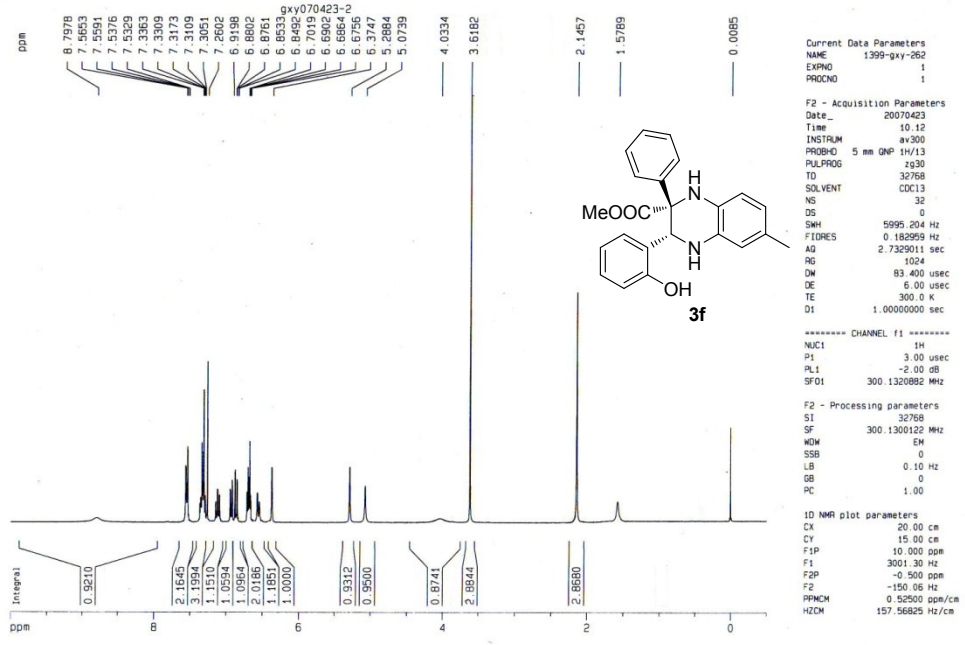
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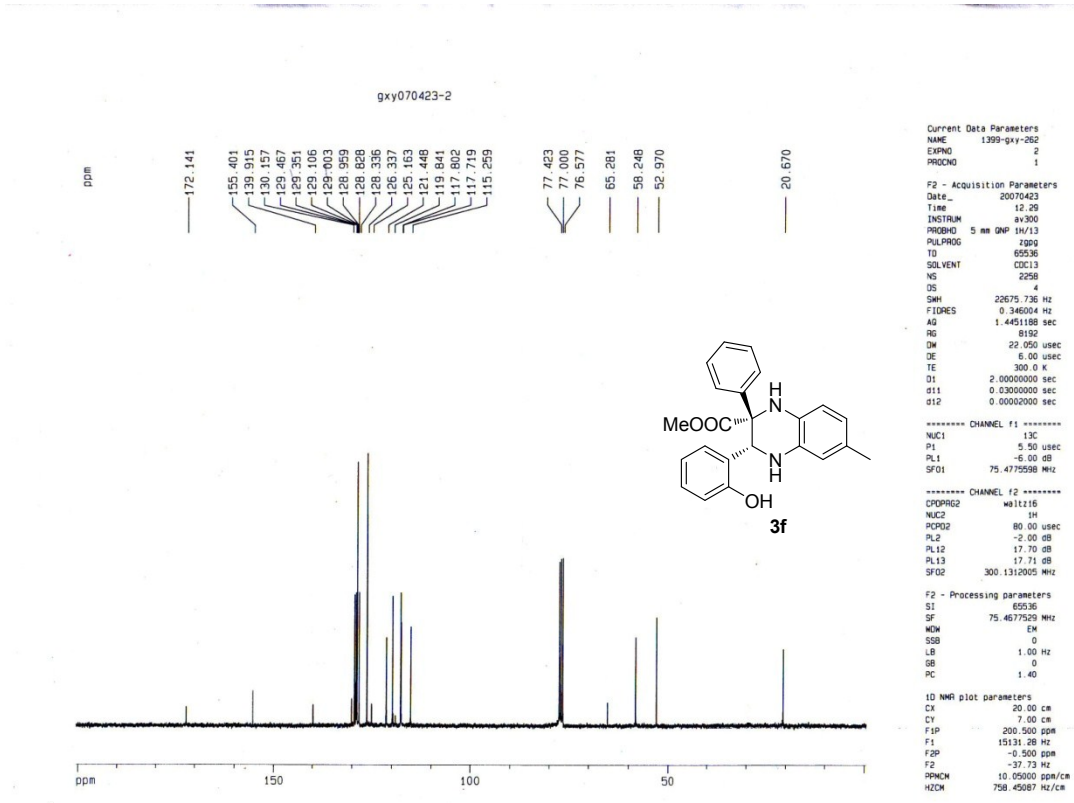
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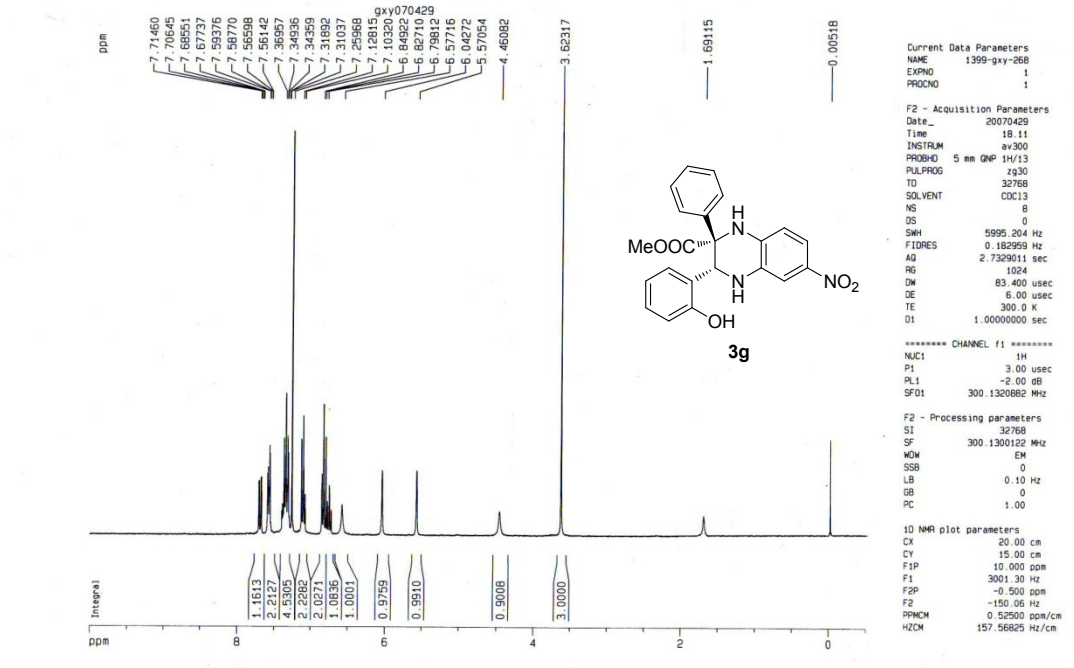
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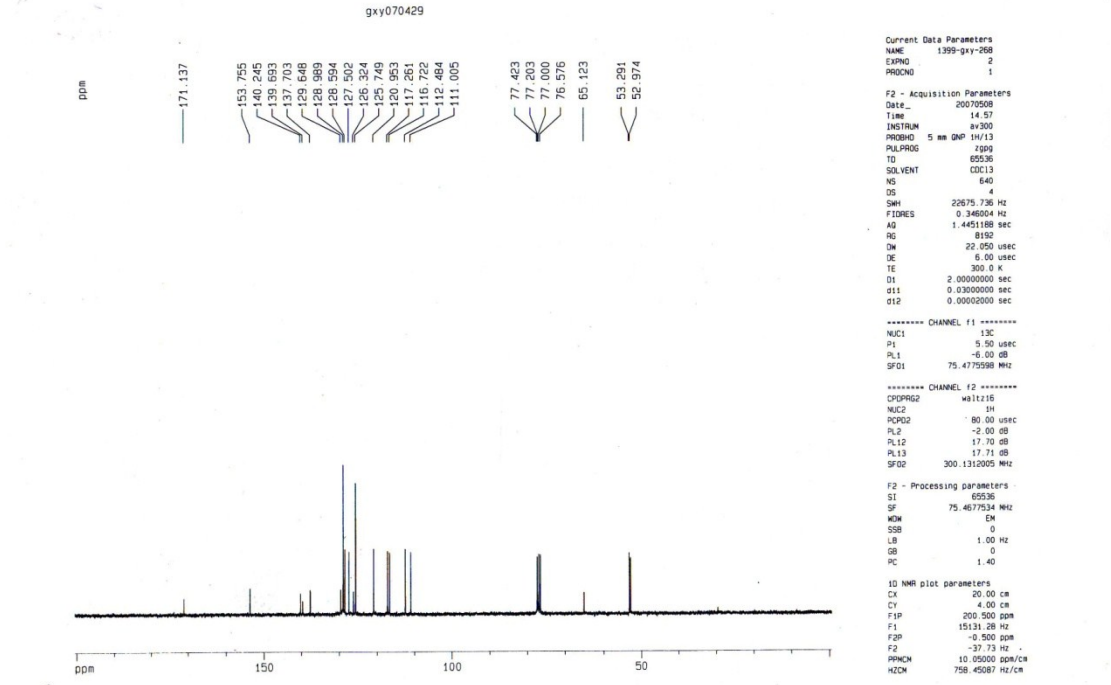
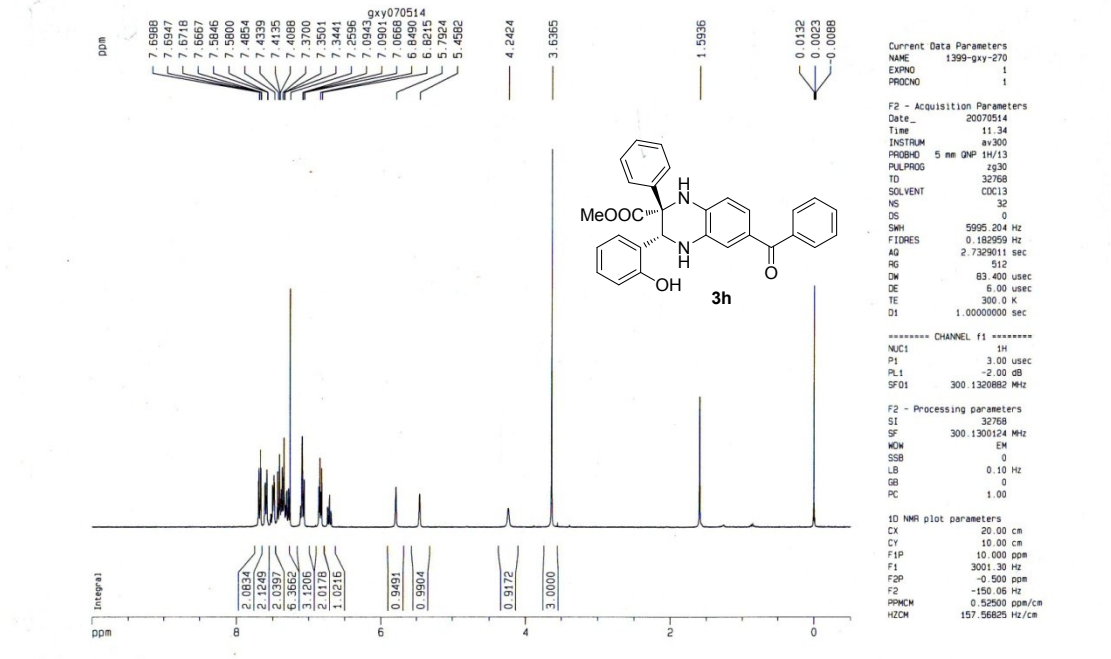
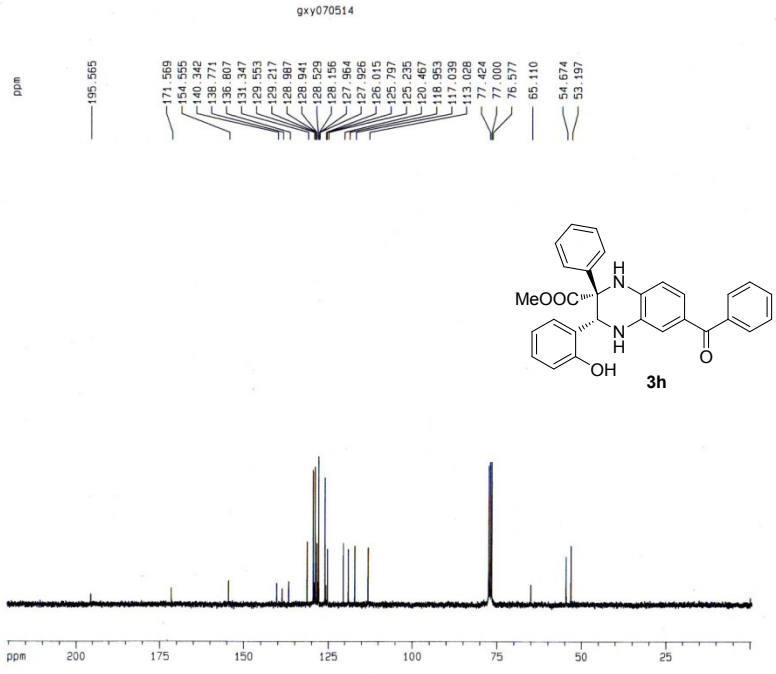
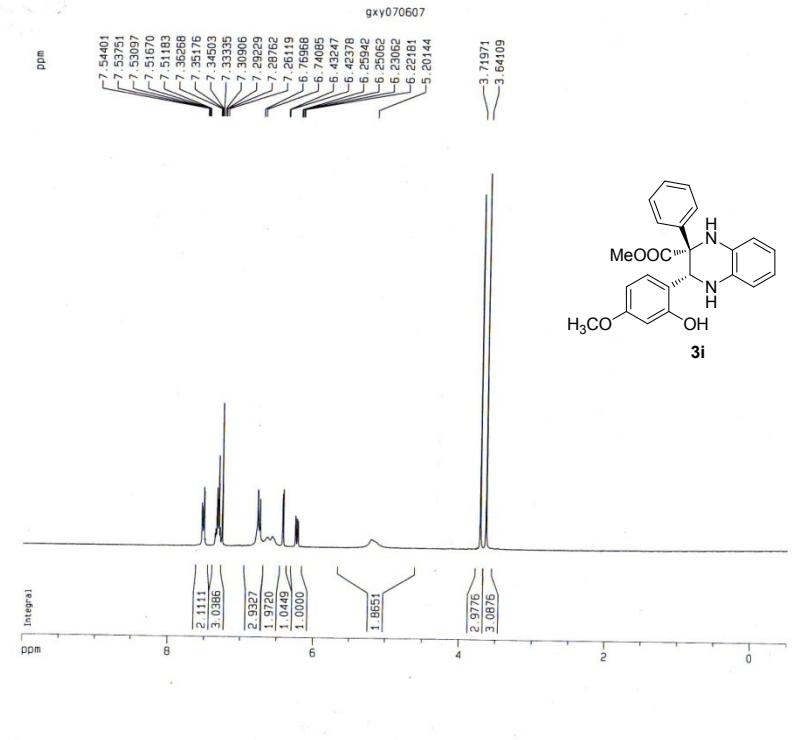
Time 18.11
INSTRUM av300
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1024
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm


gxy070429
ppm
171.137
153.755
140.245
139.693
137.703
129.648
128.989
128.594
127.502
126.324
125.749
120.953
117.261
116.722
112.484
111.005
77.423
77.203
77.000
76.576
65.123
53.291
52.974
ppm
150
100
50
Current Data Parameters
NAME 1399-gxy-268
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070508
Time 14.57
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 640
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677534 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 4.00 cm
F1P 200.500 ppm
F1 15131.28 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 10.05000 ppm/cm
HZCM 758.45087 Hz/cm

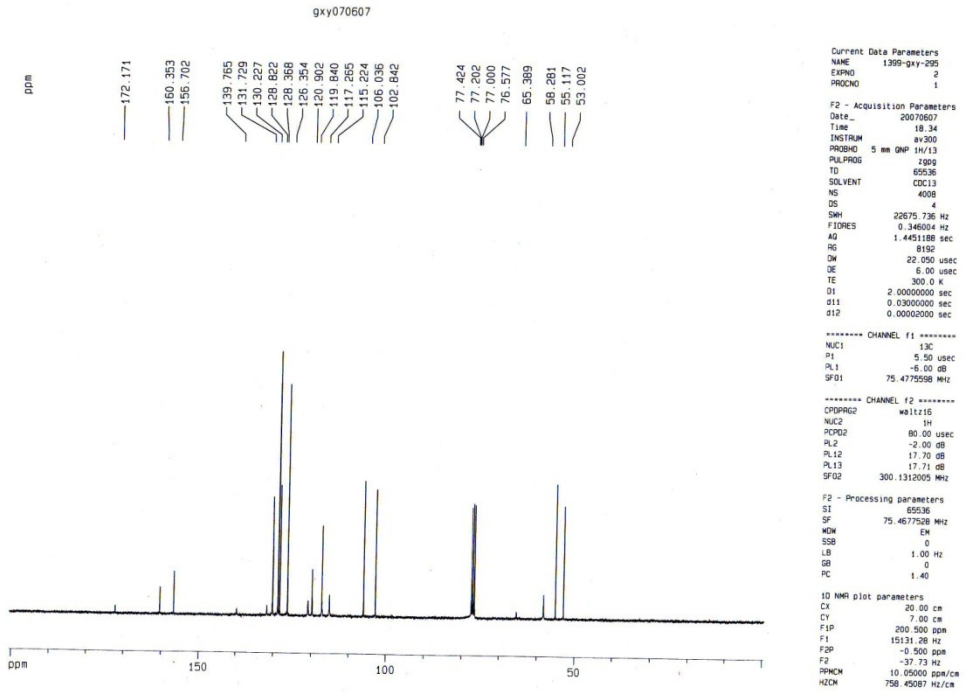

gxy070514
ppm
7.6988
7.6947
7.6718
7.6667
7.5846
7.5800
7.4854
7.4339
7.4135
7.4088
7.3700
7.3501
7.3441
7.2596
7.0943
7.0901
7.0668
6.8490
6.8215
5.7924
5.4582
4.2424
3.6365
1.5936
0.0132
0.0023
-0.0088
MeOOC
H
N
N
H
O
OH
3h
Integral
2.0834
2.1249
2.0397
6.3662
3.1206
2.0178
1.0216
0.9491
0.9904
0.9172
3.0000
ppm
8
6
4
2
0
Current Data Parameters
NAME 1399-gxy-270
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070514
Time 11.34
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300124 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

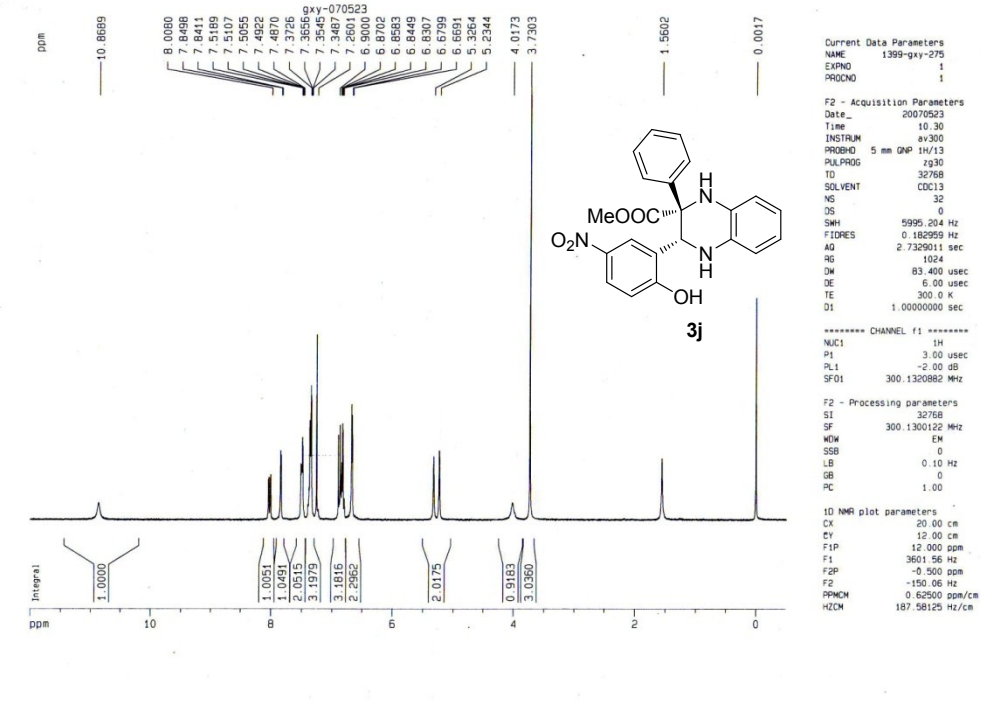


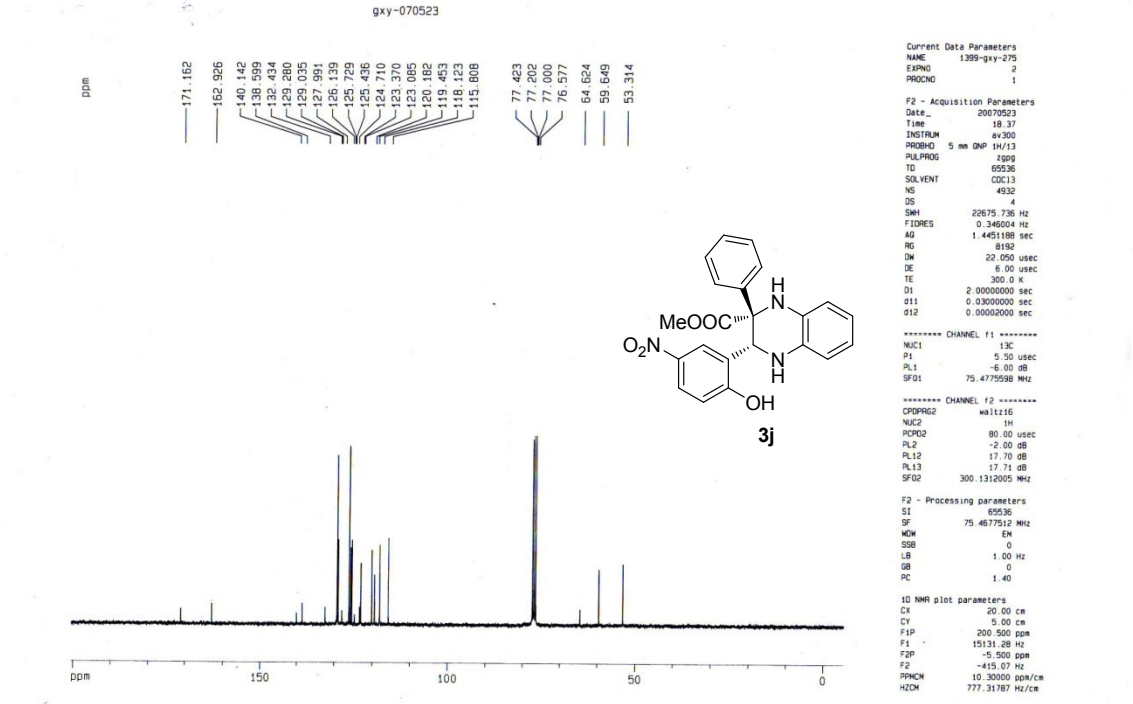

```
Current Data Parameters
NAME            1399-gxy-270
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_           20070514
Time                 12.41
INSTRUM              av300
PROBHD   5 mm QNP 1H/13
PULPROG              zgpg
TD                  65536
SOLVENT              CDCl3
NS                    2106
DS                       4
SWH             22675.736 Hz
FIDRES           0.346004 Hz
AQ               1.4451188 sec
RG                    8192
DW                 22.050 usec
DE                  6.00 usec
TE                 300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                   5.50 usec
PL1                 -6.00 dB
SFO1           75.4775598 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2              80.00 usec
PL2                 -2.00 dB
PL12                17.70 dB
PL13                17.71 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                      65536
SF             75.4677518 MHz
WDW                        EM
SSB                         0
LB                   1.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   4.00 cm
F1P                220.500 ppm
F1               16640.64 Hz
F2P                 -0.500 ppm
F2                 -37.73 Hz
PPMCM            11.05000 ppm/cm
HZCM             833.91864 Hz/cm
```

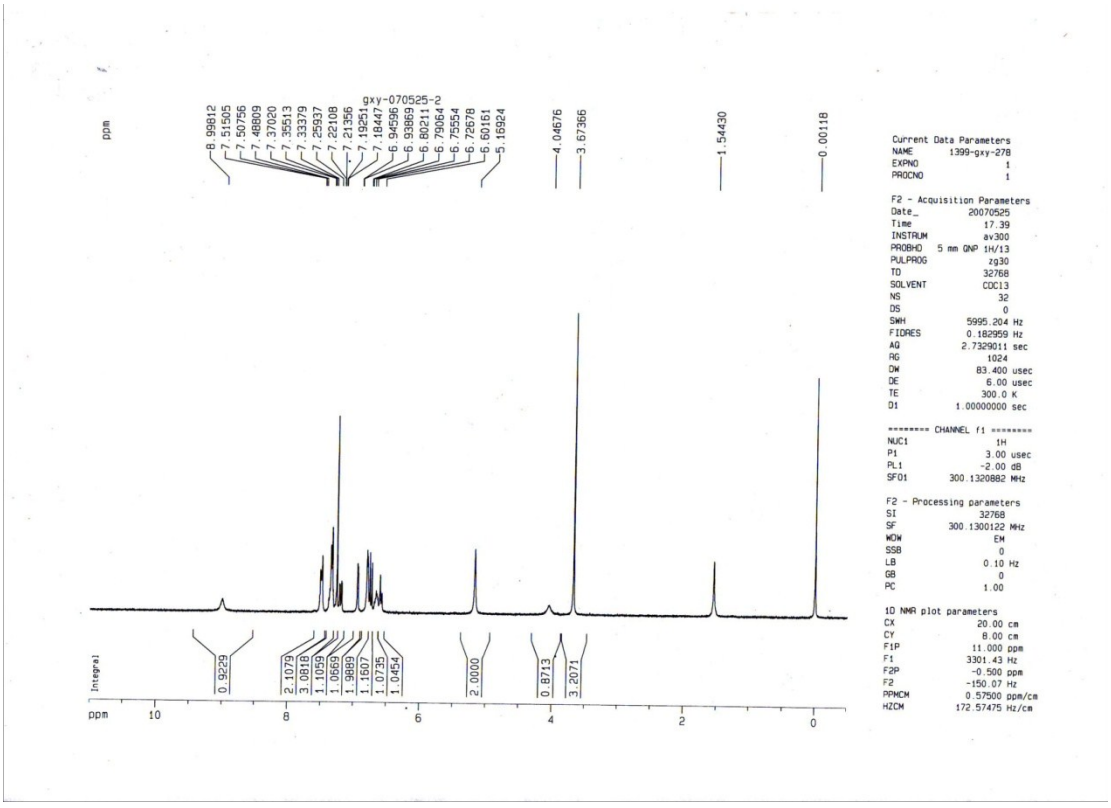


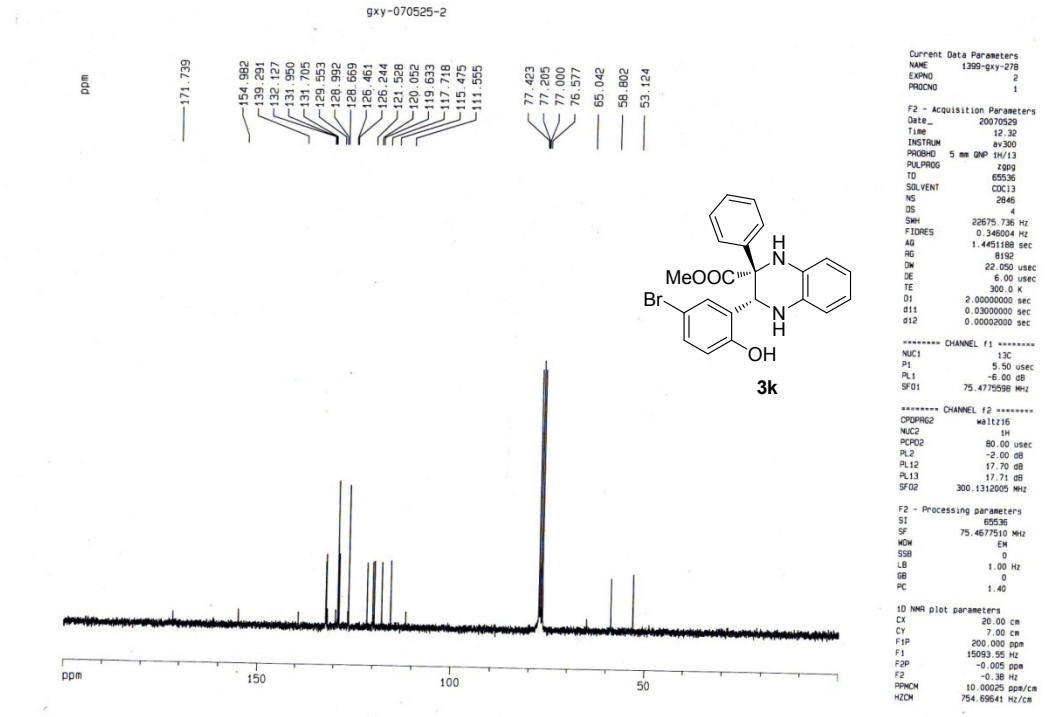

```
Current Data Parameters
NAME            1399-gxy-295
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_           20070607
Time                 16.39
INSTRUM              av300
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT              CDCl3
NS                      32
DS                       0
SWH              5995.204 Hz
FIDRES           0.182959 Hz
AQ               2.7329011 sec
RG                     512
DW                 83.400 usec
DE                  6.00 usec
TE                 300.0 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   3.00 usec
PL1                 -2.00 dB
SFO1           300.1320882 MHz

F2 - Processing parameters
SI                      32768
SF             300.1300122 MHz
WDW                        EM
SSB                         0
LB                   0.10 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                  20.00 cm
CY                  10.00 cm
F1P                10.000 ppm
F1               3001.30 Hz
F2P                 -0.500 ppm
F2                -150.06 Hz
PPMCM            0.52500 ppm/cm
HZCM             157.56825 Hz/cm
```

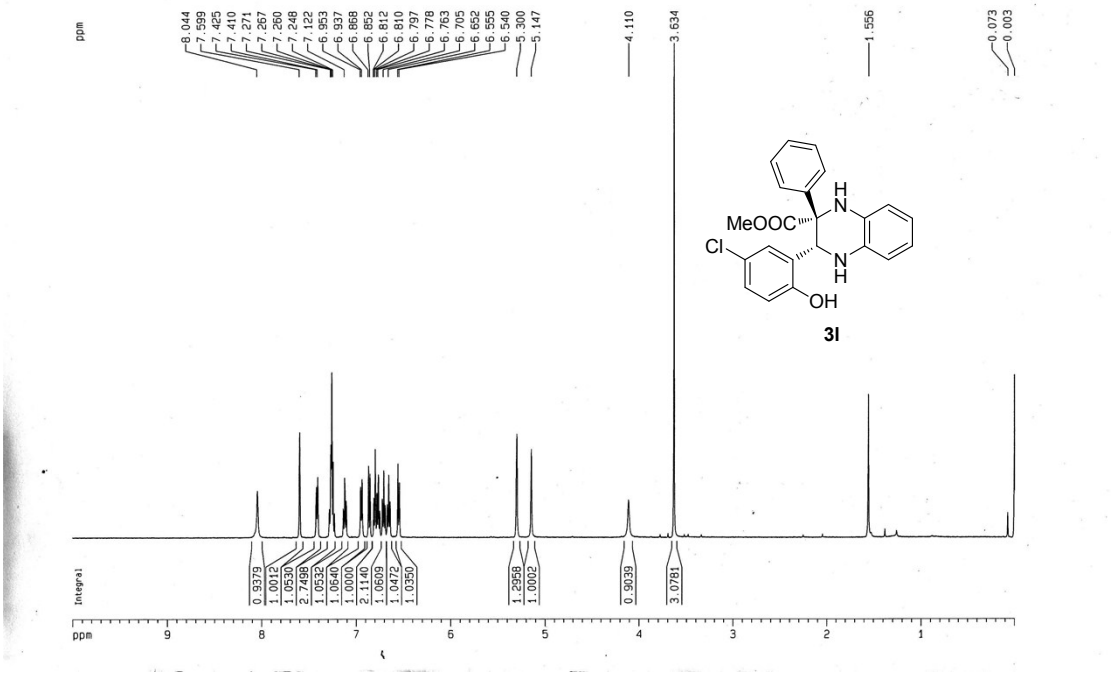

gxy070607
ppm
172.171
160.353
156.702
139.765
131.729
130.227
128.822
128.368
126.354
120.902
119.840
117.265
115.224
106.036
102.842
77.424
77.202
77.000
76.577
65.389
58.281
55.117
53.002
ppm
150
100
50
Current Data Parameters
NAME 1399-gxy-295
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070607
Time 18.34
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 4008
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677528 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 7.00 cm
F1P 200.500 ppm
F1 15131.28 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 10.05000 ppm/cm
HZCM 758.45087 Hz/cm

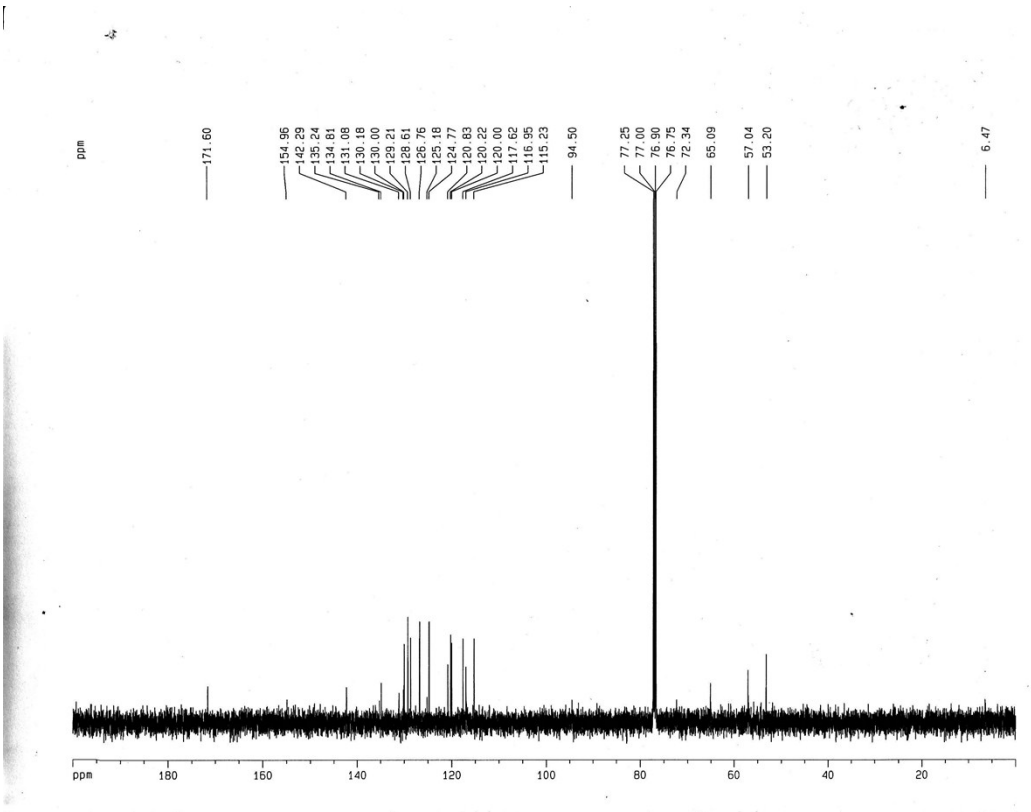

gxy-070523
ppm
10.8689
8.0080
7.8498
7.8411
7.5189
7.5107
7.5055
7.4922
7.4870
7.3726
7.3656
7.3545
7.3487
7.2601
6.9000
6.8702
6.8583
6.8449
6.8307
6.6799
6.6691
5.3264
5.2344
4.0173
3.7303
1.5602
0.0017
MeOOC
O2N
H
N
N
H
OH
3j
Integral
1.0000
1.0051
1.0491
2.0515
3.1979
3.1816
2.2962
2.0175
0.9183
3.0360
ppm
10
8
6
4
2
0
Current Data Parameters
NAME 1399-gxy-275
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070523
Time 10.30
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1024
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 12.000 ppm
F1 3601.56 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.62500 ppm/cm
HZCM 187.58125 Hz/cm

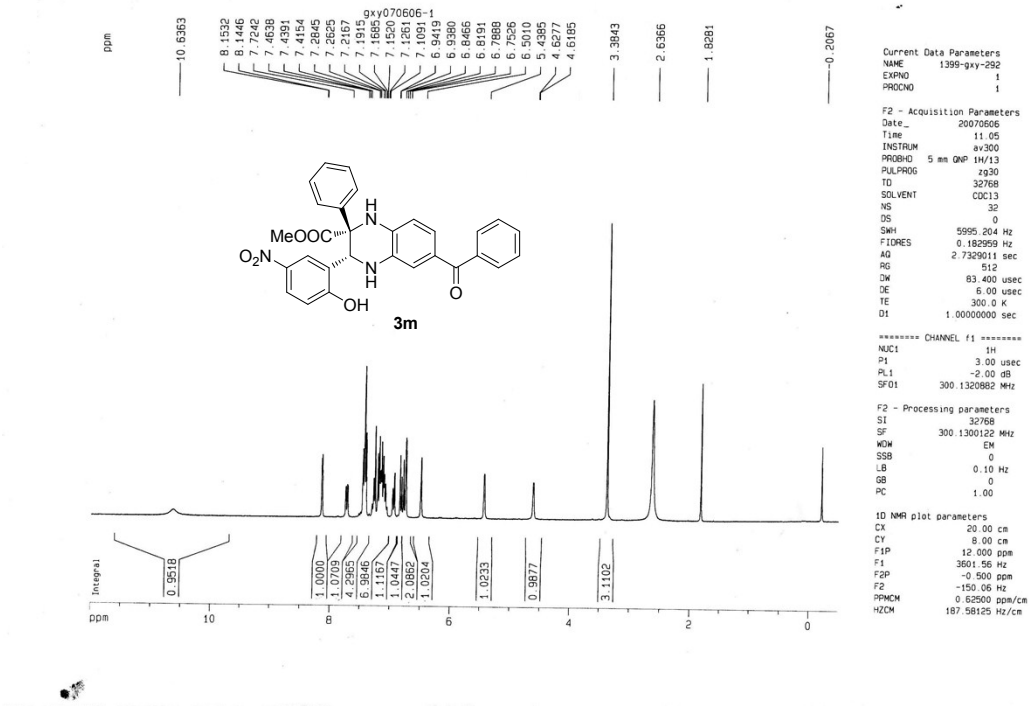

gxy-070523
ppm
171.162
162.926
140.142
138.599
132.434
129.280
129.035
127.991
126.139
125.729
125.436
124.710
123.370
123.085
120.182
119.453
118.123
115.808
77.423
77.202
77.000
76.577
64.624
59.649
53.314
MeOOC
O2N
H
N
N
H
OH
3j
ppm
150
100
50
0
Current Data Parameters
NAME 1399-gxy-275
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070523
Time 18.37
INSTRUM av300
PROBHD 5 mm GNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 4932
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677512 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 200.500 ppm
F1 15131.28 Hz
F2P -5.500 ppm
F2 -415.07 Hz
PPMCM 10.30000 ppm/cm
HZCM 777.31787 Hz/cm

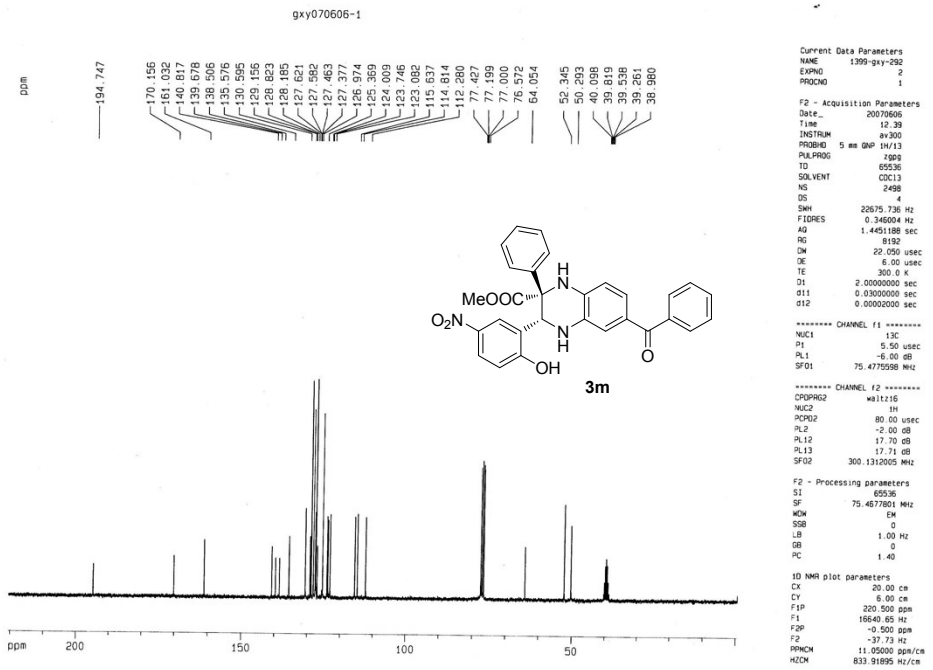

gxy-070525-2
ppm
8.99812
7.51505
7.50756
7.48809
7.37020
7.35513
7.33379
7.25937
7.22108
7.21356
7.19251
7.18447
6.94596
6.93869
6.80211
6.79064
6.75554
6.72678
6.60161
5.16924
4.04676
3.67366
1.54430
0.00118
Integral
0.9229
2.1079
3.0818
1.1059
1.0669
1.9869
1.1607
1.0735
1.0454
2.0000
0.8713
3.2071
ppm
10
8
6
4
2
0
Current Data Parameters
NAME 1399-gxy-278
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070525
Time 17.39
INSTRUM av300
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1024
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SF01 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.57500 ppm/cm
HZCM 172.57475 Hz/cm

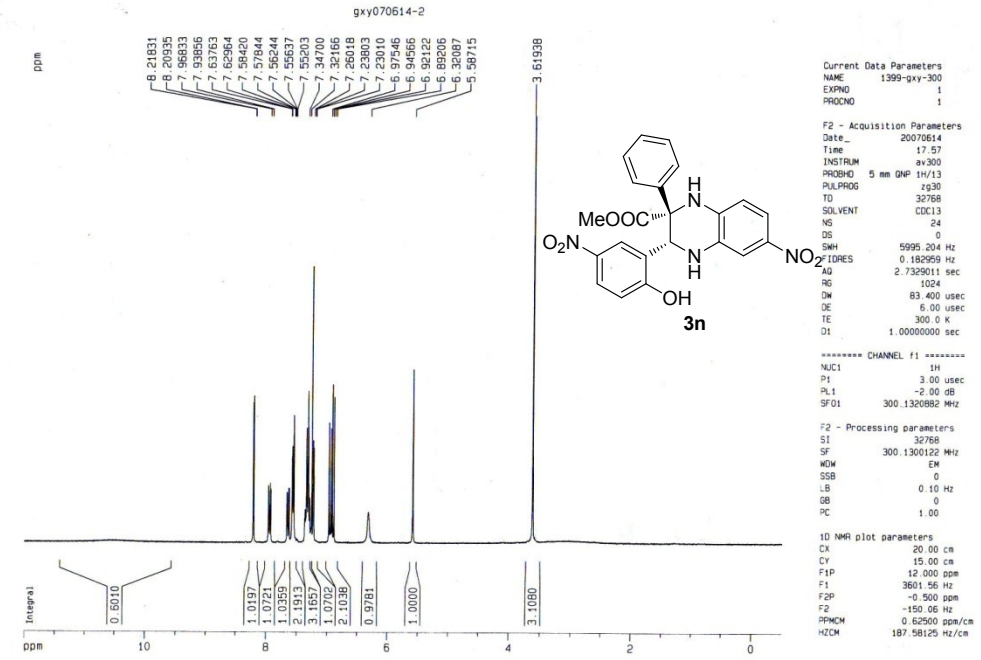

gxy-070525-2
ppm
171.739
154.982
139.291
132.127
131.950
131.705
129.553
128.992
128.669
126.461
126.244
121.528
120.052
119.633
117.718
115.475
111.555
77.423
77.205
77.000
76.577
65.042
58.802
53.124
MeOOC
Br
H
N
N
H
OH
3k
Current Data Parameters
NAME 1399-gxy-278
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070529
Time 12.32
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2846
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 7.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00025 ppm/cm
HZCM 754.69541 Hz/cm
ppm
150
100
50

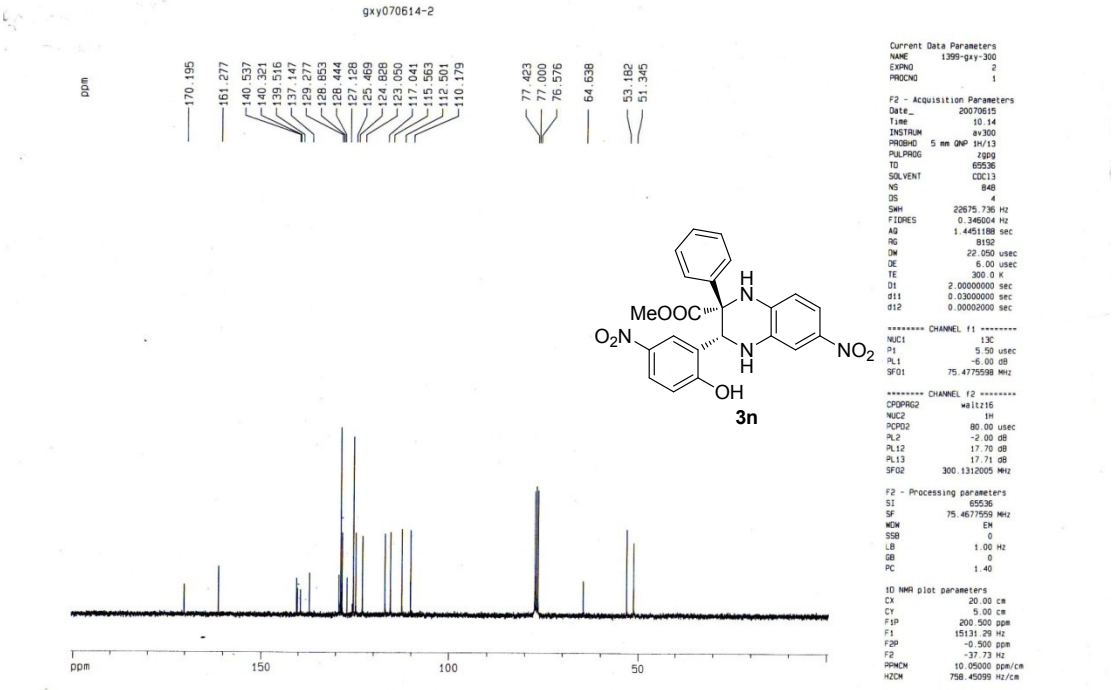

ppm
8.044
7.599
7.425
7.410
7.271
7.267
7.260
7.248
7.122
6.953
6.937
6.868
6.852
6.812
6.810
6.797
6.778
6.763
6.705
6.652
6.555
6.540
5.300
5.147
4.110
3.634
1.556
0.073
0.003
MeOOC
Cl
H
N
N
H
OH
3l
Integral
0.9379
1.0012
1.0530
2.7498
1.0532
1.0640
1.0000
2.1140
1.0609
1.0472
1.0350
1.2958
1.0002
0.9039
3.0781
ppm
9
8
7
6
5
4
3
2
1

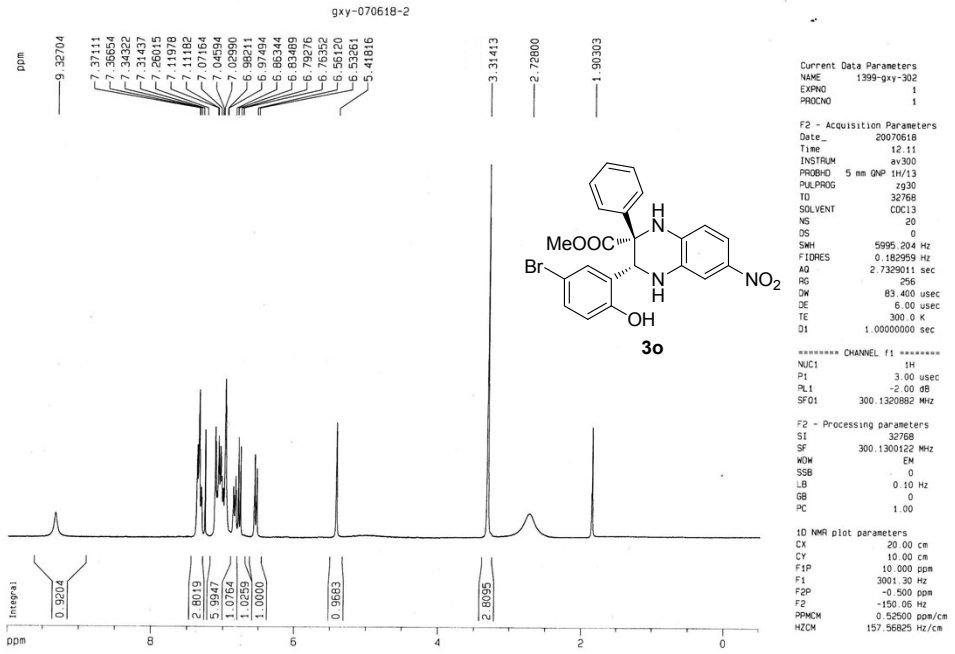

ppm
171.60
154.96
142.29
135.24
134.81
131.08
130.18
130.00
129.21
128.61
126.76
125.18
124.77
120.83
120.22
120.00
117.62
116.95
115.23
94.50
77.25
77.00
76.90
76.75
72.34
65.09
57.04
53.20
6.47
ppm
180
160
140
120
100
80
60
40
20

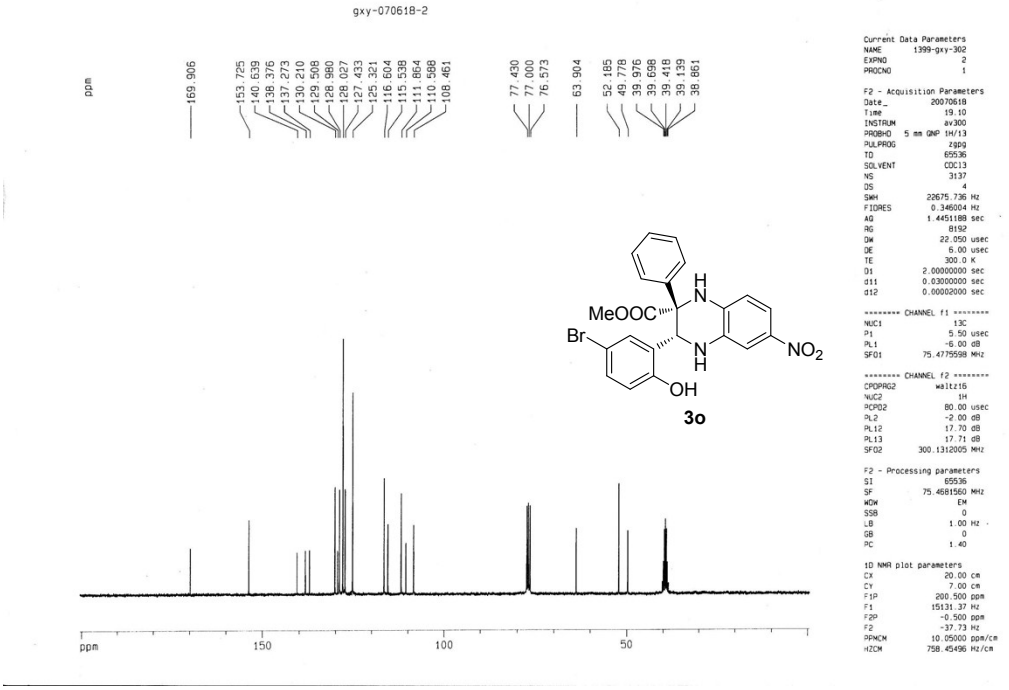

gxy070606-1
ppm
10.6363
8.1532
8.1446
7.7242
7.4638
7.4391
7.4154
7.2845
7.2625
7.2167
7.1915
7.1685
7.1520
7.1261
7.1091
6.9419
6.9380
6.8466
6.8191
6.7888
6.7526
6.5010
5.4385
4.6277
4.6185
3.3843
2.6366
1.8281
0.2067
MeOOC
O2N
OH
3m
Integral
0.9518
1.0000
1.0709
4.2965
6.9846
1.1167
1.0447
2.0852
1.0204
1.0233
0.9877
3.1102
ppm
10
8
6
4
2
0
Current Data Parameters
NAME 1399-gxy-292
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070606
Time 11.05
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 12.000 ppm
F1 3601.56 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.62500 ppm/cm
HZCM 187.58125 Hz/cm

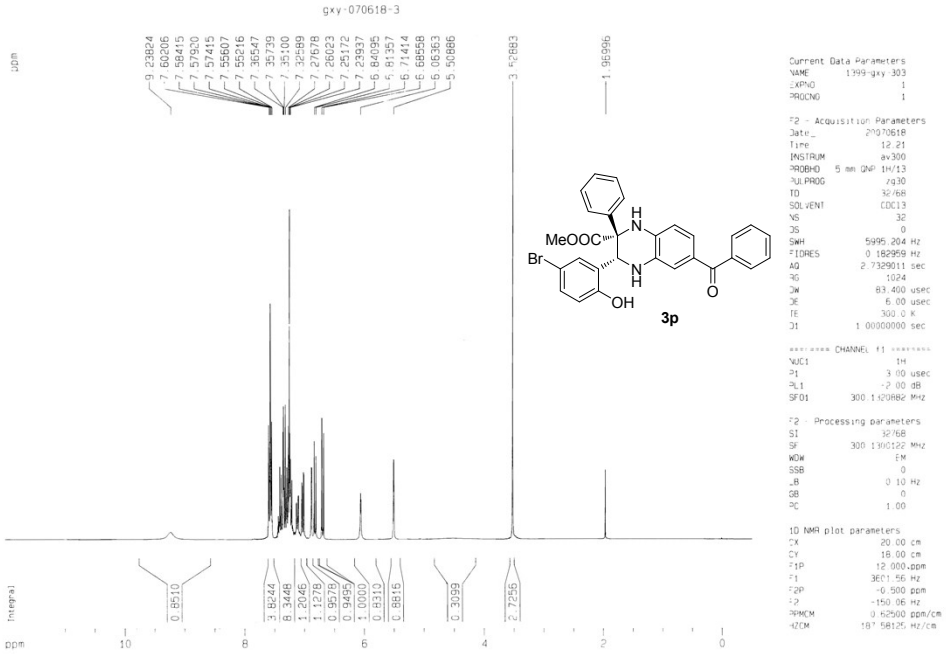

gxy070606-1
ppm
194.747
170.156
161.032
140.817
139.678
138.506
135.576
130.595
129.156
128.823
128.185
127.621
127.382
127.463
127.377
126.974
125.369
124.009
123.746
123.082
115.637
114.814
112.280
77.427
77.199
77.000
76.572
64.054
52.345
50.293
40.098
39.819
39.538
39.261
38.980
MeOOC
O2N
H
N
N
H
OH
O
3m
ppm
200
150
100
50
Current Data Parameters
NAME 1399-gxy-292
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070606
Time 12.39
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2498
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677801 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 220.500 ppm
F1 16640.65 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 11.05000 ppm/cm
HZCM 833.91895 Hz/cm

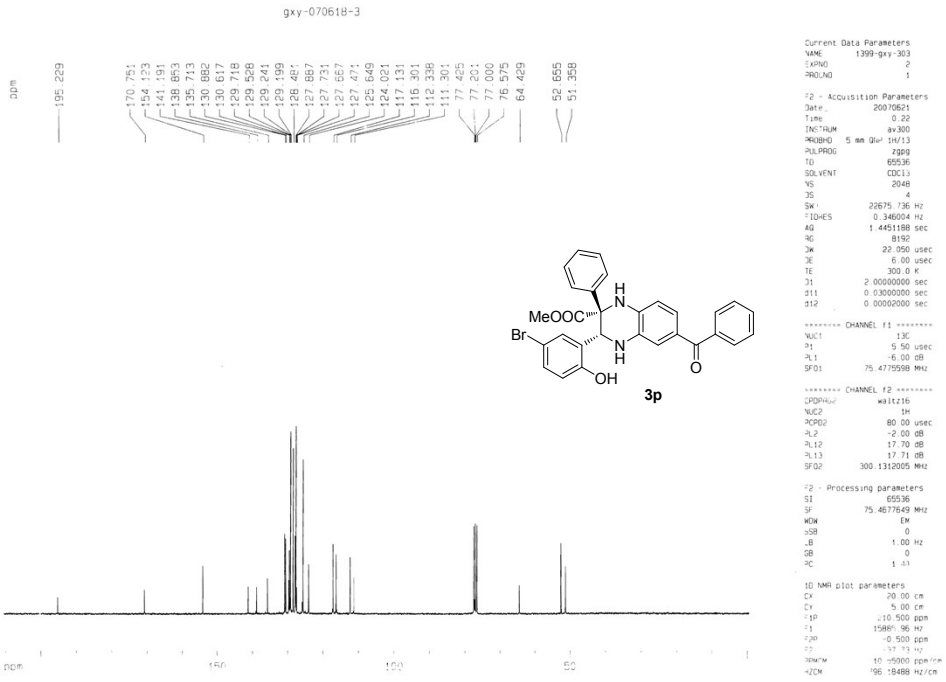

gxy070614-2
ppm
8.21831
8.20935
7.96833
7.93856
7.63763
7.62964
7.58420
7.57844
7.56244
7.55637
7.55203
7.34700
7.32166
7.26018
7.23803
7.23010
6.97546
6.94566
6.92122
6.89206
6.32087
5.58715
3.61938
MeOOC
O2N
NO2
H
N
N
H
OH
3n
Integral
0.6010
1.0197
1.0721
1.0359
2.1913
3.1657
1.0702
2.1038
0.9781
1.0000
3.1080
ppm
10
8
6
4
2
0
Current Data Parameters
NAME 1399-gxy-300
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070614
Time 17.57
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1024
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 12.000 ppm
F1 3601.56 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.62500 ppm/cm
HZCM 187.58125 Hz/cm

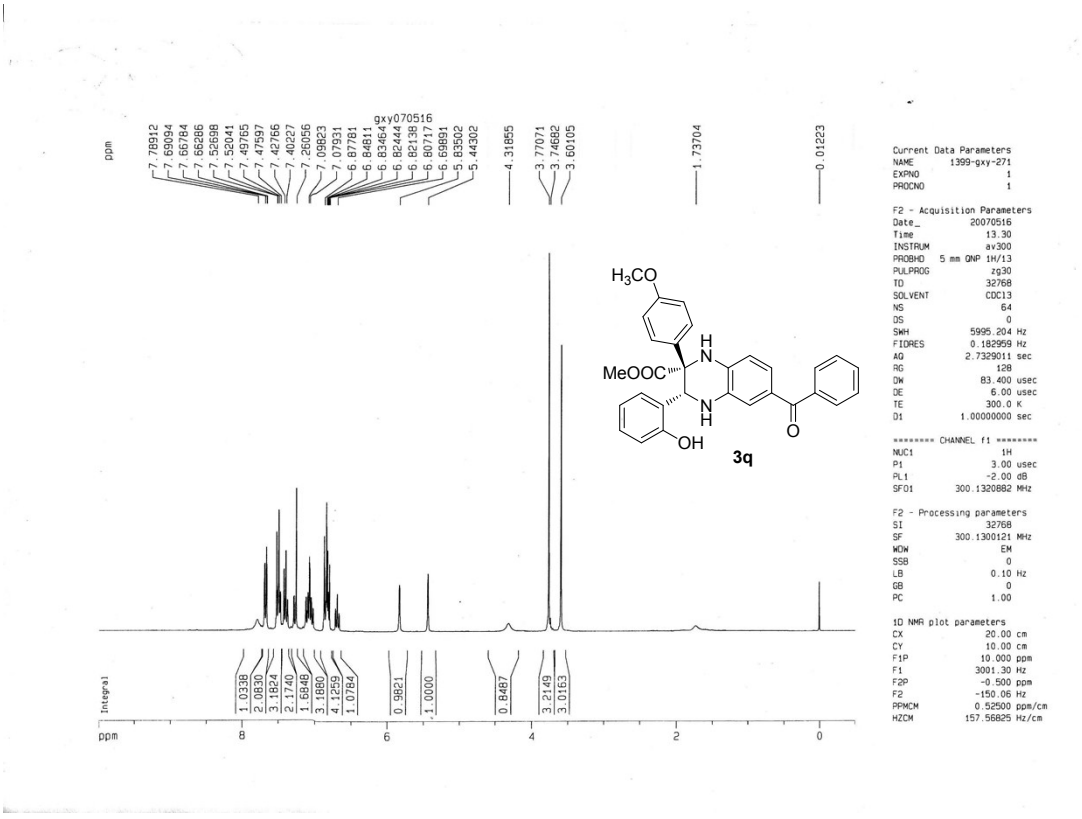

gxy070614-2
ppm
170.195
161.277
140.537
140.321
139.516
137.147
129.277
128.853
128.444
127.128
125.469
124.828
123.050
117.041
115.563
112.501
110.179
77.423
77.000
76.576
64.638
53.182
51.345
MeOOC
O2N
NO2
OH
3n
ppm
150
100
50
Current Data Parameters
NAME 1399-gxy-300
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070615
Time 10.14
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 848
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677559 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 200.500 ppm
F1 15131.29 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 10.05000 ppm/cm
HZCM 758.45099 Hz/cm

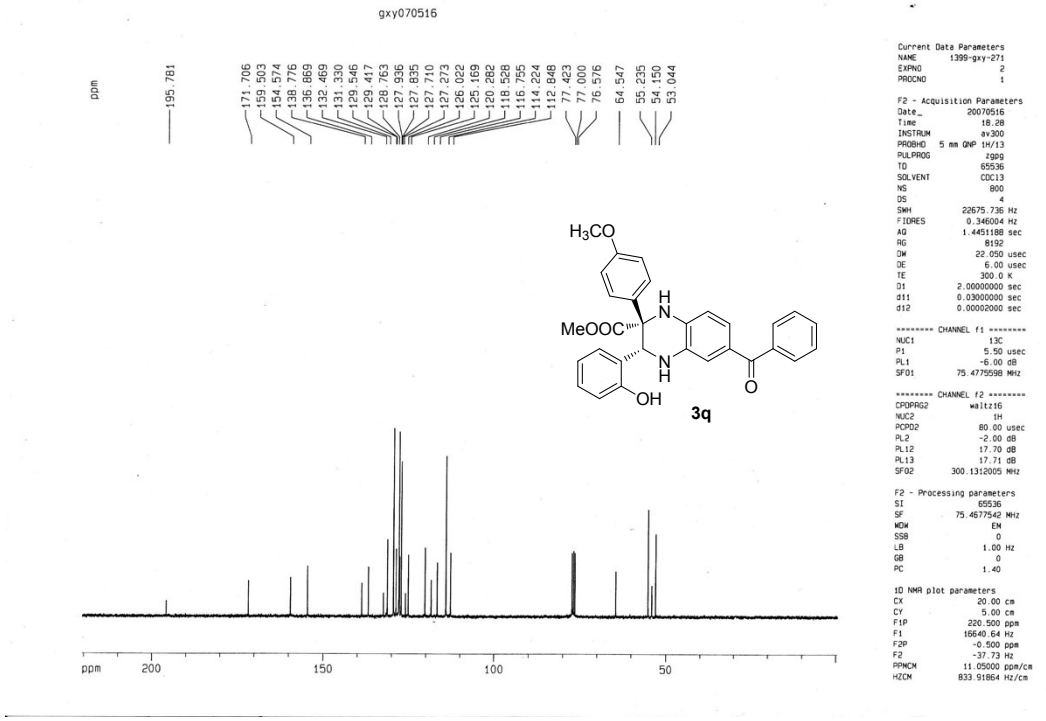

gxy-070618-2
ppm
9.32704
7.37111
7.36654
7.34322
7.31437
7.26015
7.11978
7.11182
7.07164
7.04594
7.02990
6.98211
6.97494
6.86344
6.83489
6.79276
6.76352
6.56120
6.53261
5.41816
3.31413
2.72800
1.90303
MeOOC
Br
NO2
OH
3o
Integral
0.9204
2.8019
5.9947
1.0764
1.0259
1.0000
0.9683
2.8095
ppm
8
6
4
2
0
Current Data Parameters
NAME 1399-gxy-302
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070618
Time 12.11
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 20
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 256
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SF01 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

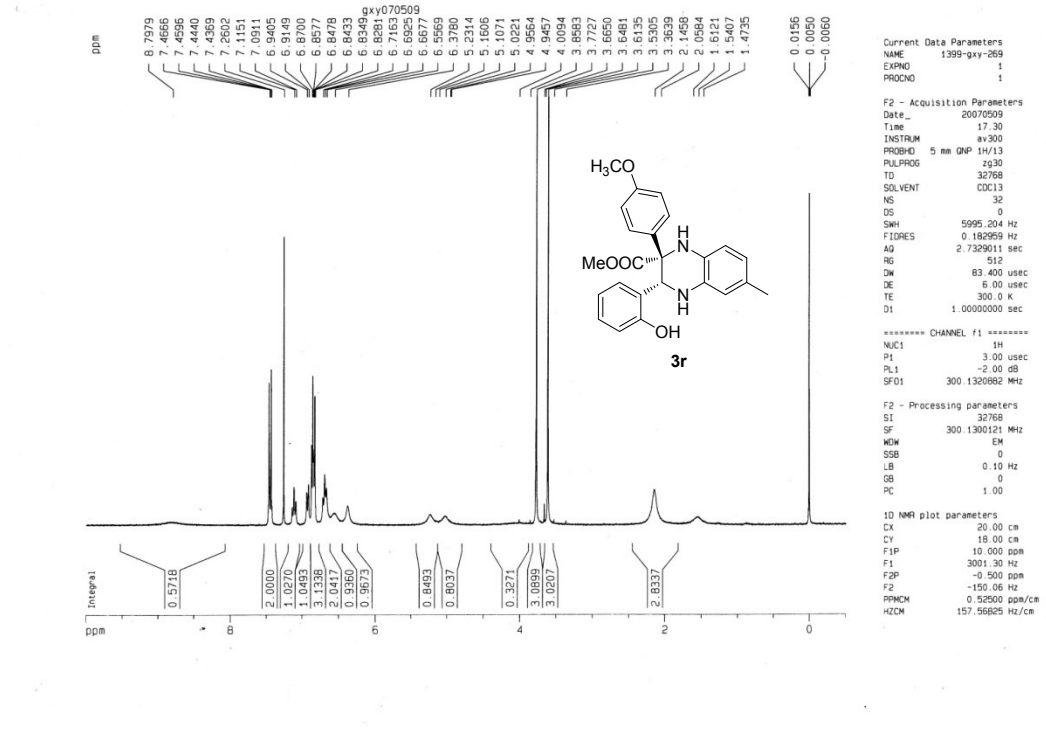

gxy-070618-2
ppm
169.906
153.725
140.639
138.376
137.273
130.210
129.508
128.980
128.027
127.433
125.321
116.604
115.538
111.864
110.988
108.461
77.430
77.000
76.573
63.904
52.185
49.778
39.976
39.698
39.418
39.139
38.861
MeOOC
Br
NO2
OH
3o
ppm
150
100
50
Current Data Parameters
NAME 1399-gxy-302
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070618
Time 19.10
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 3137
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4681560 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 7.00 cm
F1P 200.500 ppm
F1 15131.37 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 10.05000 ppm/cm
HZCM 758.45496 Hz/cm

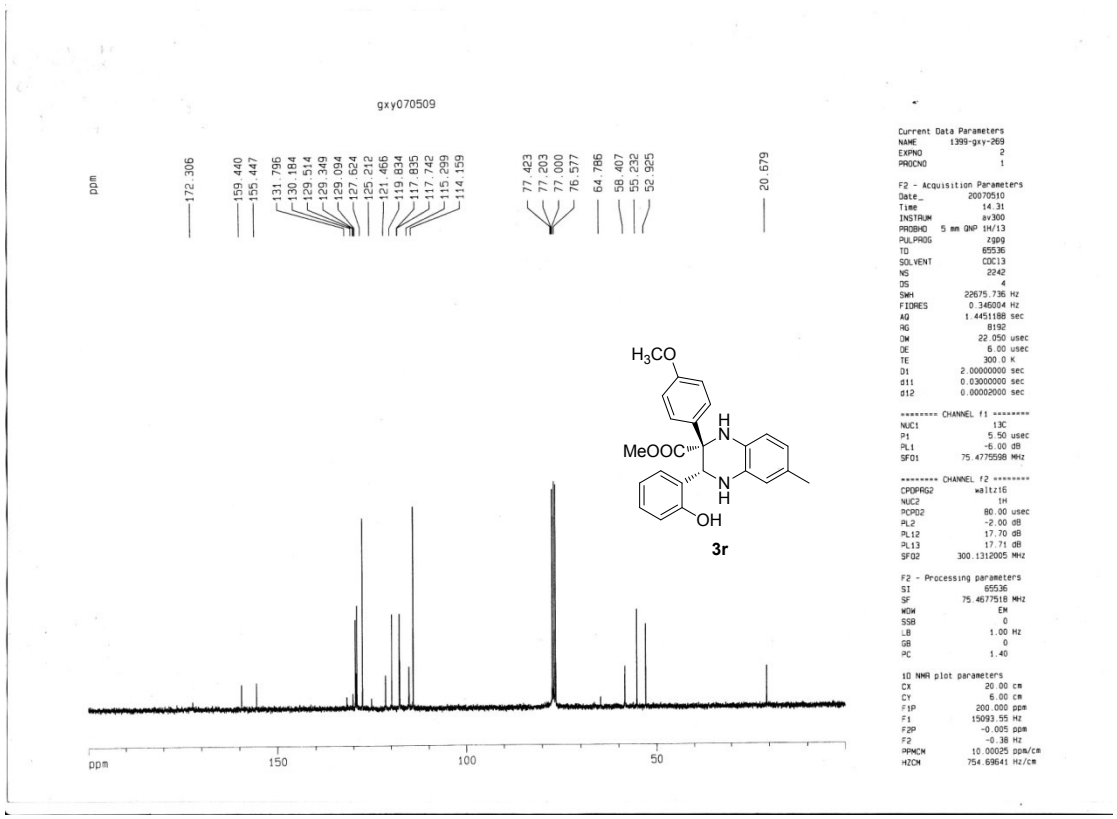

gxy-070618-3
ppm
9.23824
7.60206
7.58415
7.57920
7.57415
7.55607
7.55216
7.36547
7.35739
7.35100
7.32589
7.27678
7.26023
7.25172
7.23937
6.84095
6.81357
6.71414
6.68558
6.66363
6.50886
3.52883
1.96996
MeOOC
Br
OH
O
3p
Integral
0.8510
3.8244
8.3448
1.2046
1.1278
0.9579
0.9455
1.0000
0.8310
0.8815
0.3099
2.7256
ppm
10
8
6
4
2
0
Current Data Parameters
NAME 1399-gxy-303
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070618
Time 12.21
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 1024
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 18.00 cm
F1P 12.000 ppm
F1 3601.56 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.62500 ppm/cm
HZCM 187.58125 Hz/cm

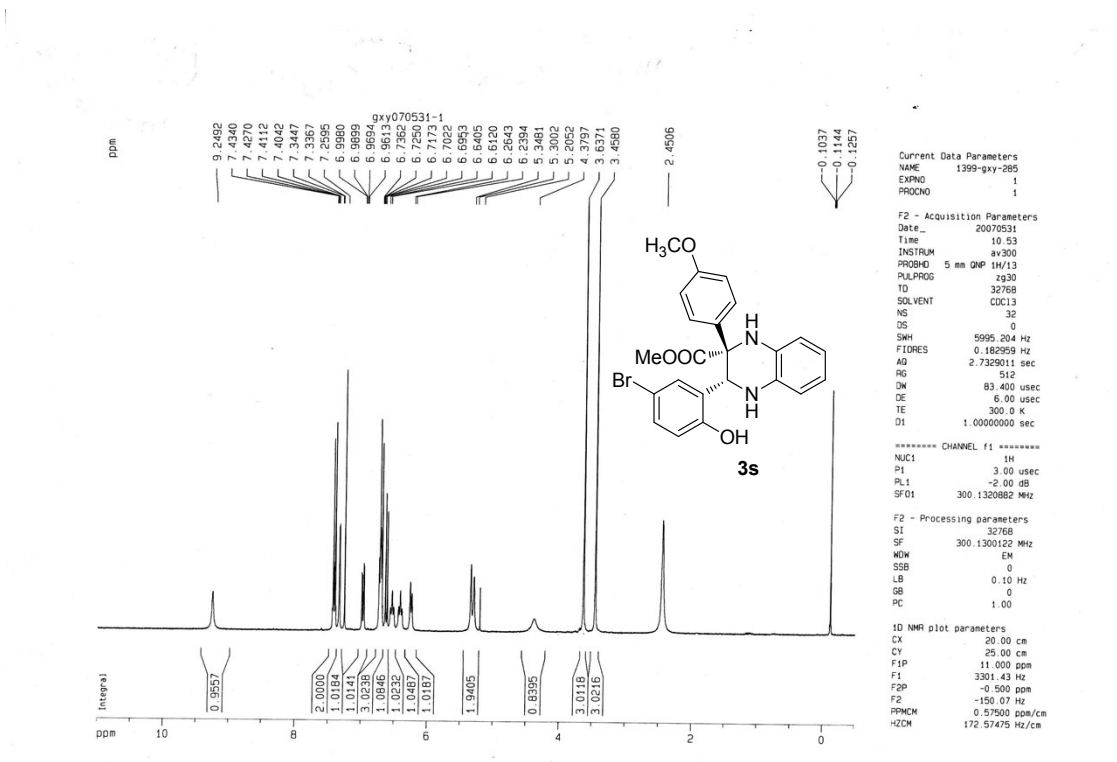

gxy-070618-3
ppm
195.229
170.751
154.123
141.191
138.853
135.713
130.882
130.617
129.718
129.528
129.241
129.199
128.481
127.867
127.731
127.667
127.471
125.649
124.021
117.131
116.301
112.338
111.301
77.425
77.201
77.000
76.575
64.429
52.655
51.358
MeOOC
Br
OH
N
H
O
3p
Current Data Parameters
NAME 1399-gxy-303
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070621
Time 0.22
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677649 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 210.500 ppm
F1 15885.96 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 10.95000 ppm/cm
HZCM 996.18488 Hz/cm


gxy070516
ppm
7.78912
7.69094
7.66784
7.66286
7.52698
7.52041
7.49765
7.47597
7.42766
7.40227
7.26056
7.09823
7.07931
6.87781
6.84611
6.83464
6.82444
6.82138
6.80717
6.69891
5.83502
5.44302
4.31855
3.77071
3.74682
3.60105
1.73704
0.01223
H3CO
MeOOC
OH
N
H
O
3q
Integral
1.0338
2.0830
3.1824
2.1740
1.6848
3.1860
4.1259
1.0784
0.9821
1.0000
0.8487
3.2149
3.0163
Current Data Parameters
NAME 1399-gxy-271
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070516
Time 13.30
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300121 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm


gxy070516
ppm
195.781
171.706
159.503
154.574
138.776
136.869
132.469
131.330
129.546
129.417
128.763
127.936
127.835
127.710
127.273
126.022
125.169
120.282
118.528
116.755
114.224
112.848
77.423
77.000
76.576
64.547
55.235
54.150
53.044
H3CO
MeOOC
H
N
N
H
OH
O
3q
ppm
200
150
100
50
Current Data Parameters
NAME 1399-gxy-271
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070516
Time 18.28
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 800
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677542 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 220.500 ppm
F1 16640.64 Hz
F2P -0.500 ppm
F2 -37.73 Hz
PPMCM 11.05000 ppm/cm
HZCM 833.91864 Hz/cm


gxy070509
ppm
8.7979
7.4666
7.4596
7.4440
7.4369
7.2602
7.1151
7.0911
6.9405
6.9149
6.8700
6.8577
6.8478
6.8433
6.8349
6.8281
6.7163
6.6925
6.6677
6.5569
6.3780
5.2314
5.1606
5.1071
5.0221
4.9564
4.9457
4.0094
3.8583
3.7727
3.6650
3.6481
3.6135
3.5305
3.3639
2.1458
2.0584
1.6121
1.5407
1.4735
0.0156
0.0050
-0.0060
H3CO
MeOOC
H
N
N
H
OH
3r
Integral
0.5718
2.0000
1.0270
1.0493
1.1336
2.0417
0.9360
0.9673
0.8493
0.8037
0.3271
3.0899
3.0207
2.8337
ppm
8
6
4
2
0
Current Data Parameters
NAME 1399-gxy-269
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070509
Time 17.30
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300121 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 18.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm


gxy070509
ppm
172.306
159.440
155.447
131.796
130.184
129.514
129.349
129.094
127.624
125.212
121.466
119.834
117.835
117.742
115.299
114.159
77.423
77.203
77.000
76.577
64.786
58.407
55.232
52.925
20.679
H3CO
MeOOC
H
N
N
H
OH
3r
ppm
150
100
50
Current Data Parameters
NAME 1399-gxy-269
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070510
Time 14.31
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2242
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677518 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -0.005 ppm
F2 -0.38 Hz
PPMCM 10.00025 ppm/cm
HZCM 754.69641 Hz/cm


gxy070531-1
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9.2492
7.4340
7.4270
7.4112
7.4042
7.3447
7.3367
7.2595
6.9980
6.9899
6.9694
6.9613
6.7362
6.7250
6.7173
6.7022
6.6993
6.6405
6.6120
6.2643
6.2394
5.3481
5.3002
5.2052
4.3797
3.6371
3.4580
2.4506
0.1037
0.1144
0.1257
H3CO
MeOOC
Br
H
N
N
H
OH
3s
Integral
0.9557
2.0000
1.0184
1.0141
3.0238
1.0846
1.0232
1.0487
1.0187
1.9405
0.8395
3.0118
3.0216
ppm
10
8
6
4
2
0
Current Data Parameters
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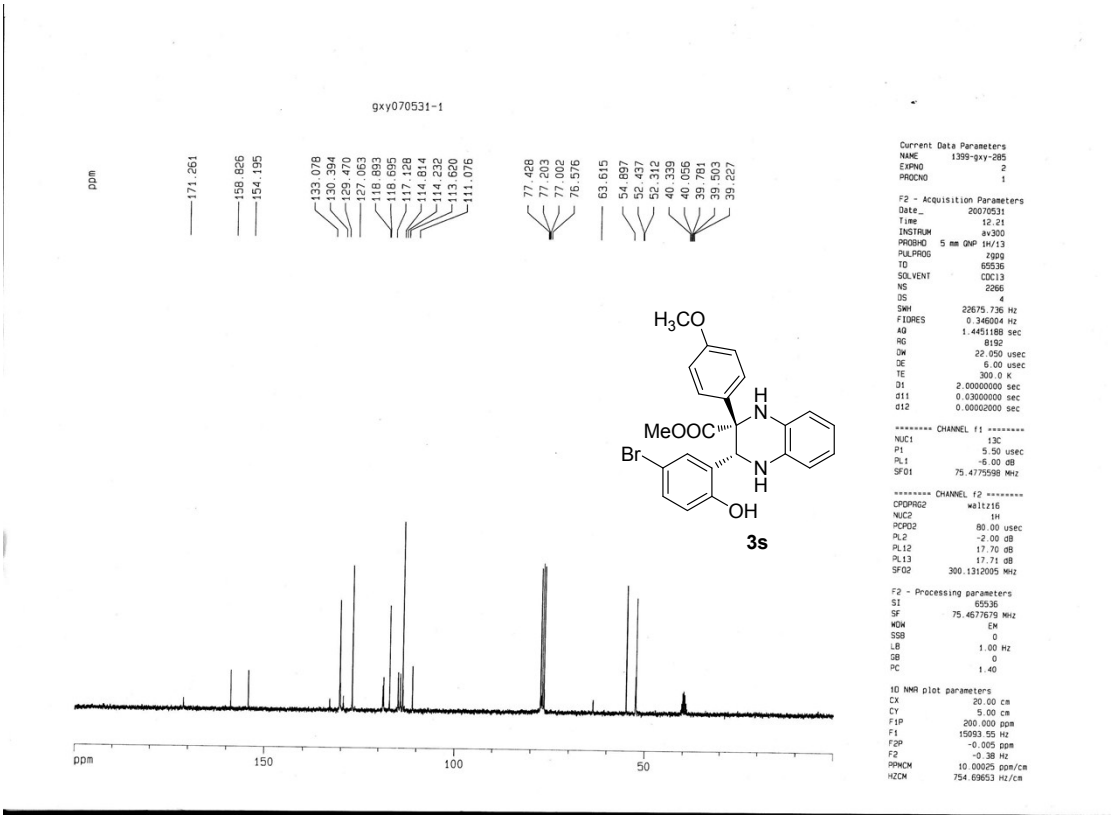
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070531
Time 10.53
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 25.00 cm
F1P 11.000 ppm
F1 3301.43 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.57500 ppm/cm
HZCM 172.57475 Hz/cm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130.394
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127.063
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118.695
117.128
114.814
114.232
113.620
111.076
77.428
77.203
77.002
76.576
63.615
54.897
52.437
52.312
40.339
40.056
39.781
39.503
39.227
H3CO
MeOOC
Br
OH
H
N
N
H
3s
ppm
150
100
50
Current Data Parameters
NAME 1399-gxy-285
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070531
Time 12.21
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 2266
DS 4
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.445188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SF01 75.4775598 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
PL13 17.71 dB
SF02 300.1312005 MHz
F2 - Processing parameters
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SF 75.4677679 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
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F1 15093.55 Hz
F2P -0.005 ppm
F2 -0.38 Hz
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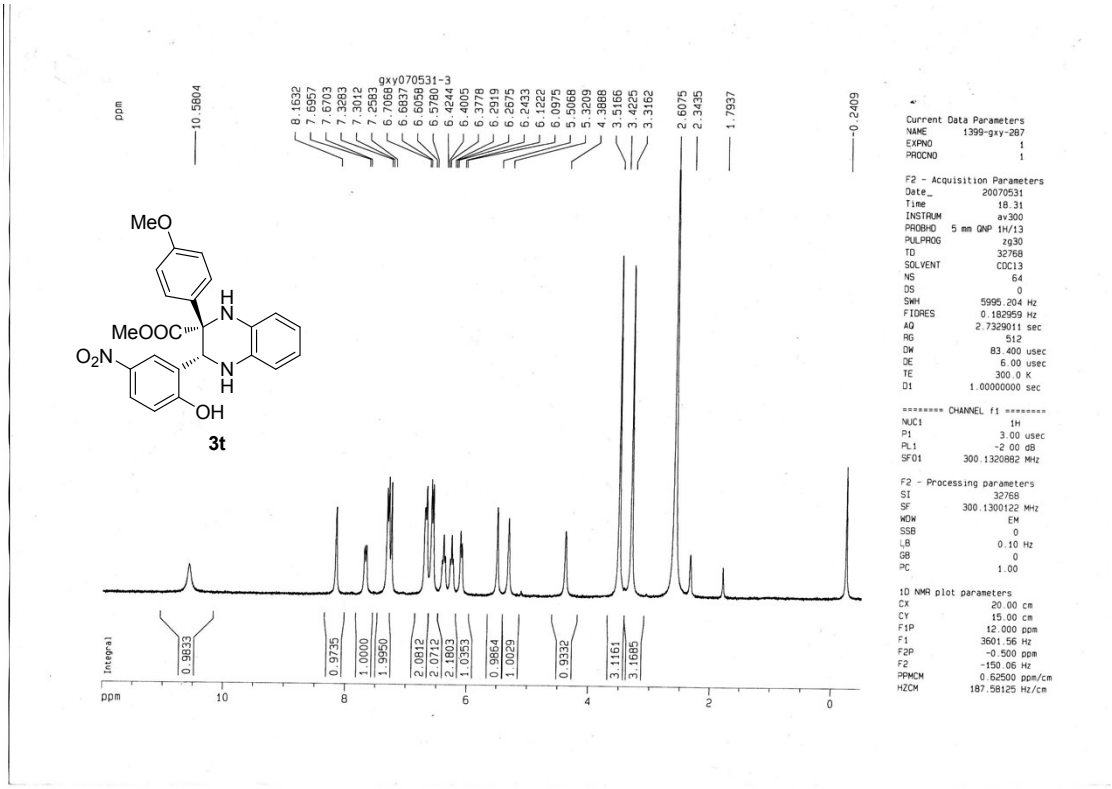
HZCM 754.69653 Hz/cm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6.6837
6.6056
6.5780
6.4244
6.4005
6.3778
6.2919
6.2675
6.2433
6.1222
6.0975
5.5068
5.3209
4.3888
3.5166
3.4225
3.3162
2.6075
2.3435
1.7937
0.2409
MeO
MeOOC
O2N
H
N
N
H
OH
3t
Integral
0.9833
0.9735
1.0000
1.9950
2.0812
2.0712
2.1803
1.0353
0.9864
1.0029
0.9332
3.1161
3.1685
ppm
10
8
6
4
2
0
Current Data Parameters
NAME 1399-gxy-287
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070531
Time 18.31
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SF01 300.1320882 MHz
F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 12.000 ppm
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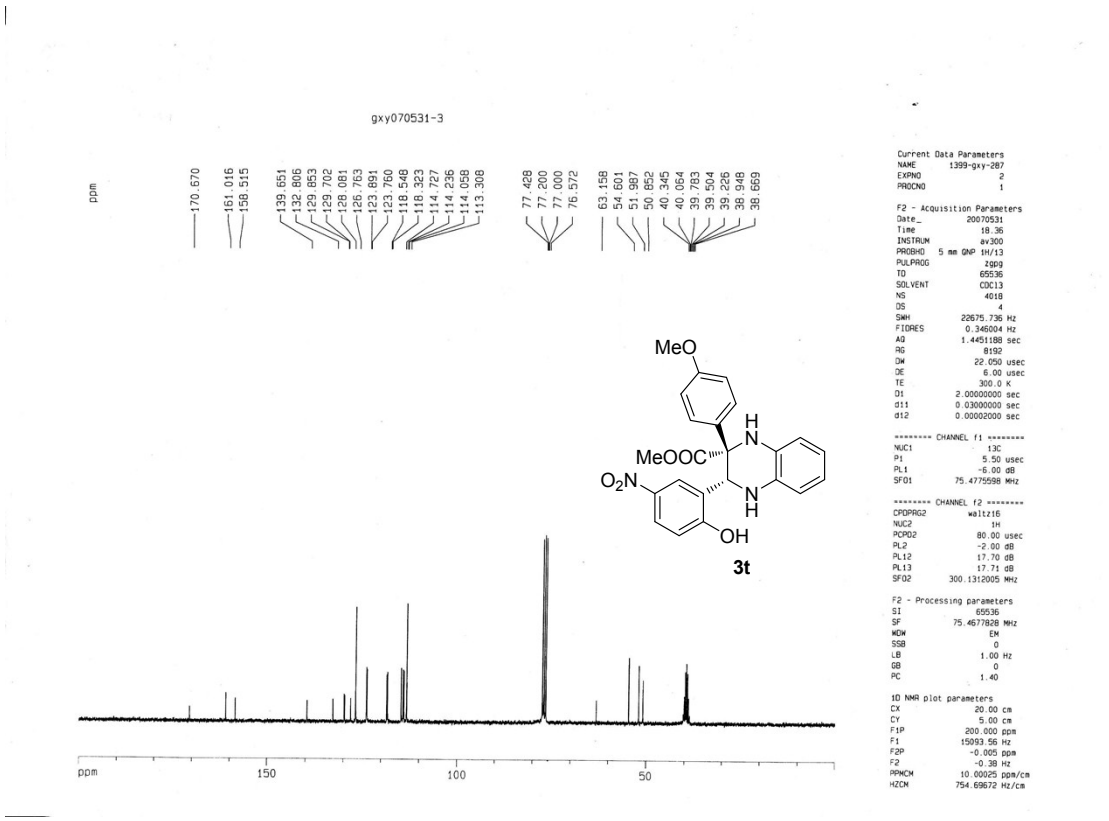
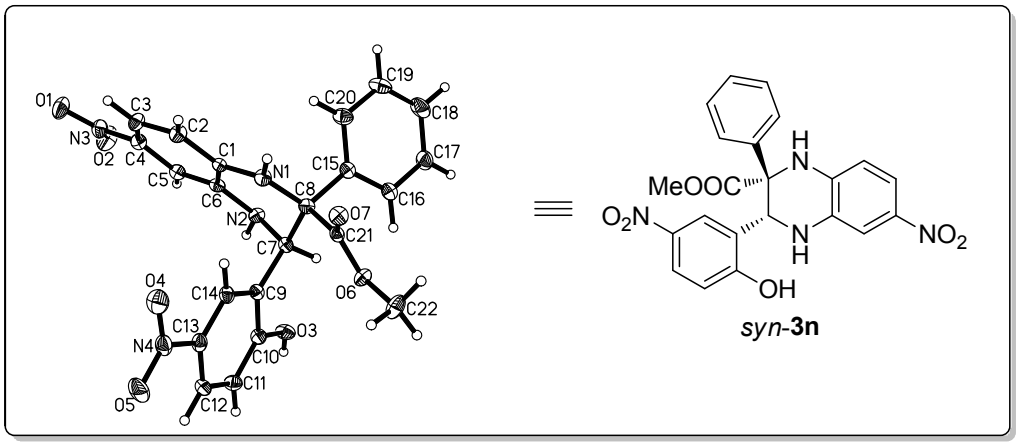
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.62500 ppm/cm
HZCM 187.98125 Hz/cm



6. X-ray diffraction parameters and data for *syn*-3n (CCDC 1898566)



X-ray data of *syn*-3n

Empirical formula	$C_{26}H_{26}N_4O_9$
Formula weight	538.51
Temperature/K	153 (2)
Crystal system	Triclinic
Space group	$P\bar{1}$

a/Å	7.9231 (9)
b/Å	12.3032 (12)
c/Å	14.0647 (13)
α/°	97.079 (3)
β/°	99.017 (3)
γ/°	105.178 (3)
Volume/Å^3	1287.3 (2)
Z	2
ρ_{calc}g/cm^3	1.389
μ/mm^{-1}	0.107
F(000)	564
Crystal size/mm^3	0.43 × 0.30 × 0.16
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.08 to 54.96
Index ranges	-10≤ h ≤ 10, -15 ≤ k ≤ 15, -17 ≤ l ≤ 18
Reflections collected	23115
Independent reflections	5851 [R_{int} = 0.0338, R_{sigma} = N/A]
Data/restraints/parameters	5851/0/368
Goodness-of-fit on F^2	1.053
Final R indexes [I>=2σ (I)]	R_1 = 0.1088, wR_2 = 0.3702
Final R indexes [all data]	R_1 = 0.1265, wR_2 = 0.3790
Largest diff. peak/hole/ e Å^{-3}	0.63/-0.50