Organocatalytic Michael addition of unprotected 3-substituted oxindoles to nitroolefins

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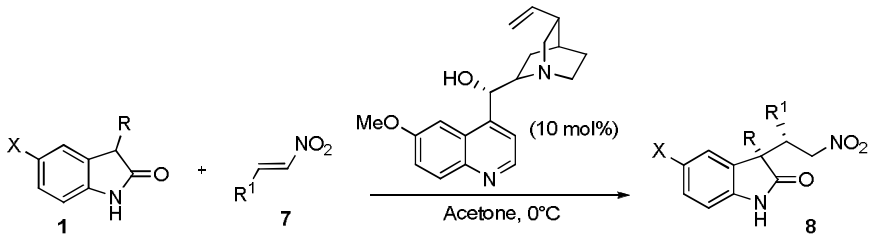
General: Reactions were monitored by thin layer chromatography using UV light to visualize the course of reaction. Purification of reaction products were carried out by flash chromatography on silica gel. Chemical yields refer to pure isolated substances. ^{1}H and ^{13}C NMR spectra were obtained using a Bruker DPX-300 spectrometer. Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard. The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, h = heptet, m = multiplet, br = broad.

All reactions were run under air except noted. Anhydrous halogenated solvents were prepared by first distilled over P_2O_5 and then from CaH_2. Anhydrous acetone was distilled over anhydrous $CaSO_4$ and stored over MS 4Å. Cinchonidine and Quinidine were purchased from Aldrich, and Bifunctional quinidine derived thiourea catalyst **10**[1] was prepared using a literature method. 3-substituted oxindoles **1**[2] and nitroolefins **7**[3] were prepared according to literature reports.

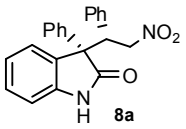
General procedure for the Michael Addition of oxindole 1 to nitroolefin 7.



To a 5 mL vial were added quinidine **QD** (0.025 mmol), 3-substituted oxindoles **1** (0.25 mmol) and 2.5 mL of anhydrous acetone, followed by **7** (0.275 mmol). The resulting mixture was stirred at 0°C till almost full conversion of **1** by TLC analysis. To determine the diastereoselectivity, 0.5 mL of crude mixture was taken for ^{1}H NMR analysis. And then the sample for analysis and the rest of the reaction mixture were

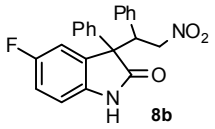
[1] B. Vakulya, S. Varga, A. Csámpai and T. Soós, *Org. Lett.,* 2005, **7**, 1967.
[2] a) P. Galzerano, G. Bencivenni, F. Pesciaioli, A. Mazzanti, B. Giannichi, L. Sambri, G. Bartoli and P. Melchiorre, *Chem. Eur. J.,* 2009, **15**, 7846; b) G. Lakshmaiah, T. Kawabata, M. Shang and K. Fuji, *J. Org. Chem.,* 1999, **64**, 1699; c) Y. Hamashima, T. Suzuki, H. Takano, Y. Shimura and M. Sodeoka, *J. Am. Chem. Soc.,* 2005, **127**, 10164.
[3] D. Lucet, S. Sabelle, O. Kostelitz, T. l. Gall and C. Mioskowski, *Eur. J. Org. Chem.,* 1999, 2583.

recombined and concentrated under reduced pressure, and then directly subjected to column chromatography to afford the desired product **8**, using CH_2Cl_2 as eluent. Of the 14 examples of product **8a-n** we obtained, the major diastereomer of nine products **8** could be obtained as pure compound after recrystallization, using a mixed solvent of petroleum and CH_2Cl_2.



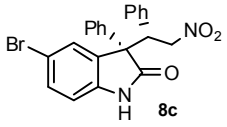
8a

Product **8a** was obtained in 96% yield as white powder, and the pure major diastereomer was obtained after recrystallization: ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.81 (s, 1H), 7.64-7.62 (m, 2H), 7.43-7.32 (m, 5H), 7.26-7.21 (m, 1H), 7.09-6.96 (m, 3H), 6.84-6.82 (m, 2H), 6.73 (d, J = 7.5 Hz, 1H), 5.03-4.95 (m, 1H), 4.83-4.70 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 177.3, 141.6, 135.7, 133.4, 129.4, 129.2, 128.2, 128.4, 128.1, 128.0, 127.7, 127.6, 126.4, 122.4, 110.7, 76.2, 60.0, 50.4; IR (KBr): 3191, 3066, 3032, 1702, 1618, 1554, 1472, 1375, 1233, 699; MS (EI): 358 (M^+, 5), 359 [$(M+H)^+$, 1], 208 (100), 91 (30), 209 (28), 77 (18), 43 (17), 180 (14), 55 (12), 104 (11); HRMS (EI): Exact mass calcd for $C_{22}H_{18}N_2O_3$: 358.1317, Found: 358.1319.



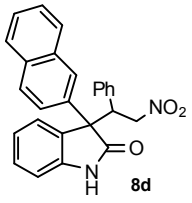
8b

Product **8b** was obtained in 87% yield as white powder. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.39-8.28 (m, 0.21H), 7.78-7.73 (m, 0.79H), 7.62-7.55 (m, 2H), 7.46-7.37 (m, 3H), 7.15-6.90 (m, 5H), 6.89-6.85 (m, 2H), 6.75-6.67 (m, 0.79H), 6.55-6.52 (m, 0.21H), 5.46-5.37 (m, 0.21H), 5.01-4.93 (m, 0.79H), 4.84-4.62 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.2, 178.0, 160.0, 157.0, 137.8, 137.2, 137.1, 135.3, 135.0, 133.5, 133.4, 133.2, 133.1, 133.0, 129.6, 129.2, 128.8, 128.7, 128.6, 128.4, 128.3, 128.2, 128.1, 128.0, 127.3, 126.4, 126.3, 116.2, 116.0, 115.0, 114.7, 114.3, 114.0, 112.6, 112.5, 112.3, 111.5, 111.4, 110.8, 110.7, 75.8, 75.4, 60.8, 60.6, 50.0, 49.8, 49.7; IR (KBr): 3389, 3231, 2922, 2866, 2362, 1723, 1557, 1488, 1461, 1375; MS (EI): 376 (M^+, 4), 377 [$(M+H)^+$, 1], 226 (100), 227 (43), 198 (23), 104 (11), 170 (8), 77 (7), 228 (6), 199 (6); HRMS (EI): Exact mass calcd for $C_{22}H_{17}N_2O_3F$: 376.1223, Found: 376.1223.



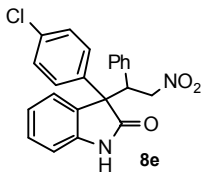
8c

Product **8c** was obtained in 95% yield as white powder, and the pure major diastereomer was obtained after recrystallization: ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.61-7.55 (m, 3H), 7.49-7.36 (m, 5H), 7.15-7.03 (m, 3H), 6.89-6.86 (m, 2H), 6.62 (d, *J* = 8.7 Hz, 1H), 5.01-4.93 (m, 1H), 4.83-4.69 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 176.6, 140.6, 134.9, 133.1, 132.4, 130.1, 129.4, 129.3, 128.9, 128.7, 128.4, 128.2, 127.4, 115.0, 112.0, 75.9, 60.2, 50.2; IR (KBr): 3409, 3243, 2923, 2852, 2361, 1721, 1617, 1556, 1476, 698; MS (EI): 436 (M^+, 4), 288 (100), 286 (93), 207 (52), 287 (42), 289 (42), 179 (29), 178 (27), 104 (24); HRMS (EI): Exact mass calcd for $C_{22}H_{17}N_2O_3Br$: 436.0423, Found: 436.0420.



8d

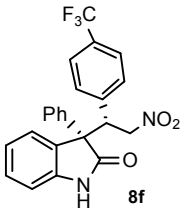
Product **8d** was obtained in 92% yield as white powder. ^{1}H NMR (300 MHz, $CDCl_3$): 7.95-7.75 (m, 5H), 7.51-7.26 (m, 6H), 7.21-6.82 (m, 5H), 6.70-6.80 (m, 0.83H), 6.65-6.55 (m, 0.17H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 177.5, 141.7, 133.4, 133.1, 133.0, 132.9, 129.5, 129.2, 128.9, 128.2, 128.0, 127.9, 127.7, 127.5, 127.3, 126.8, 126.7, 126.5, 126.4, 124.6, 122.7, 122.5, 110.8, 76.2, 60.1, 50.3; IR (KBr): 3413, 3060, 2362, 1712, 1619, 1554, 1473, 1372, 749, 700; MS (EI): 408 (M^+, 6), 258 (100), 208 (61), 43 (49), 57 (40), 55 (37), 259 (31). 41 (30), 91 (29); HRMS (EI): Exact mass calcd for $C_{26}H_{20}N_2O_3$: 408.1474, Found: 408.1479.



8e

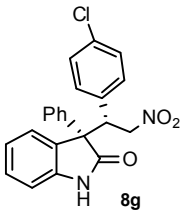
Product **8e** was obtained in 95% yield as white powder. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95-7.85 (m, 0.17H), 7.59-7.55 (m, 1.83H), 7.40-7.24 (m, 5H), 7.17-7.06 (m, 4H), 6.85-6.82 (m, 2H), 6.75-6.72 (m, 0.83H), 6.62-6.60 (m, 0.17H), 5.40-5.30 (m, 0.17H), 5.02-4.93 (m, 0.83H), 4.77-4.59 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 176.7, 141.5, 134.7, 134.2, 133.2, 129.7, 129.3, 129.0, 128.9, 128.3, 128.1, 127.2, 126.3, 122.7, 110.8, 76.1, 59.5, 50.6; IR (KBr): 3422, 2361, 1710, 1619, 1553, 1491, 1472, 1377, 1097, 1014; MS (EI): 392 (M^+, 5), 242 (100), 207 (34), 243 (32), 244 (29), 43 (23), 104 (21), 57 (18), 41 (17); HRMS (EI): Exact mass calcd for $C_{22}H_{17}N_2O_3Cl$: 392.0928, Found:

392.0928.



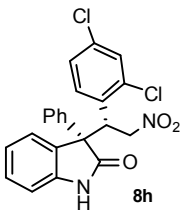
8f

Product **8f** was obtained in 95% yield as white powder, and the pure major diastereomer was obtained after recrystallization: ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.65-7.62 (m, 2H), 7.45-7.26 (m, 8H), 7.15 (s, 1H), 7.02-6.99 (m, 2H), 6.76-6.74 (m, 1H), 5.04-4.87 (m, 2H), 4.77-4.73 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 176.8, 141.4, 137.8, 135.2, 129.8, 129.4, 129.3, 128.7, 127.5, 127.1, 126.4, 125.0, 124.9, 122.7, 110.9, 75.9, 59.6, 50.1; IR (KBr): 3145, 3085, 3037, 2894, 2838, 2361, 1707, 1620, 1594, 1473; MS (EI): 426 (M^+, 1), 208 (100), 209 (21), 180 (19), 57 (15), 43 (15), 149 (10), 71 (10), 41 (9); HRMS (EI): Exact mass calcd for $C_{23}H_{17}N_2O_3F_3$: 426.1191, Found: 426.1190.



8g

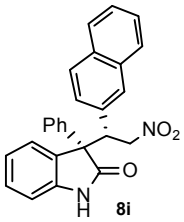
Product **8g** was obtained in 92% yield as white powder, and the pure major diastereomer was obtained after recrystallization: ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.64-7.61 (m, 2H), 7.45-7.34 (m, 5H), 7.28-7.23 (m, 2H), 7.05-7.02 (m, 2H), 6.82-6.75 (m, 3H), 4.98-4.90 (m, 1H), 4.83-4.69 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 176.9, 141.5, 135.4, 134.2, 132.1, 130.3, 129.7, 129.3, 128.6, 128.3, 127.5, 127.3, 126.4, 122.6, 110.8, 76.0, 59.8, 49.8; IR (KBr): 3395, 3254, 3060, 2362, 1721, 1700, 1619, 1551, 1473, 1375; MS (EI): 392 (M^+, 1), 208 (100), 209 (26), 180 (20), 152 (10), 77 (9), 43 (8), 138 (7), 153 (7); HRMS (EI): Exact mass calcd for $C_{22}H_{17}N_2O_3Cl$: 392.0928, Found: 392.0931.



8h

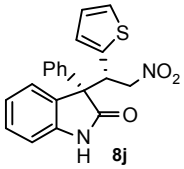
Product **8h** was obtained in 85% yield as white powder, and the pure major diastereomer was obtained after recrystallization: ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.72-7.65 (m, 3H), 7.44-7.24 (m, 7H), 6.88-6.83 (m, 2H), 6.33 (d, J = 8.7 Hz, 1H), 5.56-5.49 (m, 1H), 4.78 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 176.4, 141.6, 137.2, 135.3, 134.6, 131.2, 120.0, 129.9, 129.1, 128.6, 127.7, 127.4, 126.7, 126.5, 122.7, 110.9,

76.6, 58.9, 44.6; IR (KBr): 3395, 3141, 3087, 3035, 2893, 2841, 2361, 1712, 1620, 1552, 1473; MS (EI): 426 (M^+, 2), 208 (100), 209 (23), 43 (20), 180 (17), 91 (16), 59 (15), 69 (14), 74 (14); HRMS (EI): Exact mass calcd for $C_{22}H_{16}N_2O_3Cl_2$: 426.0538, Found: 426.0539.



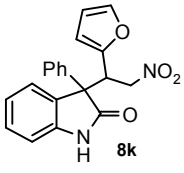
8i

Product **8i** was obtained in 92% yield as white powder, and the pure major diastereomer was obtained after recrystallization: ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.71-7.69 (m, 3H), 7.59-7.30 (m, 9H), 6.95-6.87 (m, 2H), 6.64-6.61 (m, 1H), 5.15-4.97 (m, 2H), 4.93-4.72 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 176.5, 141.5, 135.6, 132.8, 132.7, 131.1, 129.4, 129.2, 128.9, 128.8, 128.4, 127.7, 127.6, 127.3, 126.5, 126.2, 126.1, 125.9, 122.4, 110.5, 76.6, 59.9, 50.6; IR (KBr): 3387, 3143, 3059, 3030, 2361, 1705, 1619, 1548, 1471, 1377; MS (EI): 408 (M^+, 2), 208 (100), 209 (76), 154 (40), 180 (32), 152 (30), 153 (22), 77 (11), 57 (11); HRMS (EI): Exact mass calcd for $C_{26}H_{20}N_2O_3$: 408.1474, Found: 408.1471.



8j

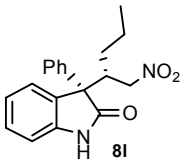
Product **8j** was obtained in 95% yield as white powder, and the pure major diastereomer was obtained after recrystallization: ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.63-7.61 (m, 2H), 7.43-7.24 (m, 7H), 7.03-7.01 (m, 1H), 6.83 (d, J = 7.5 Hz, 1H), 6.75-6.74 (m, 2H), 5.21-5.16 (m, 1H), 4.91-4.83 (m, 1H), 4.73-4.68 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 177.0, 142.1, 136.1, 135.4, 129.7, 129.2, 128.5, 128.4, 127.6, 127.4, 126.6, 126.2, 125.9, 122.6, 110.7, 78.0, 59.8, 46.5; IR (KBr): 3248, 3063, 3028, 2960, 2361, 1715, 1681, 1619, 1551, 1471; MS (EI): 364 (M^+, 0.3), 365 [$(M+H)^+$, 0.2], 208 (100), 209 (53), 180 (23), 110 (13), 152 (12), 77 (8), 210 (7), 181 (6); HRMS (EI): Exact mass calcd for $C_{20}H_{16}N_2O_3S$: 364.0882, Found: 364.0886.



8k

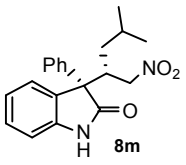
Product **8k** was obtained in 75% yield as white powder. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.92-7.50 (m, 3H), 7.35-7.15 (m, 6H), 7.08-7.05 (m, 1H), 6.84-6.80 (m, 1H), 6.10-6.09 (m, 1H), 5.98-5.87

(m, 1H), 5.16-4. 4.85 (m, 2H), 4.69-4.40 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.0, 178.2, 148.6, 148.4, 142.4, 142.2, 141.3, 140.1, 137.0, 135.3, 129.8, 129.3, 129.2, 129.1, 128.8, 128.5, 128.1, 128.0, 127.4, 126.7, 126.2, 125.4, 122.8, 122.5, 110.7, 110.4, 110.3, 110.2, 109.4, 108.8, 76.6, 74.6, 74.3, 59.3, 59.1, 44.3, 44.0; IR (KBr): 3399, 3234, 3063, 2922, 2361, 1715, 1681, 1620, 1554, 1473; MS (EI): 348 (M^+, 0.3), 208 (100), 209 (39), 180 (18), 152 (10), 94 (9), 181 (6), 77 (5), 153 (5); HRMS (EI): Exact mass calcd for $C_{20}H_{16}N_2O_4$: 348.1110, Found: 348.1109.



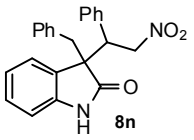
8l

Product **8l** was obtained in 95% yield as white powder, and the pure major diastereomer was obtained after recrystallization: ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.49-8.44 (s, br, 1H), 7.48-7.45 (m, 2H), 7.37-7.29 (m, 4H), 7.19-7.12 (m, 2H), 7.02-6.99 (m, 1H), 4.47-4.36 (m, 2H), 3.75-3.69 (m, 1H), 1.33-1.03 (m, 4H), 0.79-0.74 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.2, 141.3, 137.1, 129.1, 129.0, 128.8, 128.3, 127.4, 125.5, 122.8, 110.9, 77.8, 59.9, 43.5, 32.8, 20.4, 14.1; IR (KBr): 3422, 3207, 2961, 2930, 2872, 2361, 2336, 1707, 1618, 1554, 1471; MS (EI): 324 (M^+, 10), 325 [$(M+H)^+$, 2], 208 (100), 209 (21), 180 (15), 152 (7), 181 (4), 77 (4); HRMS (EI): Exact mass calcd for $C_{19}H_{20}N_2O_3$: 324.1474, Found: 324.1472.



8m

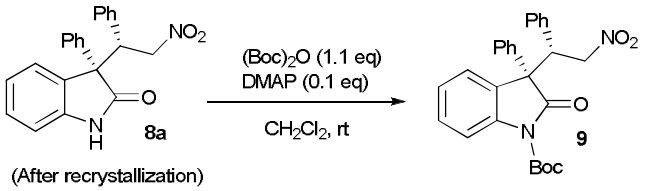
Product **8m** was obtained in 78% yield as white powder, and the pure major diastereomer was obtained after recrystallization: ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.72 (s, 1H), 7.49-7.47 (m, 2H), 7.36-7.29 (m, 4H), 7.16-7.14 (m, 2H), 7.02-6.99 (m, 1H), 4.49-4.33 (m, 2H), 3.81-3.75 (m, 1H), 1.42-1.40 (m, 1H), 1.12-0.98 (m, 2H), 0.87 (d, J = 6.3 Hz, 3H), 0.76 (d, J = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.4, 141.3, 137.1, 129.1, 129.0, 128.7, 128.2, 127.4, 125.4, 122.7, 111.0, 78.3, 60.0, 41.6, 40.0, 25.6, 23.7, 21.6; IR (KBr): 3391, 3201, 3064, 2958, 2926, 2361, 1706, 1618, 1553, 1471; MS (EI): 338 (M^+, 8), 339 [$(M+H)^+$, 2], 208 (100), 209 (25), 180 (16), 152 (7), 41 (5), 91 (5), 181 (4); HRMS (EI): Exact mass calcd for $C_{20}H_{22}N_2O_3$: 338.1630, Found: 338.1630.



8n

Product **8n** was obtained in 61% yield as white powder. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.92 (m, 0.4H), 7.62 (m, 0.6H), 7.24-6.94 (m, 12H), 6.79-6.76 (m, 2H), 6.55-6.48 (m, 1H), 5.07-4.84 (m, 2H), 4.22-4.09 (m, 1H), 3.36-2.93 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 179.9, 178.7, 141.0, 140.5, 134.7, 134.6, 134.5, 134.4, 129.8, 129.3, 129.1,. 129.0, 128.9, 128.6, 128.3, 128.2, 128.1, 127.7, 127.6, 126.7, 125.4, 124.4, 122.3, 122.1, 110.0, 109.9, 76.2, 75.5, 57.0, 56.9, 50.2, 49.7, 42.0, 40.1; IR (KBr): 3191, 3086, 2920, 2361, 1899, 1727, 1620, 1558, 1470, 1378; MS (EI): 372 (M^+, 6), 373 [$(M+H)^+$, 3], 43 (100), 91 (78), 83 (42), 149 (38), 44 (36), 55 (35), 57 (33), 41 (33); HRMS (EI): Exact mass calcd for $C_{23}H_{20}N_2O_3$: 372.1474, Found: 372.1475.

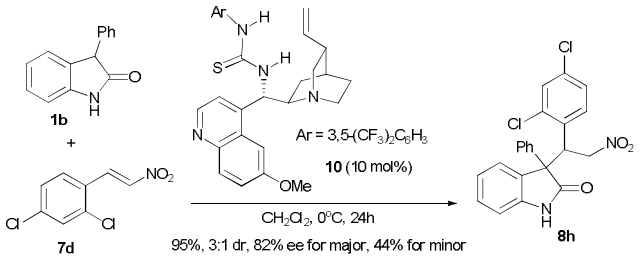
The determination of the relative configuration.



Under an atmosphere of nitrogen, to a Schlenk tube were added the pure major diastereomer of Michael adduct **8a** obtained after recrystallization (47 mg, 0.13 mmol), $(Boc)_2O$ (1.1 eq), and DMAP (10 mol%), followed by 2 mL of anhydrous dichloromethane. The reaction was stirred at room temperature till TLC analysis indicated complete conversion of the **8a**. Then 5 mL of saturated NH_4Cl solution was added, the reaction mixture was extracted with CH_2Cl_2 (3 × 5.0 mL). The combined organic phases were washed with water and brine, and then concentrated, followed by purification on a short SiO_2 column (petroleum ether/EtOAc, 6/1) to afford the N-Boc protected oxindole **9** in 87% yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.72-7.69 (m, 1H), 7.63-7.61 (m, 2H), 7.45-7.33 (m, 6H), 7.18-7.04 (m, 3H), 6.80-6.77 (m, 2H), 5.00-4.87 (m, 2H), 4.77-4.74 (m, 1H), 1.45 (s, 9H); The ^{1}H NMR data is in accordance with the reported NMR data for N-Boc protected oxindole **9**[4].

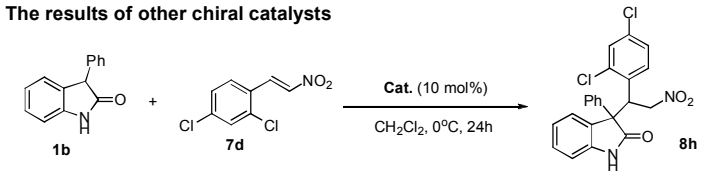
[4] R. He, S. Shirakawa and K. Maruoka, *J. Am. Chem. Soc.,* 2009, **131**, 16620.

The catalytic asymmetric Michael Addition of oxindole 1b to nitroolefin 7d.

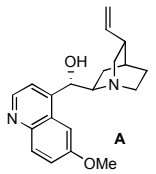


To a 5-mL vial were added catalyst **10** (6.0 mg, 0.01 mmol), oxindole **1b** (21.0 mg, 0.10 mmol) and 1.0 mL of anhydrous dichloromethane. After the reaction mixture was stirred at 0^{o}C for half an hour, nitroolefin **7d** (24.0 mg, 0.11 mmol) was added. The resulting mixture was stirred at 0°C till almost full conversion of **1** by TLC analysis. The reaction mixture was directly subjected to column chromatography to afford the desired product **8h** 41 mg in 95% yield, using CH_2Cl_2 as eluent. HPLC analysis (Chiralcel OD-H/OD-H, 5% ^{i}PrOH/hexane, 0.8 mL/min, 230 nm; for major diastereomer: t_r (minor) =27.64 min, t_r (major) =35.31 min; for minor diastereomer: t_r (minor) =39.75 min, t_r (major) =24.03 min) gave the diastereoselectivity of the product: 2.2:1.0, and isomeric composition of the major diastereomer: 85% ee.

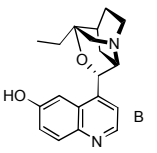
The results of other chiral catalysts



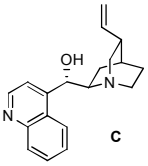
All catalysts could promote this reaction to full conversion within one day.



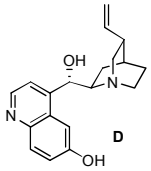
dr: 1.2/1.0, ee: 5%/5%



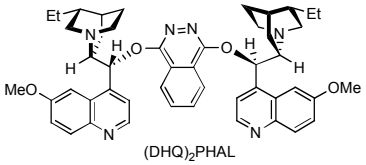
dr: 2.0/1.0, ee: 14%/8%



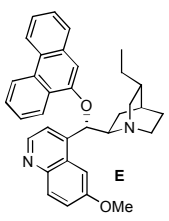
dr: 1.0/1.0, ee: 7%/22%



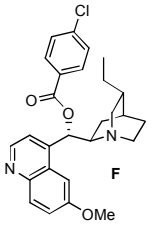
dr: 1.0/1.0, ee: 27%/46%



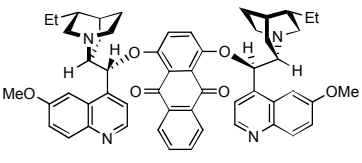
dr: 2.0/1.0, ee: 38%/16%



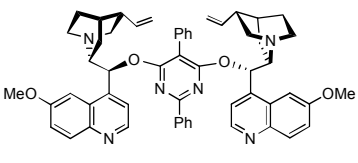
dr: 1.7/1.0, ee: 20%/43%



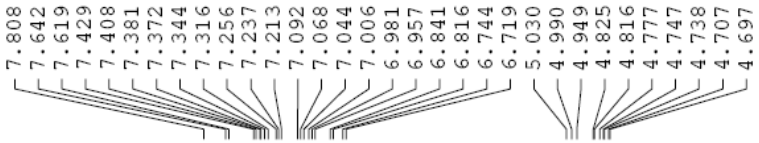
dr: 1.6/1.0, ee: 10%/50%

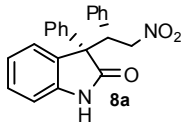


dr: 1.0/1.0, ee: 38%/0

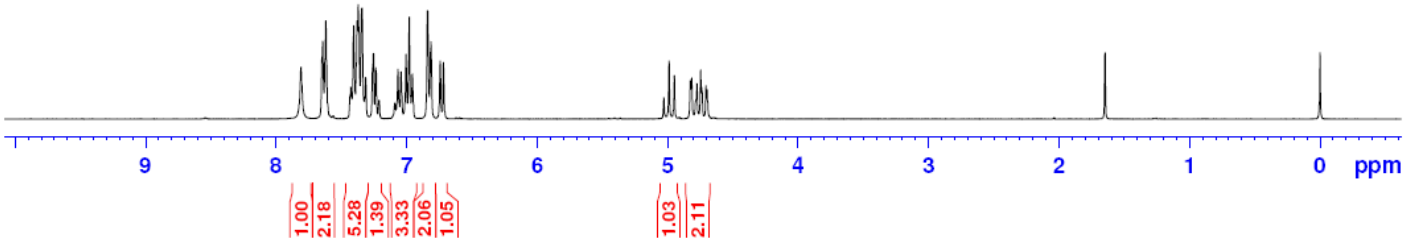


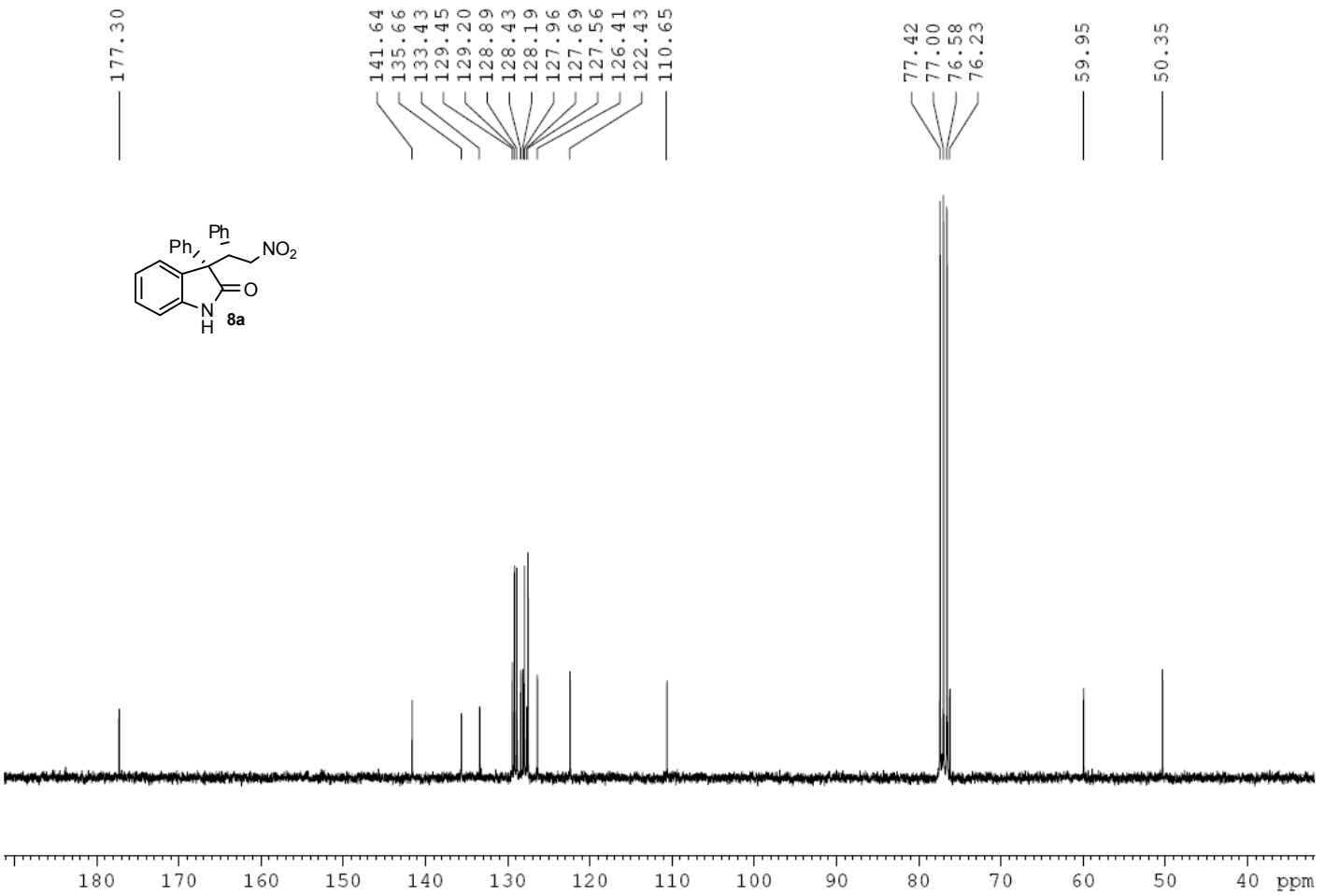
dr: 1.5/1.0, ee: 34%/19%

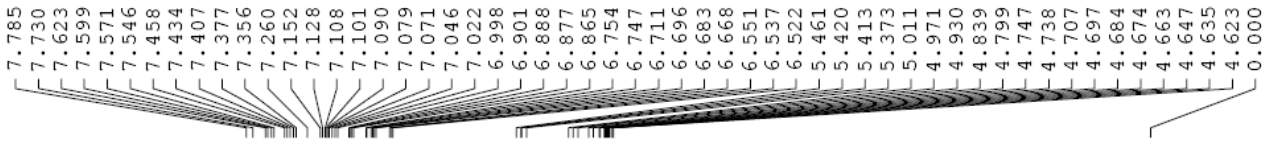


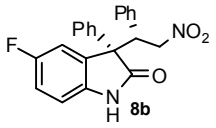


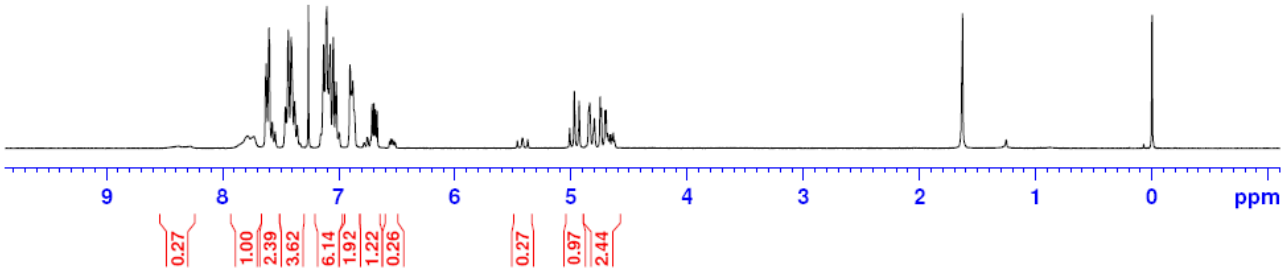
8a

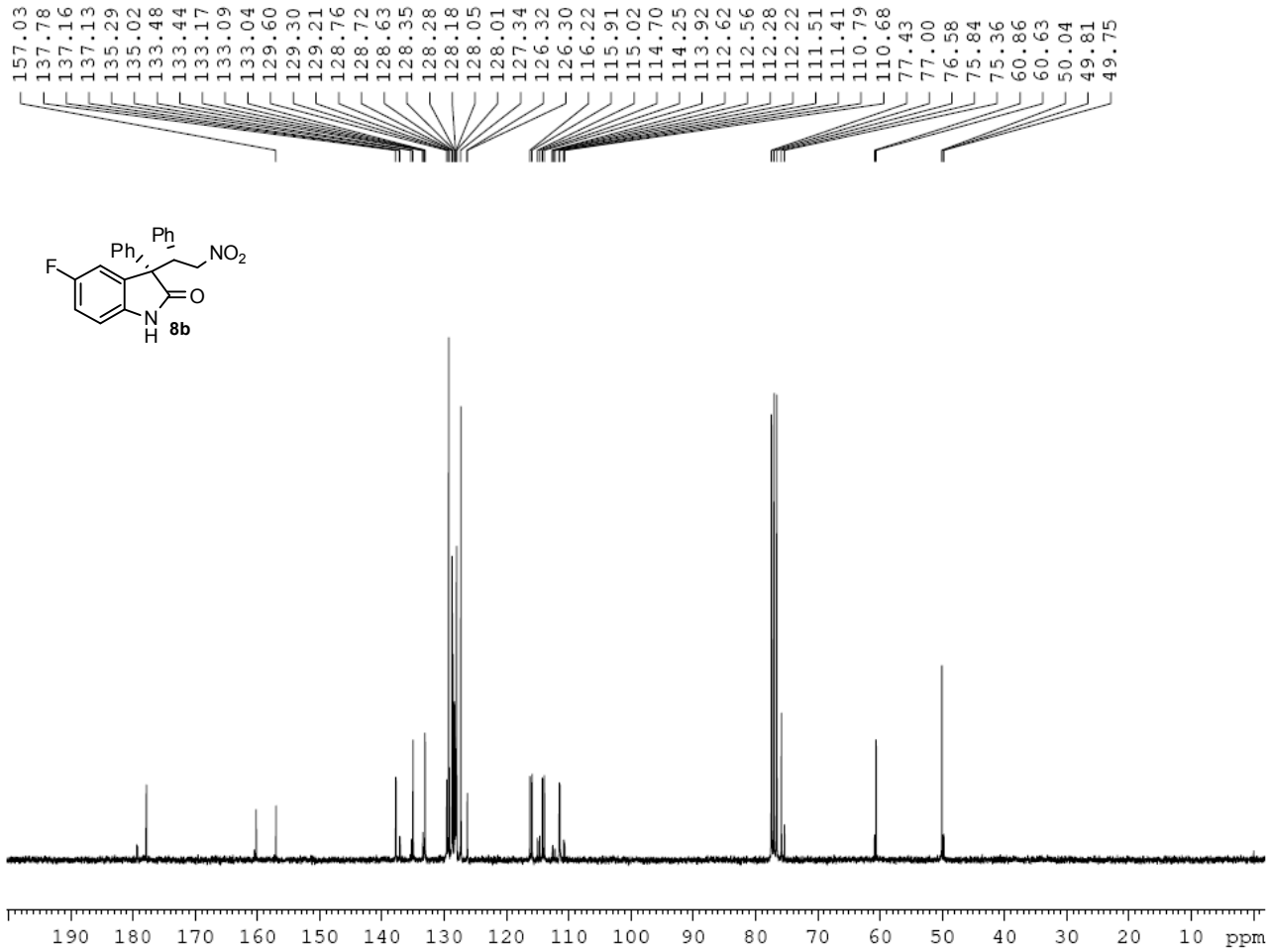


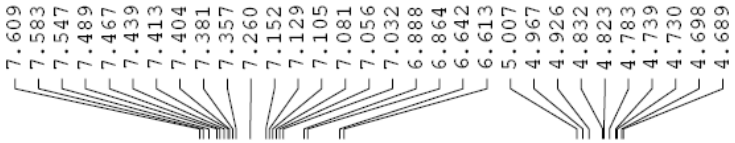


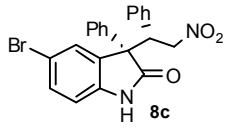




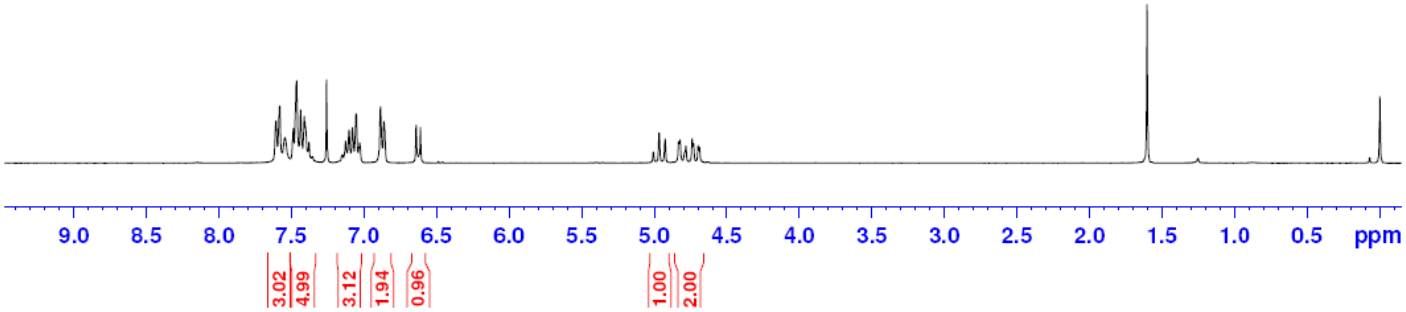


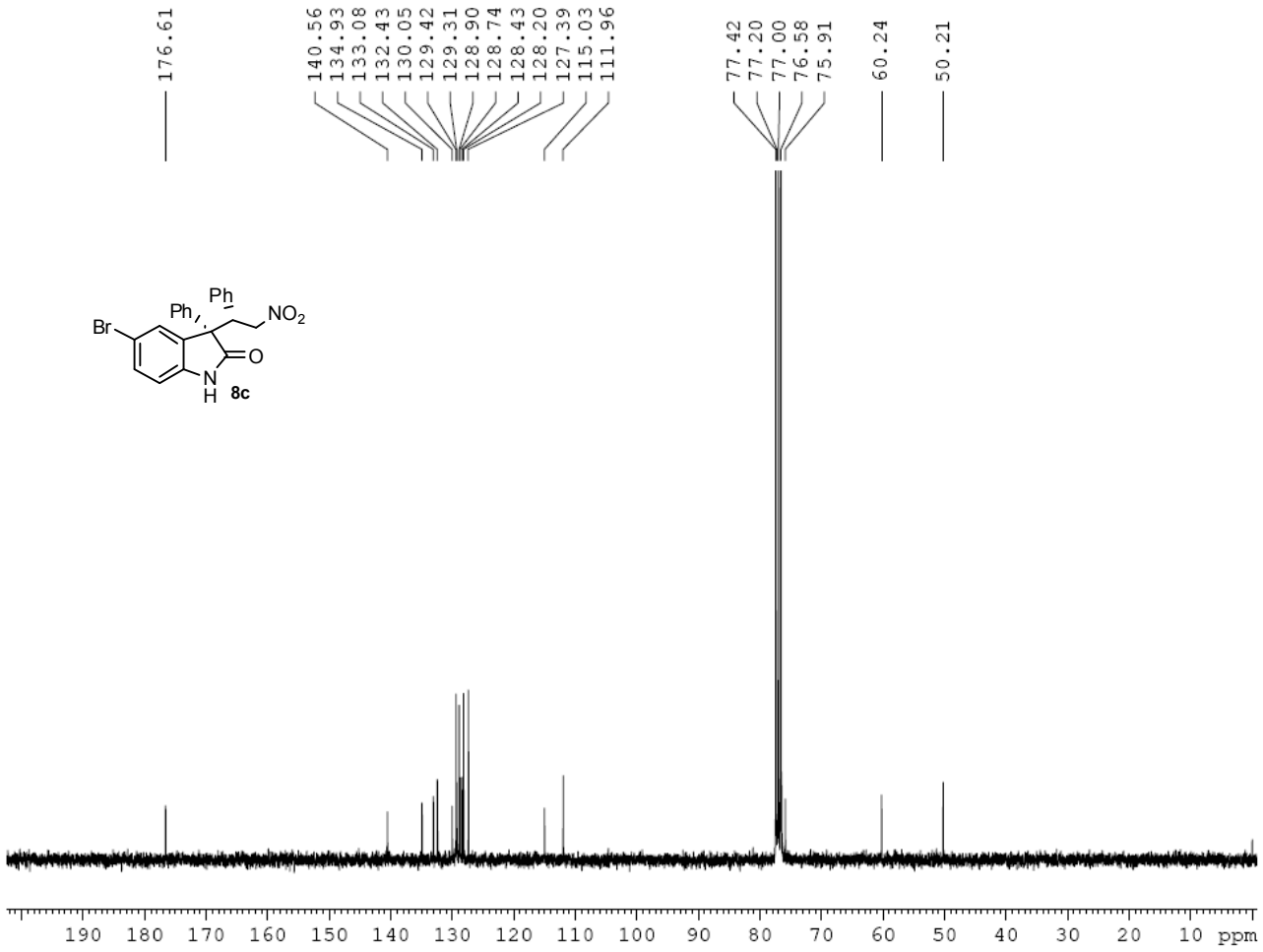


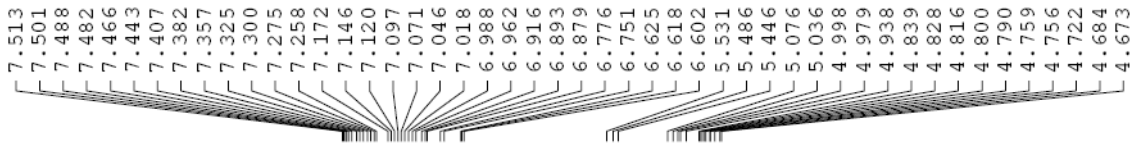


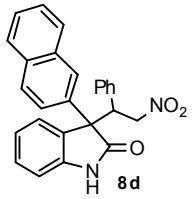


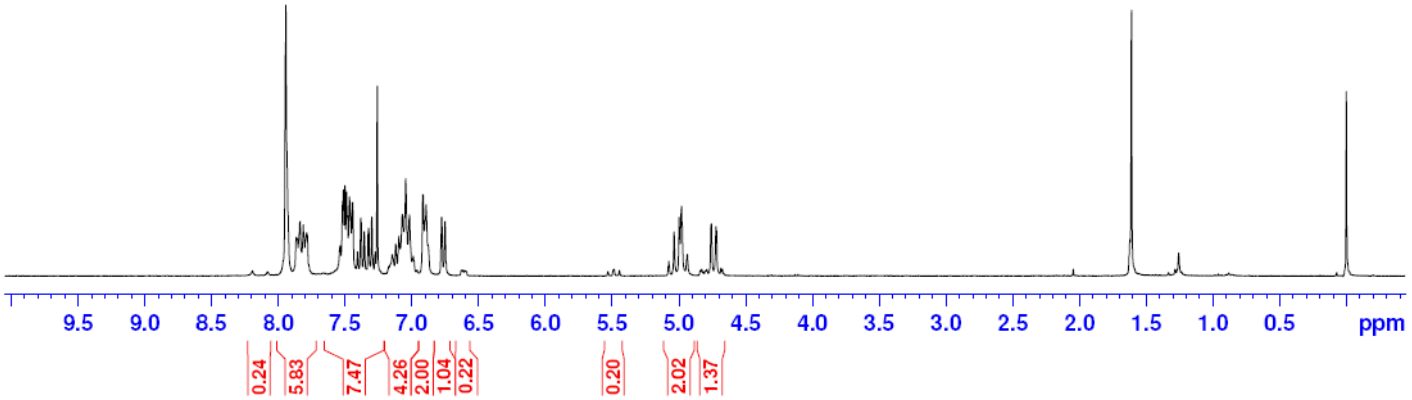
8c

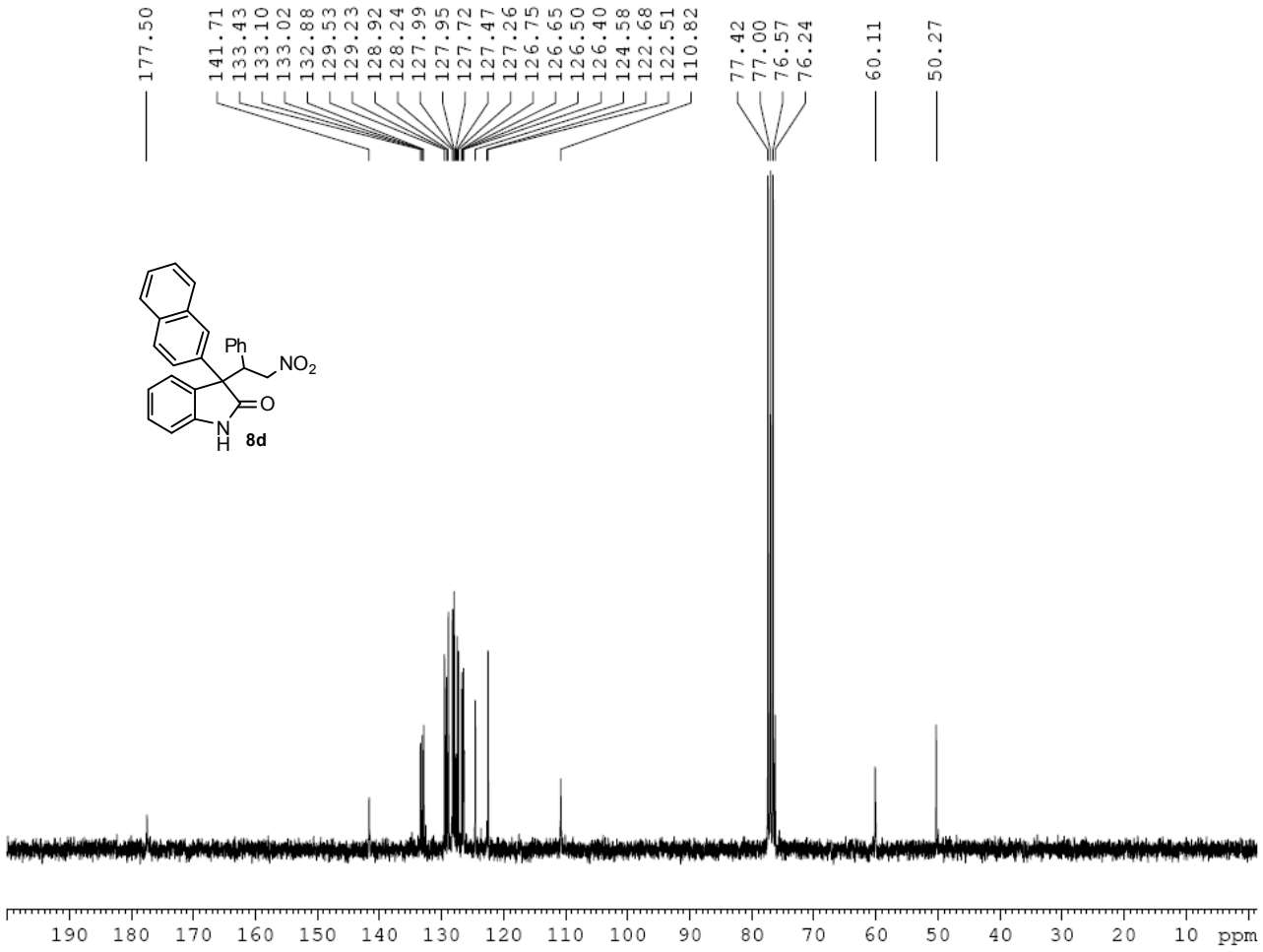


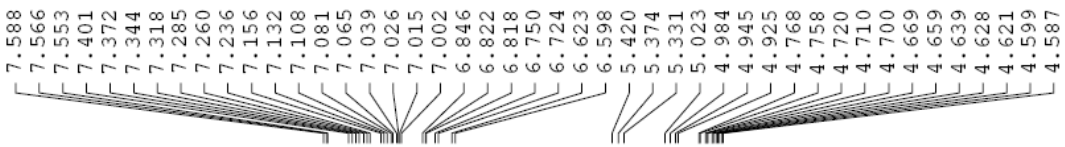


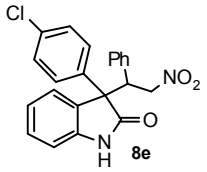


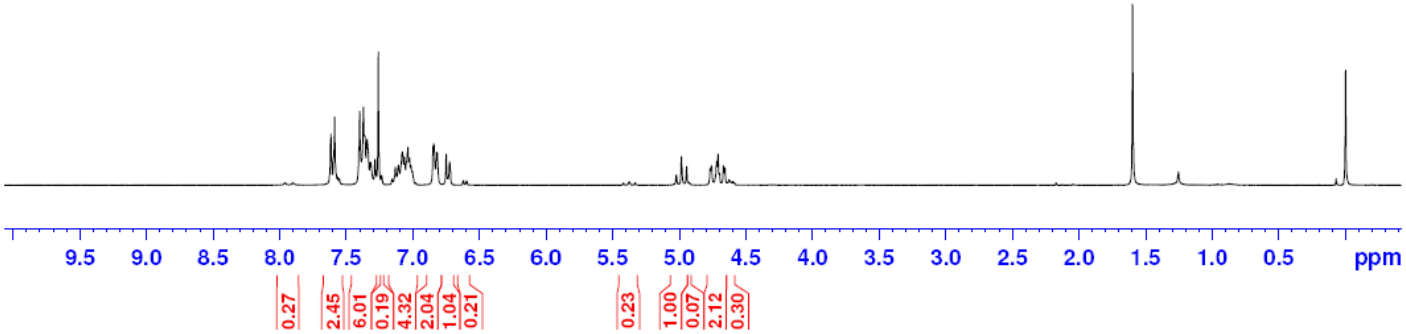


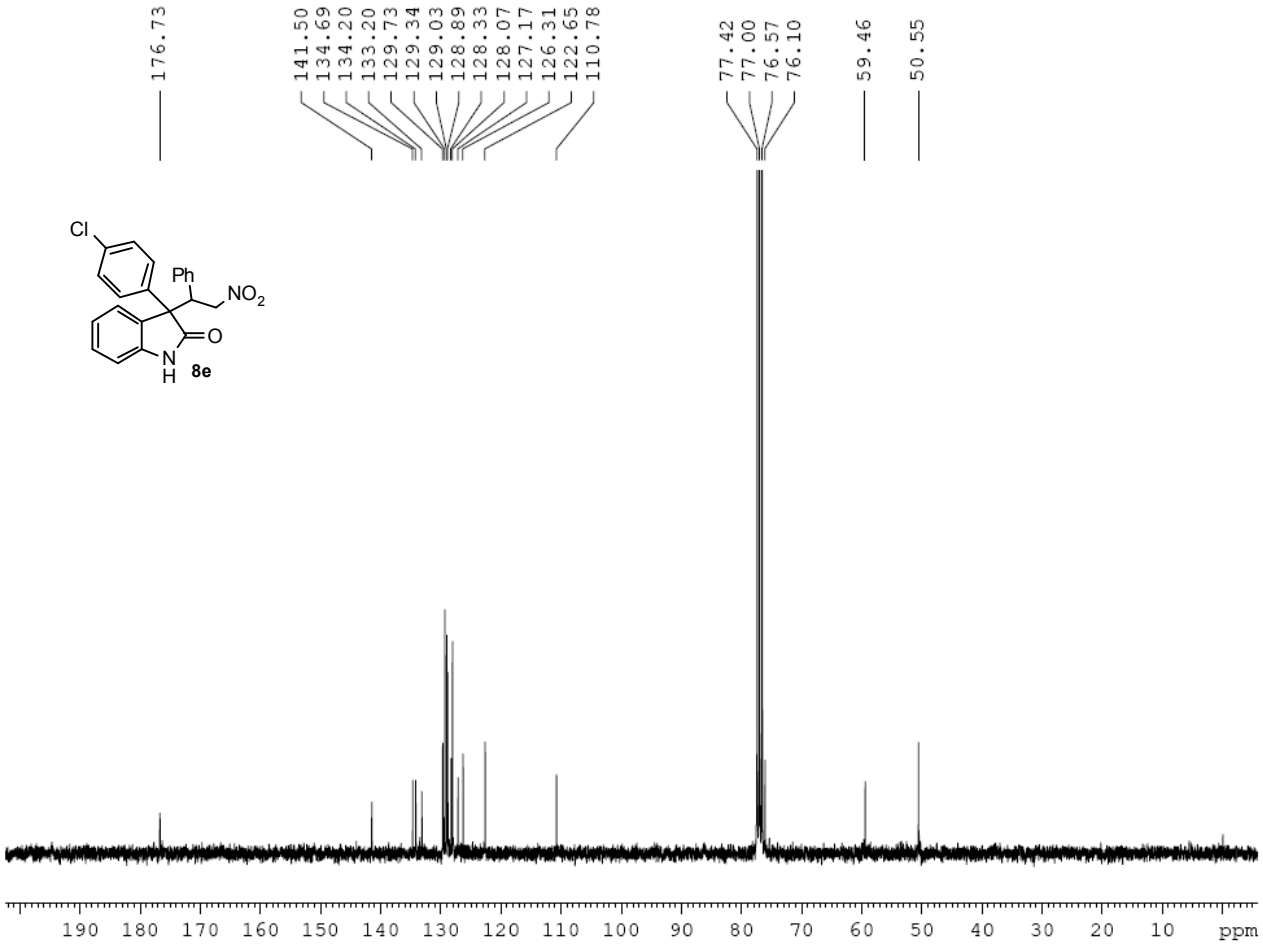


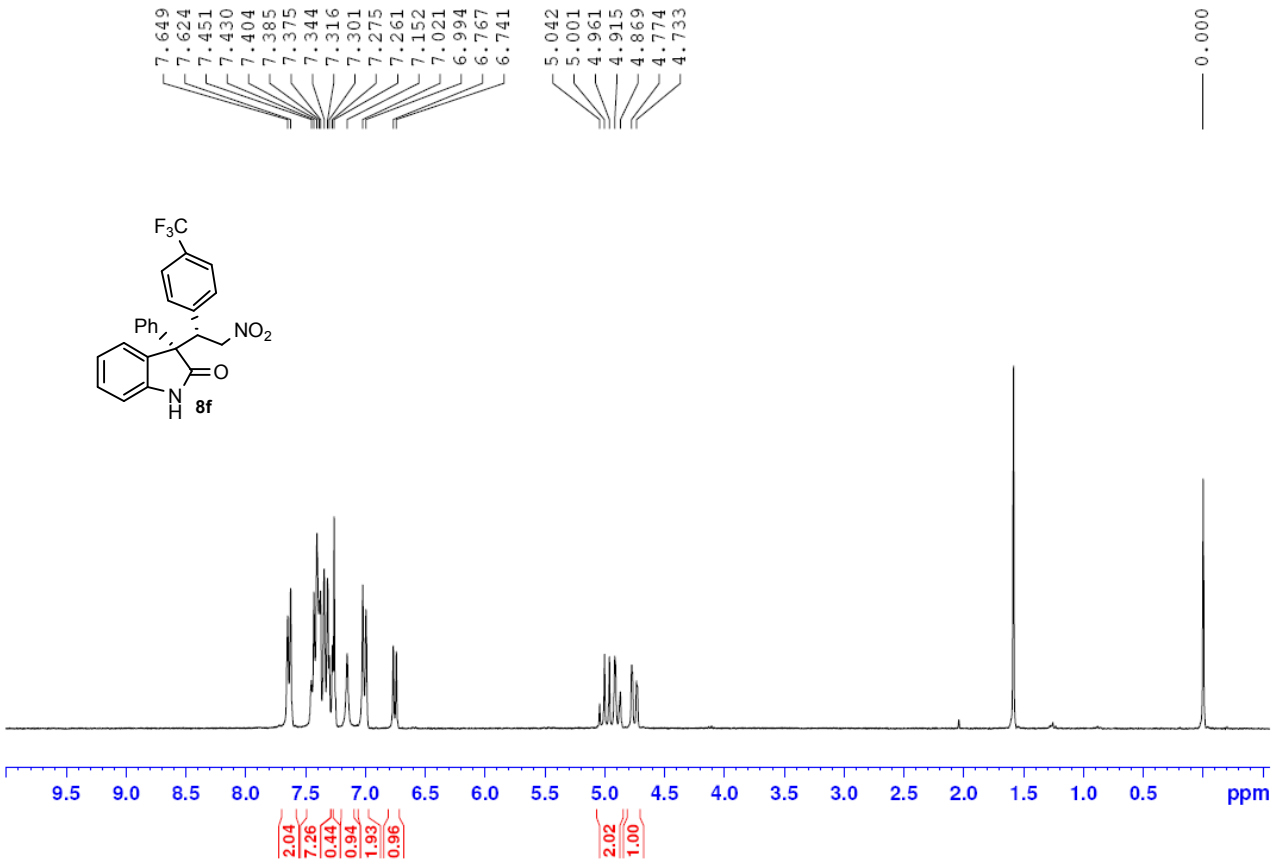


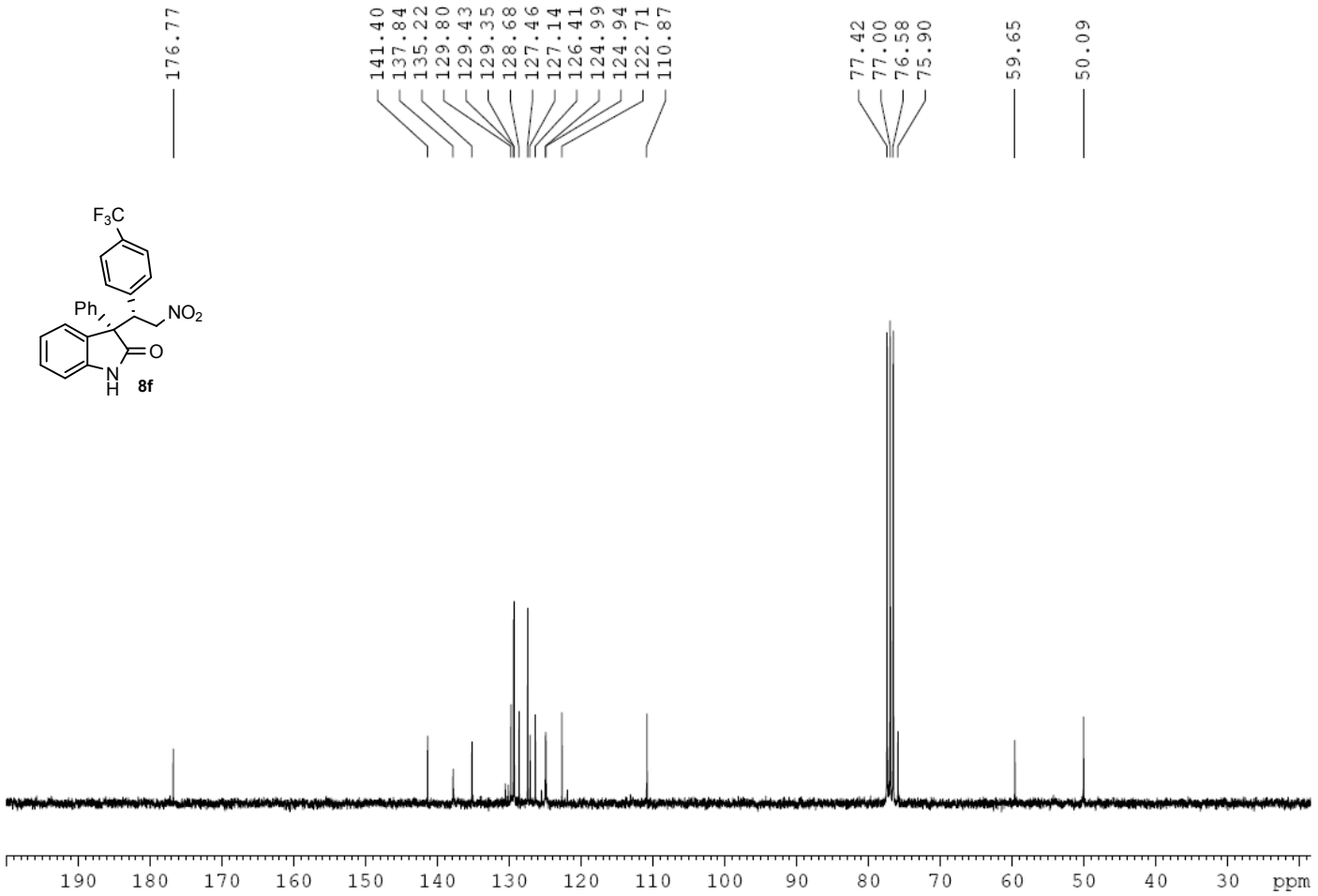


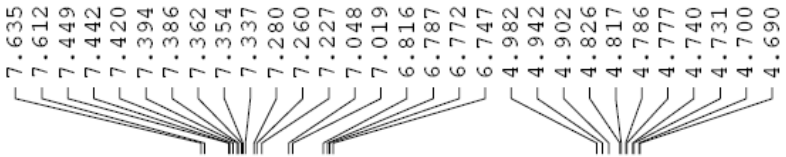


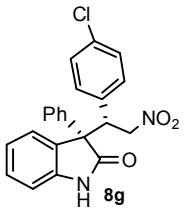




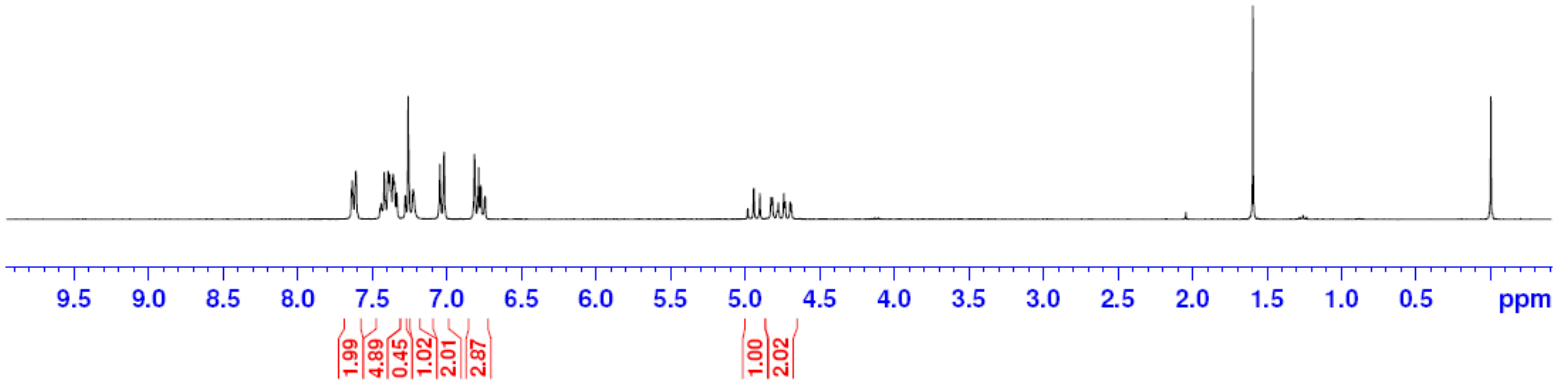


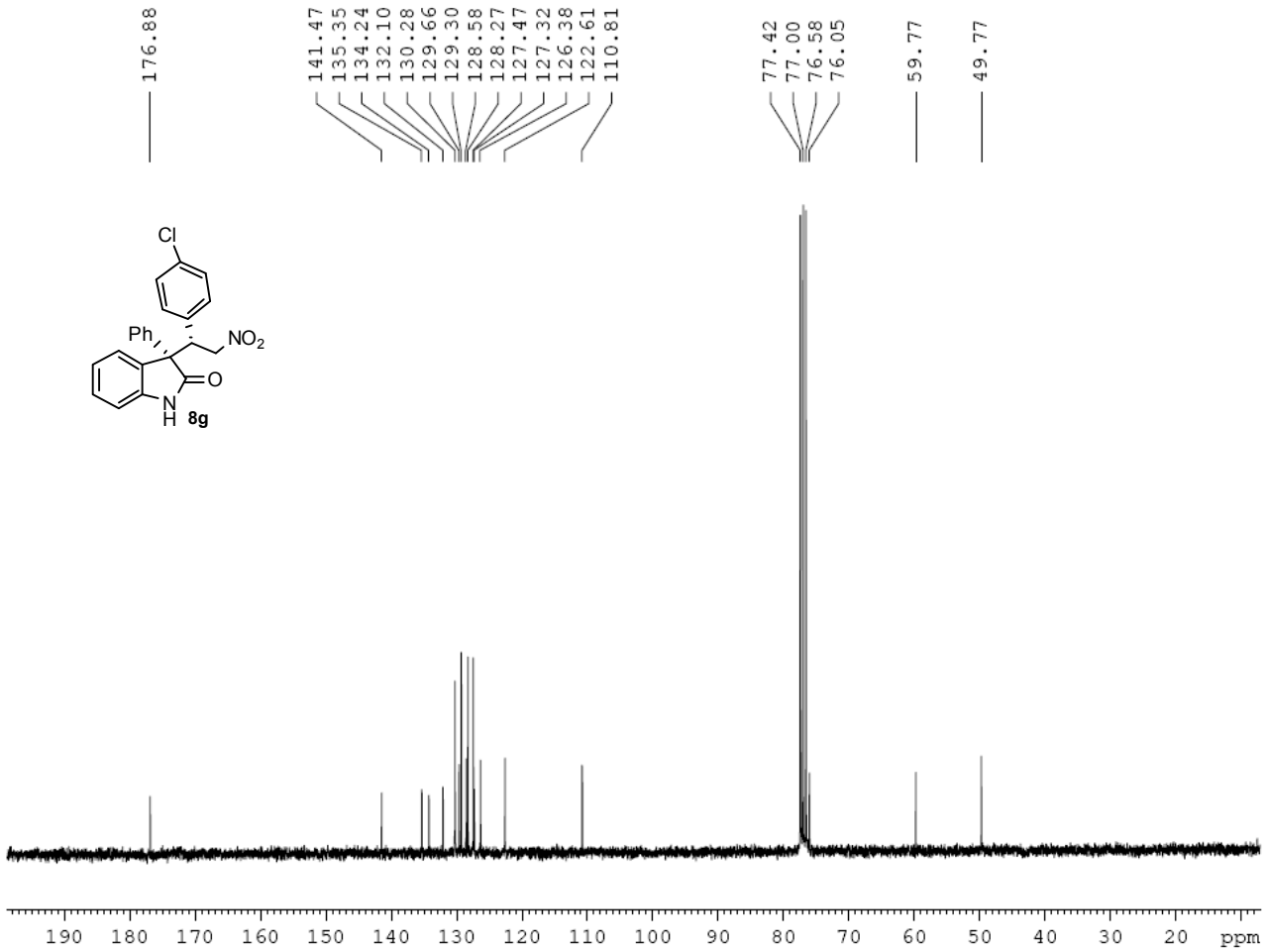


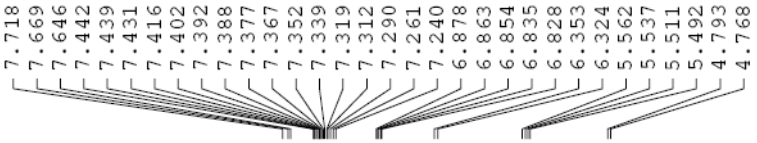


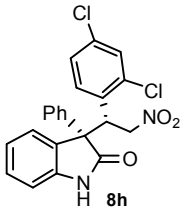


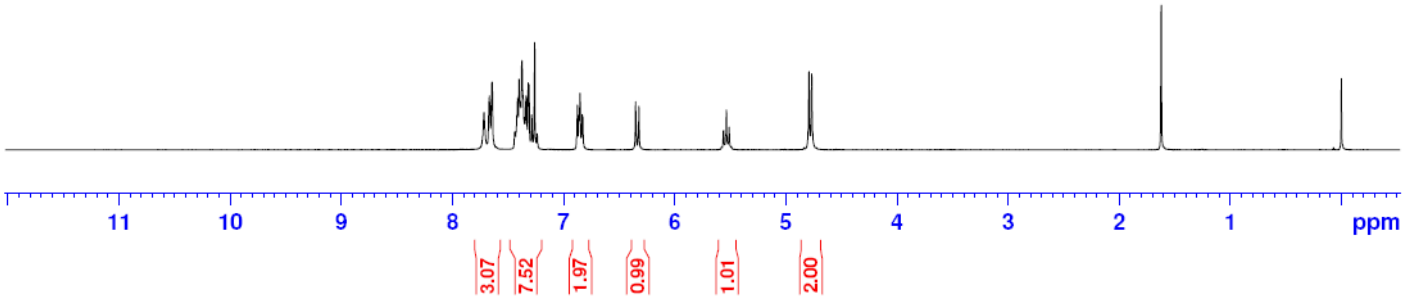
8g

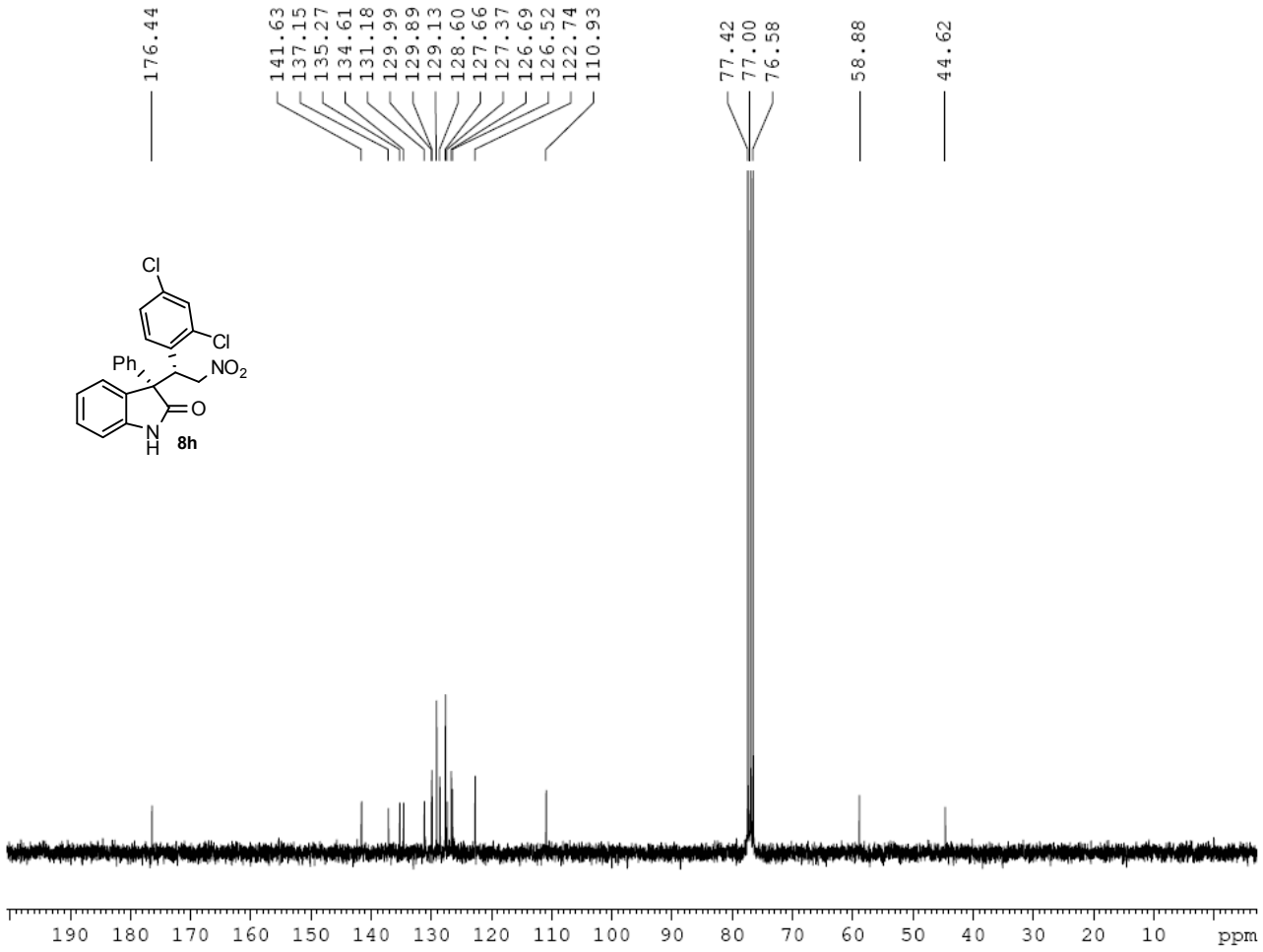


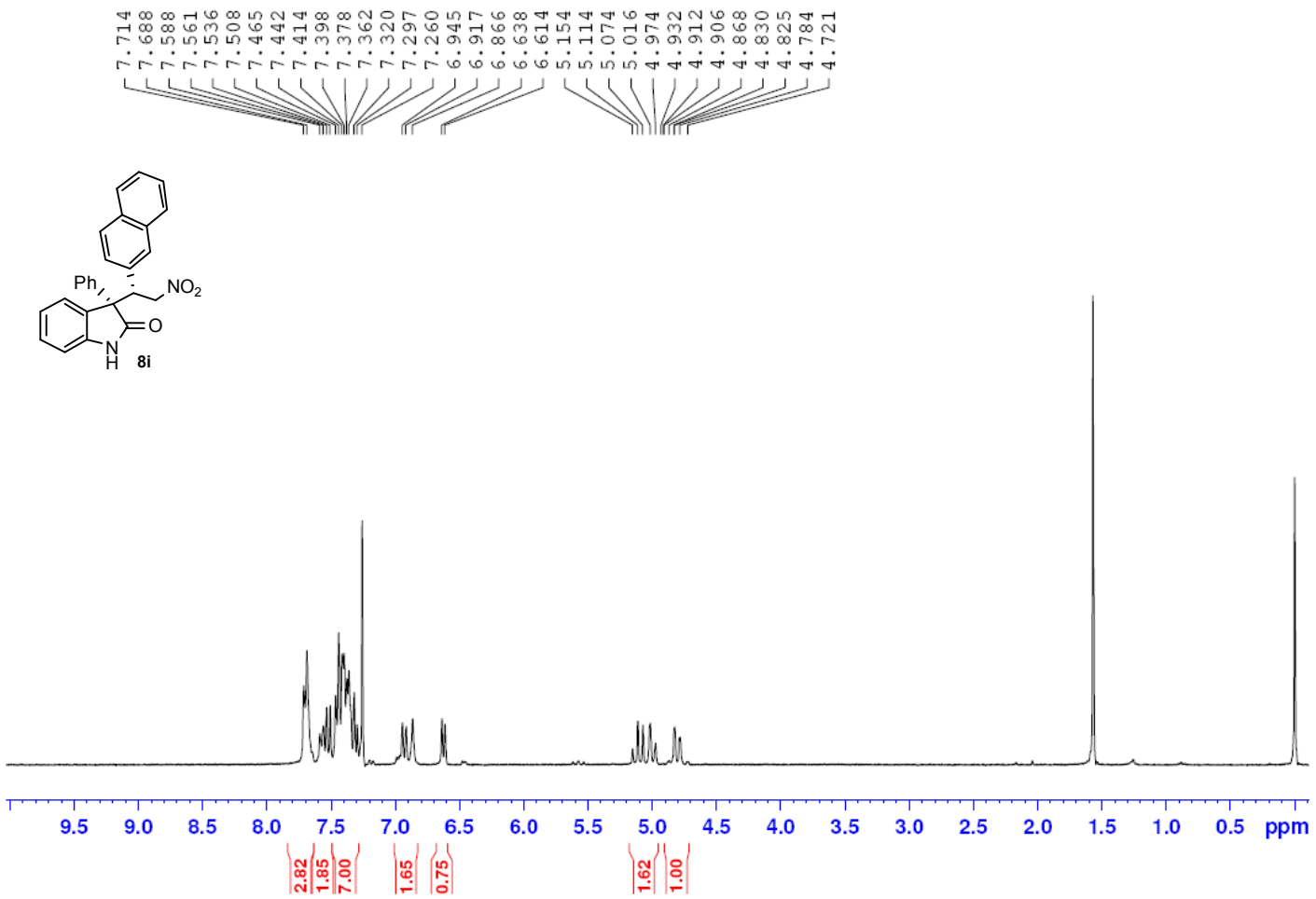


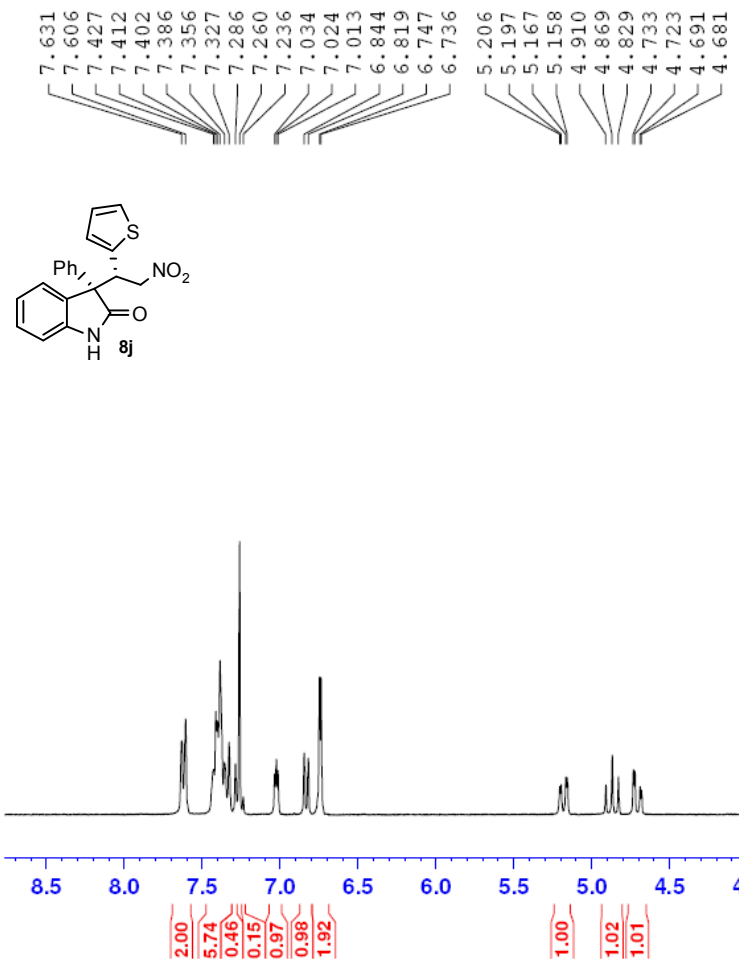


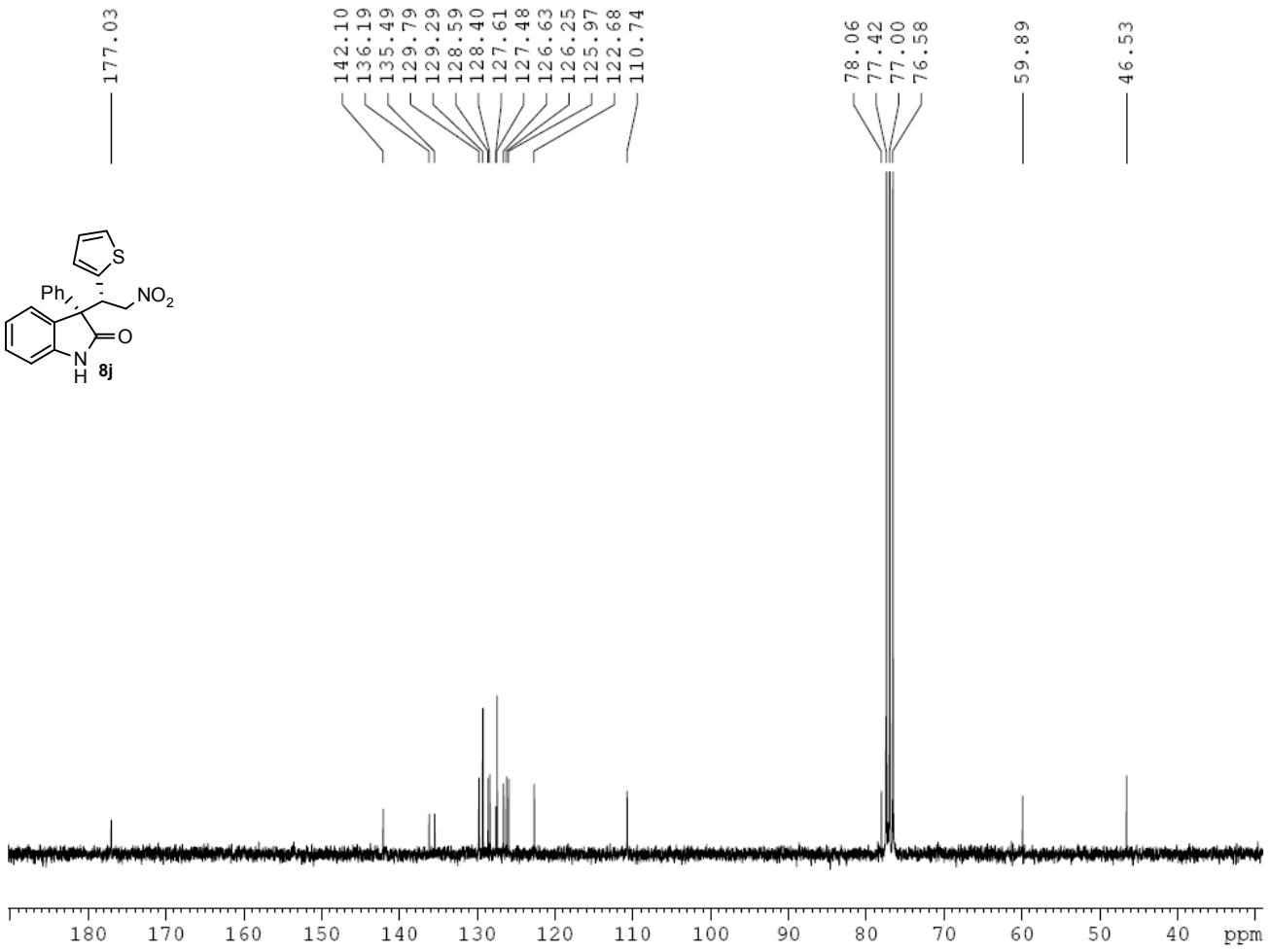


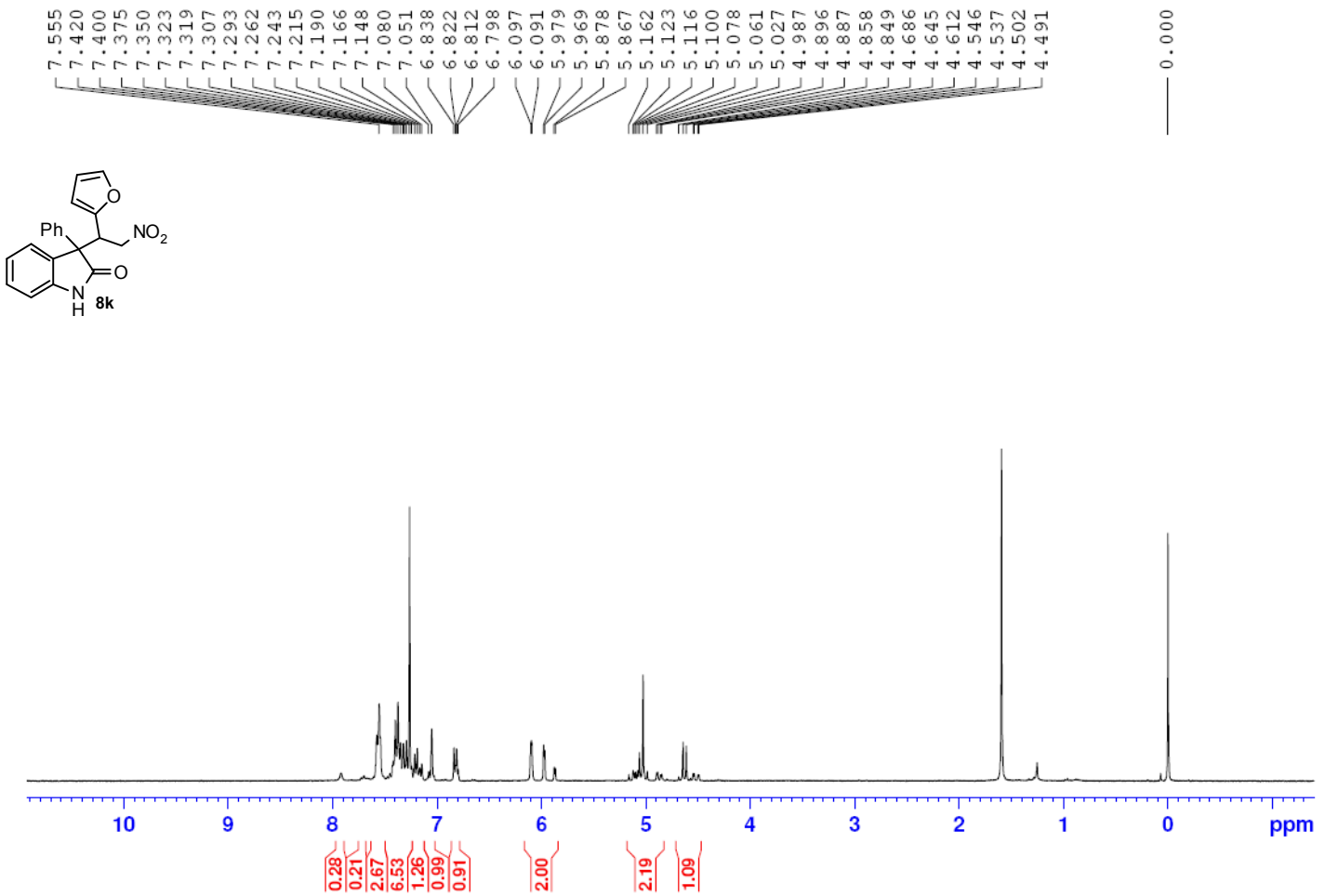


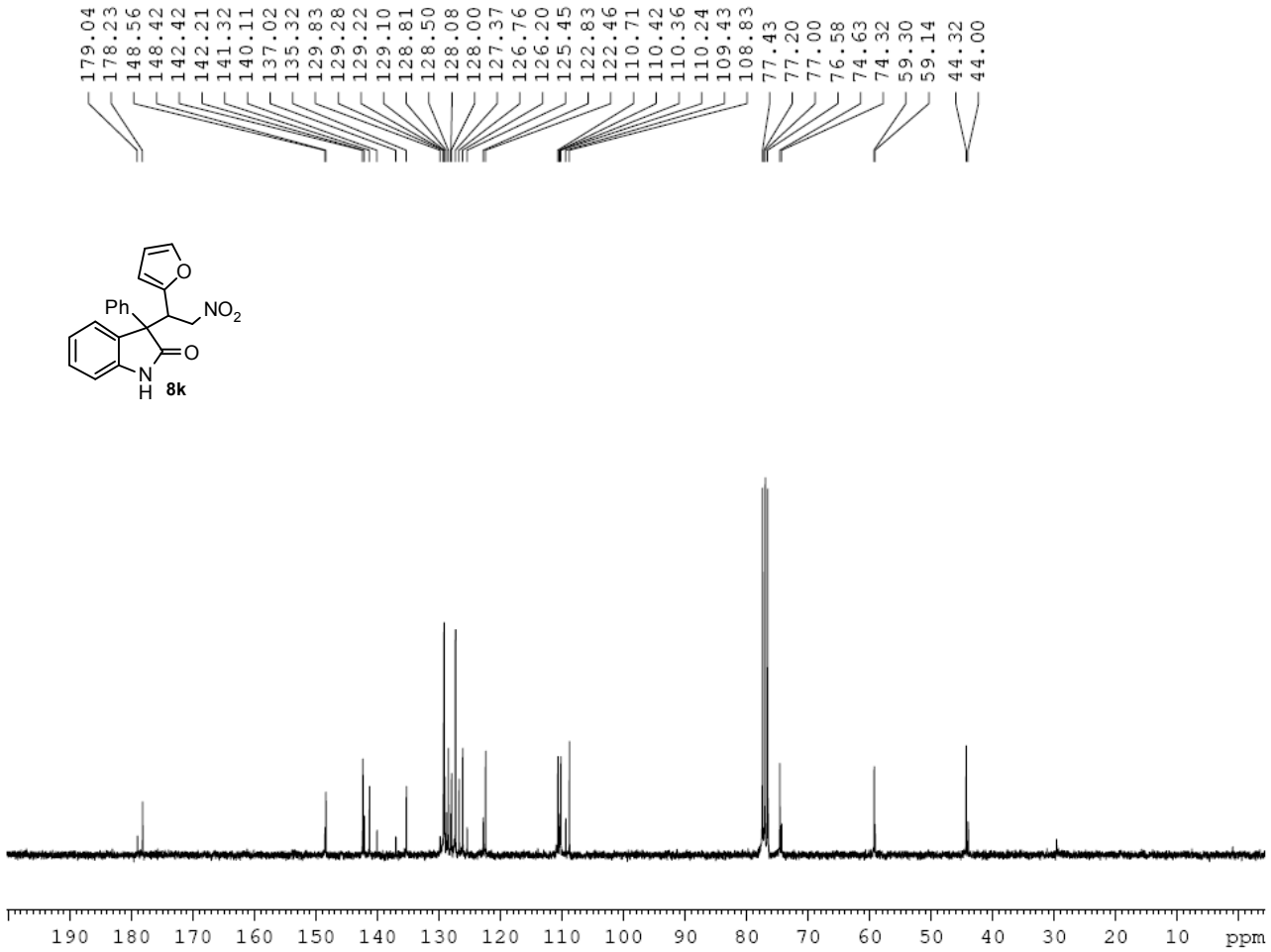


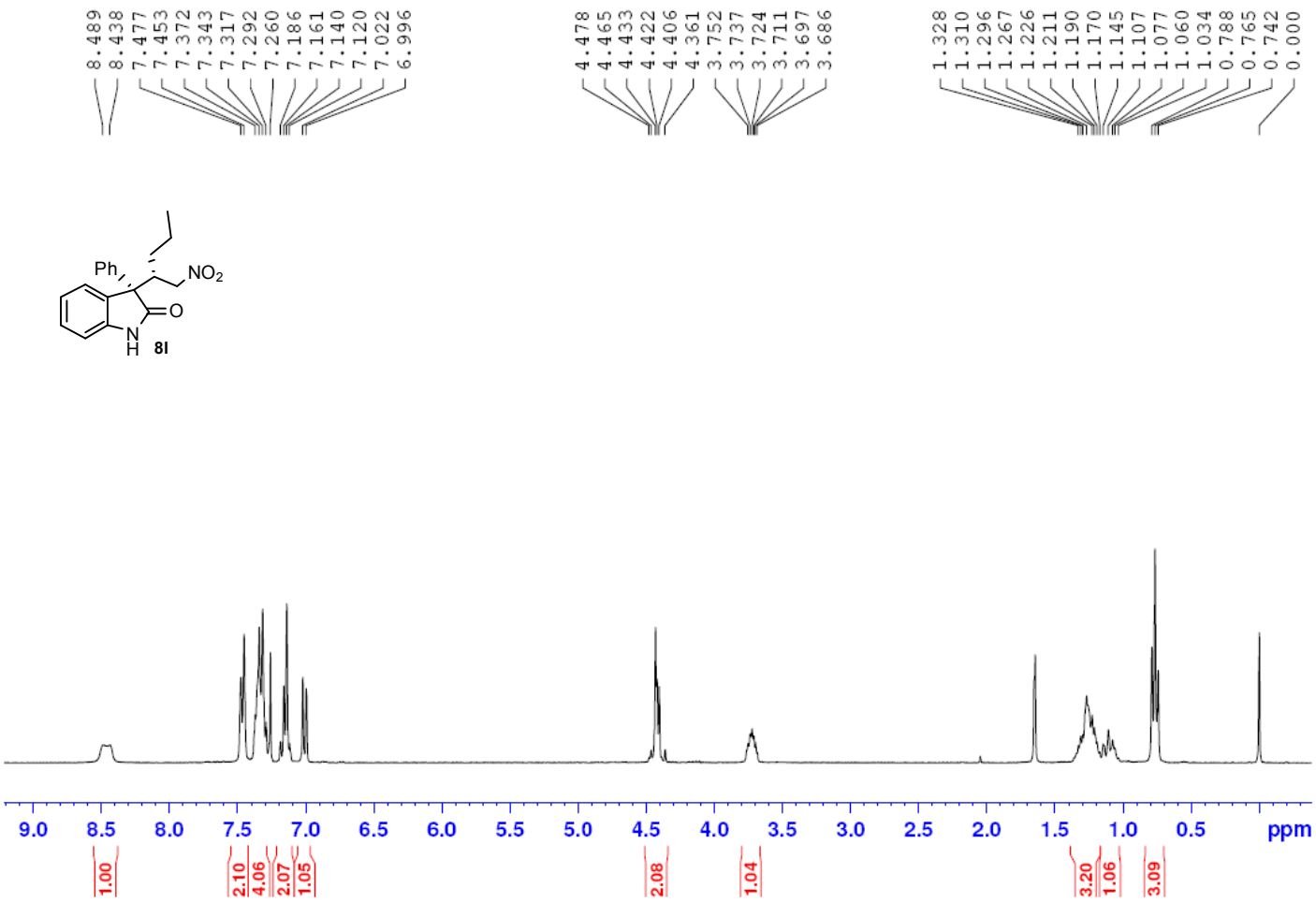


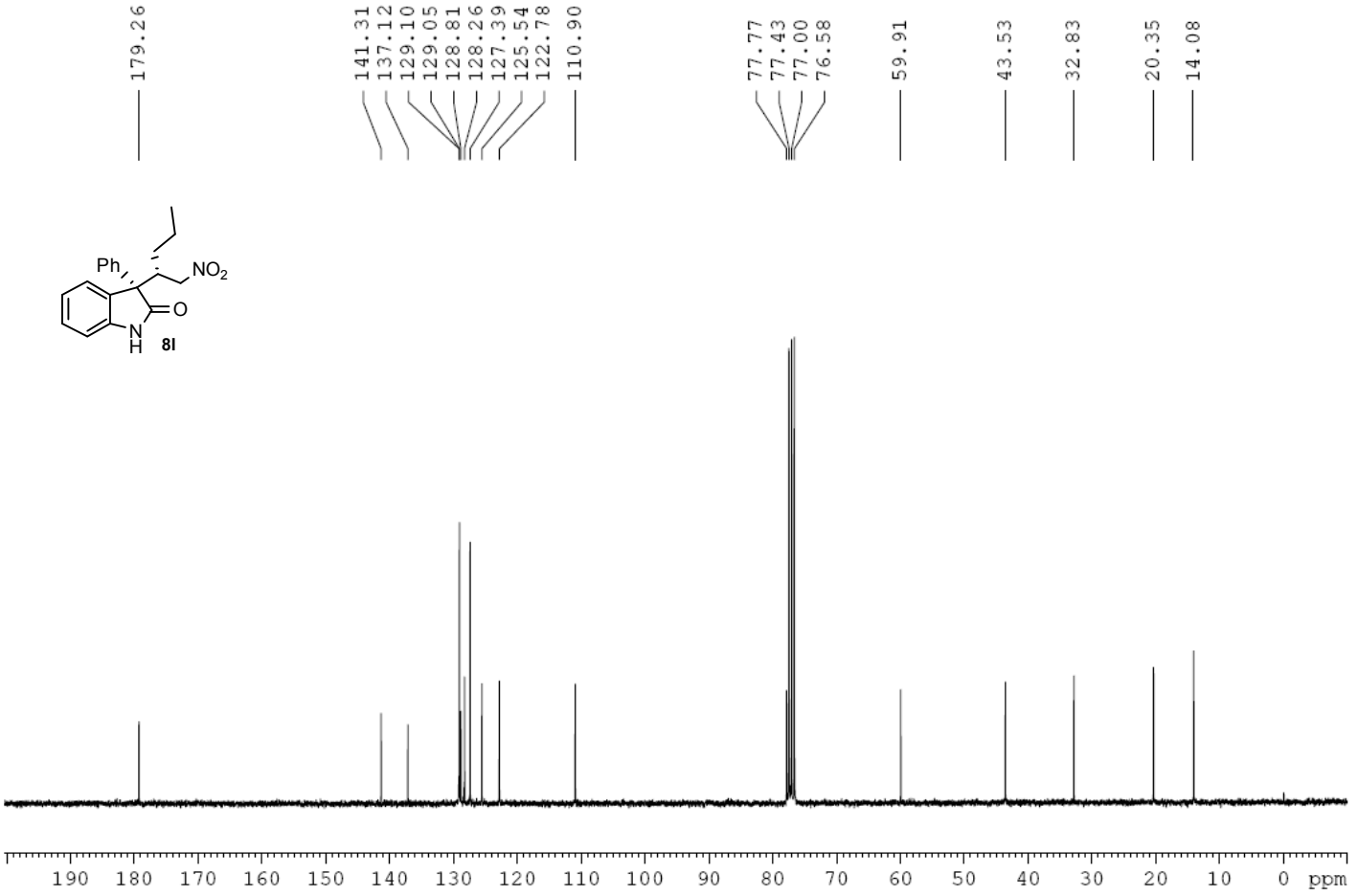


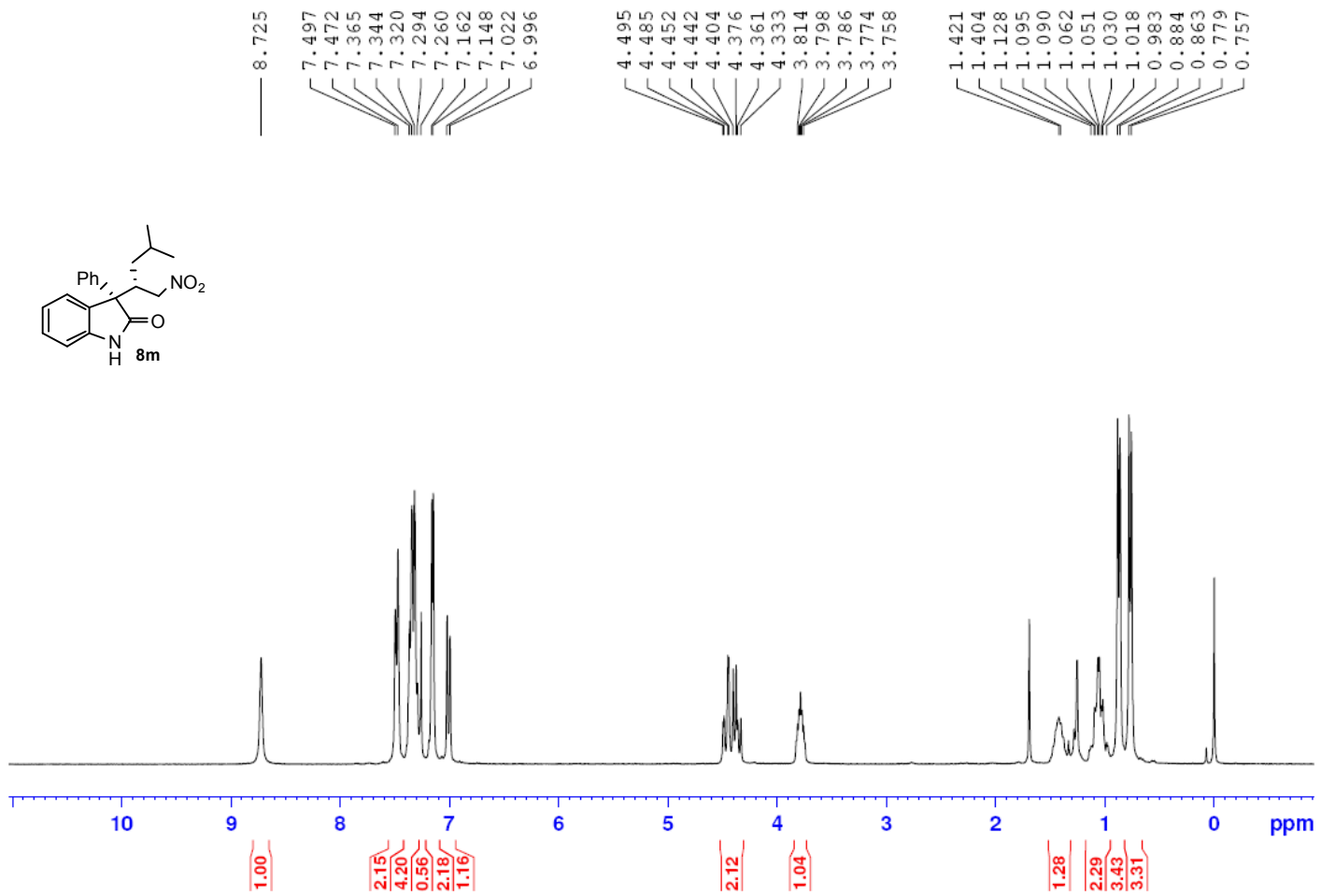


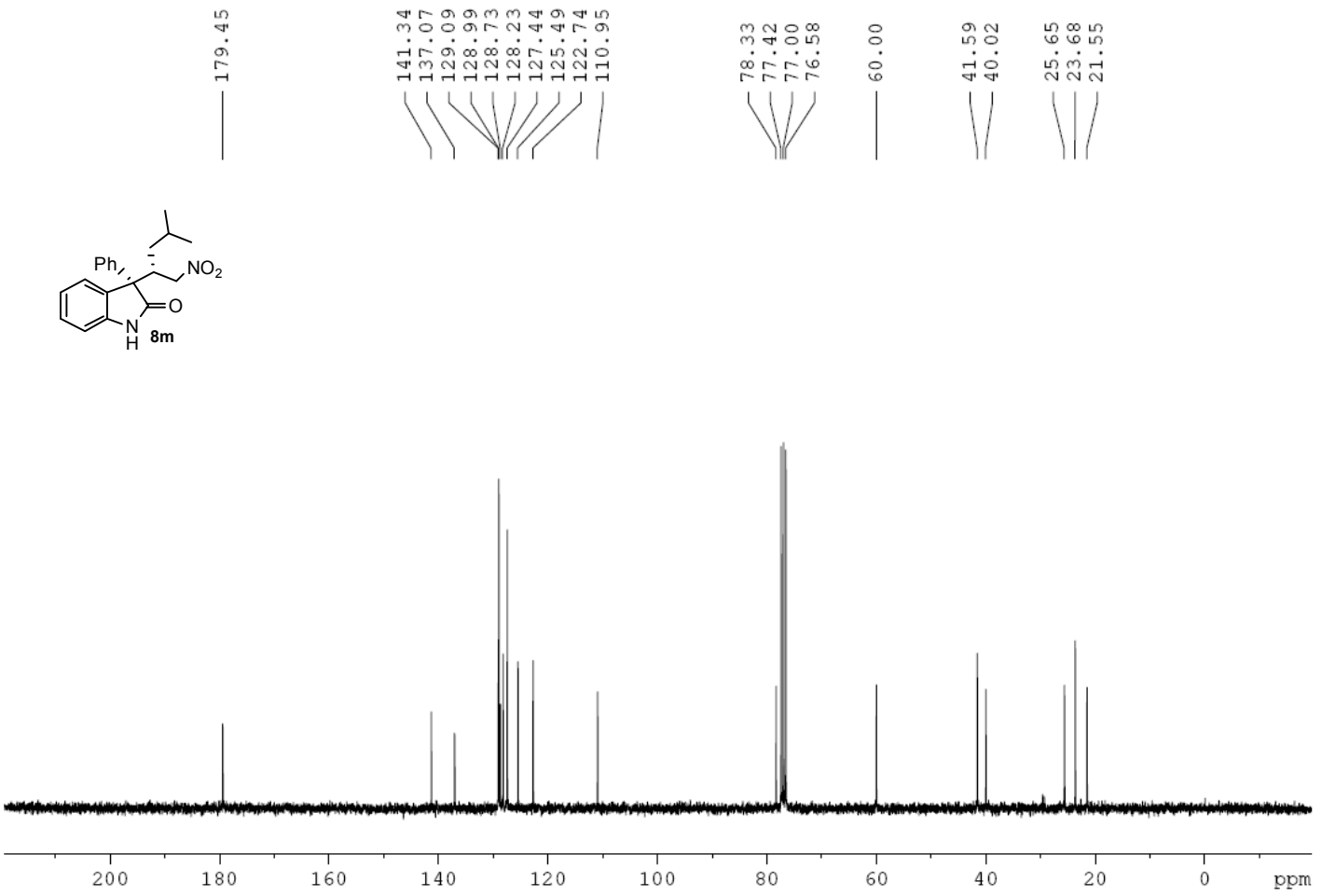


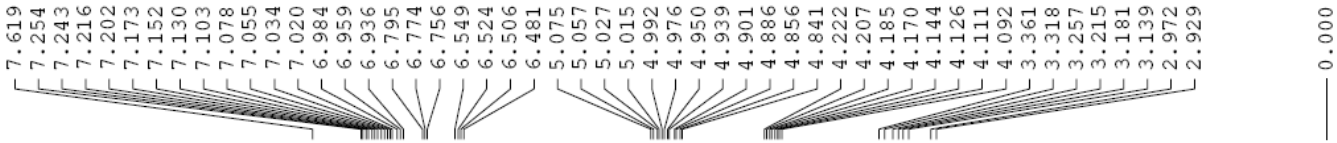


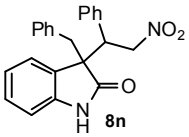












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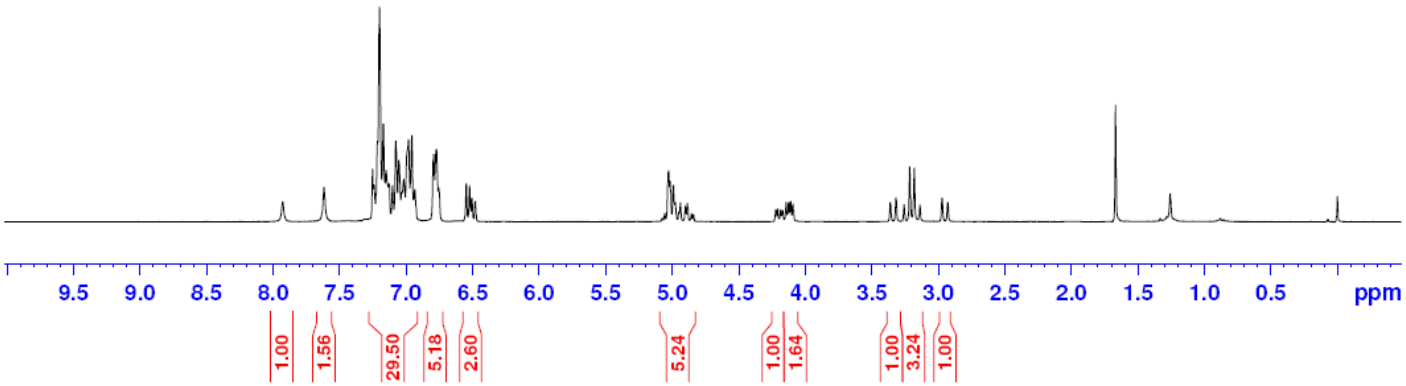

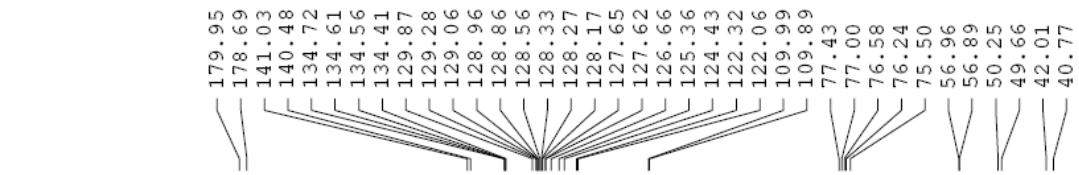

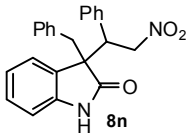

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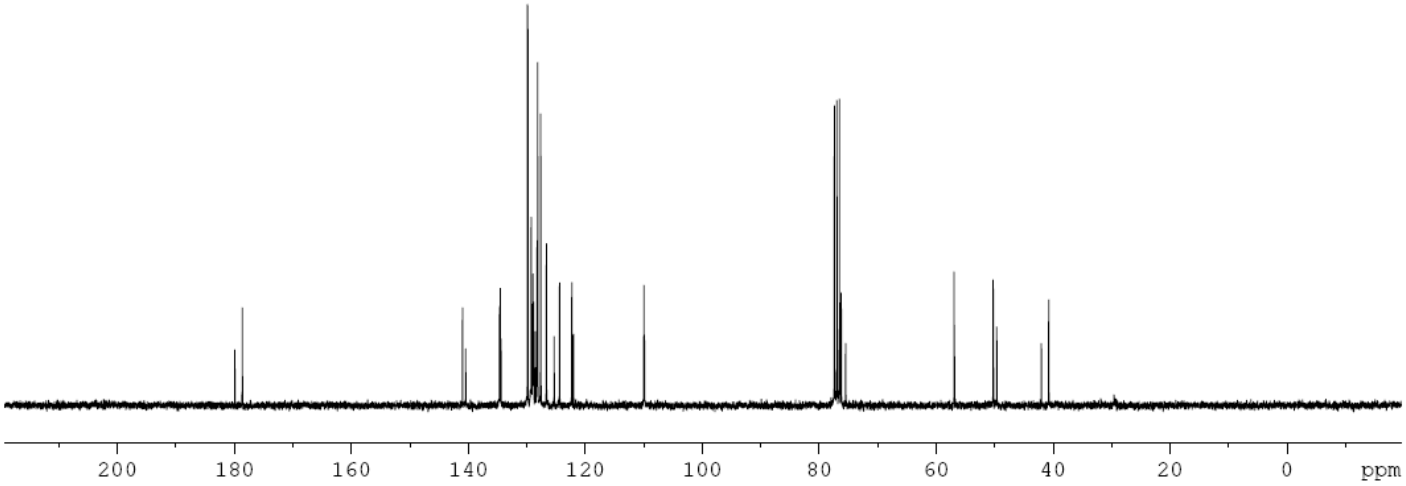

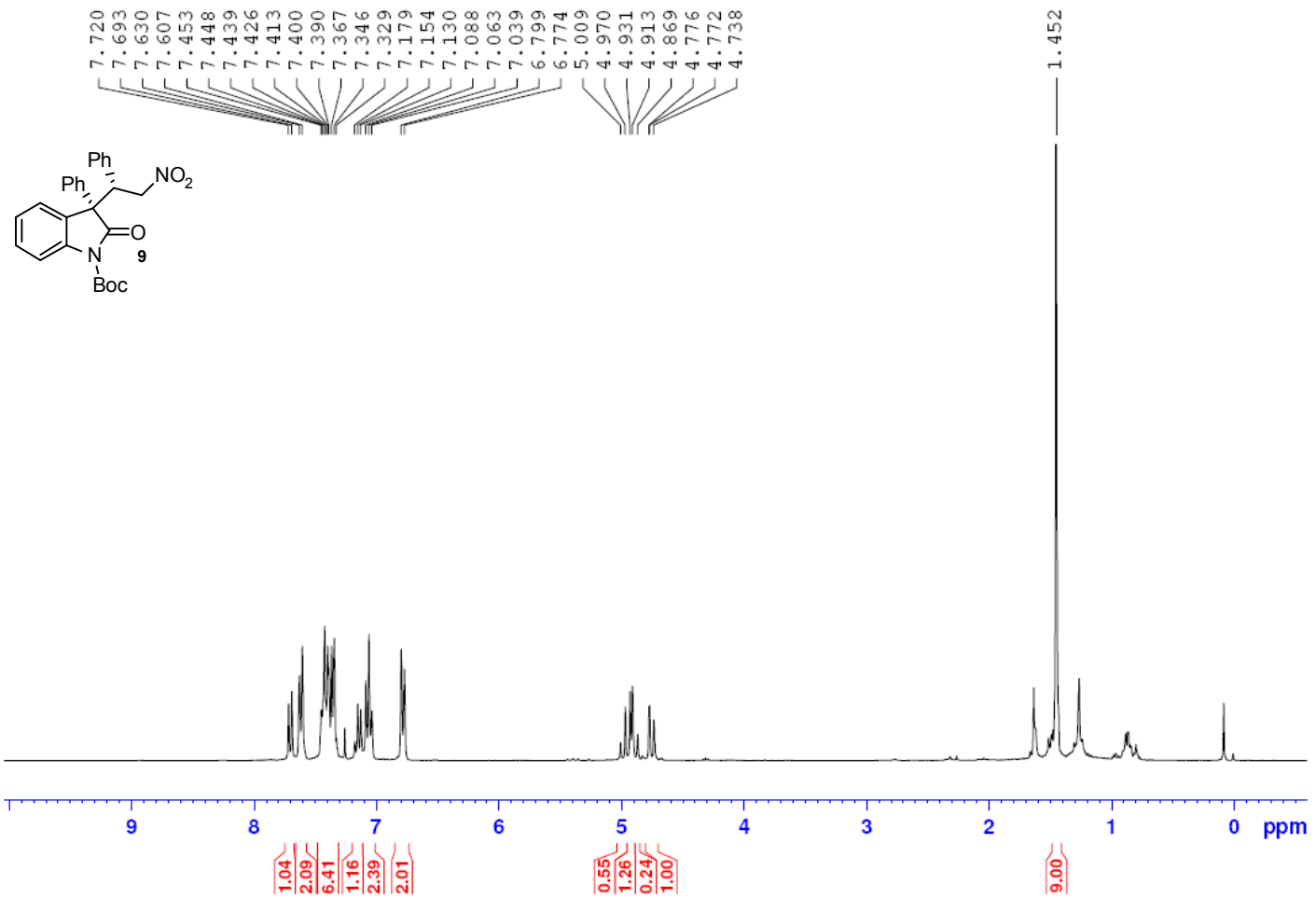

Operator:dell Timebase:U-3000 Sequence:dm

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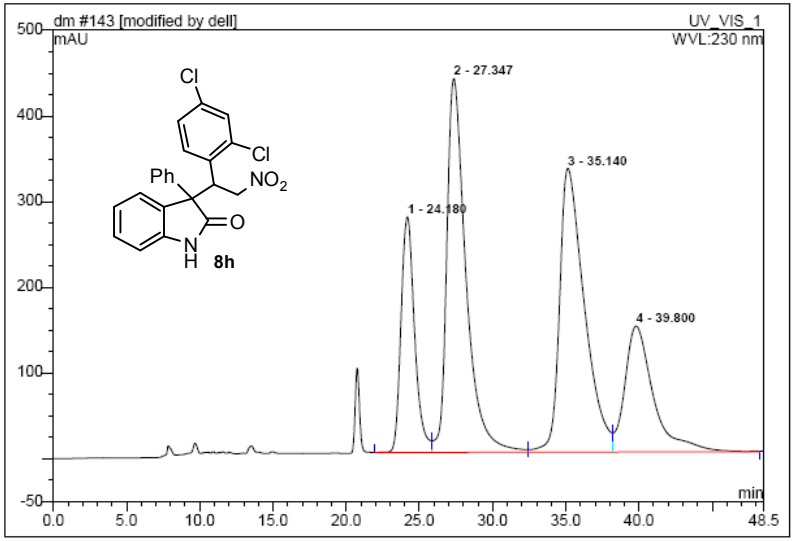
143 DIM-DA-88-RAC-ODH(Z)-ODH(J)-90/10-230

Sample Name:	**DIM-DA-88-RAC-ODH(Z)-ODH(J)-90/10-230**	*Injection Volume:*	**20.0**
Vial Number:	**153**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**230**
Control Program:	**dm**	*Bandwidth:*	**n.a.**
Quantif. Method:	**dm**	*Dilution Factor:*	**1.0000**
Recording Time:	**2010-3-6 16:21**	*Sample Weight:*	**1.0000**
Run Time (min):	**48.53**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	24.18	n.a.	274.888	296.611	15.27	n.a.	BM *
2	27.35	n.a.	436.156	662.249	34.10	n.a.	M *
3	35.14	n.a.	331.125	650.326	33.49	n.a.	M *
4	39.80	n.a.	146.900	332.926	17.14	n.a.	MB*
Total:			1189.069	1942.113	100.00	0.000	

Operator:dell Timebase:U-3000 Sequence:dm

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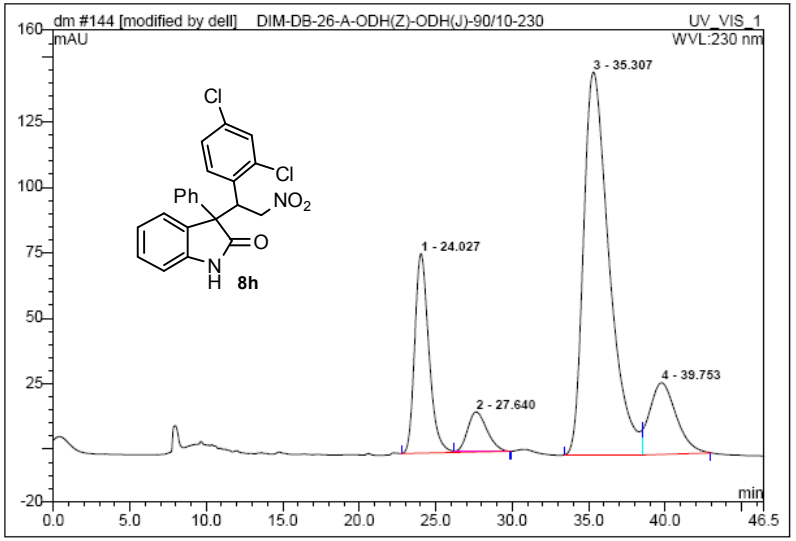
144 DIM-DB-26-A-ODH(Z)-ODH(J)-90/10-230

Sample Name:	**DIM-DB-26-A-ODH(Z)-ODH(J)-90/10-230**	*Injection Volume:*	**20.0**
Vial Number:	**154**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**230**
Control Program:	**dm**	*Bandwidth:*	**n.a.**
Quantif. Method:	**dm**	*Dilution Factor:*	**1.0000**
Recording Time:	**2010-3-6 17:11**	*Sample Weight:*	**1.0000**
Run Time (min):	**46.45**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	24.03	n.a.	76.180	82.502	18.56	n.a.	BMB
2	27.64	n.a.	14.968	21.677	4.88	n.a.	Rd
3	35.31	n.a.	146.266	285.001	64.13	n.a.	BM *
4	39.75	n.a.	27.356	55.230	12.43	n.a.	MB*
Total:			264.769	444.410	100.00	0.000	