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Asymmetric Mannich Reaction: Highly Enantioselective Synthesis of 3-Amino-oxindoles via Chiral Squaramide Based *H*-bond Donor Catalysis

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1) General method

All reactions were carried out an inert atmosphere unless otherwise indicated, following standard syringe septa techniques. Chemicals were purchased from Aldrich and used as received. Solvents used for reactions were dried before use according to standard procedures where needed. Product purification by Flash chromatography was carried out with silica gel (60-120 mesh). Technical grade solvents were used for chromatography and distilled prior to use. NMR spectra were recorded in Fourier transform mode. ^{1}H and ^{13}C NMR spectra were recorded with Bruker-Avance 300 MHz, Varian Inova 400 MHz, Varian Inova 400 MHz spectrometers using trimethylsilane as an internal standard in $CDCl_3$, CD_3OD and DMSO-d_6 solvents. Multiplicities in the ^{1}H NMR spectra are described as: s = singlet, d = doublet, t = triplet, q = quartet, qt = quintet, m = multiplet, br = broad; coupling constants are reported in Hz. IR spectra were recorded on a Thermo Nicolet Nexus 670 spectrometer and reported in cm^{-1}. Mass spectra were recorded with a Waters 2695 for low (MS) and Thermo Scientific Exactive spectrometer for HRMS. Melting points were recorded on a Toshniwal melting point apparatus. Enantiomeric excesses were determined by using HPLC analysis with Daicel Chiralpak AD-H or IC columns, as indicated. The squaramide organocatalysts **1a-c**[1a,b], **1d-e**[1c] and **1f-i**[1d] were prepared following the literature procedures.

2) General Procedure for the Enantioselective Mannich Reactions

To a solution of ketimine **2** (0.25 mmol) in CH_2Cl_2 (1.5 ml) was added squaramide bifunctional catalyst **1g** (5 mg, 0.0075 mmol, 3 mol %) and the mixture was stirred for 5 min under N_2 atmosphere. Then 1,3-dicarbonyl compound **3** (0.3 mmol) was added to the mixture. Upon consumption of ketimine substrate (monitored by TLC), the reaction mixture was concentrated and purified by silica gel column chromatography to afford the conjugate addition products **4** (or) **5**.

Compound 4a: White solid; Yield 99%; $[\alpha]^{30}_{D}$ = -11.6 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3427, 2924, 1732, 1711, 1614, 1493, 1367, 1258, 1160, 1088, 1023, 752; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.34-7.25 (m, 2H), 7.02 (t, *J* = 7.5 Hz, 1H), 6.83 (d, *J* = 7.8 Hz, 1H), 6.49 (s, 1H), 4.09 (s, 1H), 3.24 (s, 3H), 2.29 (s, 3H), 2.17 (s, 3H), 1.26 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.7, 201.3, 174.0, 153.7, 143.3, 129.6, 128.1, 123.5, 122.8, 108.4, 80.3, 68.4, 62.6, 32.2, 32.1, 28.0, 26.5; ESI-MS: m/z: 383 $(M+Na)^+$, HR-MS calcd for $C_{19}H_{24}N_2O_5Na$: 383.1577 $[M+Na]^+$, found: 383.1571; HPLC (IC, 20% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 24.1 min, t_{minor} = 19.7 min, >99% ee.

Compound 4b: White solid; Yield 90%; $[\alpha]^{30}_{D}$ = +6.5 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3363, 2975, 1725, 1669, 1620, 1472, 1398, 1363, 1165, 1060, 757; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 8.50 (s, 1H), 7.29-7.16 (m, 2H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.79 (d, *J* = 7.5 Hz, 1H), 6.73 (s, 1H), 4.12 (s, 1H), 2.34 (s, 3H), 2.16 (s, 3H), 1.32 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.3, 201.0, 175.8, 154.0, 140.6, 129.5, 123.8, 122.8, 110.6, 80.7, 67.8, 63.1, 32.5, 32.4,

28.1; ESI-MS: m/z: 369 $(M+Na)^+$, HR-MS calcd for $C_{18}H_{22}N_2O_5Na$: 369.1421 $[M+Na]^+$, found: 369.1410; HPLC (IC, 20% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 11.3 min, t_{minor} = 14.1 min, 86% ee.

Compound 4c: White solid; Yield 92%; $[\alpha]^{30}_D$ = -29.7 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3411, 2978, 1716, 1611, 1488, 1467, 1363, 1279, 1248, 1164, 755; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.33-7.27 (m, 2H), 7.01 (t, *J* = 7.6 Hz, 1H), 6.84 (d, *J* = 7.7 Hz, 1H), 6.39 (s, 1H), 4.13 (s, 1H), 3.96-3.84 (m, 1H), 3.72-3.60 (m, 1H), 2.26 (s, 3H), 2.19 (s, 3H), 1.33-1.24 (m, 12H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.6, 201.5, 173.6, 153.8, 142.5, 129.5, 128.4, 123.9, 122.6, 108.5, 80.4, 68.5, 62.5, 35.1, 32.2, 32.1, 29.6, 28.1, 12.3; ESI-MS: m/z: 397 $(M+Na)^+$, HR-MS calcd for $C_{20}H_{26}N_2O_5Na$: 397.1734 $[M+Na]^+$, found: 397.1721; HPLC (IC, 30% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 14.1 min, t_{minor} = 9.2 min, 92% ee.

Compound 4d: White solid; Yield 96%; $[\alpha]^{30}_D$ = -9.3 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3383, 2974, 1730, 1708, 1613, 1496, 1362, 1278, 1249, 1165, 748; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.41 (d, *J* = 7.6 Hz, 2H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.28 (t, *J* = 7.6 Hz, 2H), 7.18 (t, *J* = 7.7 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.72 (d, *J* = 7.7 Hz, 1H), 6.58 (s, 1H), 5.05 (d, *J* = 15.1 Hz, 1H), 4.83 (s, 1H), 4.07 (s, 1H), 2.31 (s, 3H), 2.16 (s, 3H), 1.31 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.6, 201.3, 174.2, 153.8, 142.5, 135.5, 129.4, 128.7, 127.6. 127.5, 123.6, 122.9, 109.5, 80.4, 68.5, 62.7, 44.3, 32.3, 32.2, 28.1; ESI-MS: m/z: 459 $(M+Na)^+$, HR-MS calcd for $C_{25}H_{28}N_2O_5Na$: 459.1890 $[M+Na]^+$, found: 459.1875; HPLC (IC, 5% iPrOH in Hexanes, 0.5 mL/min, 210 nm): t_{major} = 105.6 min, t_{minor} = 98.3 min, 99% ee.

Compound 4e: White solid; Yield 85%; $[\alpha]^{30}_D$ = -6.6 (c = 0.1 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3404, 2979, 1766, 1708, 1471, 1368, 1337, 1273, 1168, 759; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 8.24 (d, *J* = 8.2 Hz, 1H), 7.36 (t, *J* = 7.4, 8.2 Hz, 1H), 7.26 (d, *J* = 7.4 Hz, 1H), 7.18 (t, *J* = 7.4, 7.6 Hz, 1H), 6.64 (s, 1H), 4.05 (s, 1H), 2.70 (s, 3H), 2.24 (s, 3H), 2.18 (s, 3H), 1.26 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.8, 200.7, 175.0, 170.6, 153.6, 140.0, 130.0, 127.5, 125.4, 122.5, 116.8, 81.2, 69.5, 63.0, 32.1, 32.0, 27.9, 26.6; ESI-MS: m/z: 411 $(M+Na)^+$, HR-MS calcd for $C_{20}H_{24}N_2O_6Na$: 411.1532 $[M+Na]^+$, found: 411.1518; HPLC (IC, 20% iPrOH in Hexanes, 1 mL/min, 225 nm): t_{major} = 6.8 min, t_{minor} = 10.3 min, 33% ee.

Compound 4f: White solid; Yield 91%; $[\alpha]^{30}_D$ = -36.8 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3448, 2986, 1729, 1710, 1611, 1490, 1363, 1276, 1168, 756; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.33-7.22 (m, 2H), 7.01 (t, *J* = 7.3, 7.5 Hz, 1H), 6.83 (d, *J* = 7.7 Hz, 1H), 6.47 (s, 1H), 5.98-5.81 (m, 1H), 5.38 (d, *J* = 16.4 Hz, 1H), 5.26 (d, *J* = 10.3 Hz, 1H), 4.50 (d, *J* = 15.3 Hz, 1H), 4.23 (d, *J* = 14.7 Hz, 1H), 4.12 (s, 1H), 2.27 (s, 3H), 2.19 (s, 3H), 1.29 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.6, 201.4, 173.8, 153.8, 142.5, 131.2, 129.4, 128.1, 123.7, 122.8, 117.8, 109.3, 80.4, 68.4, 62.6, 42.7, 32.3, 32.1, 28.1; ESI-MS: m/z: 409 $(M+Na)^+$, HR-MS calcd for $C_{21}H_{26}N_2O_5Na$: 409.0716 $[M+Na]^+$, found: 409.0678; HPLC (IC, 15% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 23.2 min, t_{minor} = 18.9 min, 96% ee.

Compound 4g: White solid; Yield 93%; $[\alpha]^{30}_{D}$ = -43.6 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3440, 3266, 2978, 1730, 1710, 1612, 1489, 1363, 1278, 1259, 1168, 764, 707; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.36-7.29 (m, 2H), 7.09-7.04 (m, 2H), 6.50 (s, 1H), 4.81 (dd, *J* = 2.4, 17.7 Hz, 1H), 4.27 (dd, *J* = 2.4, 17.7 Hz, 1H), 4.13 (s, 1H), 2.30 (t, *J* = 2.4 Hz, 1H), 2.29 (s, 3H), 2.18 (s, 3H), 1.28 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.1, 173.1, 153.8, 141.4, 129.6, 128.0, 124.0, 123.3, 109.4, 80.5, 76.6, 72.6, 68.1, 62.5, 32.3, 32.1, 29.7, 28.0; ESI-MS: m/z: 407 $(M+Na)^+$, HR-MS calcd for $C_{21}H_{24}N_2O_5Na$: 407.1577 $[M+Na]^+$, found: 407.1560; HPLC (IC, 20% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 17.1 min, t_{minor} = 15.5 min, 98% ee.

Compound 5a: White solid; Yield 94%; $[\alpha]^{30}_{D}$ = -13.8 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3408, 2925, 1718, 1614, 1498, 1361, 1255, 1164, 1098, 1059, 812, 756; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.13-7.06 (m, 2H), 6.72 (d, *J* = 7.9 Hz, 1H), 6.51 (s, 1H), 4.07 (s, 1H), 3.22 (s, 3H), 2.30 (s, 6H), 2.17 (s, 3H), 1.27 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.8, 201.4, 174.0, 153.8, 141.0, 132.5, 129.8, 128.2, 124.3, 108.2, 80.3, 68.6, 62.7, 32.2, 32.1, 29.6, 28.0, 26.6, 21.1; ESI-MS: m/z: 397 $(M+Na)^+$; HPLC (IC, 30% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 22.0 min, t_{minor} = 15.0 min, 96% ee.

Compound 5b: White solid; Yield 97%; $[\alpha]^{30}_{D}$ = -23.2 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3421, 2925, 1727, 1604, 1497, 1463, 1362, 1286, 1164, 1123, 1033, 809; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 6.95-6.78 (m, 2H), 6.73 (d, *J* = 8.5 Hz, 1H), 6.50 (s, 1H), 4.09 (s, 1H), 3.76 (s, 3H), 3.22 (s, 3H), 2.30 (s, 3H), 2.18 (s, 3H), 1.28 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.5, 201.2, 173.7, 156.1, 153.7, 136.7, 129.4, 113.8, 111.1, 108.8, 80.3, 68.4, 63.0, 55.8, 32.2, 32.0, 29.6, 28.0, 26.6; ESI-MS: m/z: 413 $(M+Na)^+$; HPLC (IC, 30% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 33.2 min, t_{minor} = 16.2 min, 96% ee.

Compound 5c: White solid; Yield 92%; $[\alpha]^{30}_{D}$ = -16.5 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3431, 2981, 1735, 1707, 1622, 1495, 1361, 1275, 1164, 1123, 1059, 1022, 865, 814; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.10 (dd, *J* = 2.6, 7.9 Hz, 1H), 7.04-6.98 (m, 1H), 6.76 (dd, *J* = 4.1, 8.5 Hz, 1H), 6.43 (s, 1H), 4.12 (s, 1H), 3.24 (s, 3H), 2.30 (s, 3H), 2.19 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.2, 201.1, 173.8, 160.1, 158.2, 153.7, 139.4, 115.9, 115.7, 112.3, 112.1, 109.0, 80.6, 68.2, 62.7, 32.3, 31.9, 28.0, 26.7; ESI-MS: m/z: 401 $(M+Na)^+$; HPLC (IC, 30% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 12.3 min, t_{minor} = 9.0 min, 95% ee.

Compound 5d: White solid; Yield 95%; $[\alpha]^{30}_{D}$ = -36.6 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3407, 2924, 1737, 1703, 1610, 1487, 1423, 1359, 1267, 1168, 1055, 1022, 819; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.33-7.25 (m, 2H), 6.77 (d, *J* = 9.1 Hz, 1H), 6.46 (s, 1H), 4.10 (s, 1H), 3.23 (s, 3H), 2.30 (s, 3H), 2.19 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.1, 173.6, 153.6, 142.0, 129.7, 129.5, 128.2, 124.3, 109.4, 80.6, 68.2, 62.4, 32.2, 32.0, 28.0, 26.6; ESI-MS: m/z: 417 $(M+Na)^+$; HPLC (IC, 30% iPrOH in Hexanes, 1 mL/min, 230 nm): t_{major} = 12.1 min, t_{minor} = 8.5 min, 95% ee.

Compound 5e: White solid; Yield 91%; $[\alpha]^{30}_{D}$ = -36.4 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3403, 2980, 1736, 1694, 1607, 1487, 1423, 1357, 1266, 1167, 1055, 1022, 818; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.46-7.40 (m, 2H), 6.72 (d, *J* = 8.7 Hz, 1H), 6.45 (s, 1H), 4.09 (s, 1H), 3.23 (s, 3H), 2.30 (s, 3H), 2.19 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.2, 201.1, 173.5, 153.7, 142.5, 132.4, 130.1, 127.0, 115.5, 110.0, 80.7, 68.3, 62.4, 32.3, 32.0, 28.0, 26.7; ESI-MS: m/z: 463 $(M+Na)^+$; HPLC (IC, 30% iPrOH in Hexanes, 1 mL/min, 230 nm): t_{major} = 12.6 min, t_{minor} = 8.7 min, 95% ee.

Compound 5f: White solid; Yield 93%; $[\alpha]^{30}_{D}$ = -39.7 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3389, 2977, 1738, 1689, 1603, 1483, 1421, 1357, 1262, 1168, 1055, 1022, 816; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.63 (dd, *J* = 1.6, 8.1 Hz, 1H), 7.57 (d, *J* = 1.6 Hz, 1H), 6.62 (d, *J* = 8.2 Hz, 1H), 6.45 (s, 1H), 4.06 (s, 1H), 3.22 (s, 3H), 2.29 (s, 3H), 2.19 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.2, 201.1, 173.3, 153.6, 143.2, 138.4, 132.3, 130.3, 110.5, 85.2, 80.7, 68.2, 62.2, 32.3, 32.0, 29.6, 28.0, 26.6; ESI-MS: m/z: 509 $(M+Na)^+$; HPLC (IC, 20% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 21.4 min, t_{minor} = 15.3 min, 95% ee.

Compound 5g: White solid; Yield 99%; $[\alpha]^{30}_{D}$ = +22.7 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3413, 2924, 1738, 1706, 1604, 1459, 1361, 1252, 1167, 1107, 1055, 1024, 775; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.41 (d, *J* = 8.2 Hz, 1H), 7.19 (d, *J* = 7.4 Hz, 1H), 6.86 (t, *J* = 7.3, 8.2 Hz, 1H), 6.57 (s, 1H), 4.02 (s, 1H), 3.62 (s, 3H), 2.29 (s, 3H), 2.16 (s, 3H), 1.29 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.4, 200.8, 174.6, 153.6, 140.6, 135.2, 131.4, 124.0, 122.4, 102.8, 80.7, 68.2, 62.2, 32.5, 31.8, 30.2, 29.6, 29.3, 28.0; ESI-MS: m/z: 463 $(M+Na)^+$; HPLC (IC, 40% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 11.6 min, t_{minor} = 9.9 min, 96% ee.

Compound 5h: White solid; Yield 89%; $[\alpha]^{30}_{D}$ = +37.1 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3415, 2977, 1736, 1613, 1520, 1492, 1335, 1297, 1165, 1114, 1070, 755; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 8.34-8.18 (m, 2H), 6.93 (d, *J* = 8.6 Hz, 1H), 6.45 (s, 1H), 4.15 (s, 1H), 3.32 (s, 3H), 2.28 (s, 3H), 2.23 (s, 3H), 1.31 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.1, 200.8, 174.5, 153.7, 149.3, 143.5, 133.0, 132.1, 128.5, 126.7, 119.7, 108.0, 81.2, 68.4, 61.8, 32.1, 31.8, 28.1, 27.0; ESI-MS: m/z: 428 $(M+Na)^+$; HPLC (IC, 30% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 15.4 min, t_{minor} = 9.8 min, 92% ee.

Compound 5i: White solid; Yield 98%; $[\alpha]^{30}_{D}$ = -18.6 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3412, 2925, 1721, 1620, 1495, 1362, 1255, 1218, 1162, 1059, 1026, 758; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.24-7.16 (m, 2H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.44 (s, 1H), 4.11 (s, 1H), 3.25 (s, 3H), 2.28 (s, 3H), 2.18 (s, 3H), 1.29 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.1, 201.0, 174.0, 153.7, 144.7, 142.2, 129.5, 122.8, 118.1, 109.0, 80.8, 68.0, 62.5, 32.2, 32.0, 31.8, 29.6, 29.3, 28.0, 26.7; ESI-MS: m/z: 467 $(M+Na)^+$; HPLC (IC, 40% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 6.3 min, t_{minor} = 5.4 min, 97% ee.

Compound 5j: White solid; Yield 78%; $[\alpha]^{30}_{D}$ = +8.9 (c = 0.1 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3362, 2924, 1721, 1694, 1601, 1508, 1454, 1350, 1319, 1254, 1154, 1114, 1039, 805; ^{1}H-NMR

(300 MHz, $CDCl_3$), δ (ppm) 7.16 (d, *J* = 8.7 Hz, 1H), 6.85 (d, *J* = 8.8 Hz, 1H), 6.36 (s, 1H), 4.47 (s, 1H), 3.63 (s, 3H), 2.31 (s, 3H), 2.08 (s, 3H), 1.29 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 203.3, 202.0, 173.6, 153.0, 142.3, 133.1, 128.0, 124.0, 114.6, 80.7, 66.0, 62.6, 32.2, 31.8, 31.4, 30.1, 29.6, 29.3, 28.0; ESI-MS: m/z: 451 $(M+Na)^+$; HPLC (IC, 35% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 12.8 min, t_{minor} = 9.6 min, 94% ee.

Compound 5k: White solid; Yield 90%; $[\alpha]^{30}_D$ = -23.7 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3291, 2924, 1755, 1725, 1613, 1523, 1466, 1381, 1244, 1163, 1015, 751; ^1H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.37 (d, *J* = 7.3 Hz, 1H), 7.31 (t, *J* = 7.7 Hz, 1H), 7.03 (t, *J* = 7.6 Hz, 1H), 6.83 (d, *J* = 7.7 Hz, 1H), 6.32 (s, 1H), 3.96 (s, 1H), 3.70 (s, 3H), 3.69 (s, 3H), 3.25 (s, 3H), 1.27 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 173.8, 166.3, 166.0, 153.7, 144.0, 129.6, 127.6, 123.8, 122.6, 108.2, 80.3, 60.7, 55.2, 53.0, 28.0, 26.6; ESI-MS: m/z: 415 $(M+Na)^+$; HPLC (IC, 30% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 18.4 min, t_{minor} = 13.9 min, 97% ee.

Compound 5l: White solid; Yield 93%; $[\alpha]^{30}_D$ = -23.5 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3433, 2981, 1724, 1682, 1610, 1451, 1372, 1348, 1287, 1246, 1162, 1026, 758, 692; ^1H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.87 (d, *J* = 7.3 Hz, 2H), 7.68 (d, *J* = 7.3 Hz, 2H), 7.56-7.51 (m, 1H), 7.48 (t, *J* = 7.3, 7.4 Hz, 2H), 7.41 (t, *J* = 7.4, 8.2 Hz, 2H), 7.35 (t, *J* = 7.4, 8.2 Hz, 2H), 7.27-7.23 (m, 1H), 6.96 (t, *J* = 8.1 Hz, 1H), 6.79 (s, 1H), 6.75 (d, *J* = 7.6 Hz, 1H), 5.87 (s, 1H), 2.98 (s, 3H), 1.26 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 192.5, 192.2, 174.2, 153.8, 143.0, 136.6, 136.4, 133.7, 129.2, 128.8, 128.6, 128.4, 125.4, 122.8, 108.1, 80.1, 63.8, 57.1, 29.6, 28.0, 26.1; ESI-MS: m/z: 507 $(M+Na)^+$; HPLC (AD-H, 30% iPrOH in Hexanes, 1 mL/min, 254 nm): t_{major} = 10.2 min, t_{minor} = 9.2 min, 96% ee.

Compound 5m: White solid; Yield 91%; $[\alpha]^{30}_D$ = -25.5 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3428, 2925, 1710, 1609, 1491, 1370, 1341, 1280, 1158, 1055, 1027, 765; ^1H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.39 (d, *J* = 7.3 Hz, 0.5H), 7.31 (t, *J* = 7.7 Hz, 1.5H), 7.02 (t, *J* = 7.7 Hz, 1H), 6.83 (d, *J* = 7.8 Hz, 1H), 6.36 (s, 0.5H), 6.29 (s, 0.5H), 4.08 (s, 0.5H), 3.96 (s, 0.5H), 3.71 (s, 1.5H), 3.68 (s, 1.5H), 3.25 (s, 1.5H), 3.24 (s, 1.5H), 2.26 (s, 1.5H). 2.17 (s, 1.5H), 1.27 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 200.0, 174.1, 166.5, 153.6, 143.9, 129.6, 127.8, 123.9, 122.7, 108.3, 80.3, 61.6, 60.4, 52.9, 32.1, 31.1, 29.6, 28.0, 26.6; ESI-MS: m/z: 399 $(M+Na)^+$; HPLC (IC, 30% iPrOH in Hexanes, 1.0 mL/min, 254 nm): major diastereomer: t_{major} = 28.5 min, t_{minor} = 15.6 min, 97% ee, minor diastereomer: t_{major} = 34.1 min, t_{minor} = 17.3 min, 95% ee.

Compound 5n: White solid; Yield 95%; $[\alpha]^{30}_D$ = -40.1 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3415, 2978, 1722, 1612, 1494, 1369, 1250, 1166, 1093, 1022, 755; ^1H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.42 (d, *J* = 7.4 Hz, 0.5H), 7.31 (dd, *J* = 7.9, 16.6 Hz, 1.5H), 7.02 (t, *J* = 7.4 Hz, 1H), 6.83 (d, *J* = 7.8 Hz, 1H), 6.42 (s, 0.5H), 6.34 (s, 0.5H), 4.17-4.09 (m, 2.5H), 3.97 (s, 0.5H), 3.24 (s, 3H), 2.27 (s, 1.5H), 2.18 (s, 1.5H), 1.26 (s, 9H), 1.16 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 200.6, 174.0, 166.0, 153.5, 144.0, 129.5, 128.0, 123.8, 122.5, 108.1, 80.1, 62.0, 60.6, 32.0, 31.0, 29.5, 28.0, 26.4, 13.7; ESI-MS: m/z: 413 $(M+Na)^+$; HPLC (AD-H, 5% iPrOH in

Hexanes, 1.0 mL/min, 254 nm): major diastereomer: t_{major} = 54.7 min, t_{minor} = 52.7 min, 99% ee, minor diastereomer: t_{major} = 40.9 min, t_{minor} = 29.7 min, 97% ee.

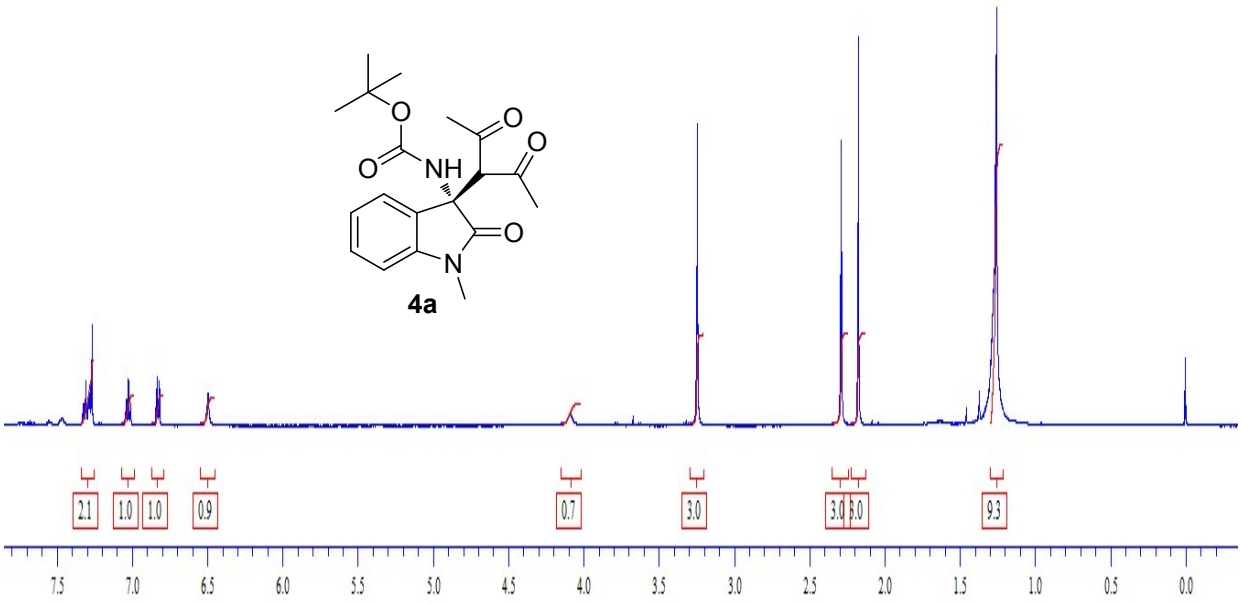
Compound 5o: White solid; Yield 92%; $[\alpha]^{30}_{D}$ = -48.2 (c = 0.4 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3433, 2980, 1715, 1611, 1498, 1368, 1282, 1253, 1162, 1130, 1025, 756; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.46 (d, *J* = 7.5 Hz, 0.5H), 7.35 (d, *J* = 7.5 Hz, 0.5H), 7.30 (t, *J* = 7.7 Hz, 1H), 7.02 (t, *J* = 7.5 Hz, 1H), 6.81 (d, *J* = 7.7 Hz, 1H), 6.36 (s, 0.5H), 6.26 (s, 0.5H), 3.97 (s, 0.5H), 3.86 (s, 0.5H), 3.24 (s, 3H), 2.28 (s, 1.5H), 2.21 (s, 1.5H), 1.31 (s, 9H), 1.27 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 201.3, 174.1, 164.9, 153.6, 143.9, 129.4, 124.6, 124.0, 122.6, 122.5, 108.1, 83.4, 80.2, 63.0, 62.3, 61.7, 31.6, 30.8, 28.1, 27.5, 26.5; ESI-MS: m/z: 441 $(M+Na)^+$; HPLC (IC, 25% iPrOH in Hexanes, 1.0 mL/min, 254 nm): major diastereomer: t_{major} = 23.5 min, t_{minor} = 12.4 min, 89% ee, minor diastereomer: t_{major} = 31.2 min, t_{minor} = 17.1 min, 90% ee.

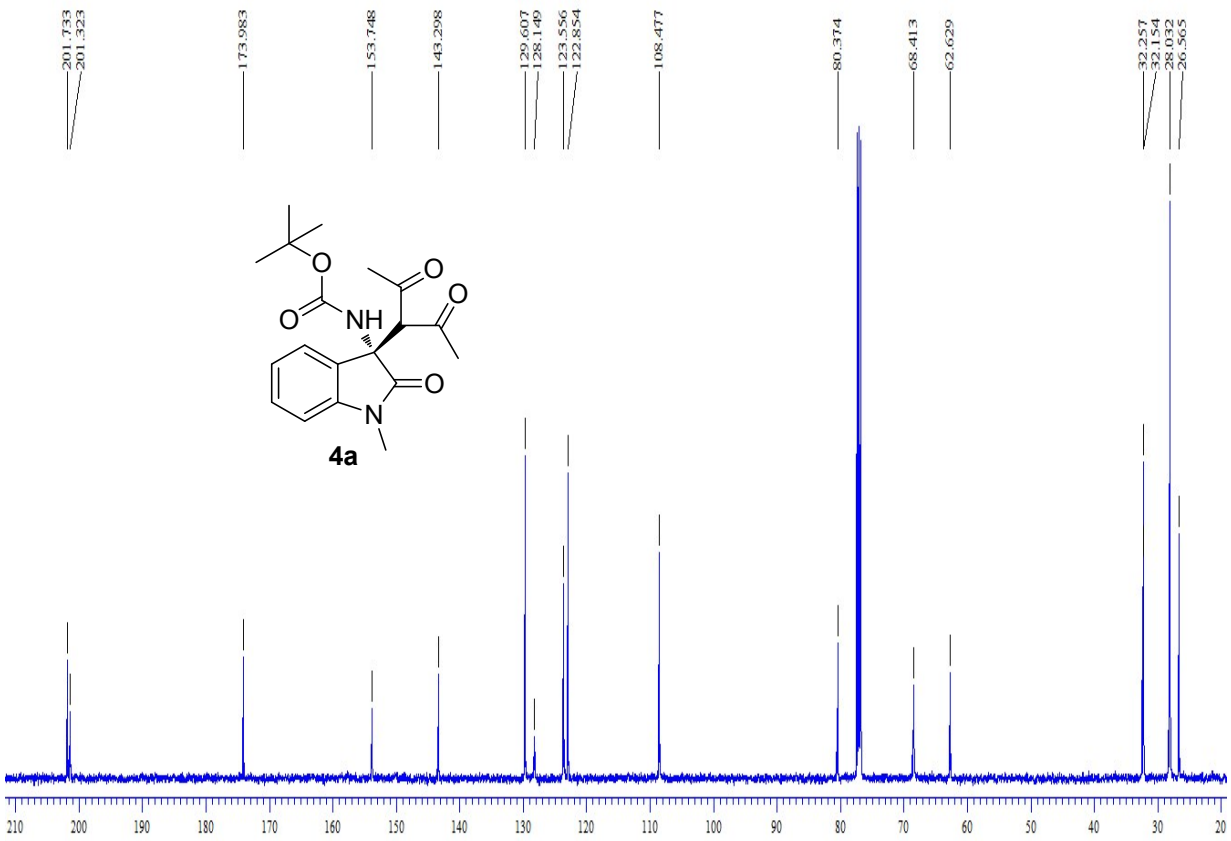
Compound 5p: White solid; Yield 96%; $[\alpha]^{30}_{D}$ = -62.7 (c = 0.5 in $CHCl_3$); IR (KBr) ν_{max} (cm^{-1}) 3403, 2925, 1713, 1674, 1611, 1455, 1353, 1254, 1171, 1025, 754, 691; ^{1}H-NMR (300 MHz, $CDCl_3$), δ (ppm) 7.76 (d, *J* = 7.3 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 2H), 7.26-7.23 (m, 1H), 7.18 (d, *J* = 7.4 Hz, 1H), 7.03 (s, 1H), 6.91 (t, *J* = 7.4 Hz, 1H), 6.78 (d, *J* = 7.7 Hz, 1H), 4.80 (s, 1H), 3.12 (s, 3H), 2.37 (s, 3H), 1.27 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$), δ (ppm) 200.9, 194.1, 173.9, 153.7, 143.1, 136.5, 134.2, 129.4, 128.8, 128.6, 123.9, 122.8, 108.2, 80.3, 63.5, 31.3, 28.0, 26.4; ESI-MS: m/z: 445 $(M+Na)^+$; HPLC (IC, 25% iPrOH in Hexanes, 1.0 mL/min, 254 nm): major diastereomer: t_{major} = 35.8 min, t_{minor} = 15.7 min, 95% ee, minor diastereomer: t_{major} = 13.0 min, t_{minor} = 11.6 min, 82% ee.

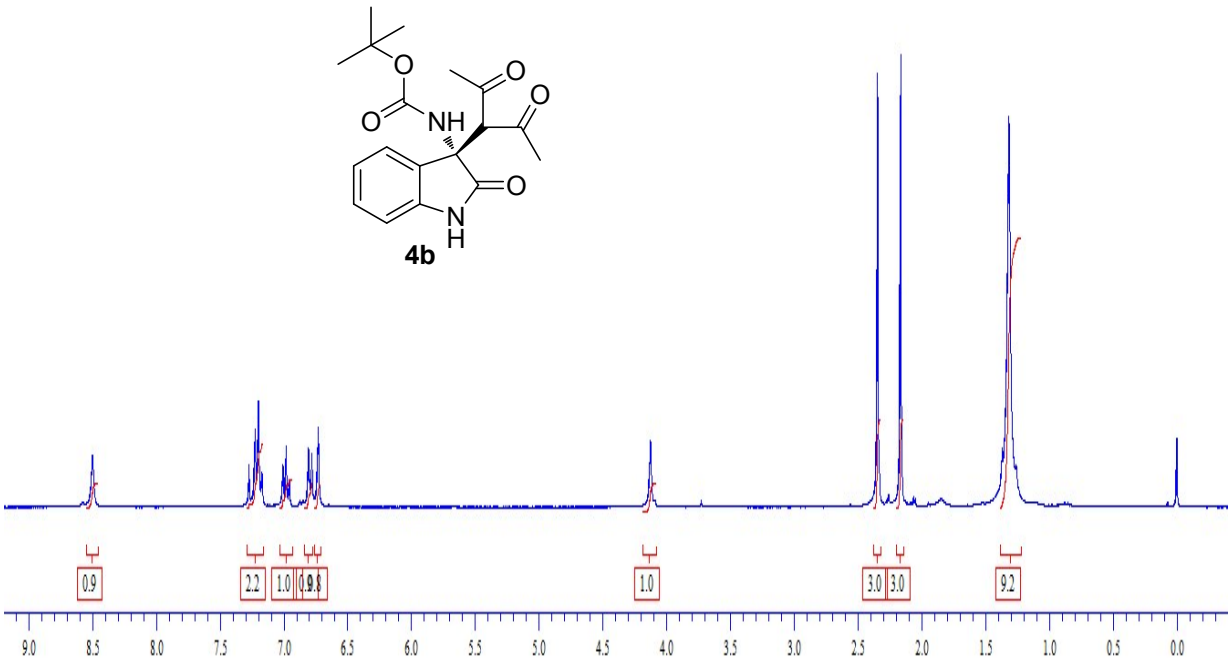
References:

1) (a) J. P. Malerich, K. Hagihara and V. H. Rawal, *J. Am. Chem. Soc*., 2008, **130**, 14416; (b) W. Yang and D. -M. Du, *Org. Lett*., 2010, **12**, 5450; (c) K. S. Rao, R. Trivedi and M. L. Kantam, *Synlett,* 2015, **26**, 221; (d) K. S. Rao, P. Ramesh, R. Trivedi and M. L. Kantam, *Tetrahedron Lett.,* 2016, **57**, 1227.

3) ^{1}H NMR, ^{13}C NMR spectra of products 4a-g and 5a-p



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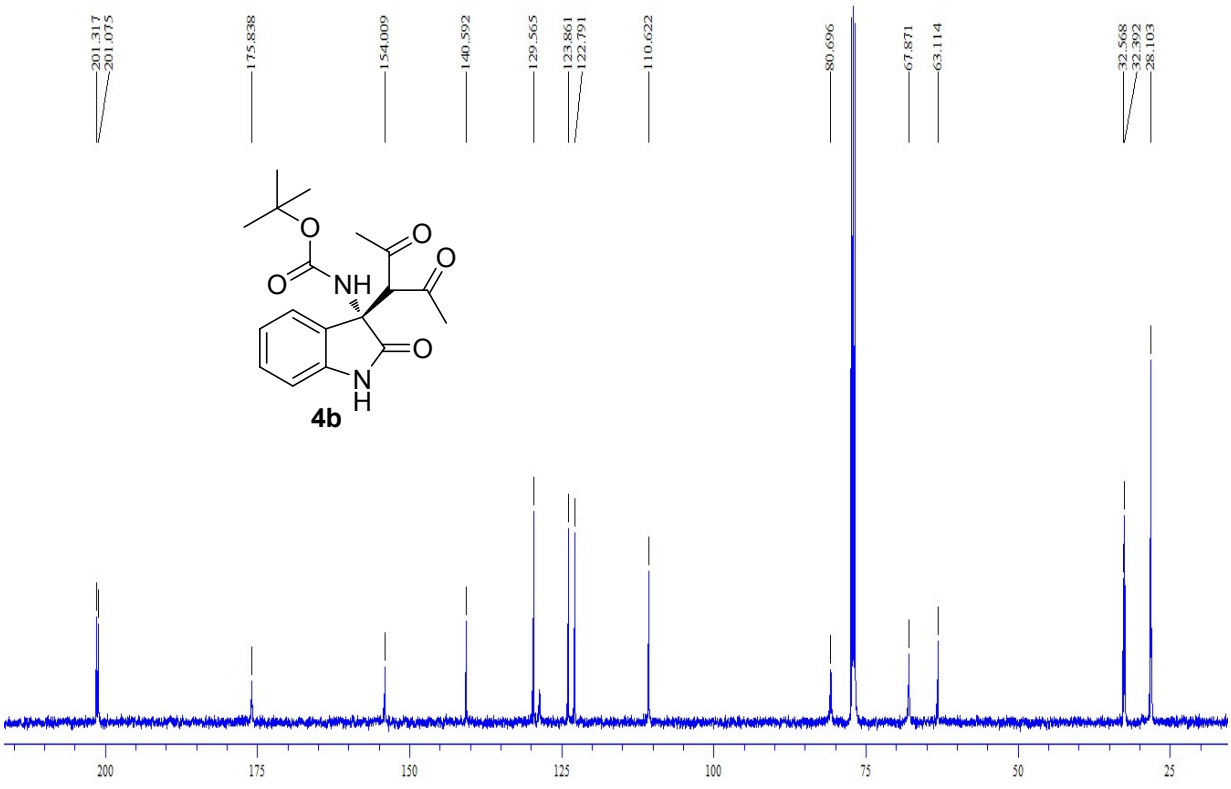
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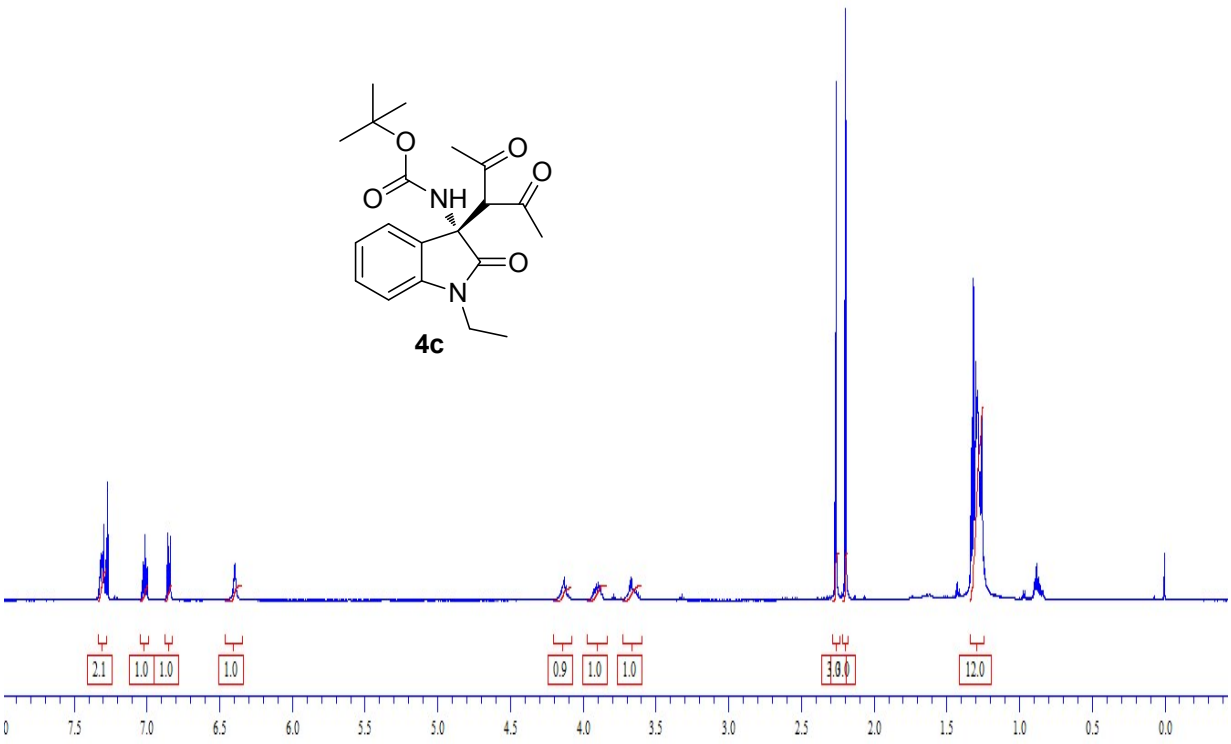
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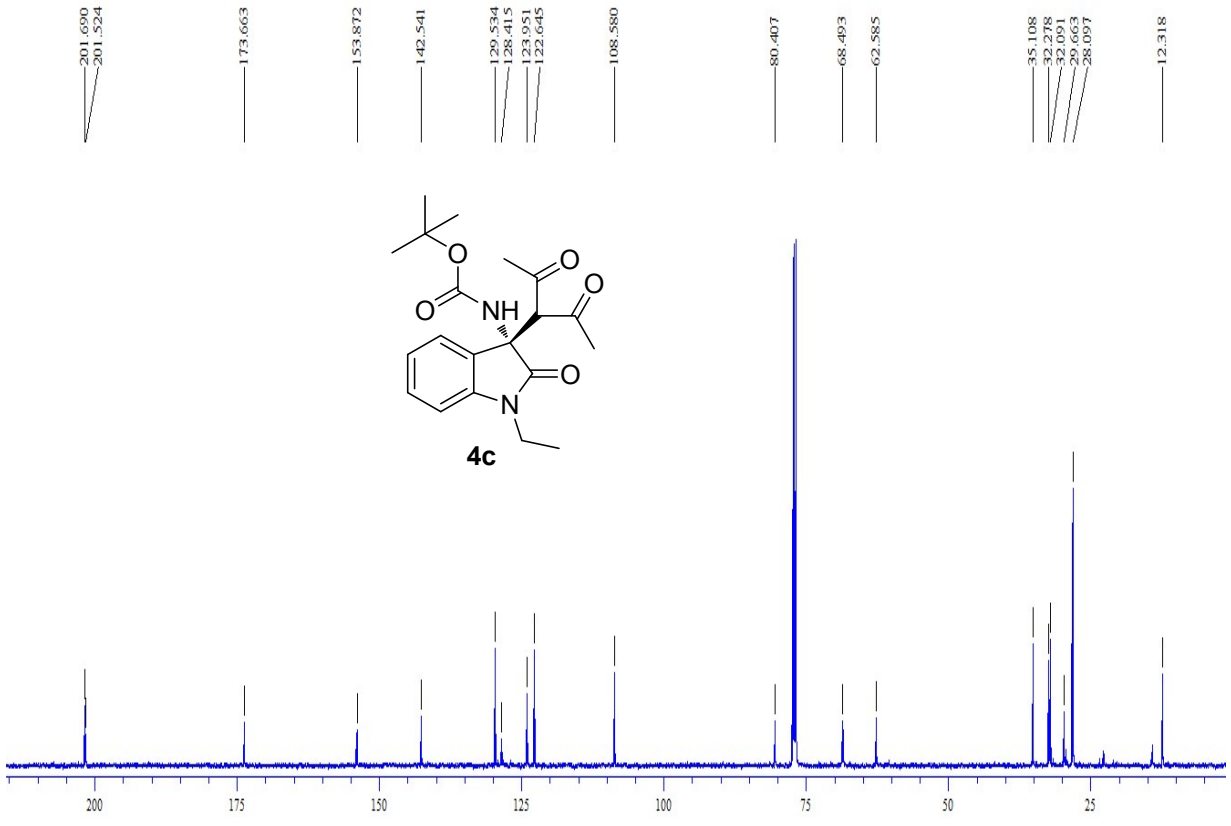
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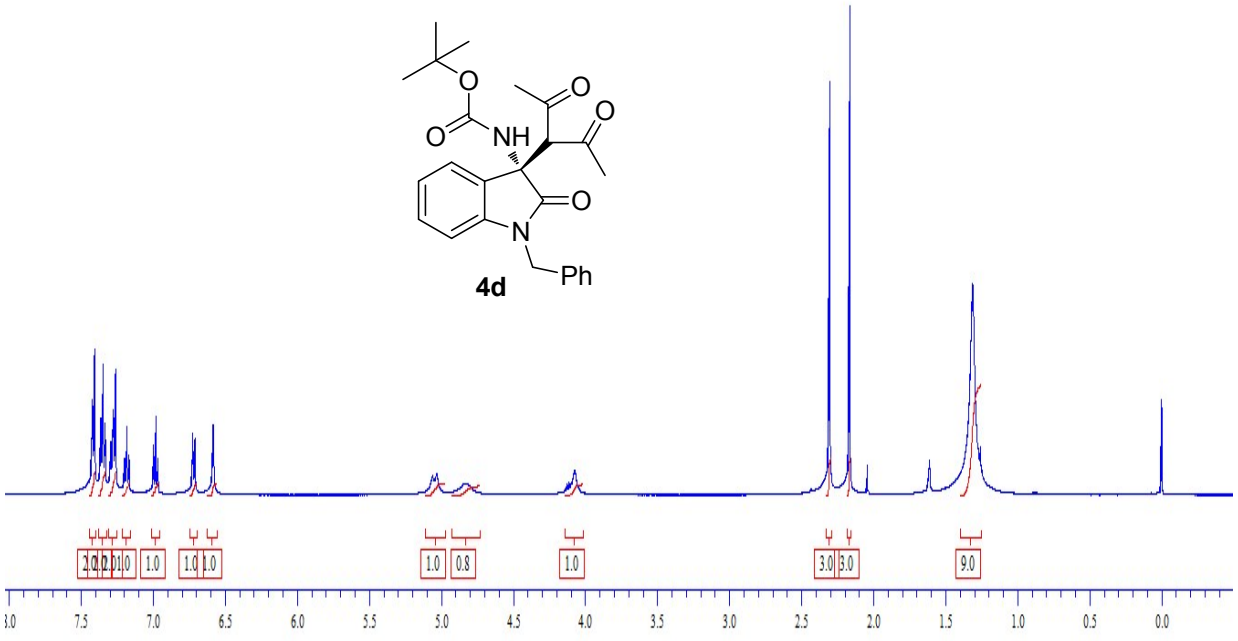
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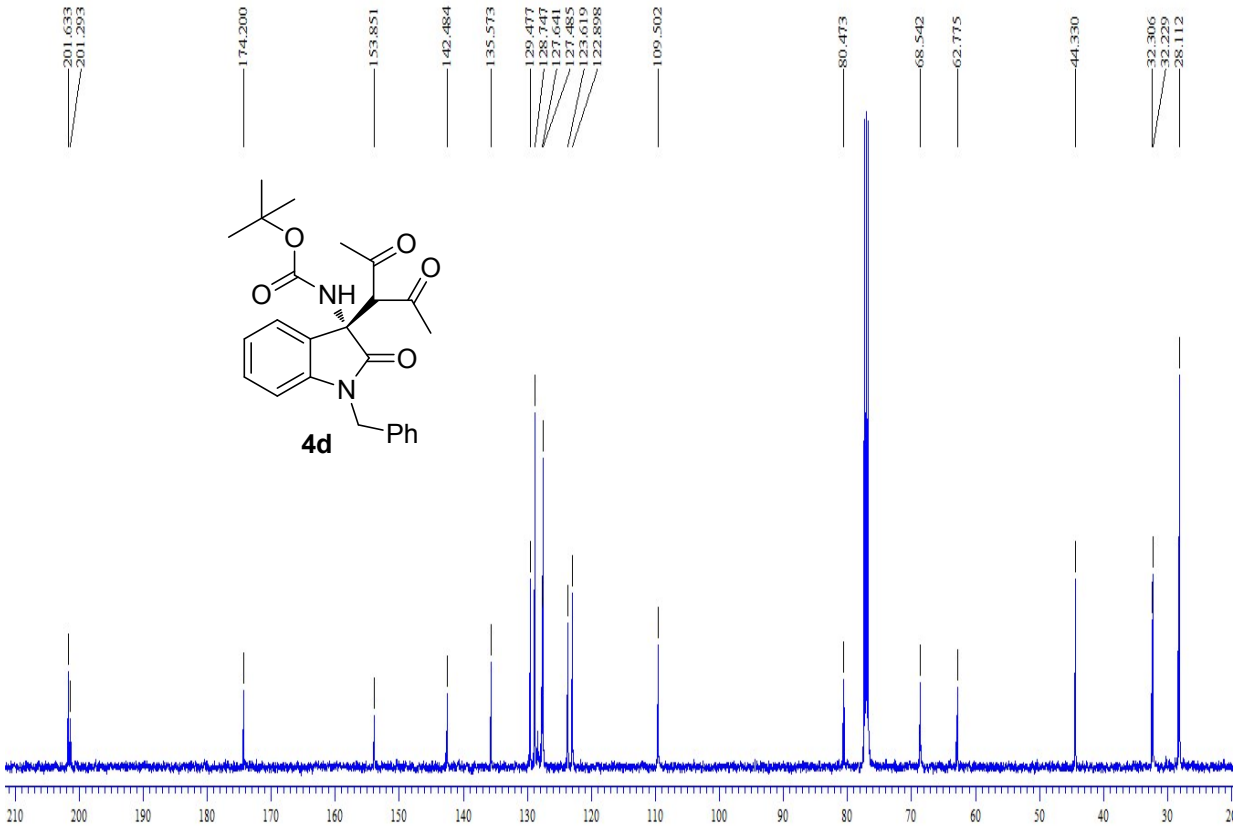
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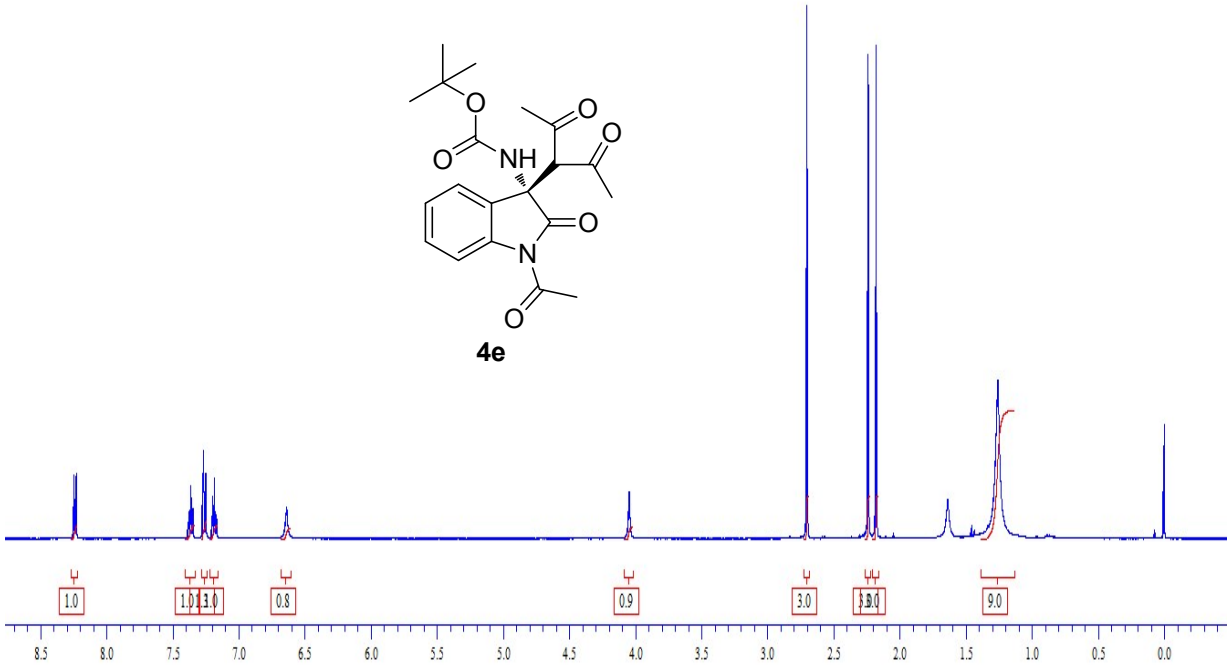
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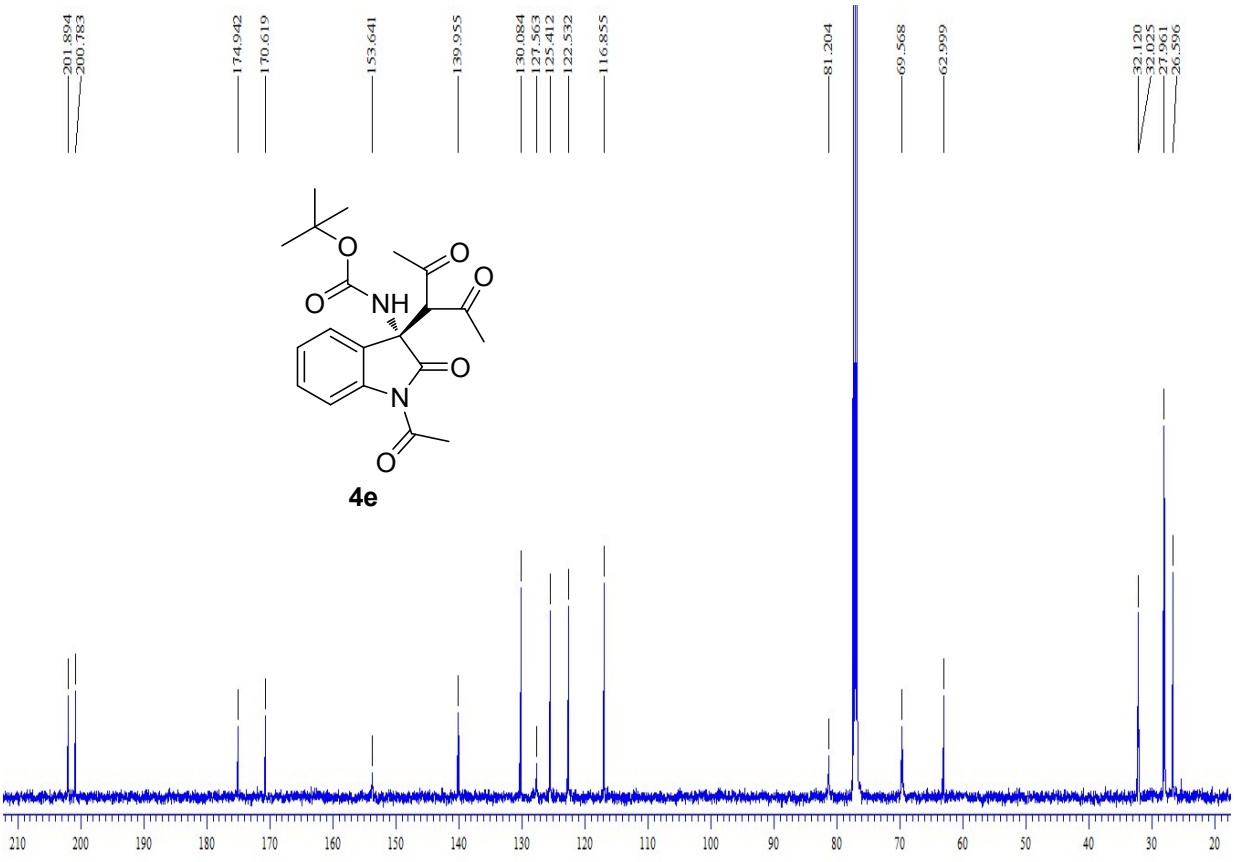
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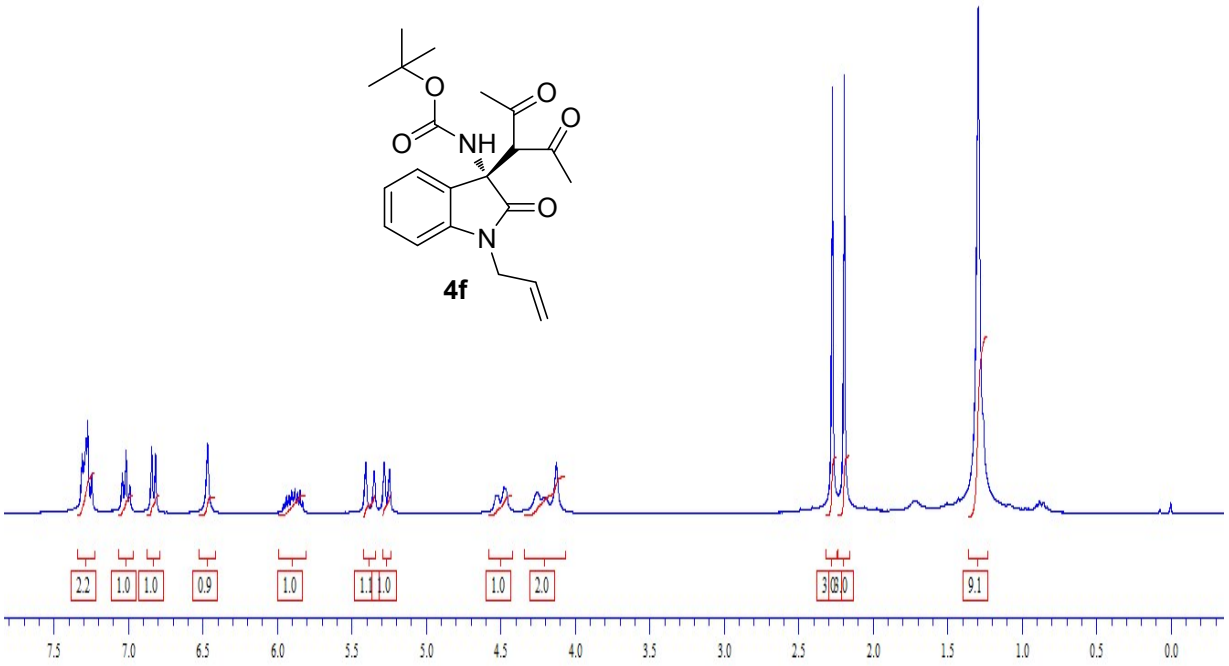
4e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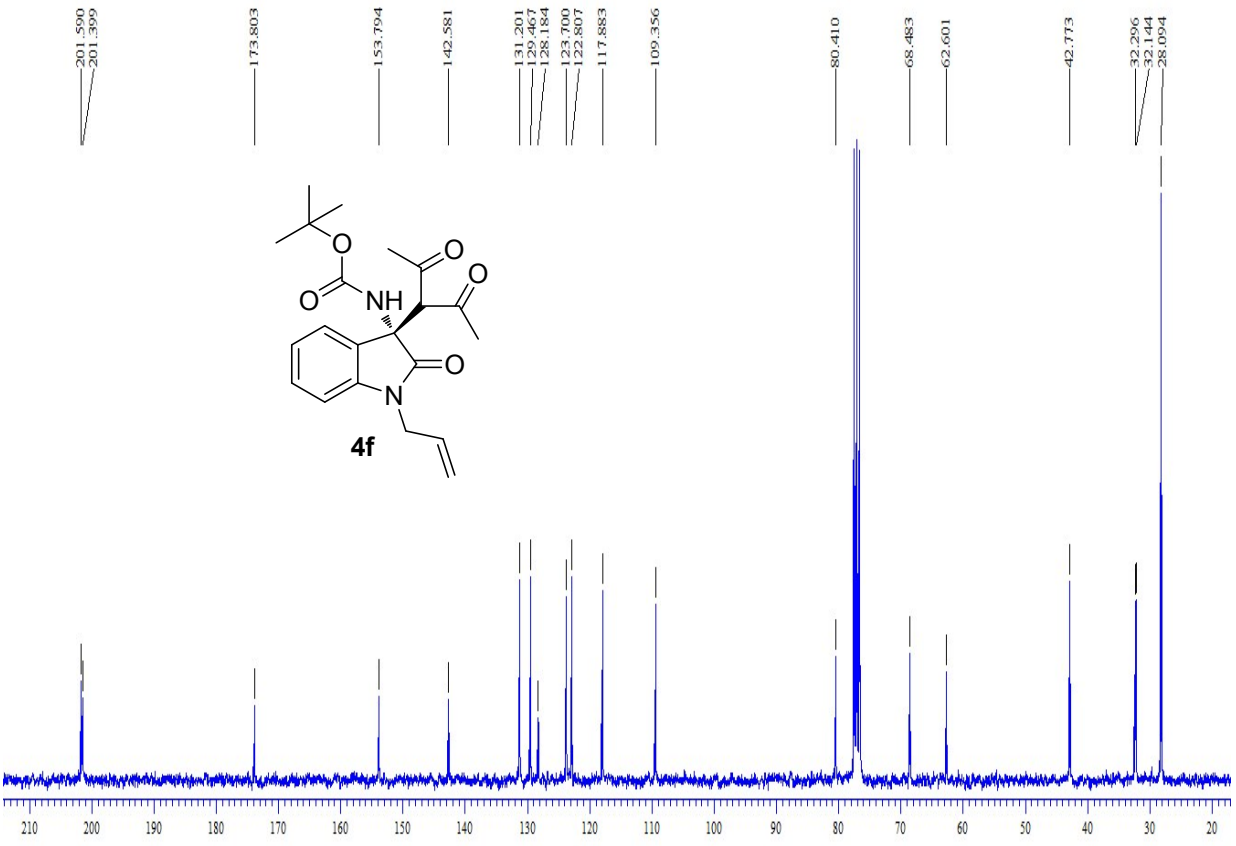
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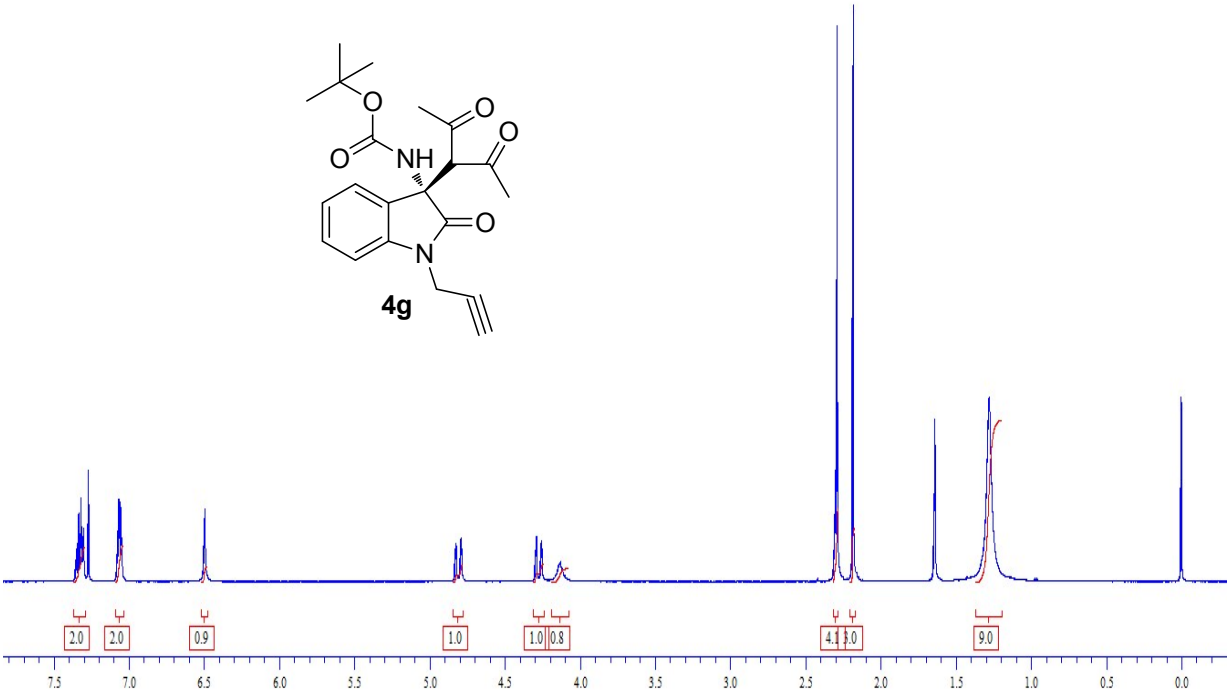
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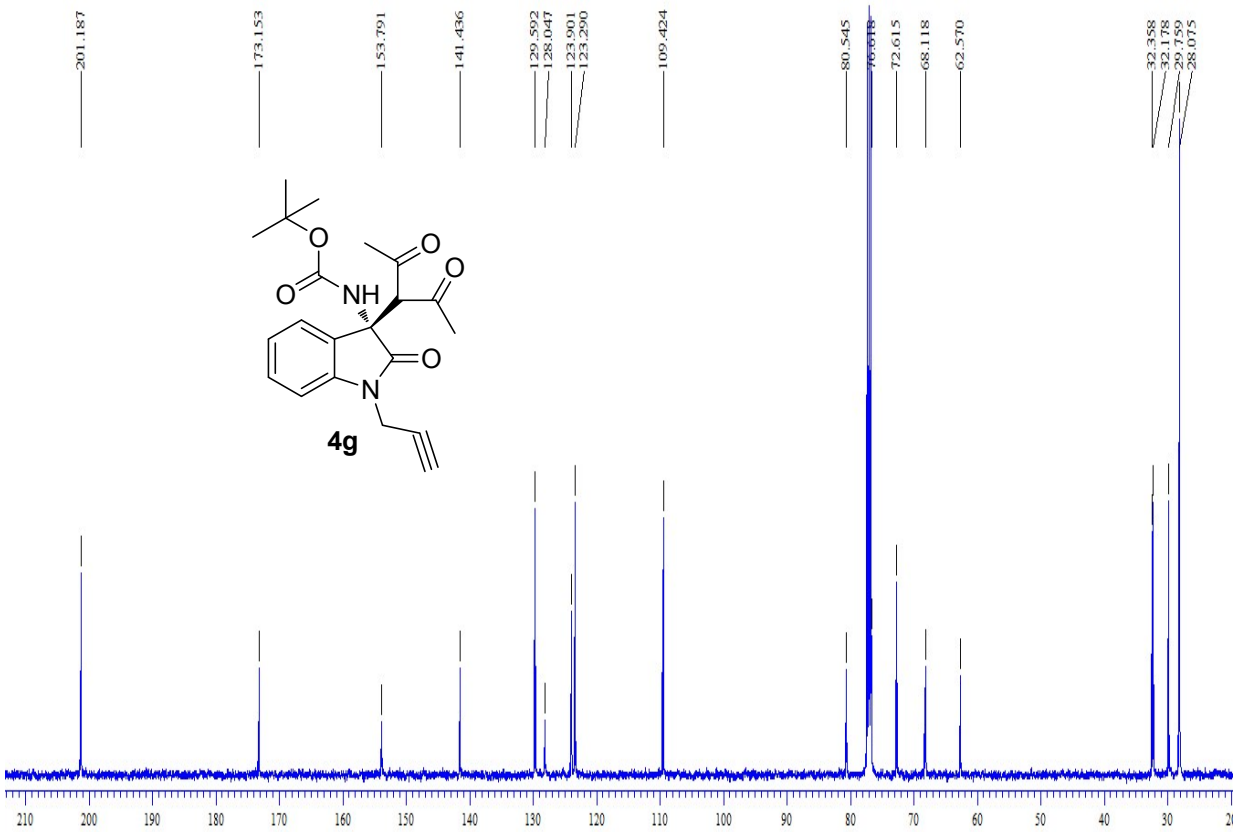
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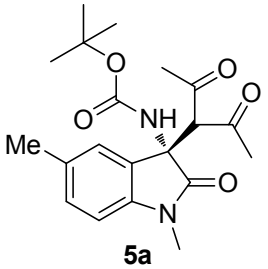
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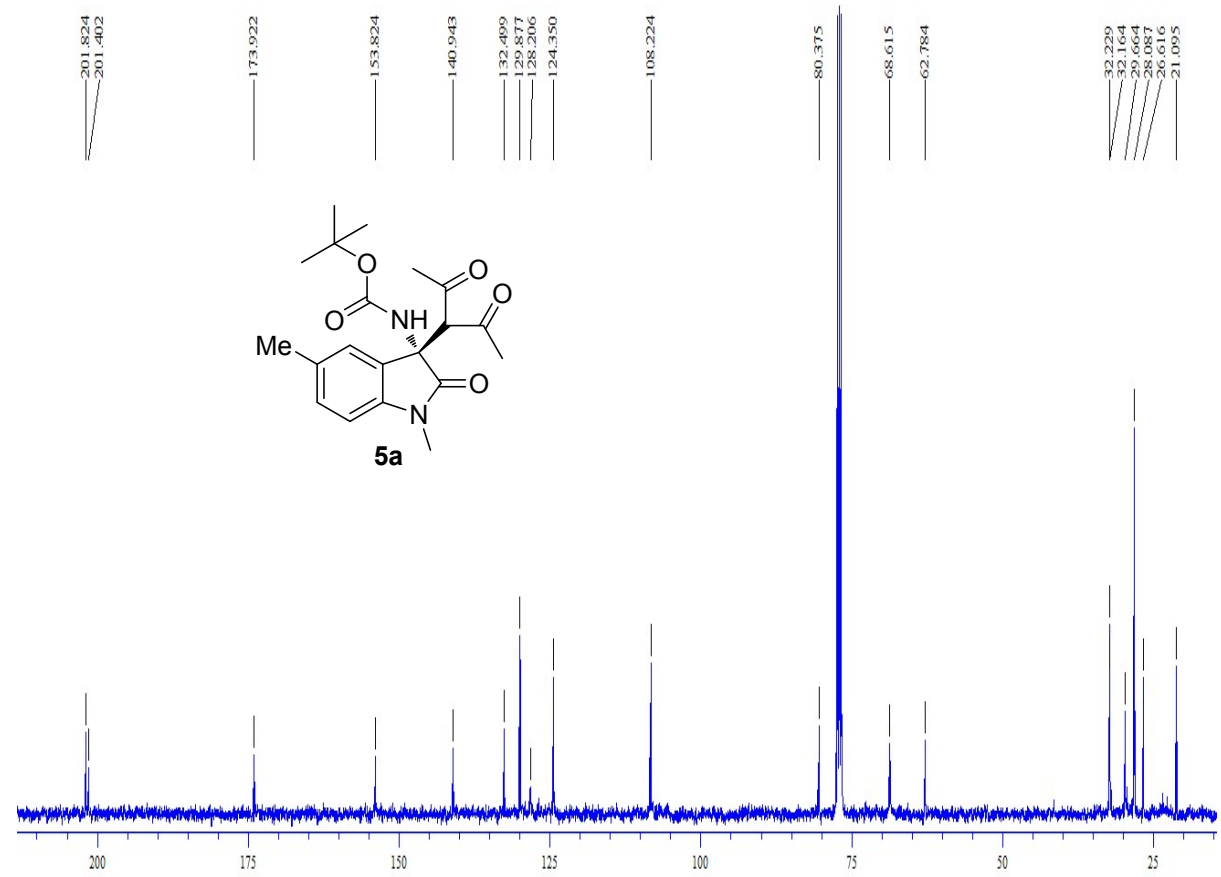
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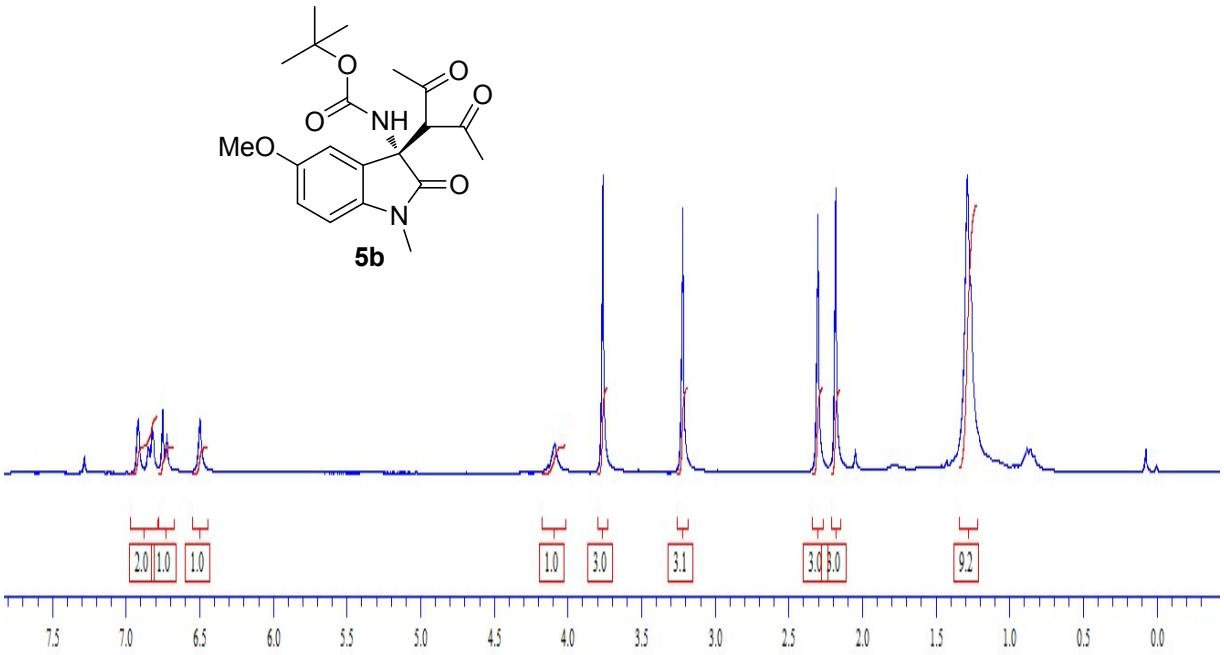
5a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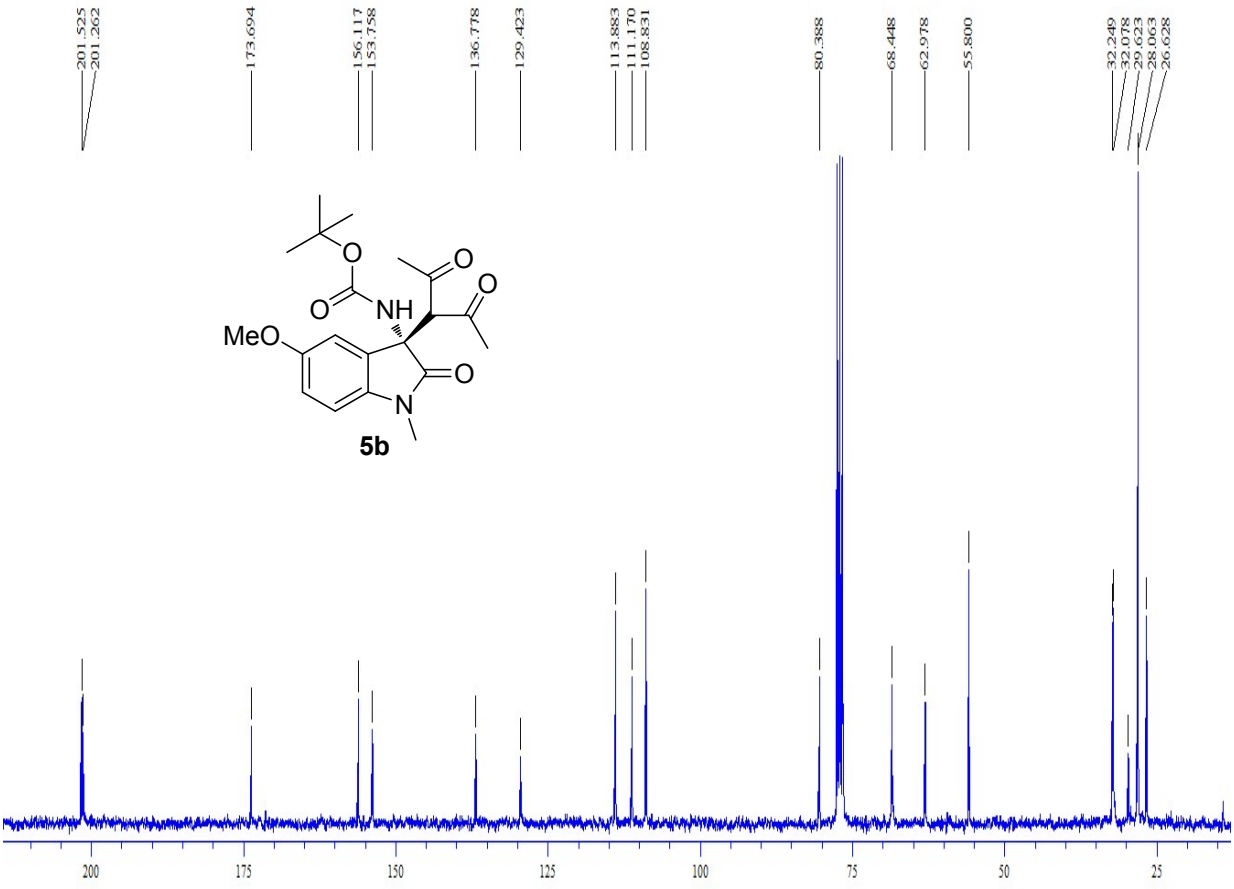
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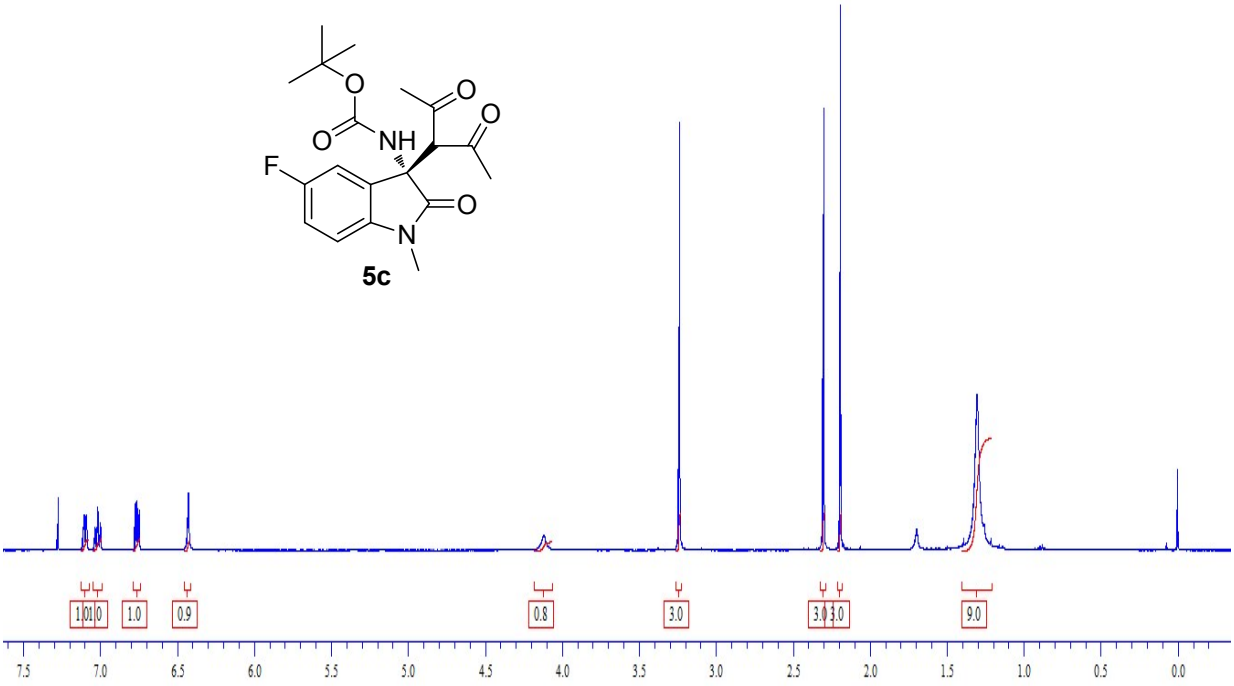
5b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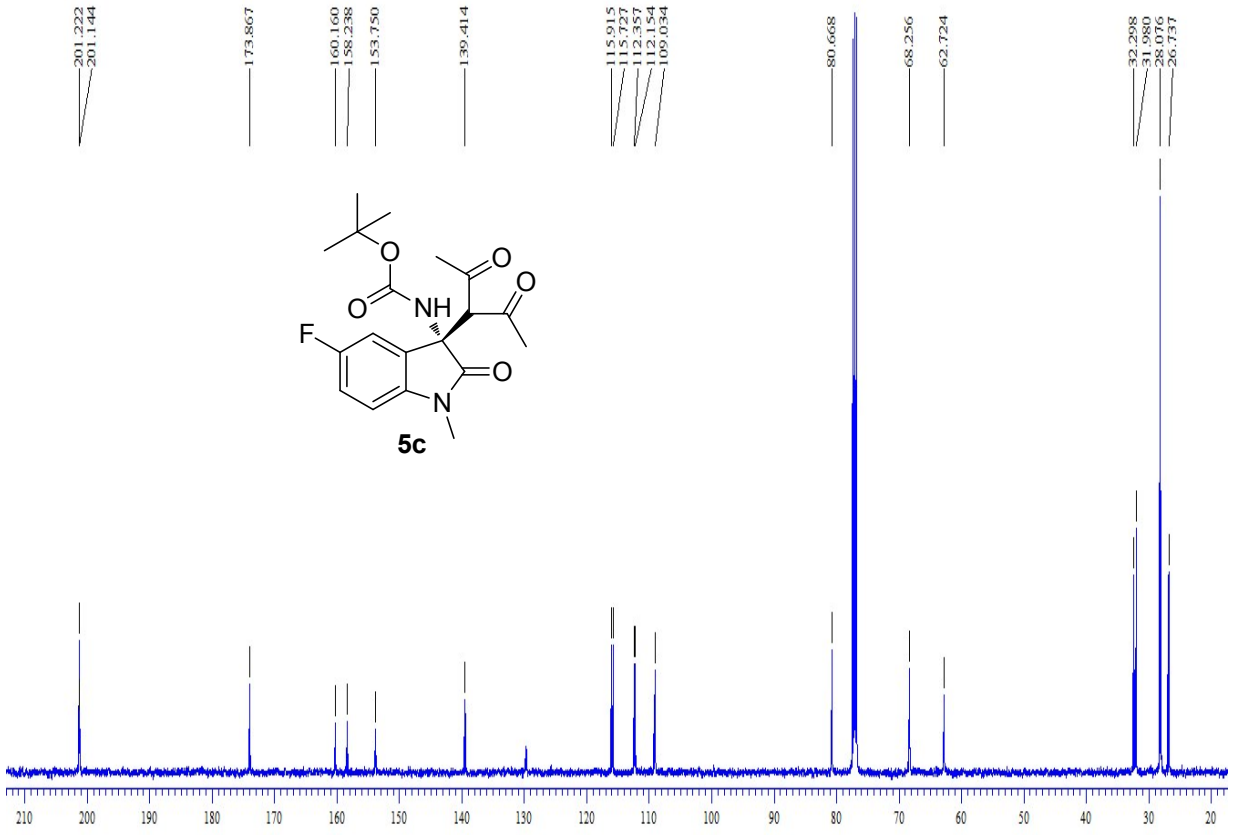
5c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153.750
139.414
115.915
115.727
112.357
112.154
109.034
80.668
68.256
62.724
32.298
31.980
28.076
26.737
O
O
NH
O
O
F
O
N
210
200
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160
150
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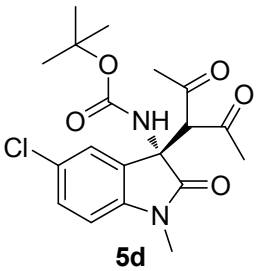
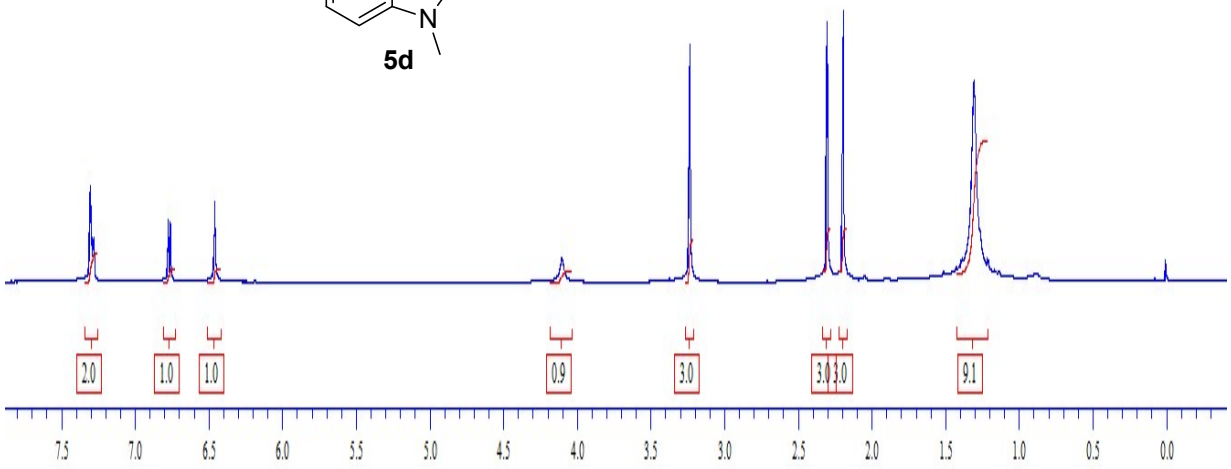
120
110
100
90
80
70
60
50
40
30
20

5c


O
O
NH
O
O
Cl
O
N

5d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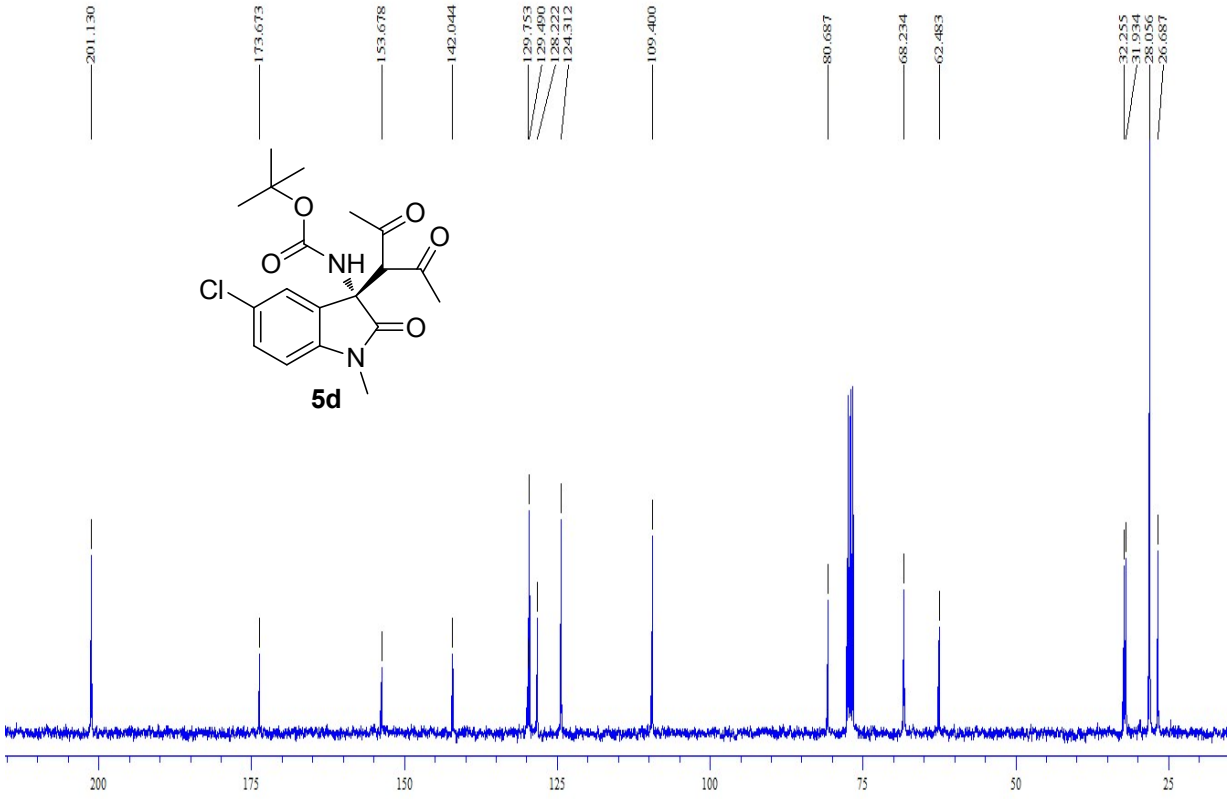
3.0
3.0
3.0
9.1
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0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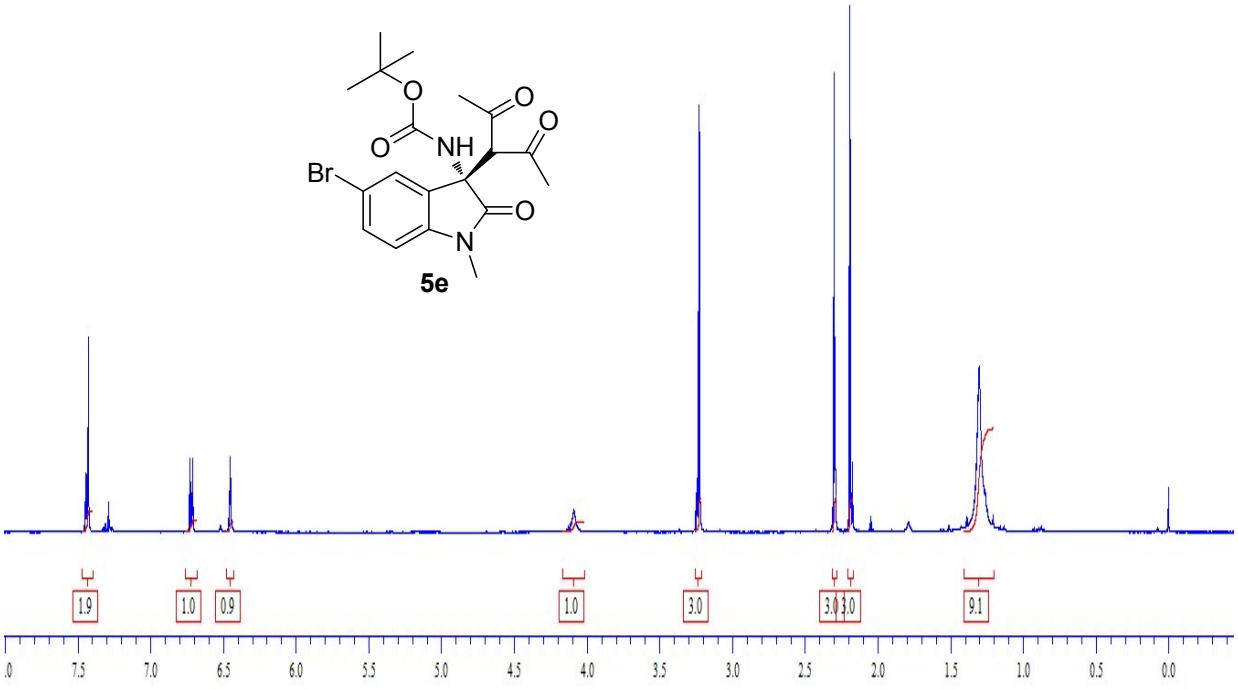
173.673
153.678
142.044
129.753
129.490
128.232
124.312
109.400
80.687
68.234
62.483
32.255
31.934
28.056
26.687
200
175
150
125
100
75
50
25

O
O
NH
O
O
Cl
O
N

5d


Br
NH
O
O
O
O
O
N
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0.9
1.0
3.0
3.0
3.0
9.1
.0
7.5
7.0
6.5
6.0
5.5
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4.0
3.5
3.0
2.5
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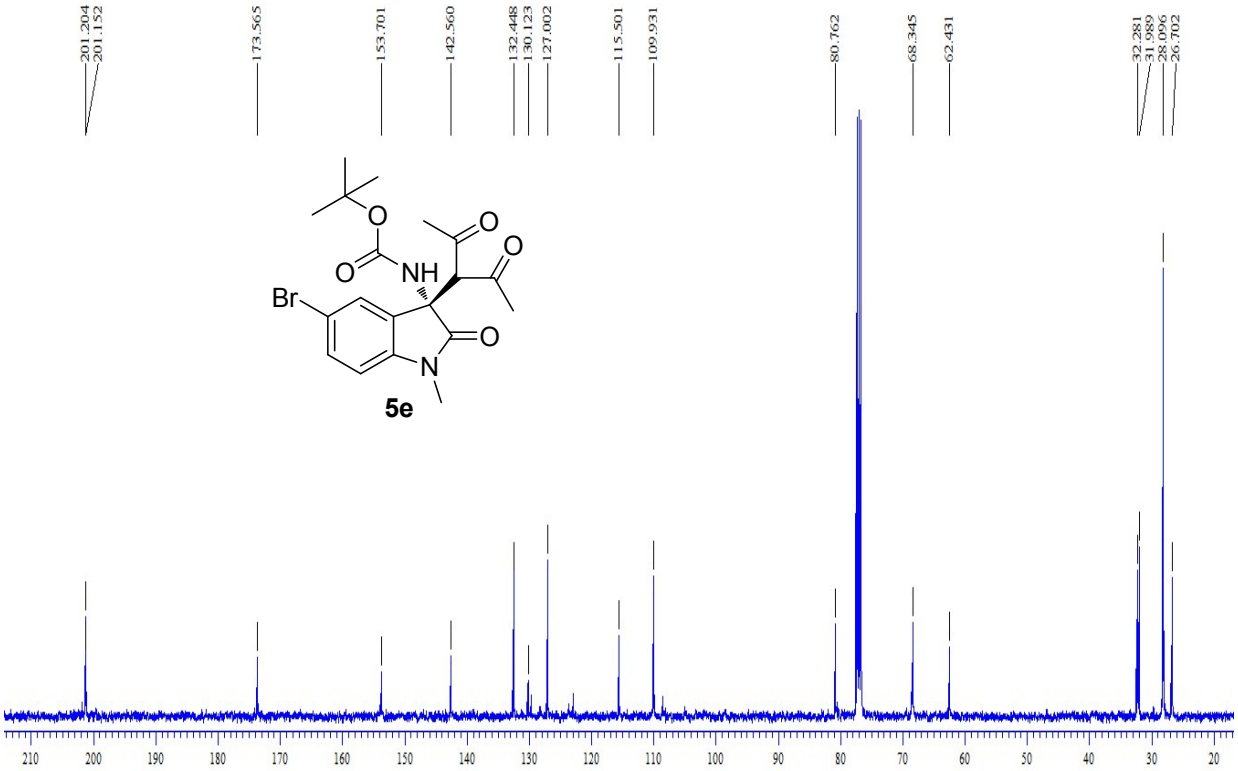
1.5
1.0
0.5
0.0

5e


201.204
201.152
173.565
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142.560
132.448
130.123
127.002
115.501
109.931
80.762
68.345
62.431
32.281
31.989
28.096
26.702
Br
NH
O
O
O
O
O
N
210
200
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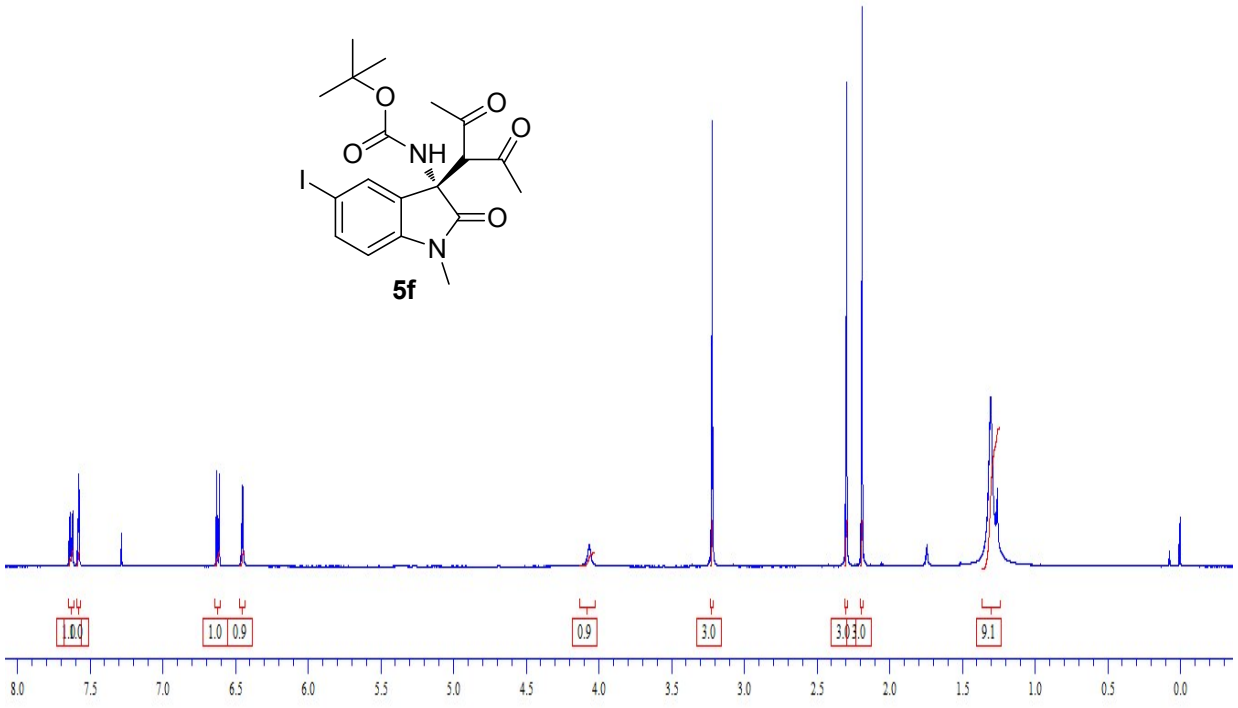
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

5e


O
O
NH
O
O
I
O
N
1.0
1.0
0.9
0.9
3.0
3.0
3.0
9.1
8.0
7.5
7.0
6.5
6.0
5.5
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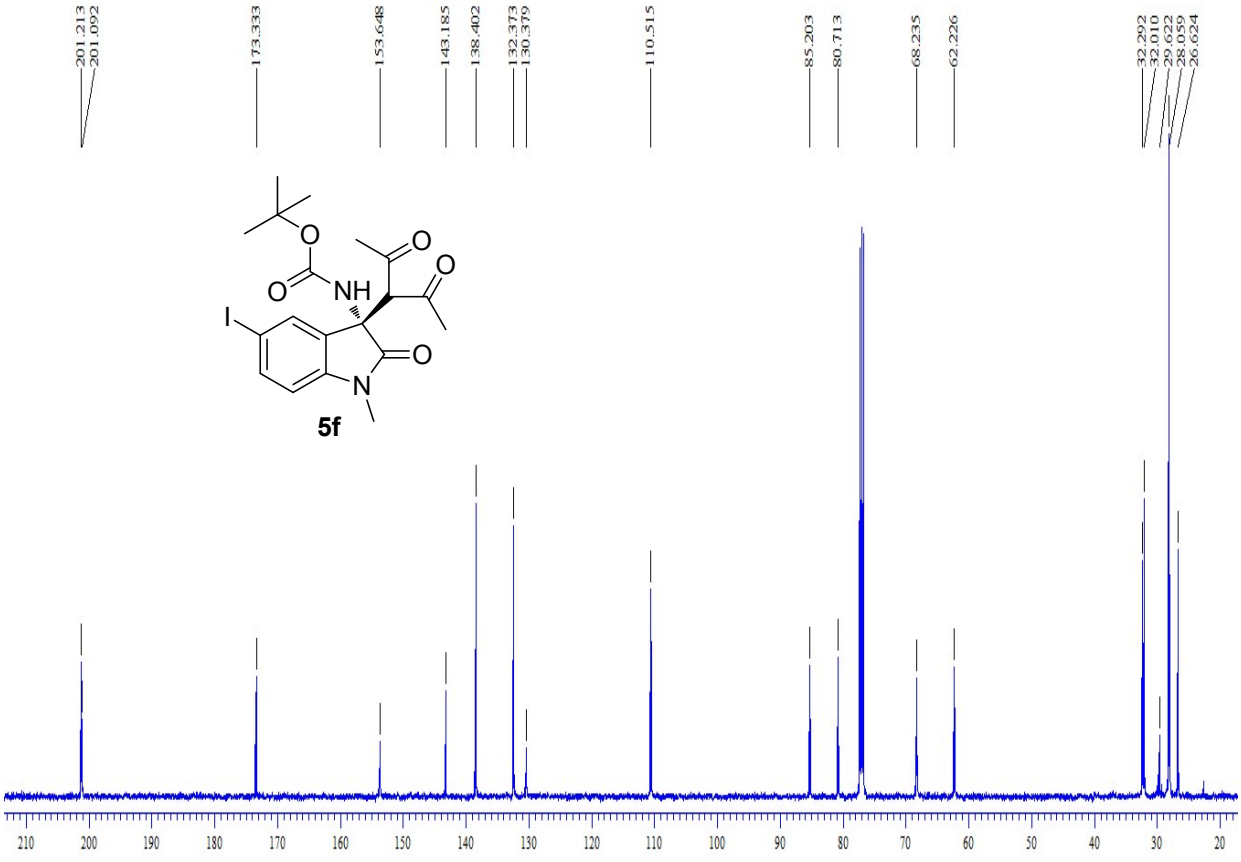
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

5f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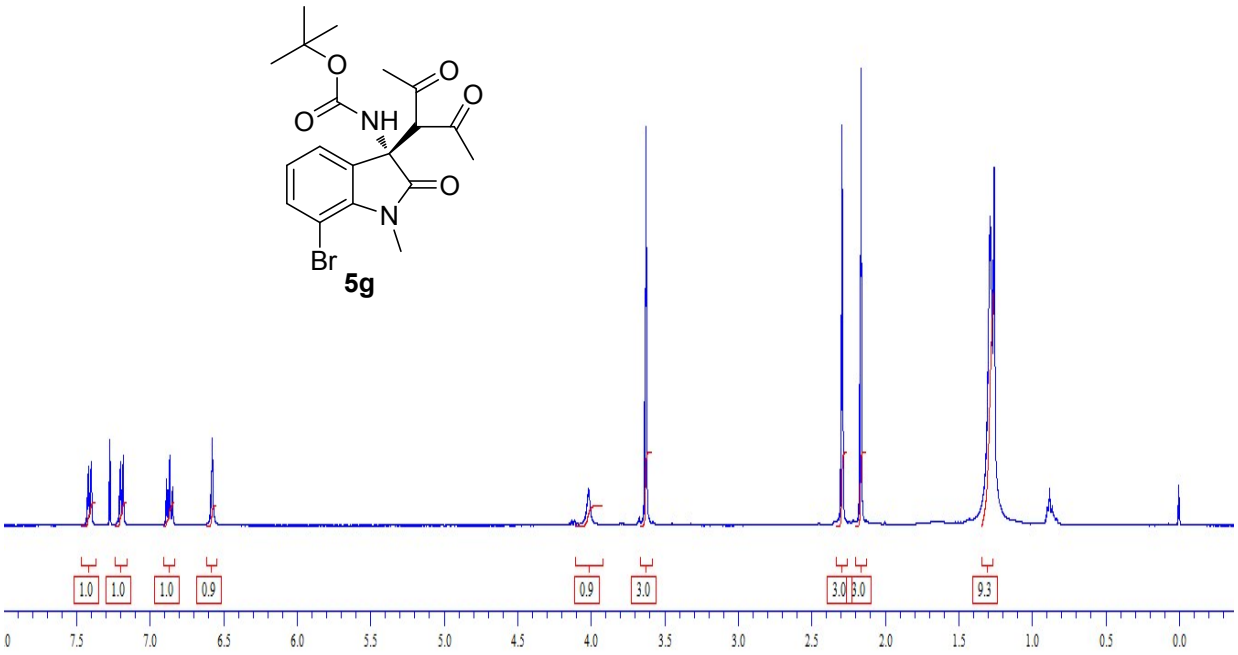
201.092
173.333
153.648
143.185
138.402
132.373
130.379
110.515
85.203
80.713
68.235
62.226
32.292
32.010
29.622
28.059
26.624
O
O
NH
O
O
I
O
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

5f


O
O
NH
O
O
O
N
Br
5g
1.0
1.0
1.0
0.9
0.9
3.0
3.0
3.0
9.3
0
7.5
7.0
6.5
6.0
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4.0
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3.0
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2.0
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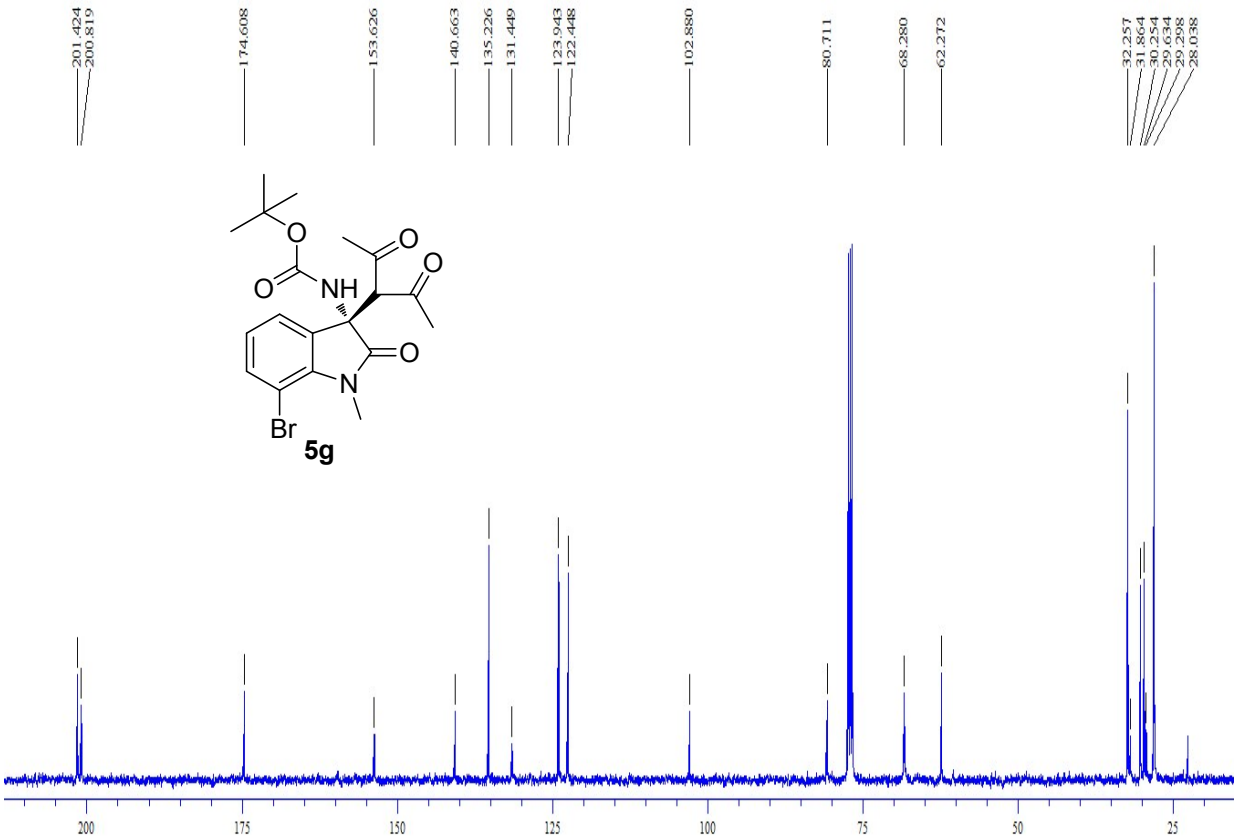
0.0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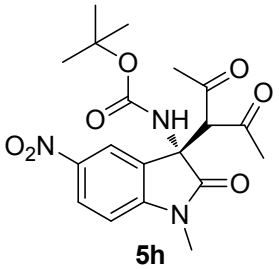
200.819
174.608
153.626
140.663
135.226
131.449
123.943
122.448
102.880
80.711
68.280
62.272
32.257
31.864
30.254
29.634
29.298
28.038
O
O
NH
O
O
O
N
Br
5g
200
175
150
125
100
75
50
25


O
O
NH
O
O
O2N
O
N

5h

2.0
1.0
1.0
1.0
3.0
2.0
9.3
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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2.0
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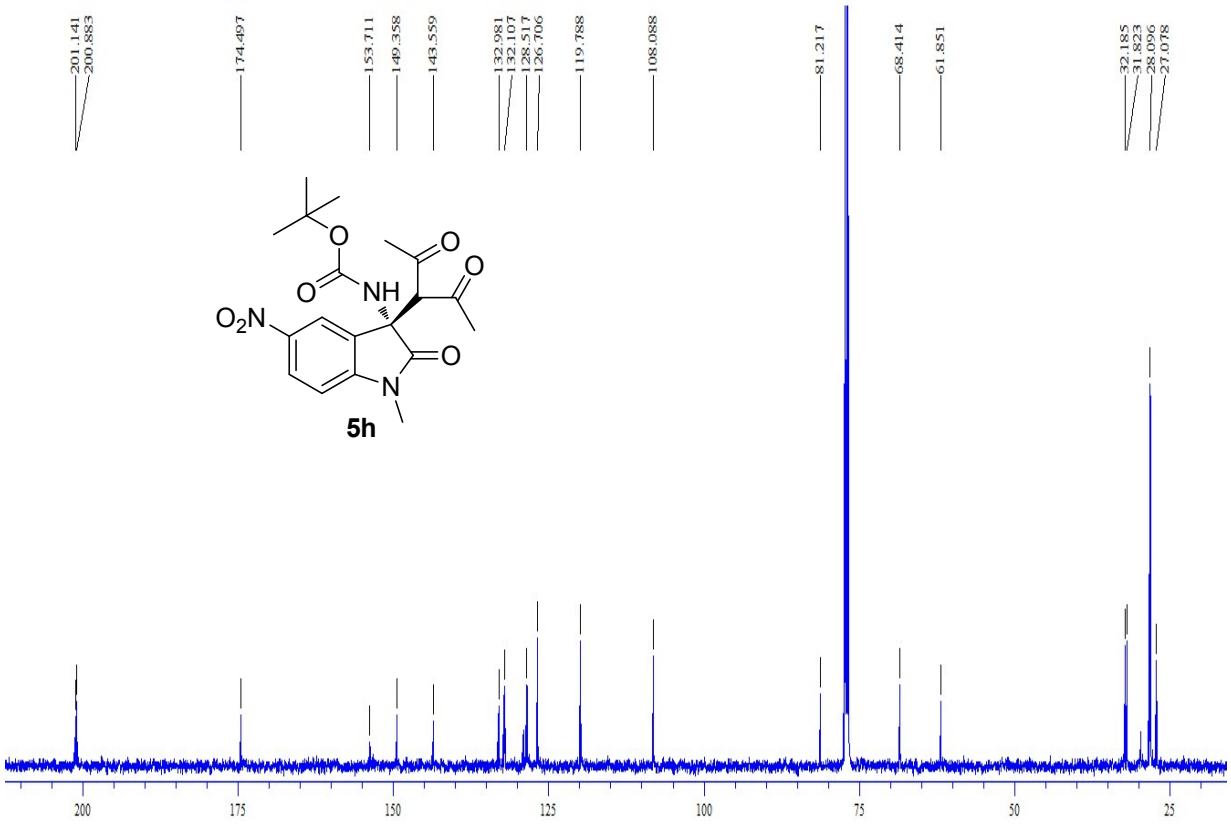
1.0
0.5
0.0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132.981
132.107
128.517
126.706
119.788
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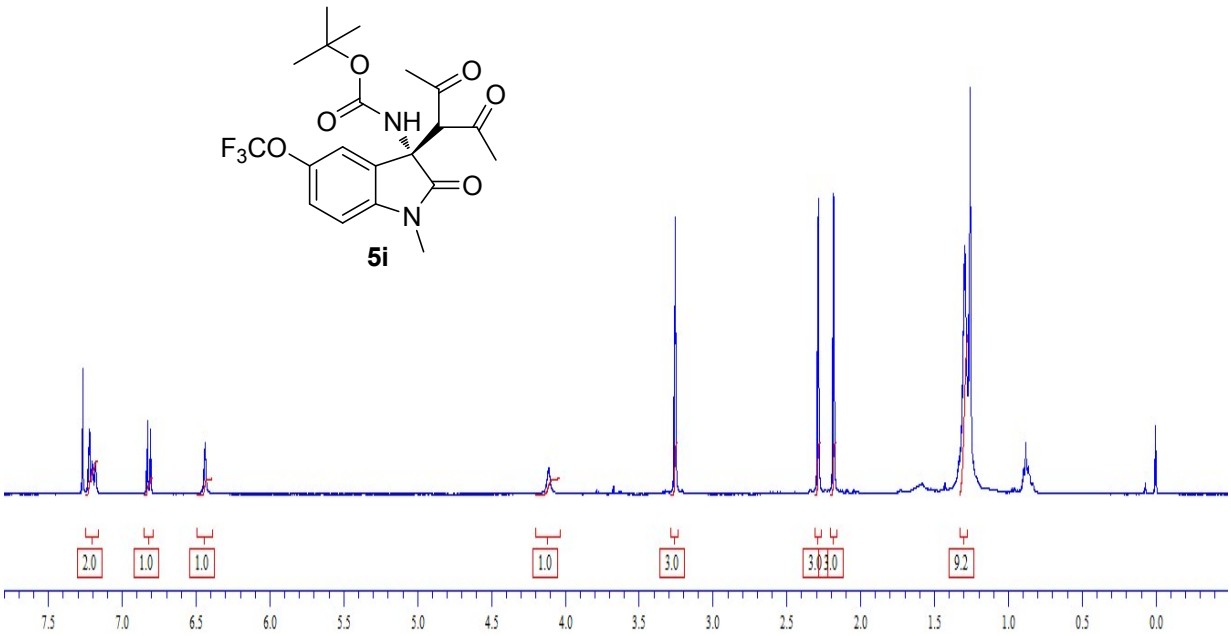
81.217
68.414
61.851
32.185
31.823
28.096
27.078
200
175
150
125
100
75
50
25

O
O
NH
O
O
O2N
O
N

5h


O
O
NH
O
O
F3CO
O
N
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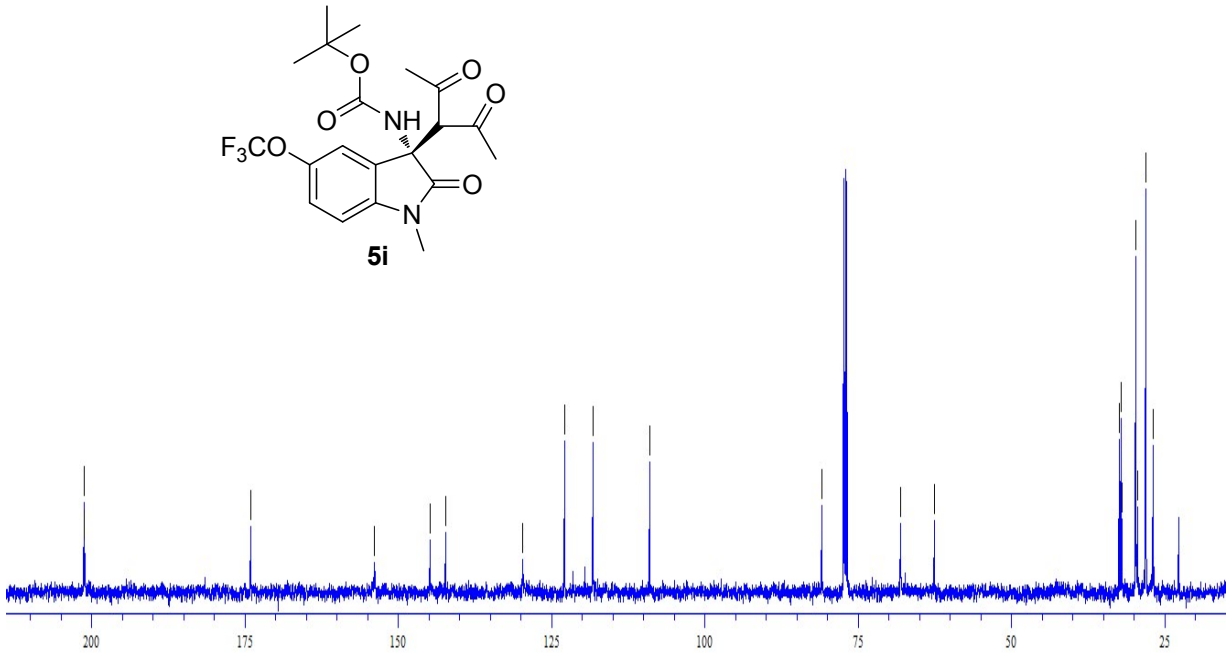
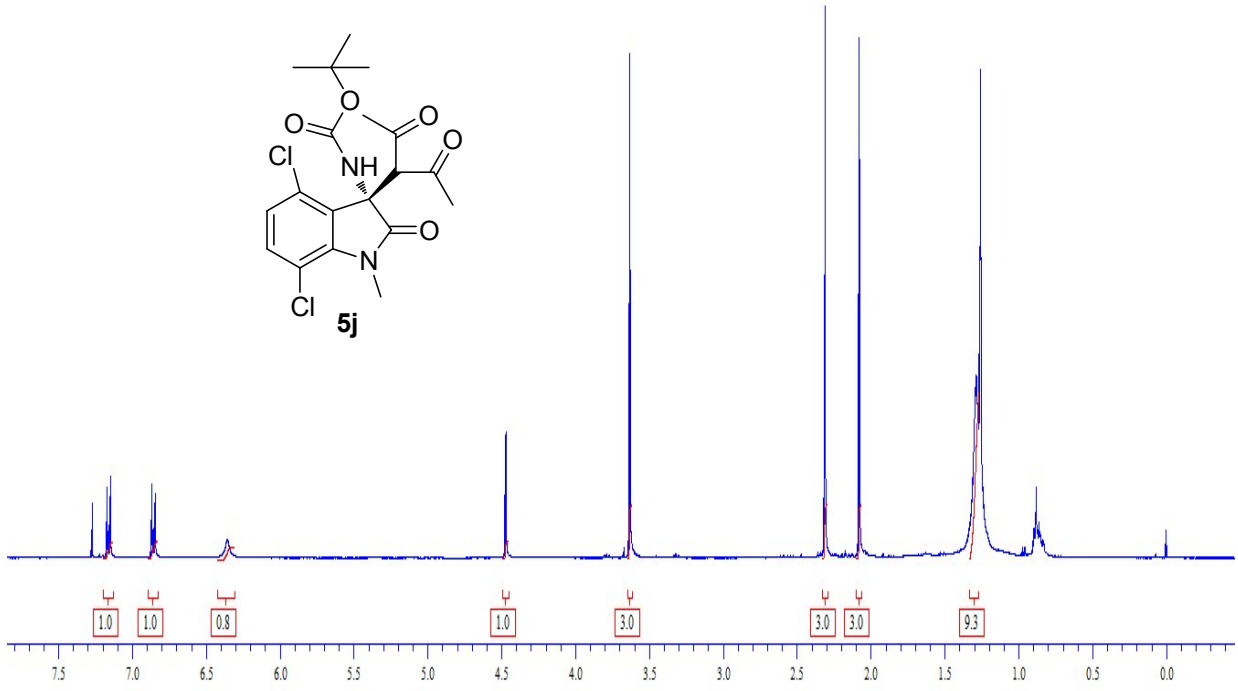
1.0
1.0
1.0
3.0
3.0
3.0
9.2
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

5i


201.126
201.055
173.976
153.770
144.708
142.223
129.587
122.811
118.155
108.933
80.807
68.015
62.522
32.254
31.975
31.880
29.651
29.309
28.004
26.762


O
O
NH
O
O
F3CO
O
N
200
175
150
125
100
75
50
25

5i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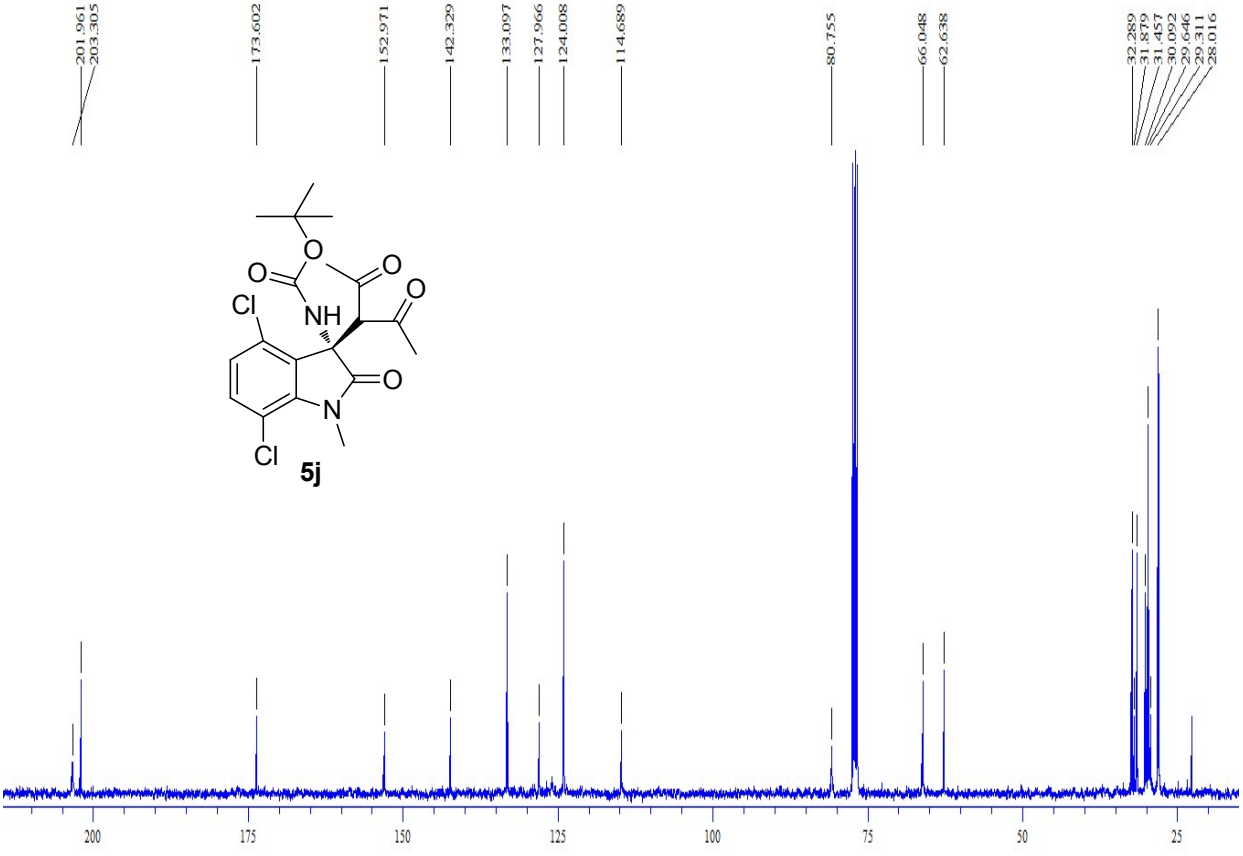
1.0
0.8
1.0
3.0
3.0
3.0
9.3
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

5j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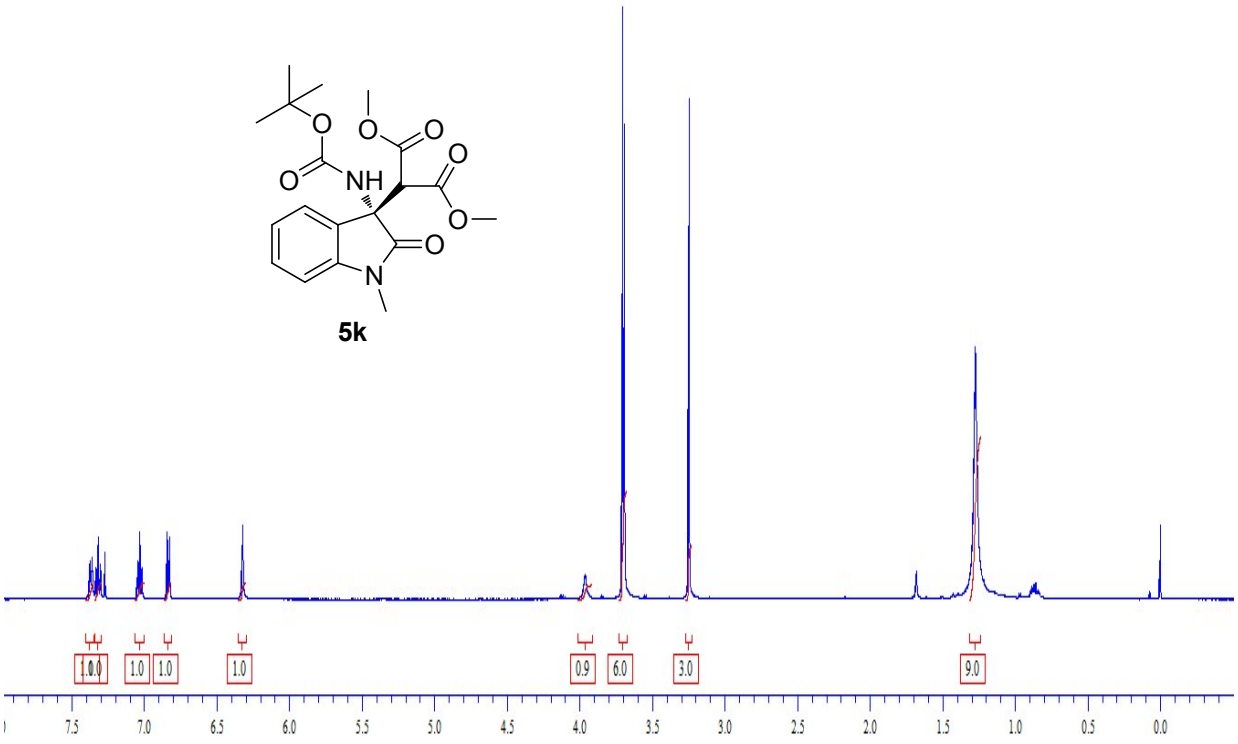
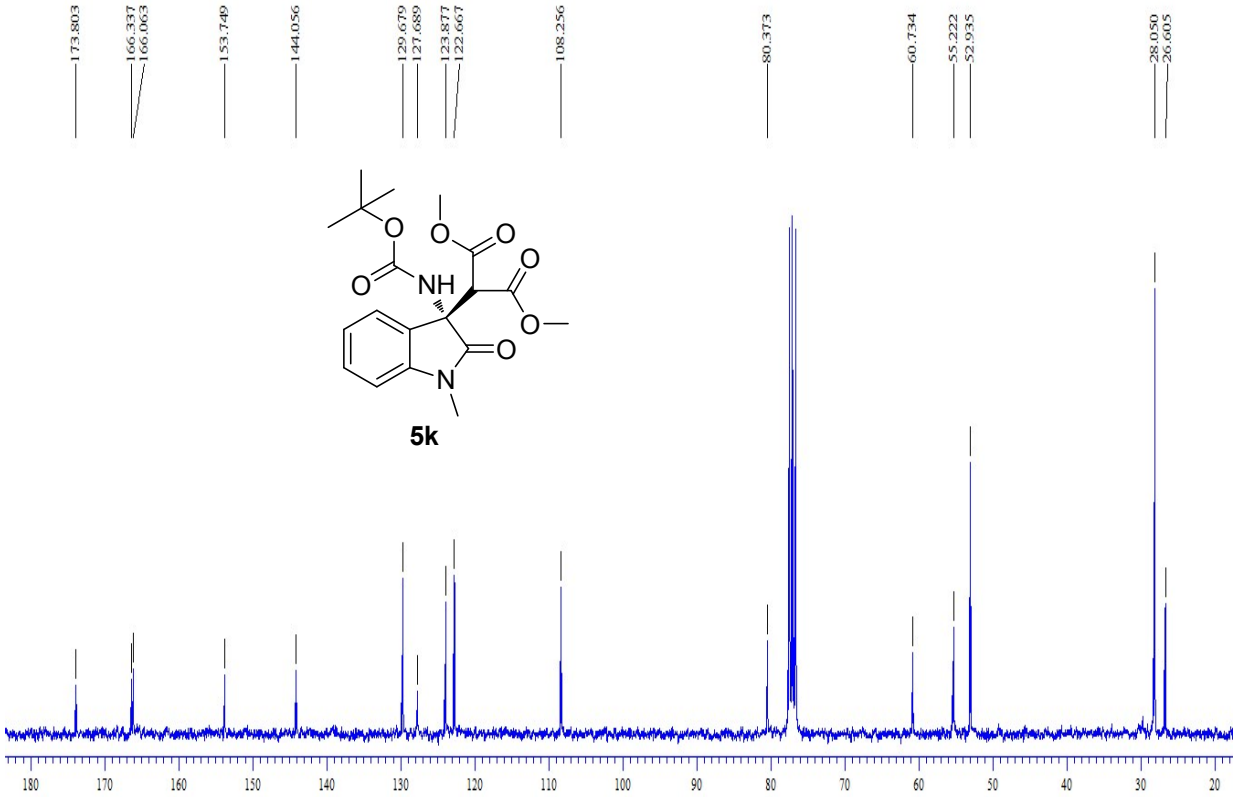
203.305
173.602
152.971
142.329
133.097
127.966
124.008
114.689
80.755
66.048
62.638
32.289
31.879
31.457
30.092
29.646
29.311
28.016
200
175
150
125
100
75
50
25

5j


1.0
1.0
1.0
1.0
0.9
6.0
3.0
9.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

5k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166.063
153.749
144.056
129.679
127.689
123.877
122.667
108.256
80.373
60.734
55.222
52.935
28.050
26.605
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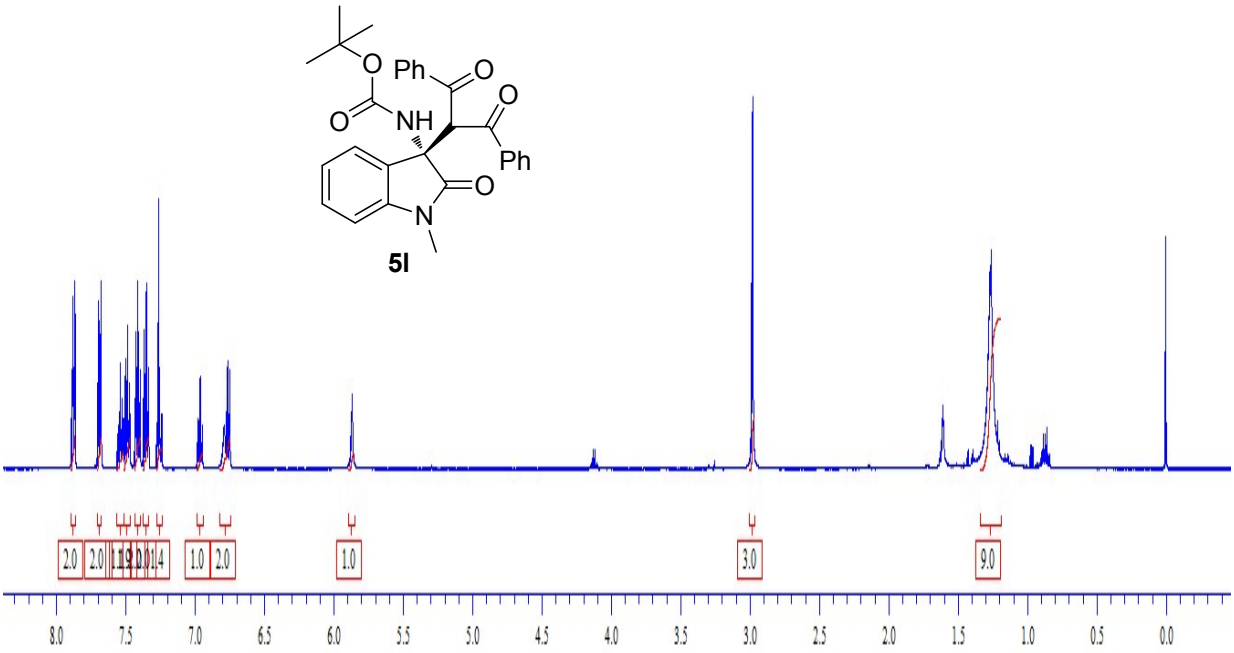
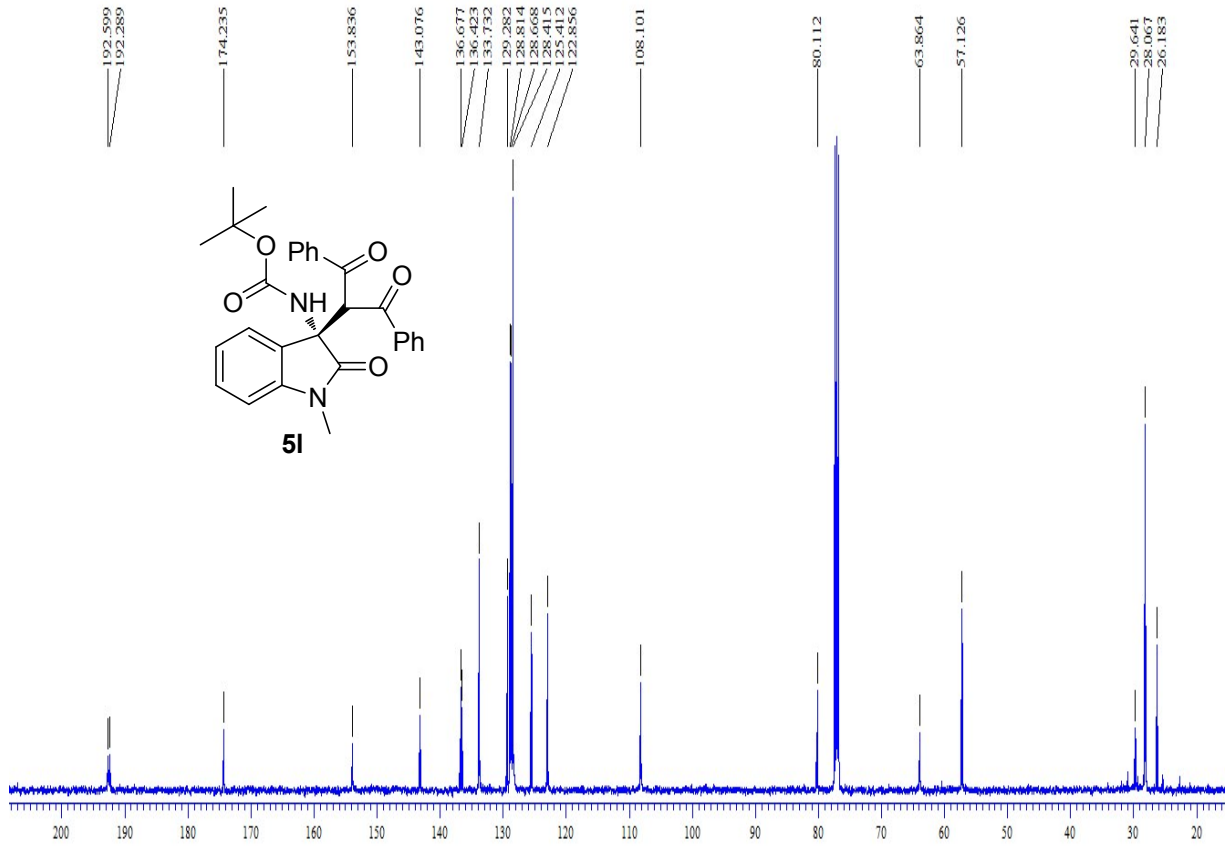
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

5k


Ph
O
O
O
NH
Ph
O
N
2.0
2.0
1.0
2.0
1.0
3.0
9.0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

5l


192.599
192.289
174.235
153.836
143.076
136.677
136.423
133.732
129.282
128.814
128.668
128.415
125.412
122.856
108.101
80.112
63.864
57.126
29.641
28.067
26.183
Ph
O
O
O
NH
Ph
O
N
200
190
180
170
160
150
140
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120
110
100
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80
70
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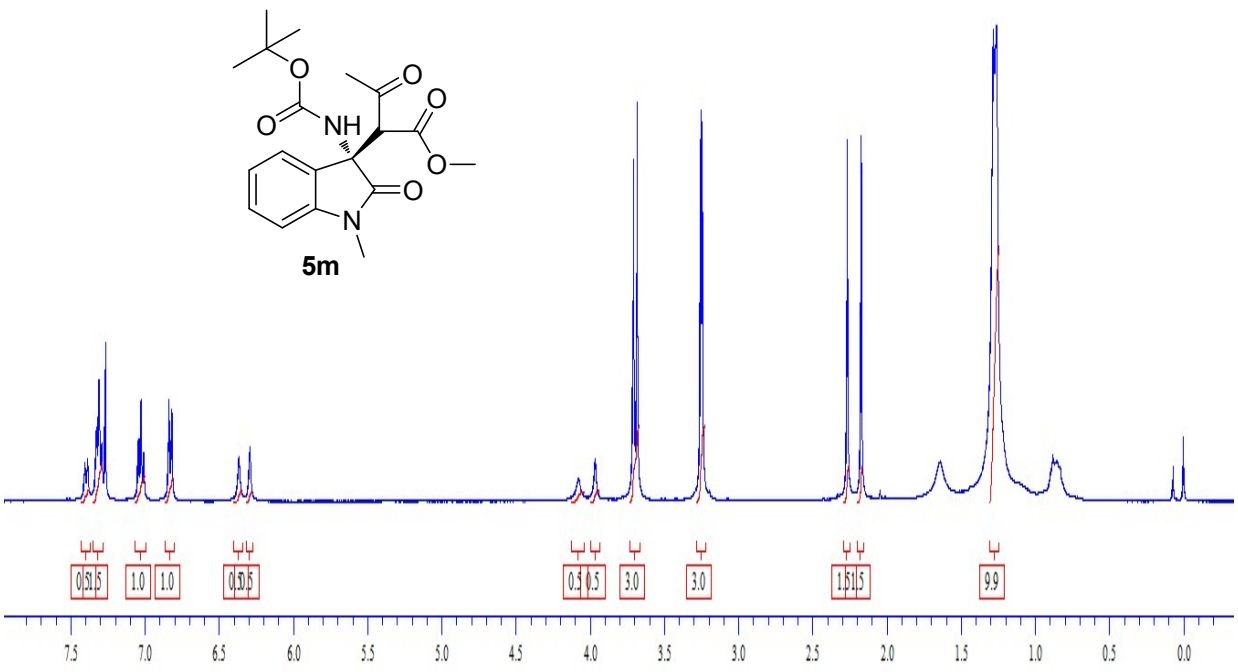
50
40
30
20

5l


5m
0.51.5
1.0
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0.51.5
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0.5
3.0
3.0
1.51.5
9.9
7.5
7.0
6.5
6.0
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4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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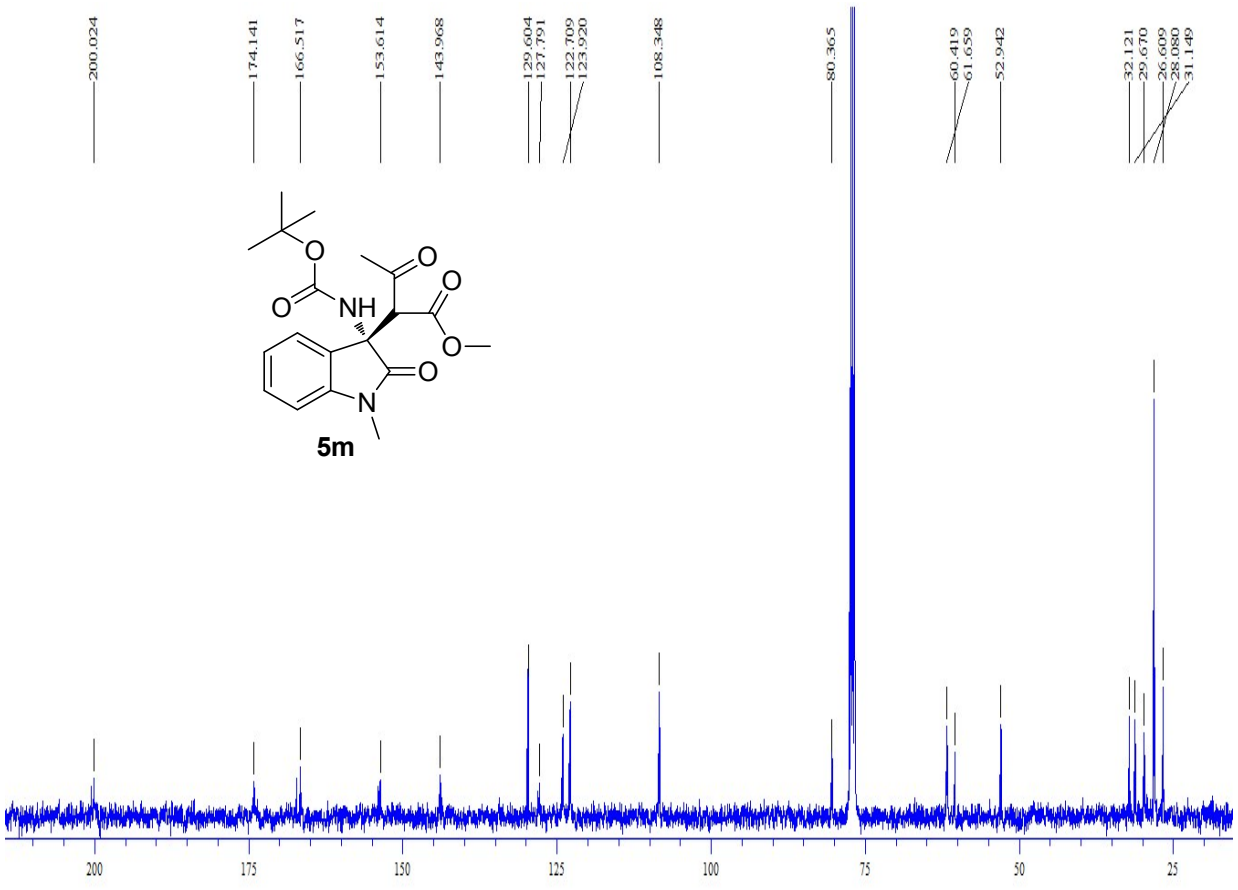
0.0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129.604
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122.709
123.920
108.348
80.365
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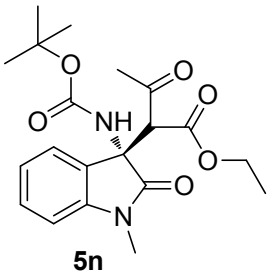
61.659
52.942
32.121
29.670
26.609
28.080
31.149
5m
200
175
150
125
100
75
50
25



5n

0.5 1.5
1.0
1.0
0.5 0.5
2.6 0.5
3.0
1.5 1.5
9.7 3.1
0
7.5
7.0
6.5
6.0
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5.0
4.5
4.0
3.5
3.0
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2.0
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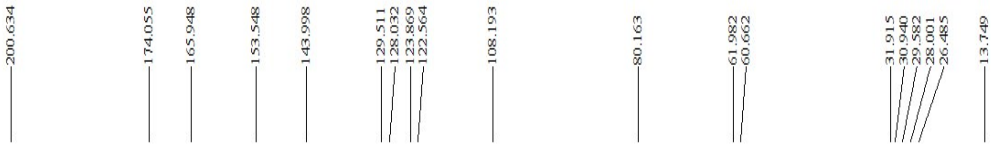
0.0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123.869
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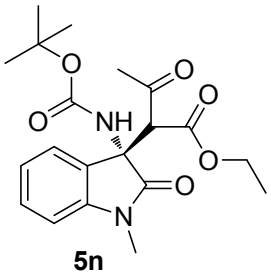
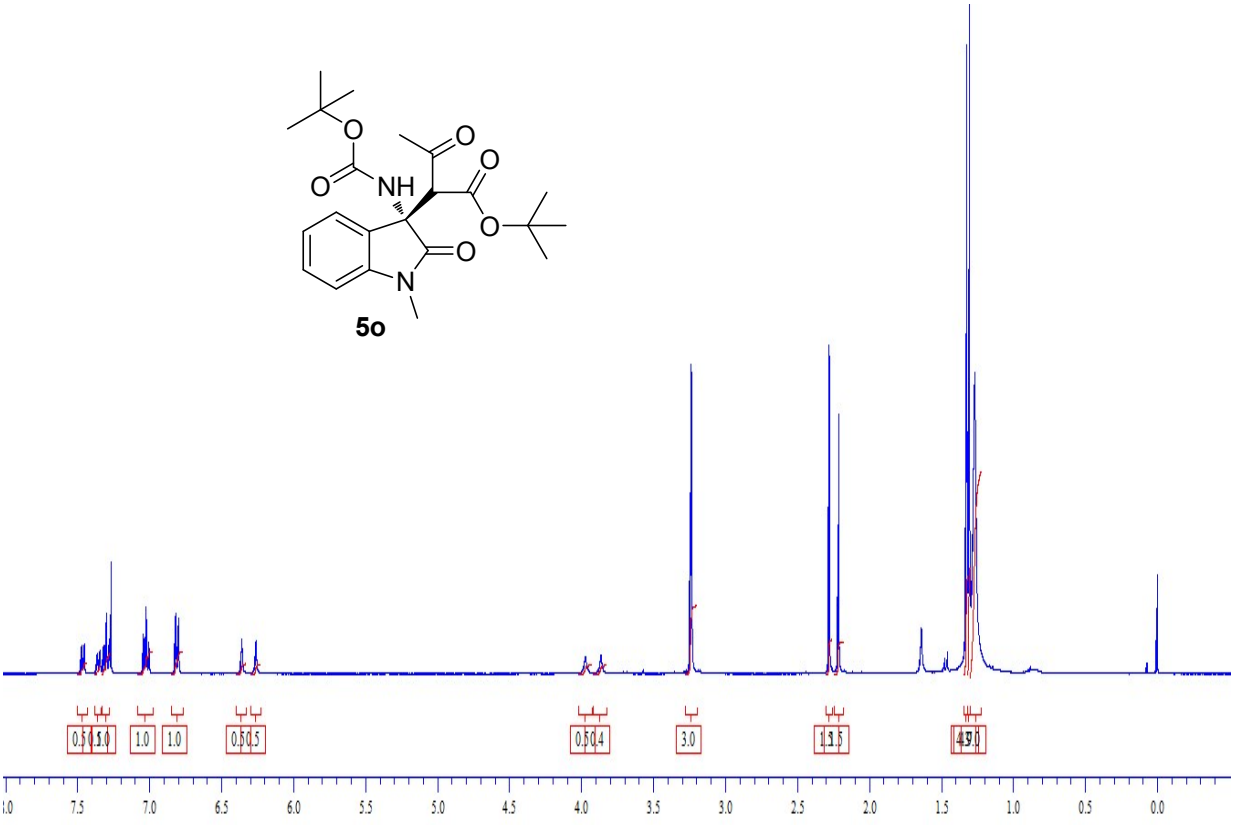
31.915
30.940
29.582
28.001
26.485
13.749



5n

200
175
150
125
100
75
50
25
0


O
O
NH
O
O
O
O
O
N
5o
0.5
0.5
1.0
1.0
1.0
0.5
0.5
0.5
0.4
3.0
1.5
1.5
4.7
4.0
7.5
7.0
6.5
6.0
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4.0
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3.0
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2.0
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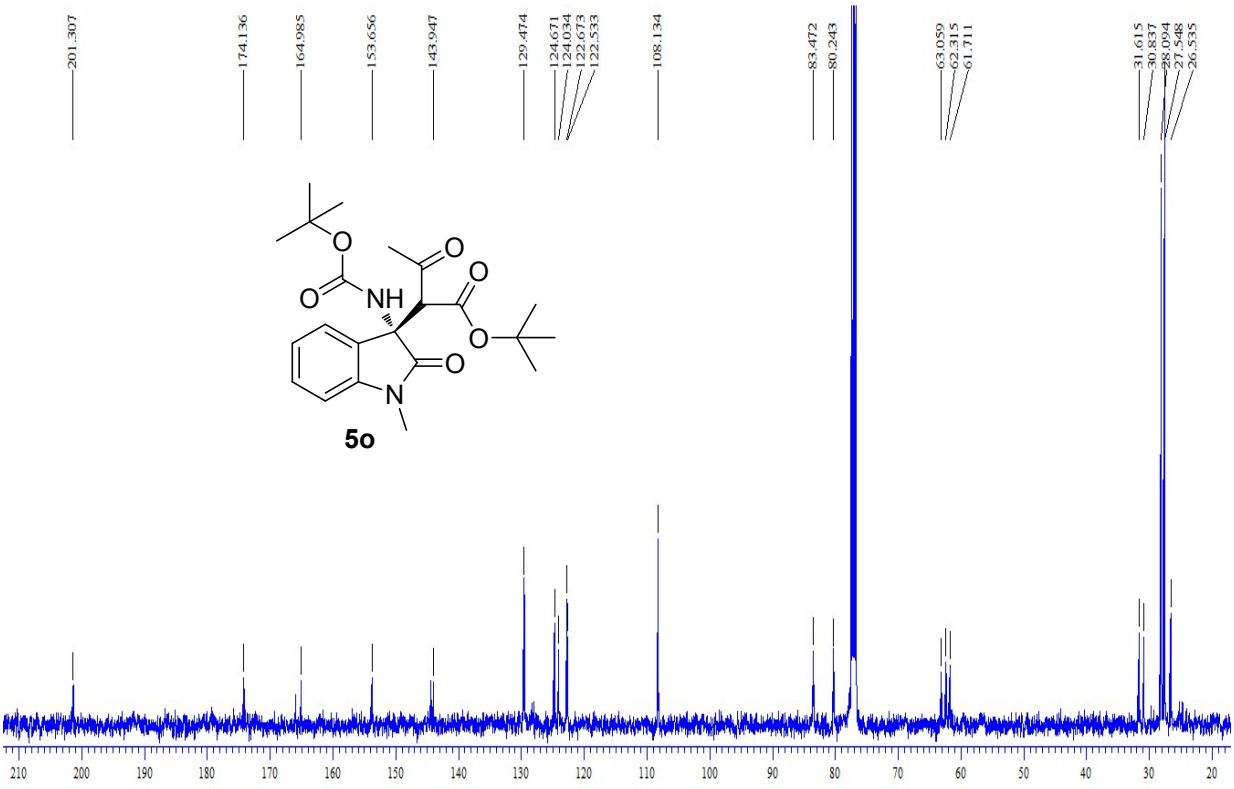
0.0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129.474
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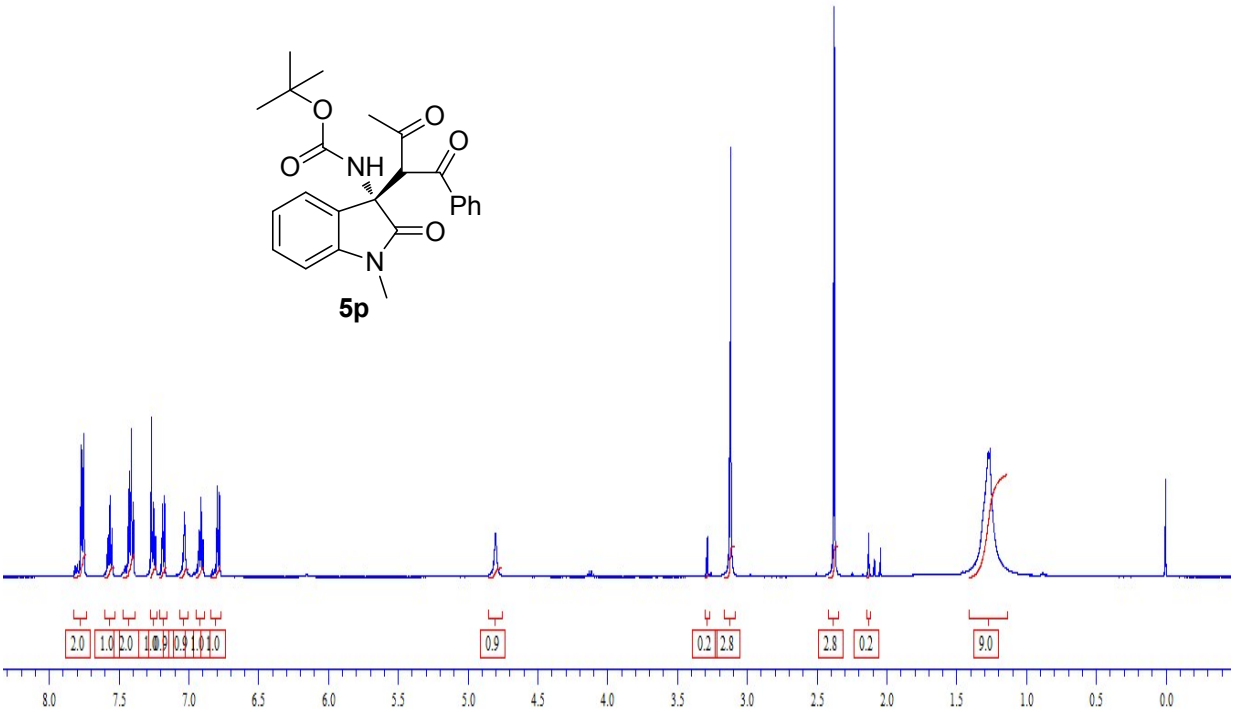
124.034
122.673
122.533
108.134
83.472
80.243
63.059
62.315
61.711
31.615
30.837
28.094
27.548
26.535
O
O
NH
O
O
O
O
O
N
5o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20


O
O
NH
O
O
Ph
O
N
2.0
1.0
2.0
0.9
1.0
1.0
0.9
0.2
2.8
2.8
0.2
9.0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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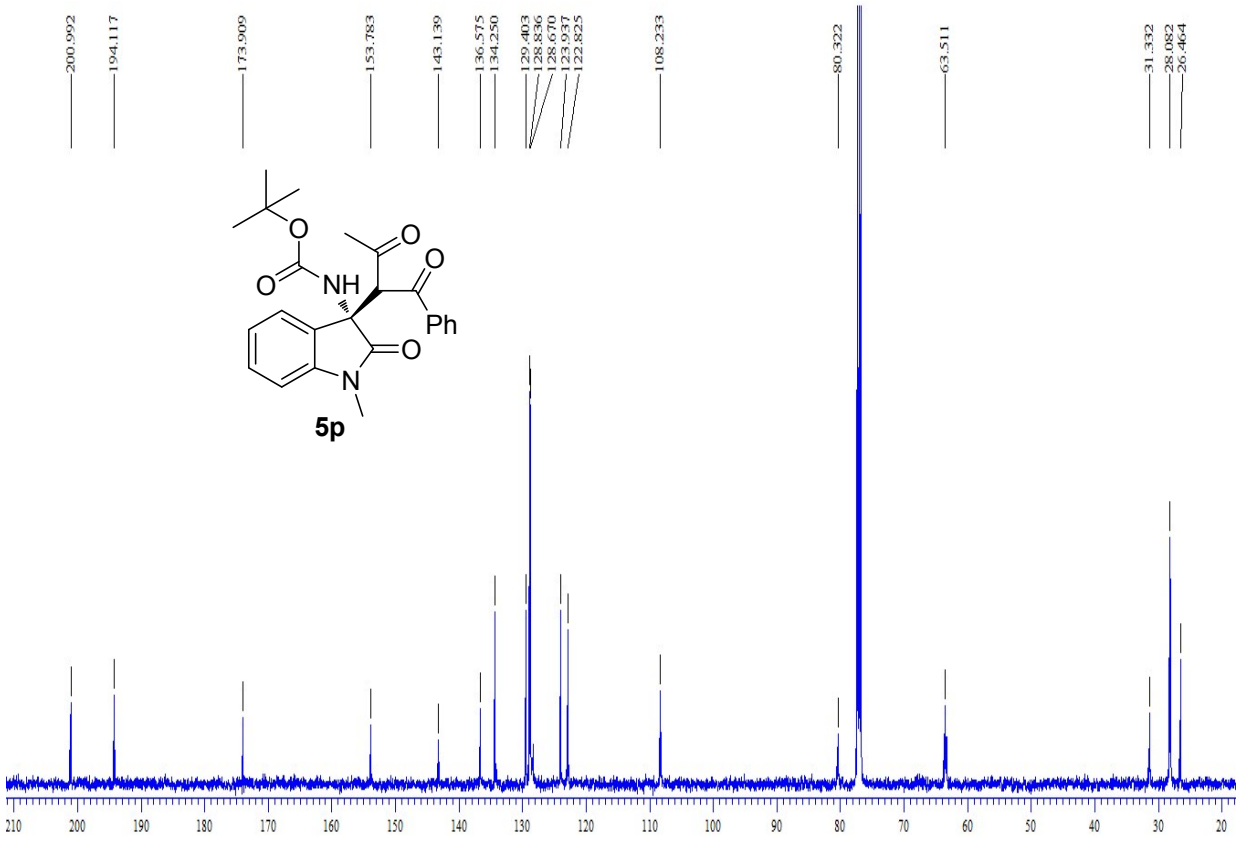
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

5p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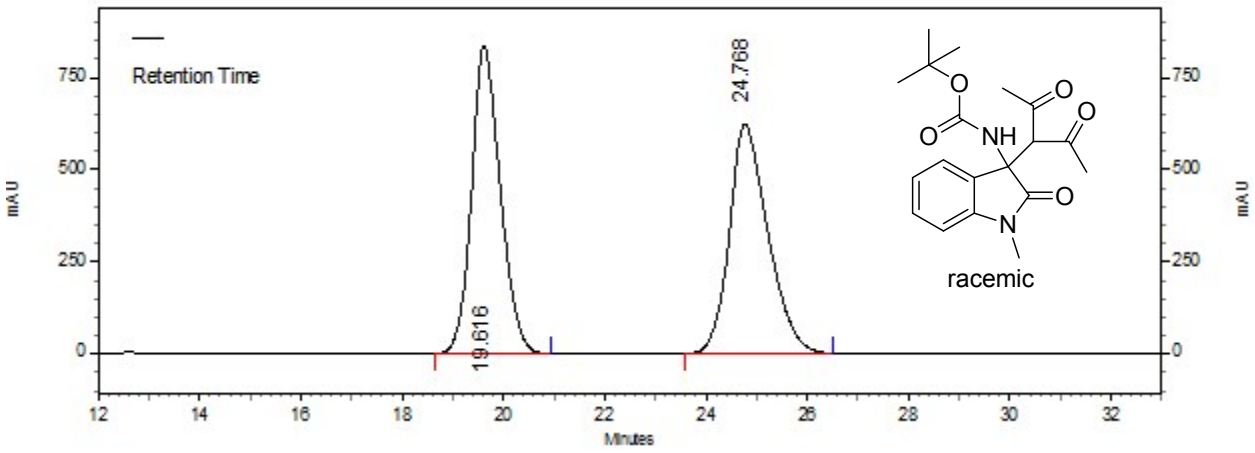
153.783
143.139
136.575
134.250
129.403
128.836
128.670
123.937
122.825
108.233
80.322
63.511
31.332
28.082
26.464
O
O
NH
O
O
Ph
O
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

5p

4) HPLC spectra of product 4 and 5



3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
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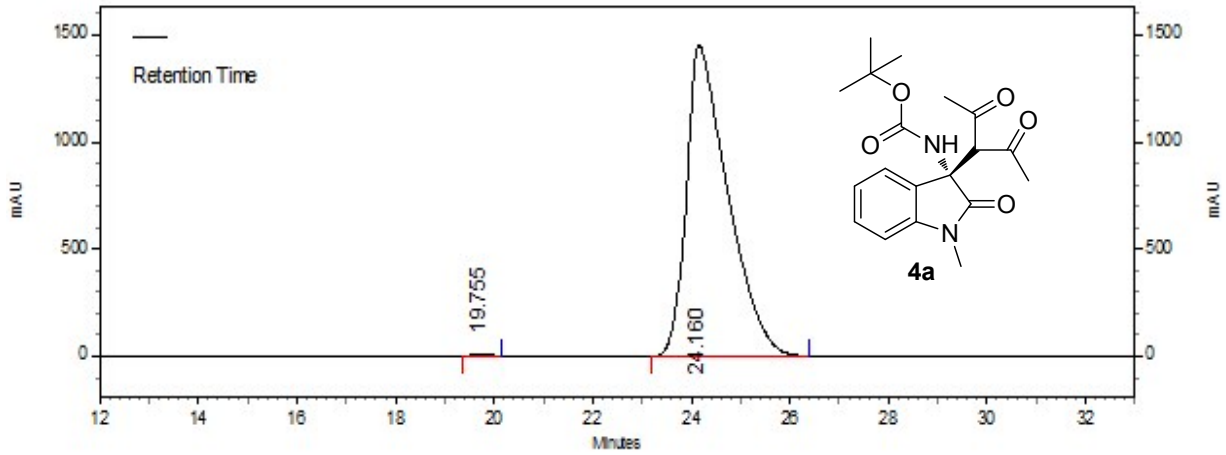
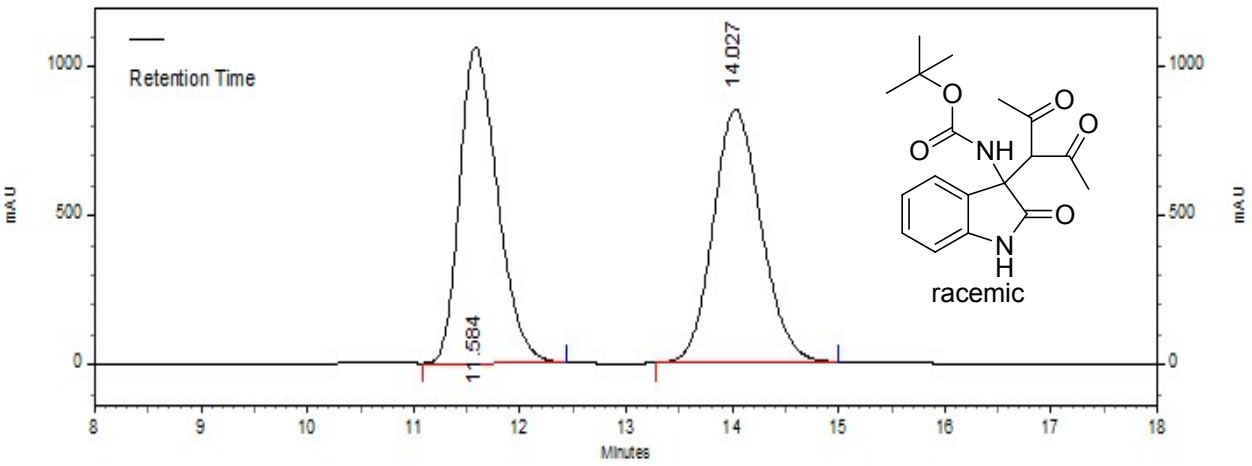
2	24.768	32946595	49.97	621980
Totals		65926404	100.00	1457391



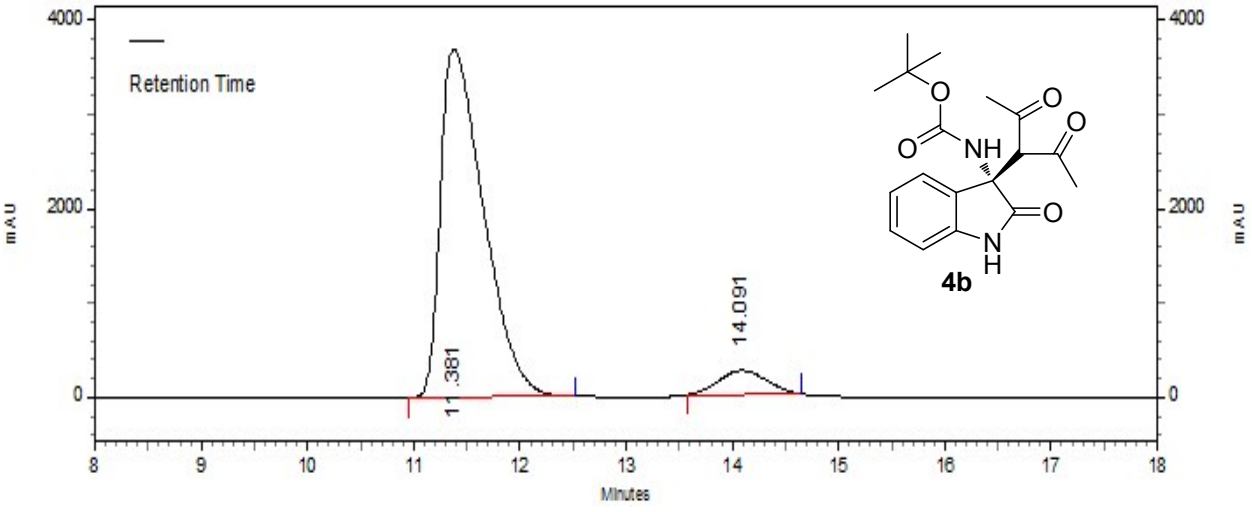
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	19.755	202446	0.24	7518
2	24.160	84743334	99.76	1452775
Totals		84945780	100.00	1460293



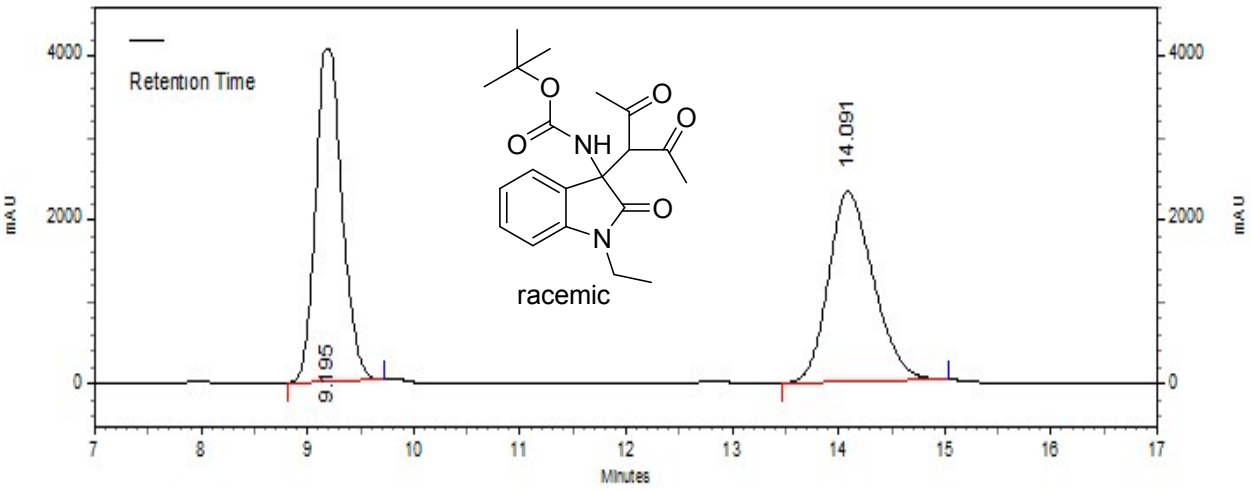
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	11.584	26179125	49.54	1058311
2	14.027	26668310	50.46	847394
Totals		52847435	100.00	1905705



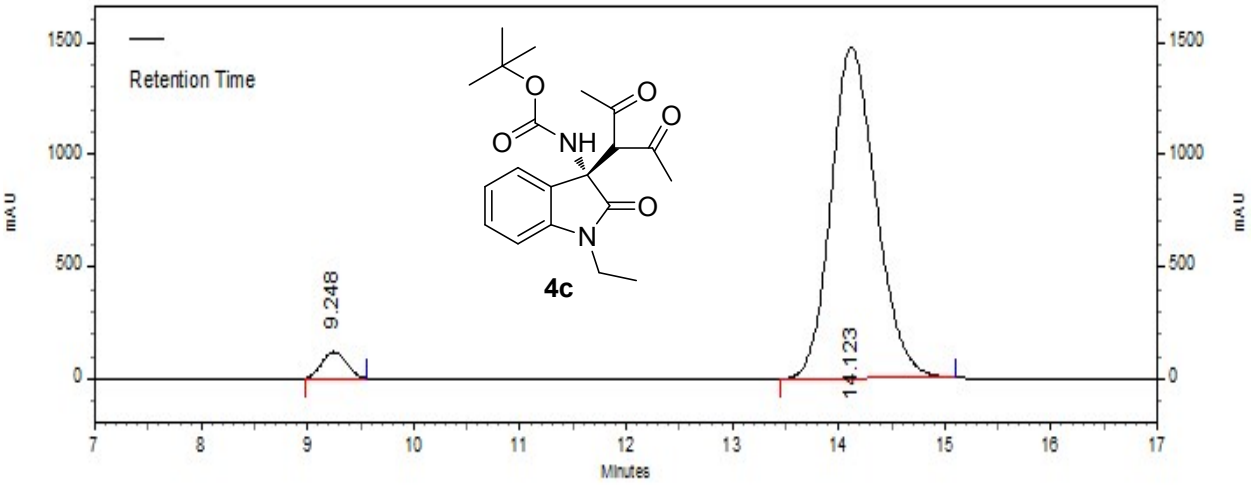
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	11.381	104133881	92.89	3687906
2	14.091	7969189	7.11	253536
Totals		112103070	100.00	3941442



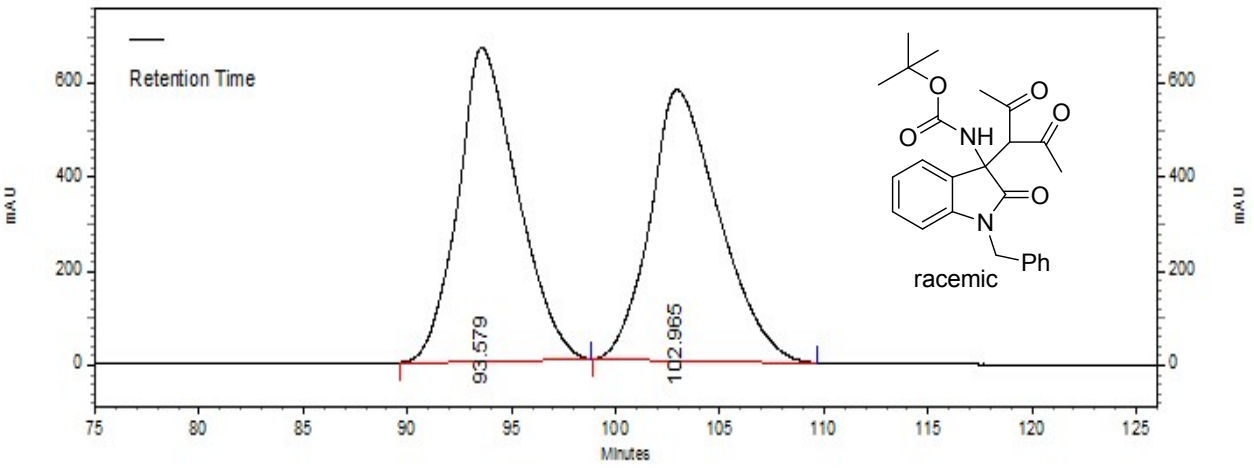
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	9.195	70559858	50.87	4059689
2	14.091	68133701	49.13	2323158
Totals		138693559	100.00	6382847



3: 254 nm, 8 nm

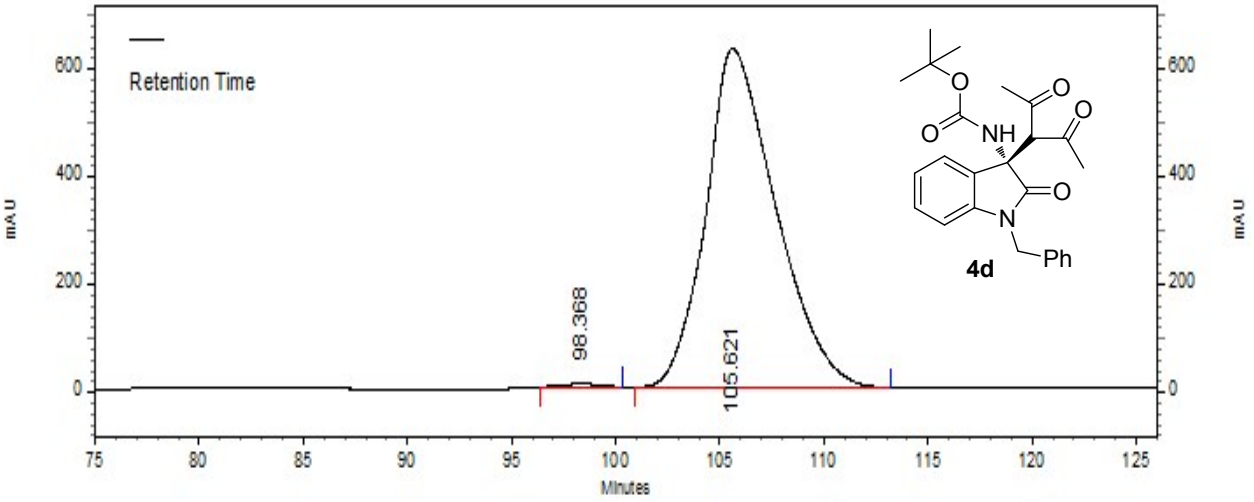
Pk #	Retention Time	Area	Area Percent	Height
1	9.248	1887166	4.15	116785
2	14.123	43626059	95.85	1476354
Totals		45513225	100.00	1593139



1: 210 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	93.579	132342715	49.90	666735
2	102.965	132888471	50.10	576540

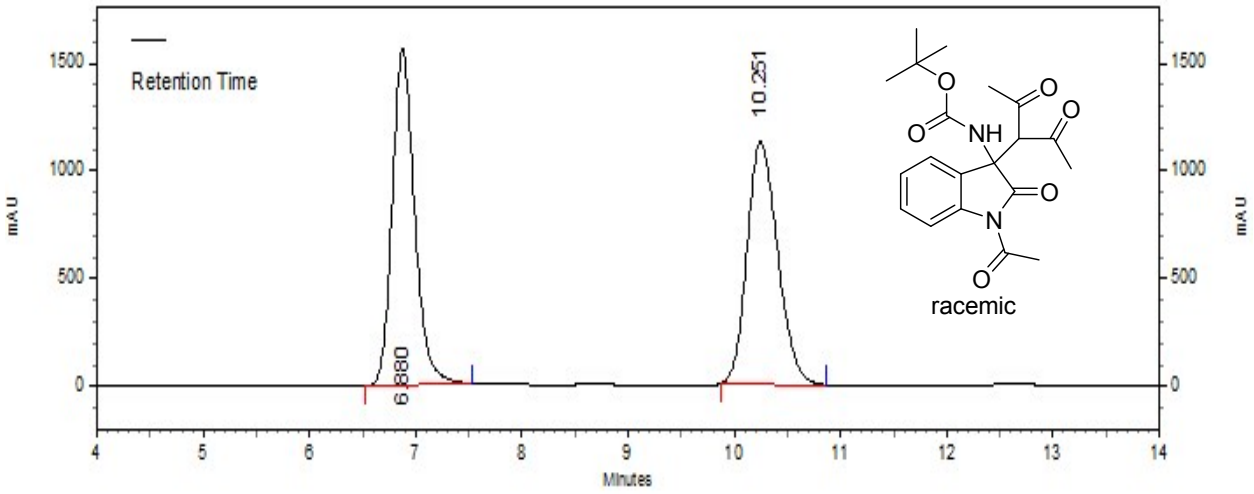
Totals				
		265231186	100.00	1243275



1: 210 nm, 8 nm

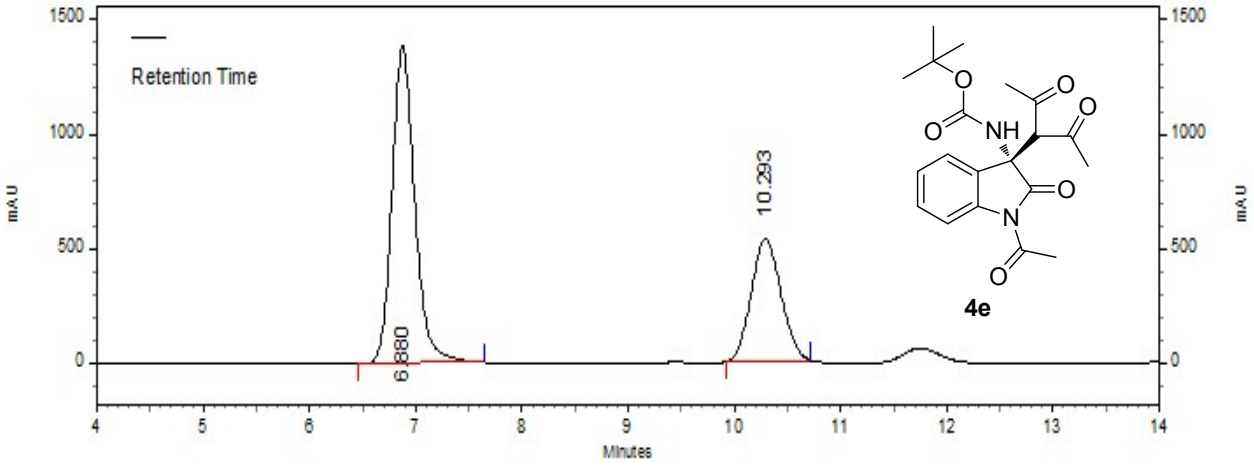
Pk #	Retention Time	Area	Area Percent	Height
1	98.368	798969	0.54	6056
2	105.621	147033369	99.46	629449

Totals				
		147832338	100.00	635505



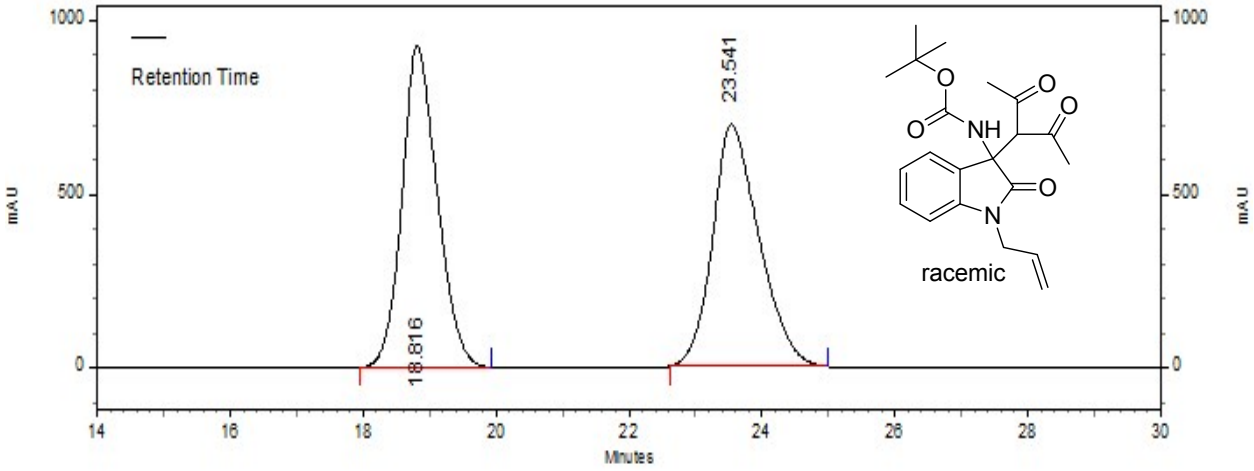
4: 225 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	6.880	22853715	50.37	1561106
2	10.251	22520855	49.63	1128095
Totals		45374570	100.00	2689201



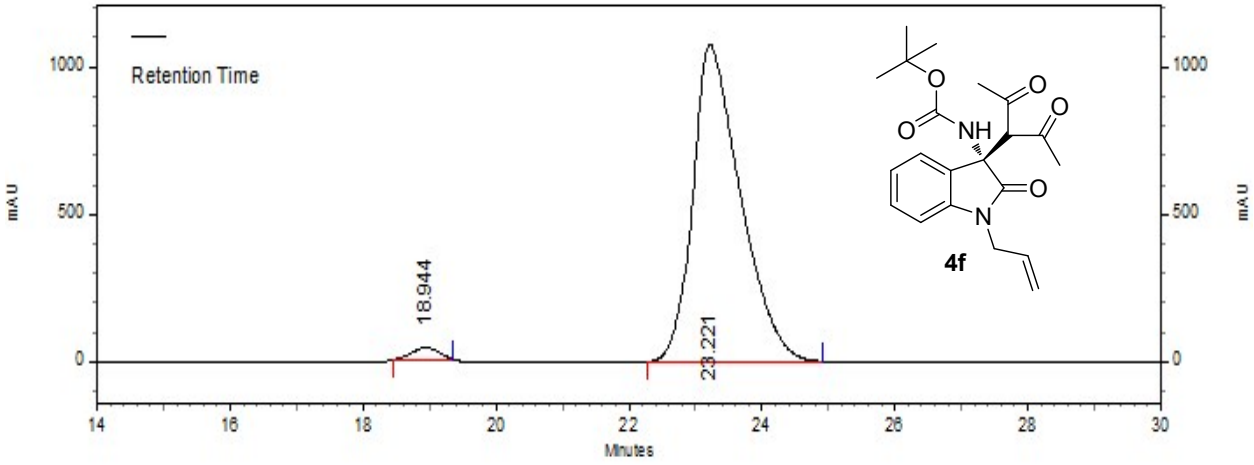
4: 225 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	6.880	20574875	66.45	1384126
2	10.293	10386913	33.55	532709
Totals		30961788	100.00	1916835



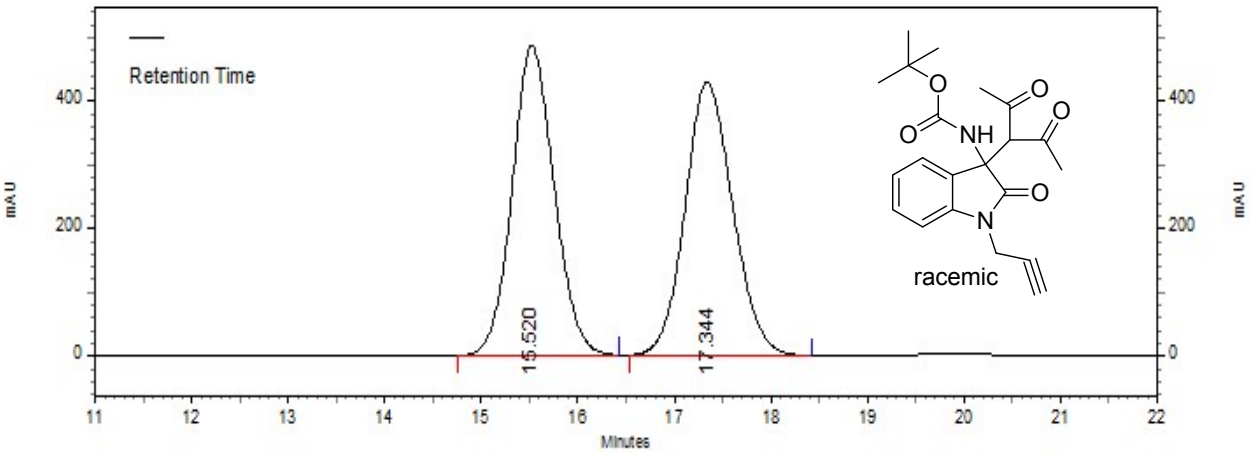
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	18.816	34145838	50.21	925578
2	23.541	33856953	49.79	695877
Totals		68002791	100.00	1621455



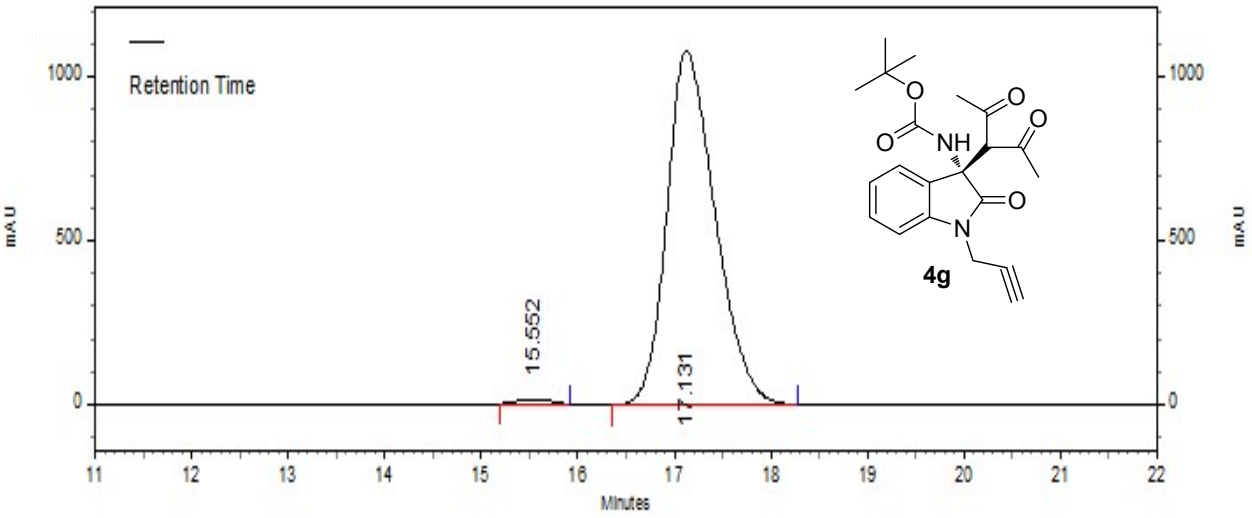
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	18.944	1167691	2.13	40618
2	23.221	53717087	97.87	1074548
Totals		54884778	100.00	1115166



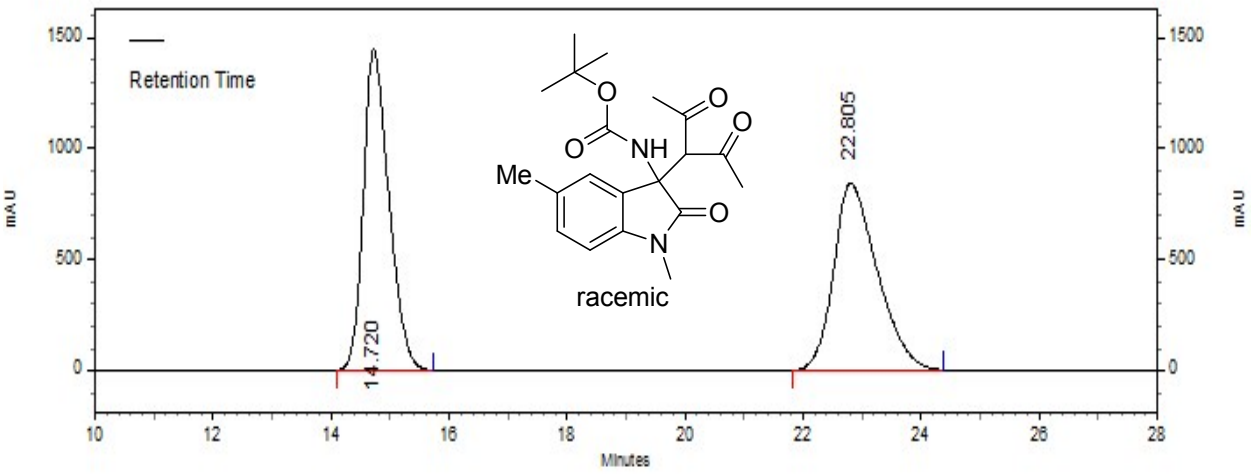
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	15.520	14757940	50.11	486169
2	17.344	14695668	49.89	427721
Totals		29453608	100.00	913890



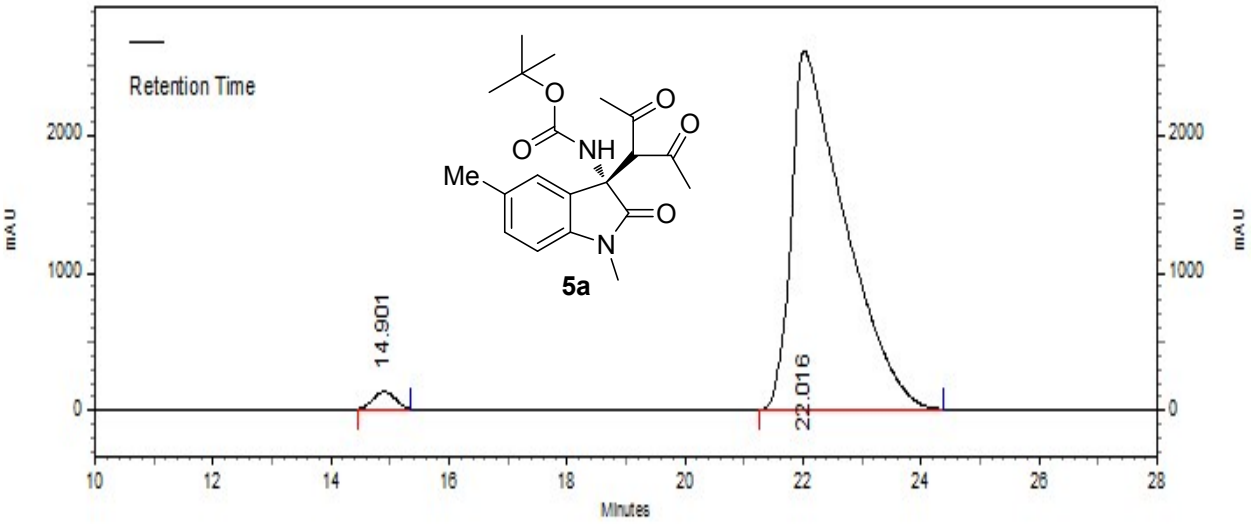
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	15.552	376004	0.99	15792
2	17.131	37524484	99.01	1078000
Totals		37900488	100.00	1093792



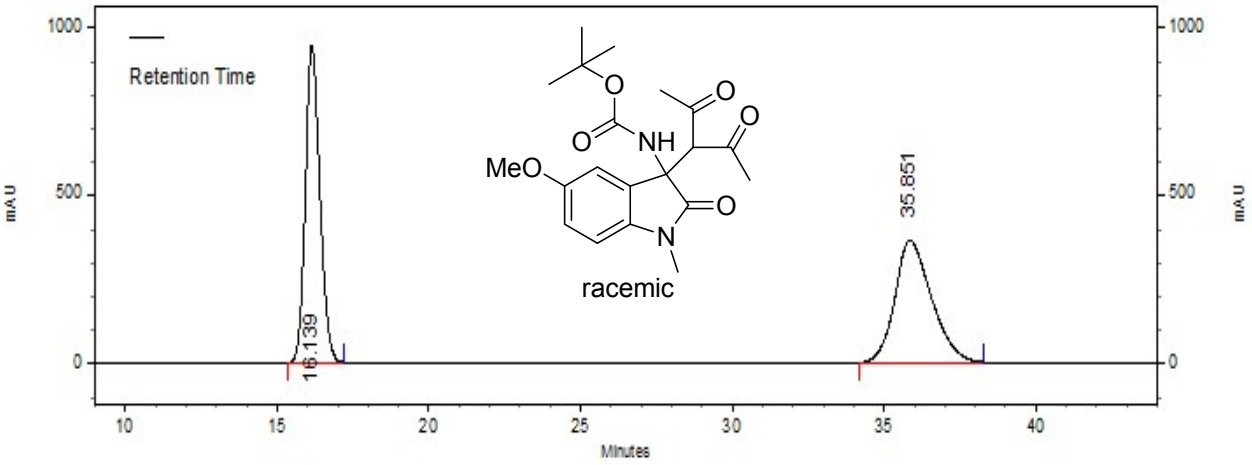
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	14.720	43557630	50.06	1447991
2	22.805	43461005	49.94	843954
Totals		87018635	100.00	2291945



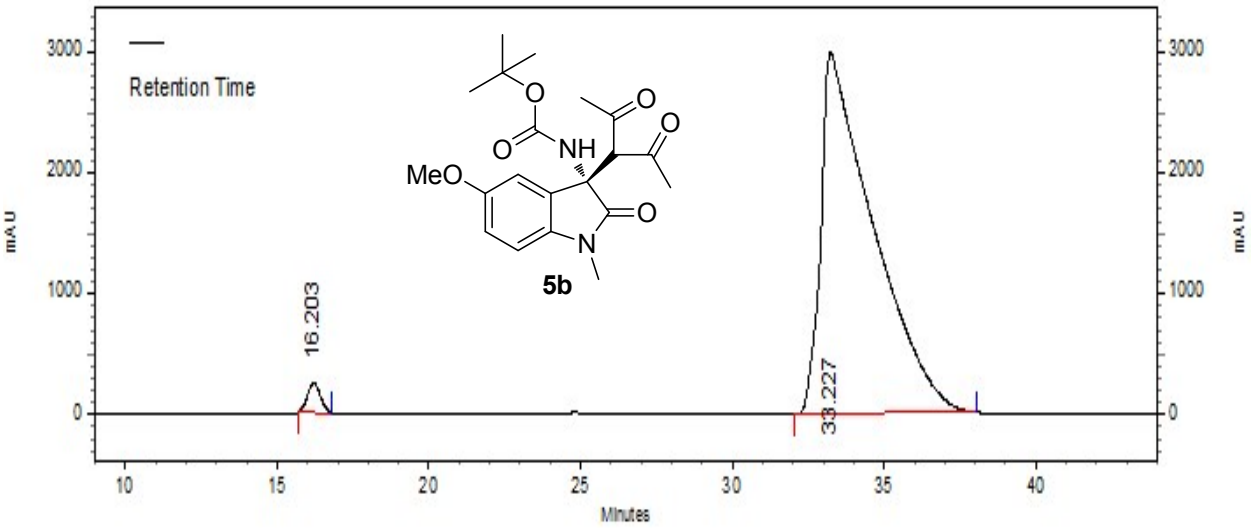
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	14.901	3251926	1.94	125660
2	22.016	164629718	98.06	2604545
Totals		167881644	100.00	2730205



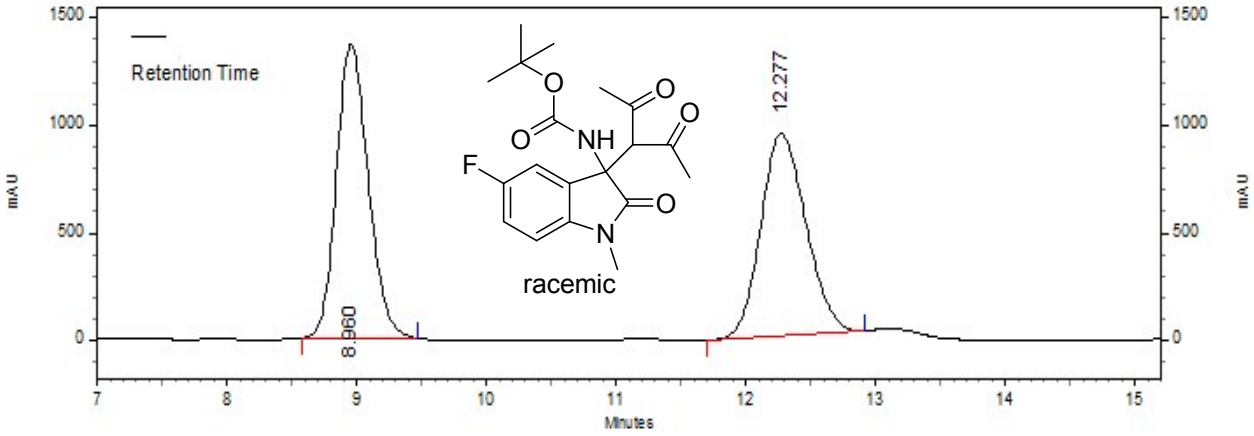
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	16.139	31446637	50.59	944672
2	35.851	30719192	49.41	365804
Totals		62165829	100.00	1310476



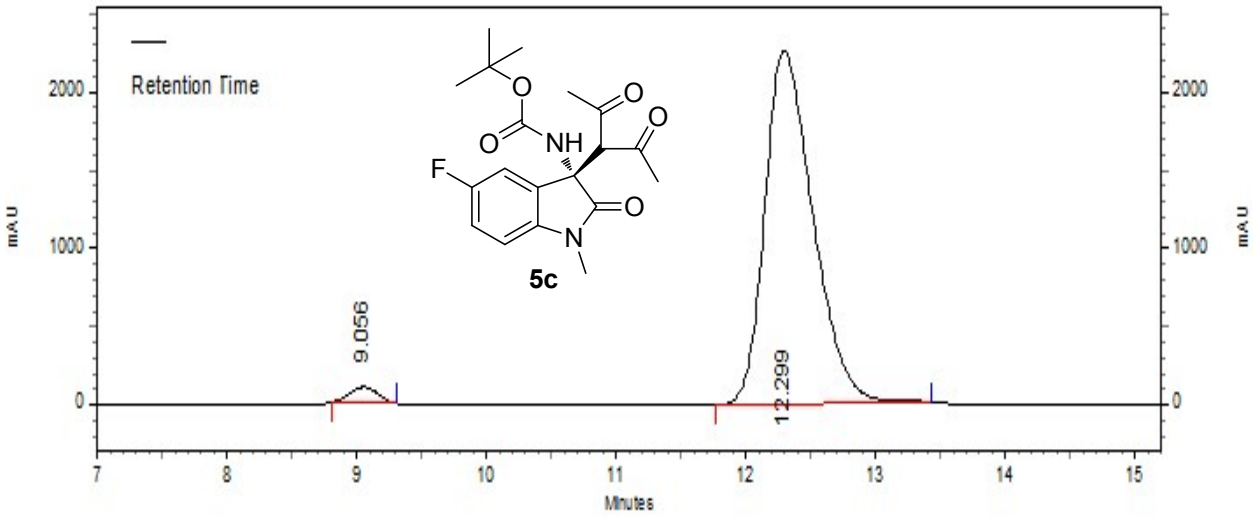
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	16.203	7289824	1.99	242188
2	33.227	359356069	98.01	2997645
Totals		366645893	100.00	3239833



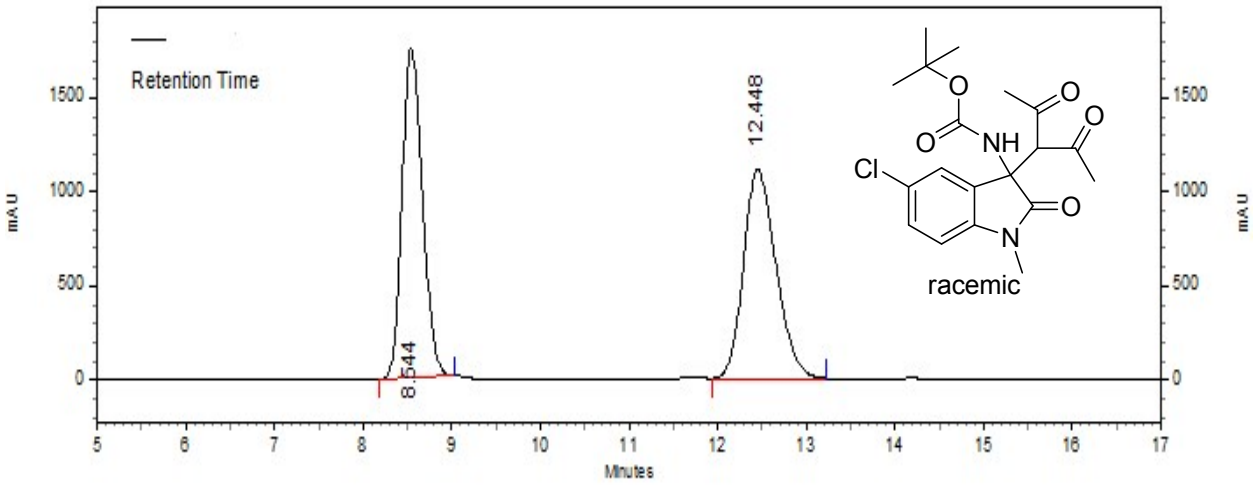
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	8.960	23246556	50.72	1364831
2	12.277	22587951	49.28	937886
Totals		45834507	100.00	2302717



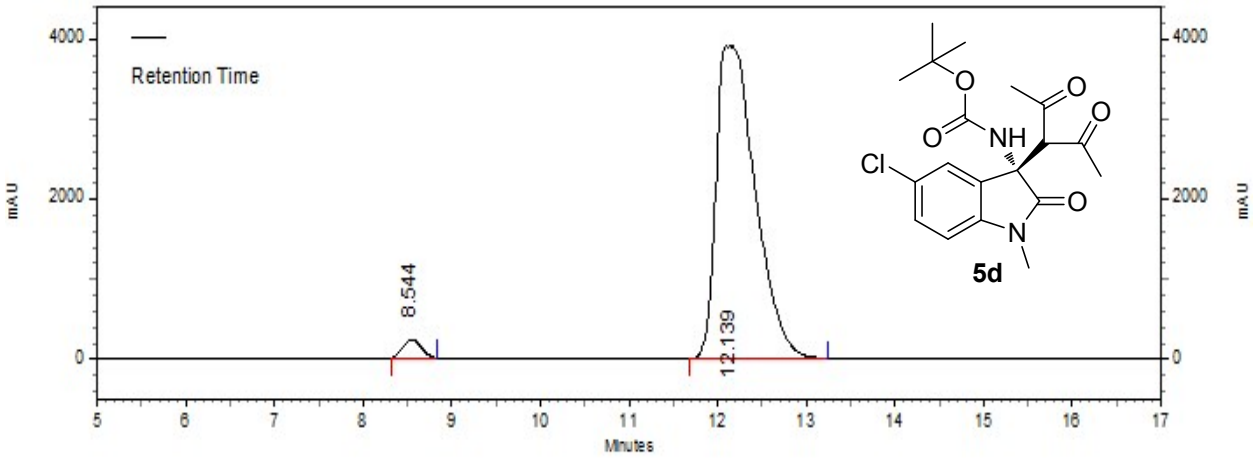
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	9.056	1495242	2.50	99176
2	12.299	58219469	97.50	2253746
Totals		59714711	100.00	2352922



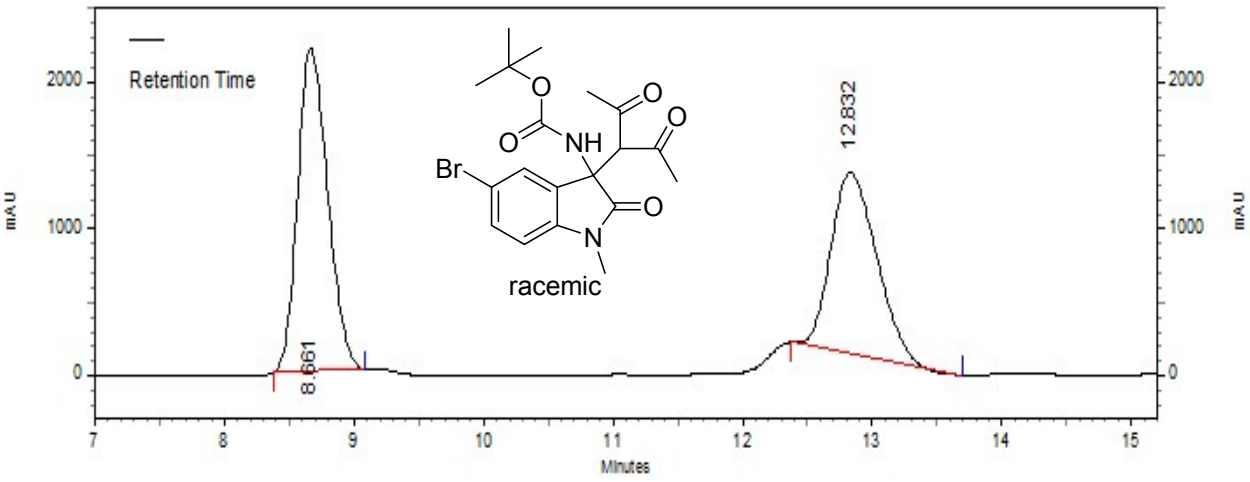
2: 230 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	8.544	28021404	49.66	1753908
2	12.448	28408220	50.34	1115255
Totals		56429624	100.00	2869163



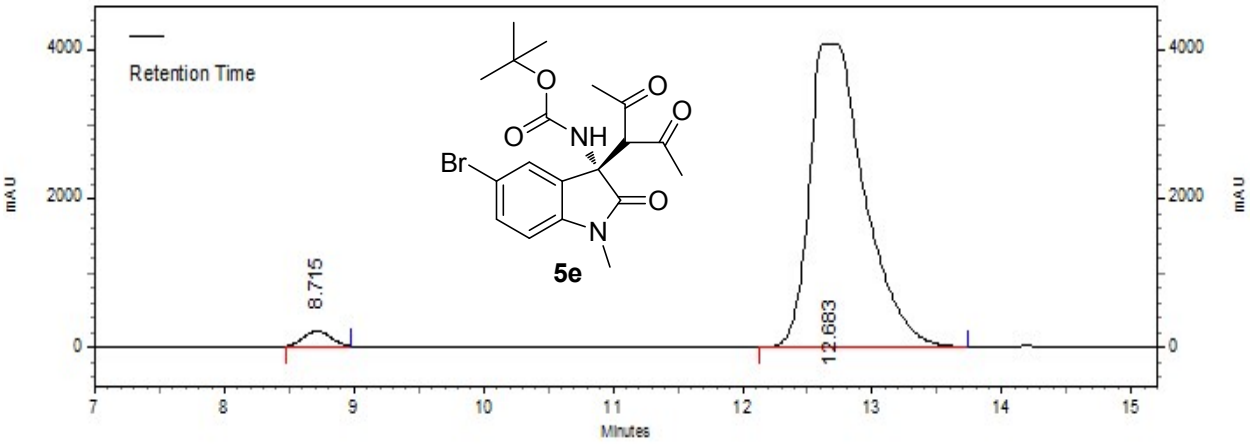
2: 230 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	8.544	3332382	2.70	229771
2	12.139	120293919	97.30	3923303
Totals		123626301	100.00	4153074



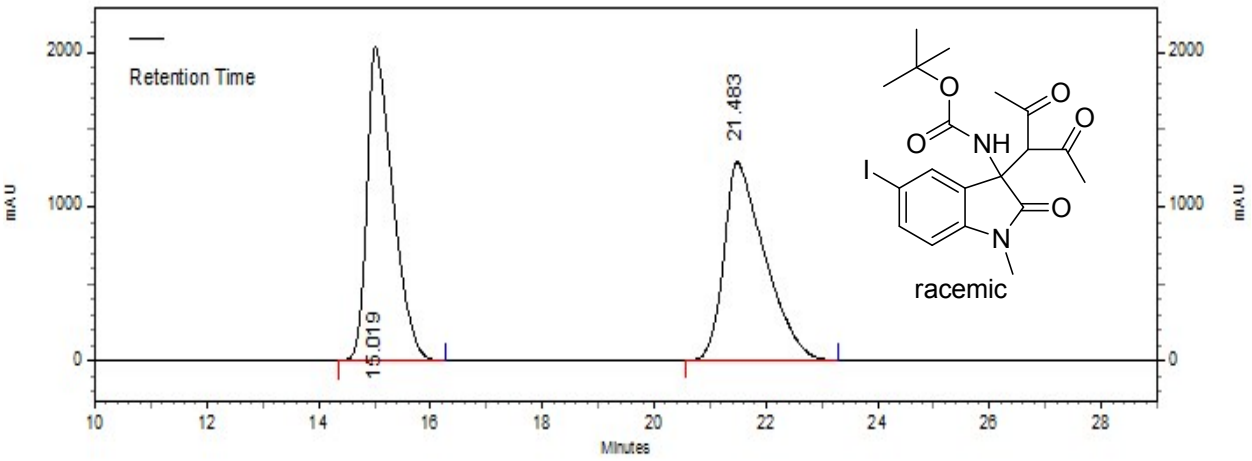
2: 230 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	8.661	35415666	54.04	2196729
2	12.832	30119066	45.96	1230810
Totals		65534732	100.00	3427539



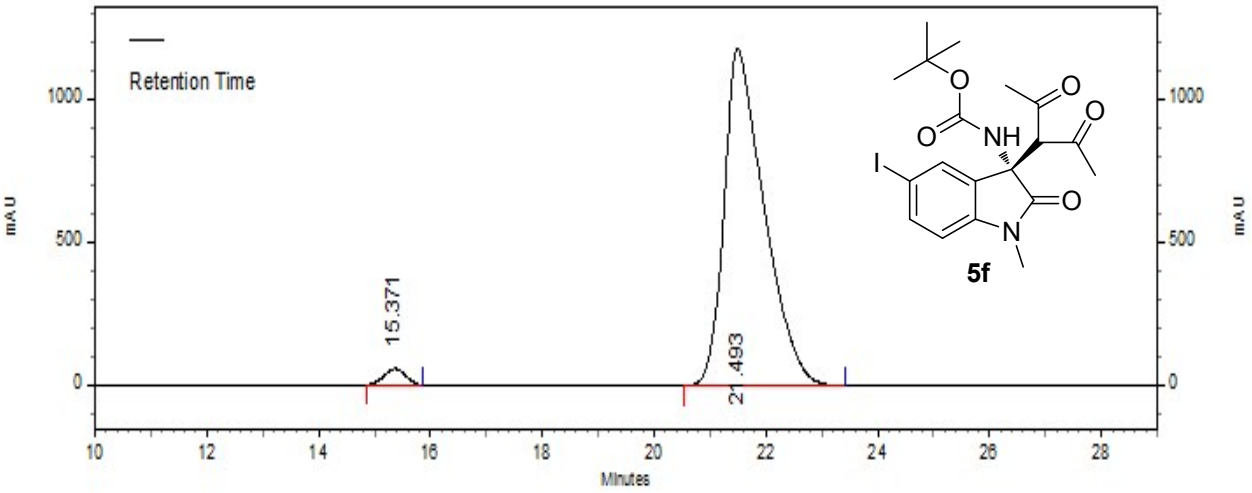
2: 230 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	8.715	3052056	2.48	205699
2	12.683	120171421	97.52	4080972
Totals		123223477	100.00	4286671



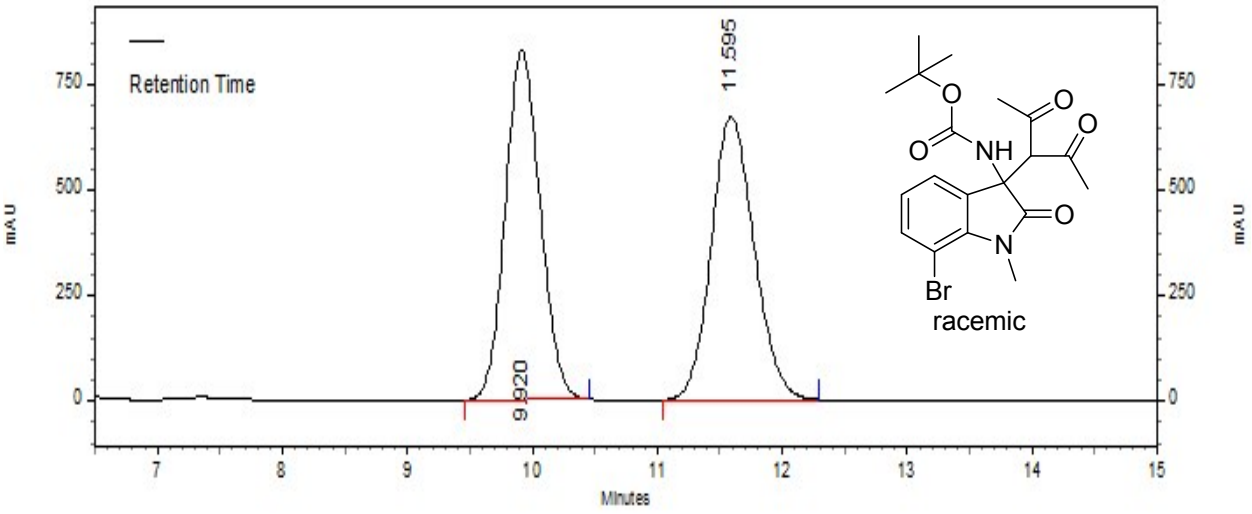
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	15.019	65252576	49.88	2037089
2	21.483	65573119	50.12	1284800
Totals		130825695	100.00	3321889



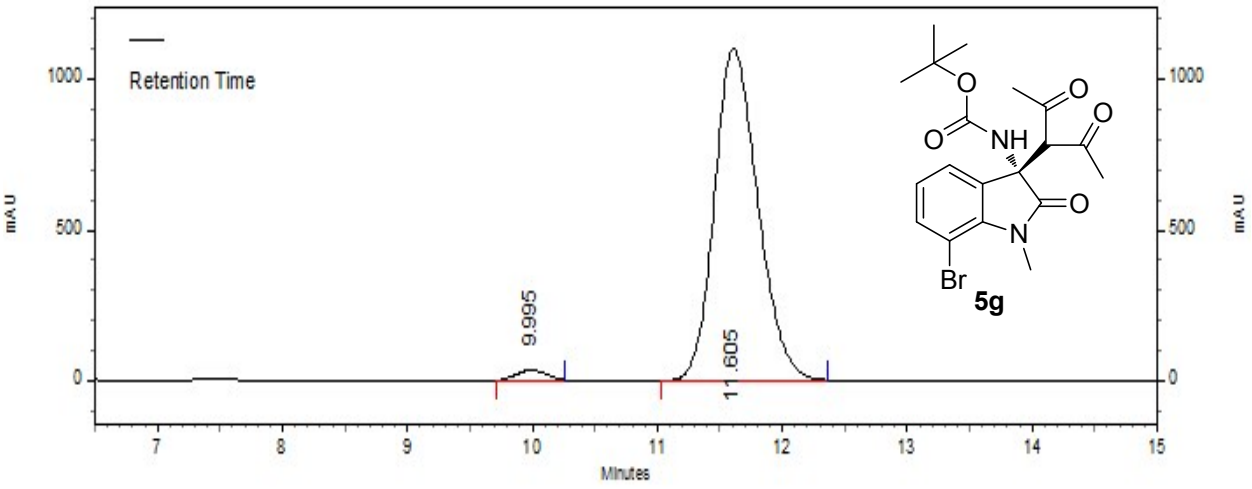
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	15.371	1568232	2.59	57491
2	21.493	58900679	97.41	1178503
Totals		60468911	100.00	1235994



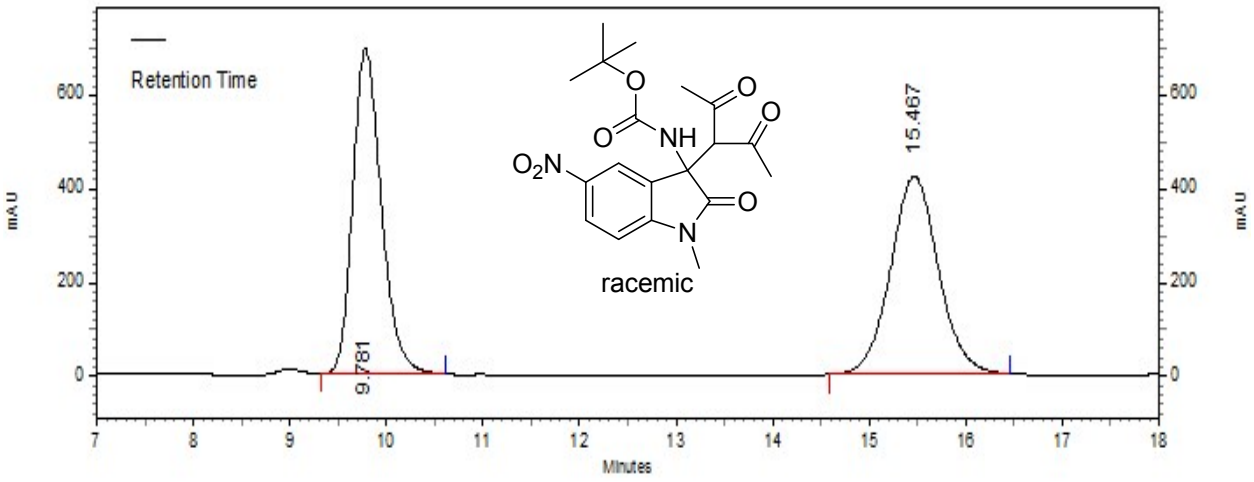
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	9.920	16118672	50.03	830502
2	11.595	16102533	49.97	672020
Totals		32221205	100.00	1502522



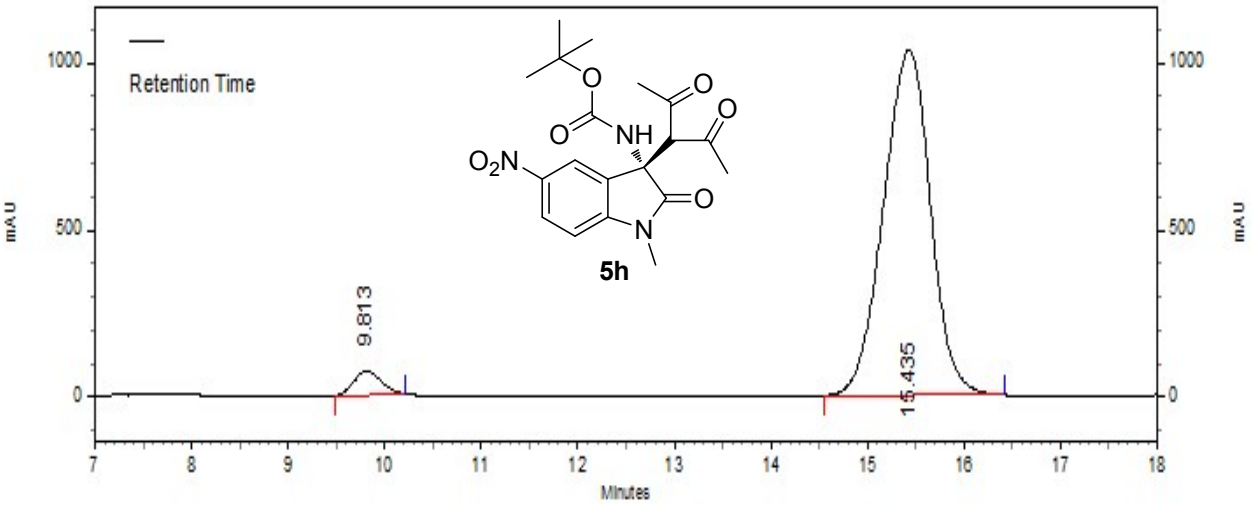
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	9.995	553701	2.05	32743
2	11.605	26504330	97.95	1097171
Totals		27058031	100.00	1129914



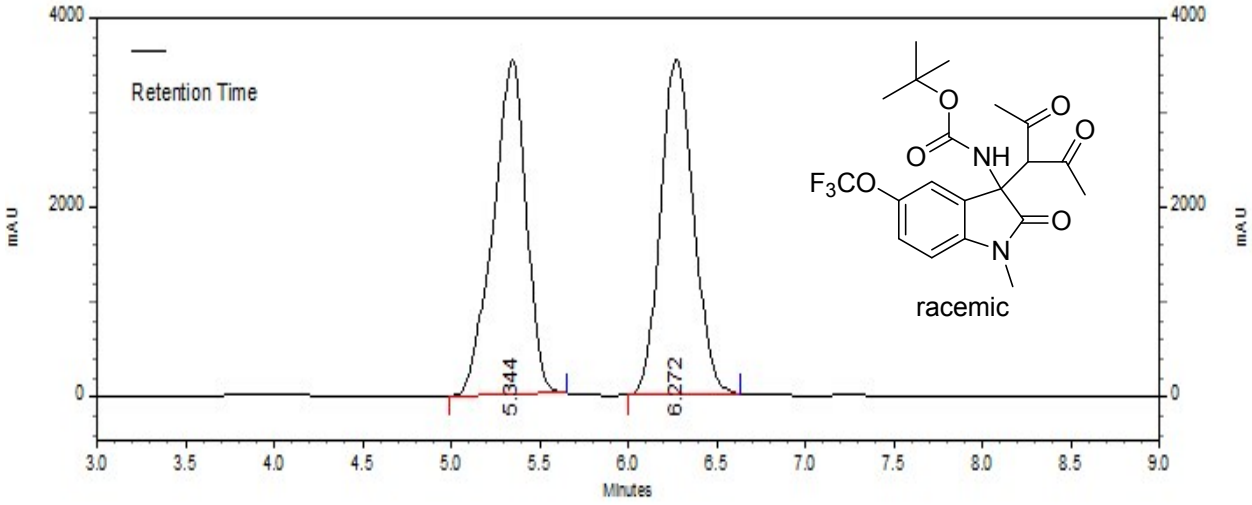
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	9.781	14299677	49.59	695807
2	15.467	14534447	50.41	422601
Totals		28834124	100.00	1118408



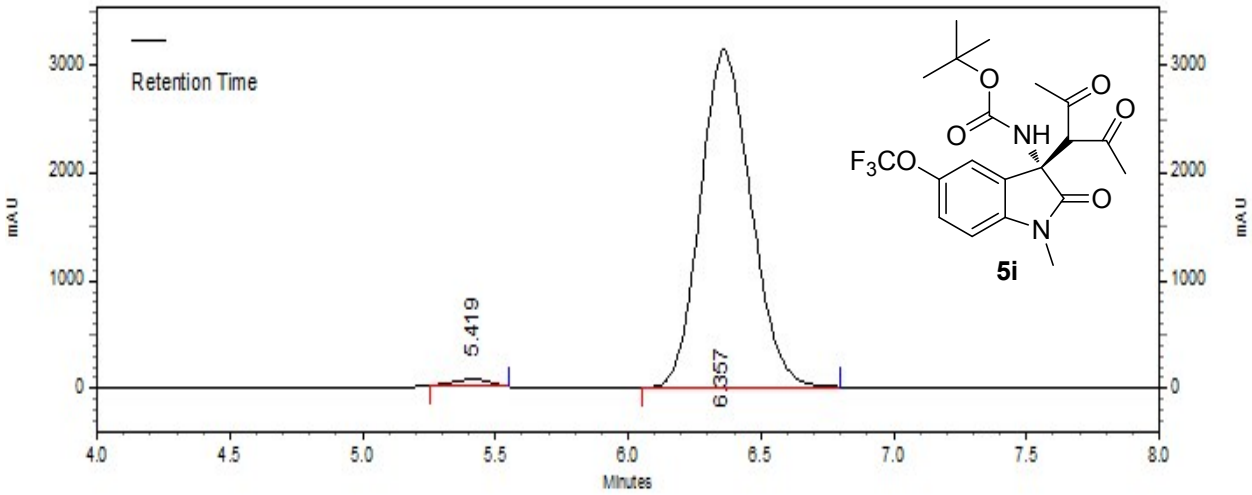
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	9.813	1403601	3.85	72150
2	15.435	35020641	96.15	1034898
Totals		36424242	100.00	1107048



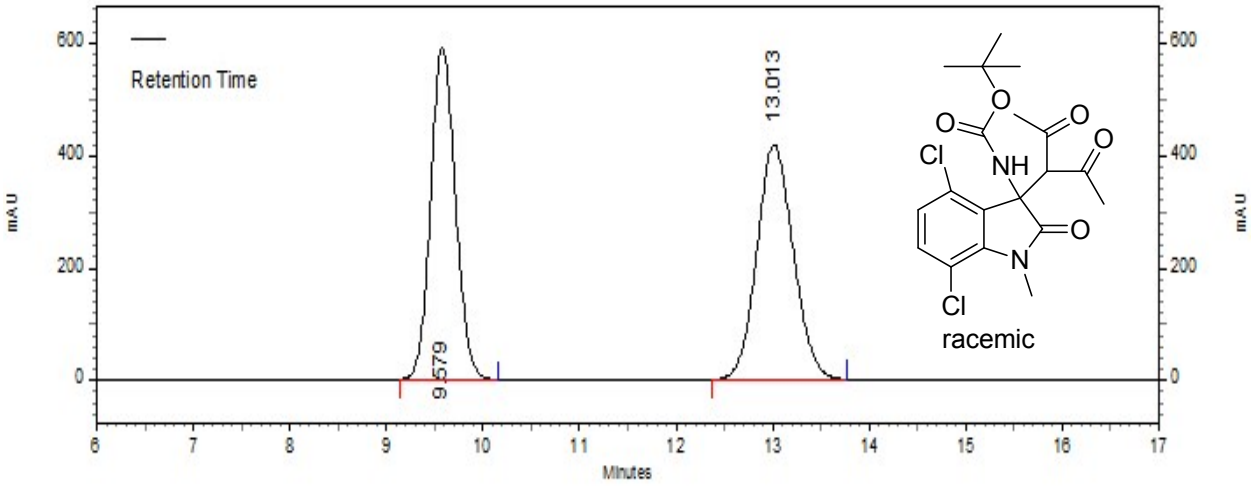
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	5.344	45839186	50.12	3532645
2	6.272	45620121	49.88	3530175
Totals		91459307	100.00	7062820



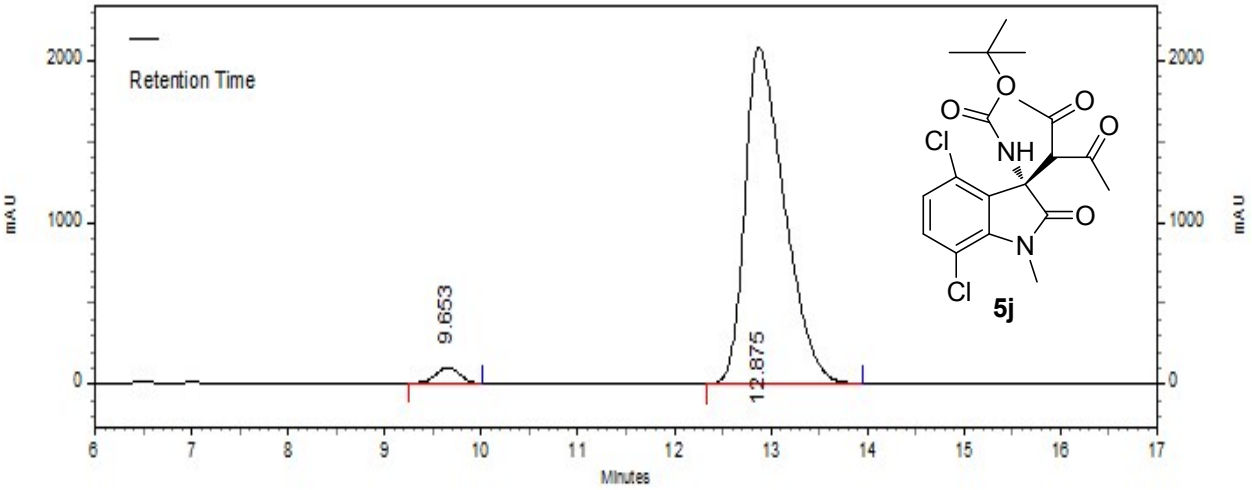
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	5.419	531869	1.26	58723
2	6.357	41545765	98.74	3147689
Totals		42077634	100.00	3206412



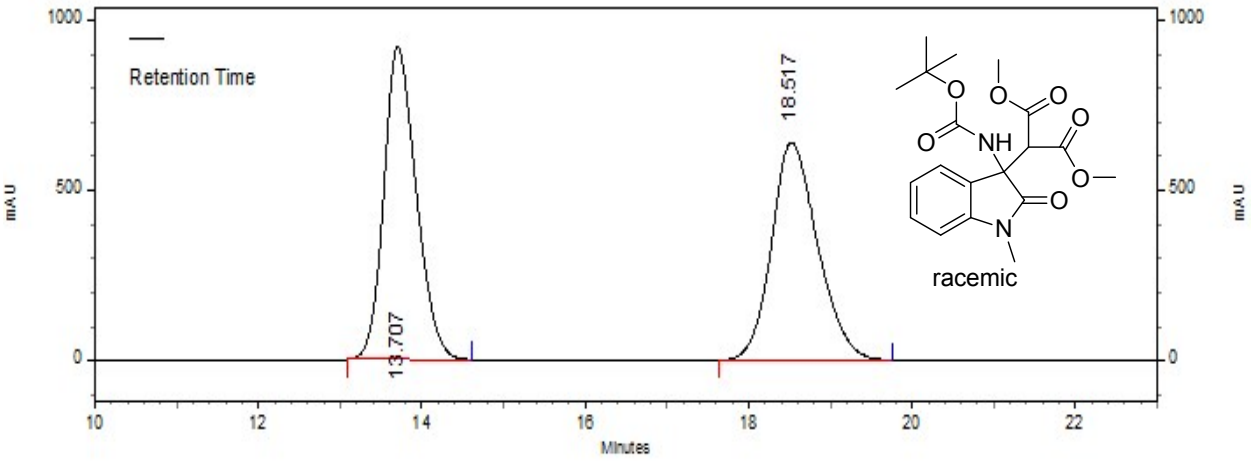
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	9.579	10490136	48.87	590511
2	13.013	10974343	51.13	417974
Totals		21464479	100.00	1008485



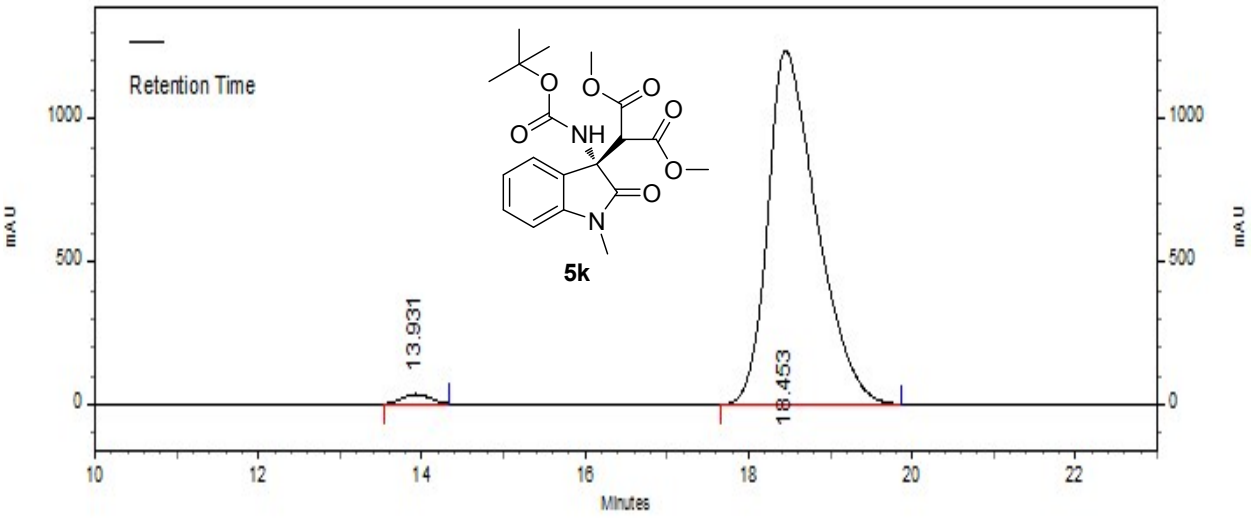
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	9.653	1807176	2.97	101445
2	12.875	59096648	97.03	2080374
Totals		60903824	100.00	2181819



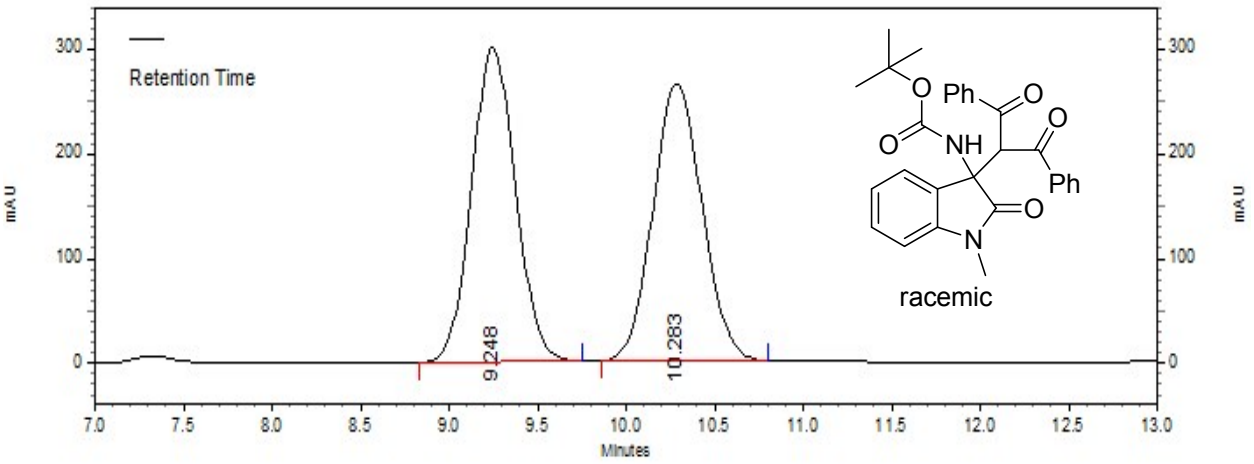
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	13.707	25210458	49.87	918573
2	18.517	25341270	50.13	639767
Totals		50551728	100.00	1558340



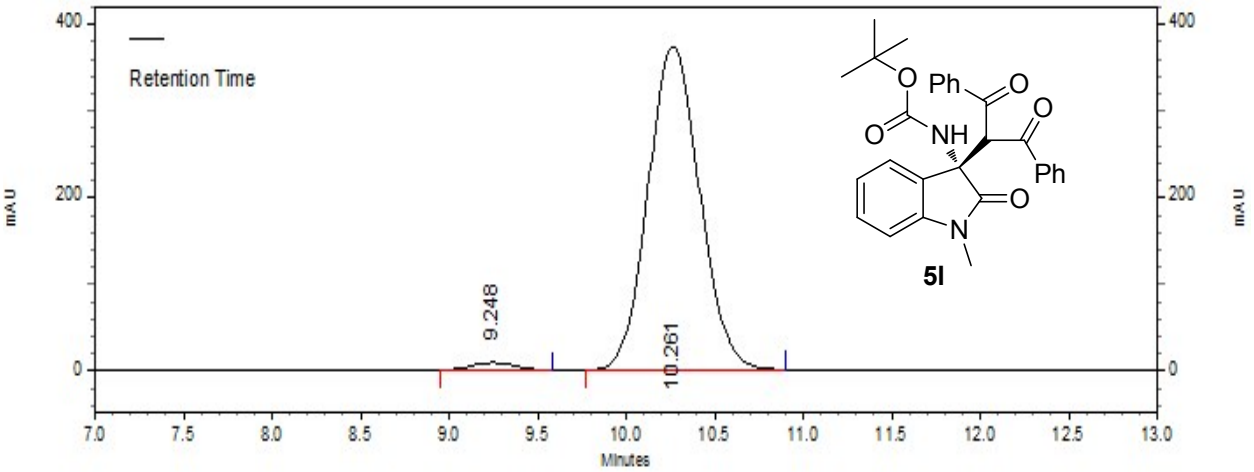
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	13.931	803910	1.53	33640
2	18.453	51910730	98.47	1236224
Totals		52714640	100.00	1269864



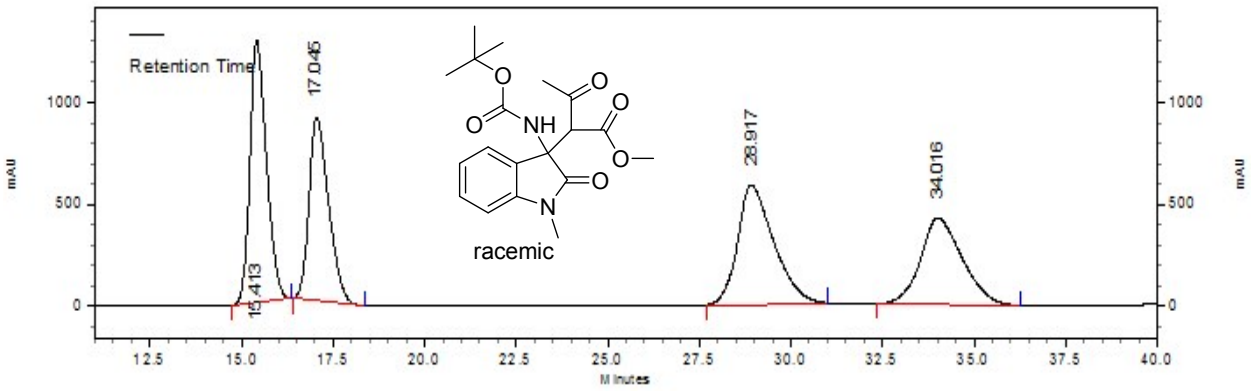
3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	9.248	5205964	50.38	300916
2	10.283	5127438	49.62	264495
Totals		10333402	100.00	565411



3: 254 nm, 8 nm

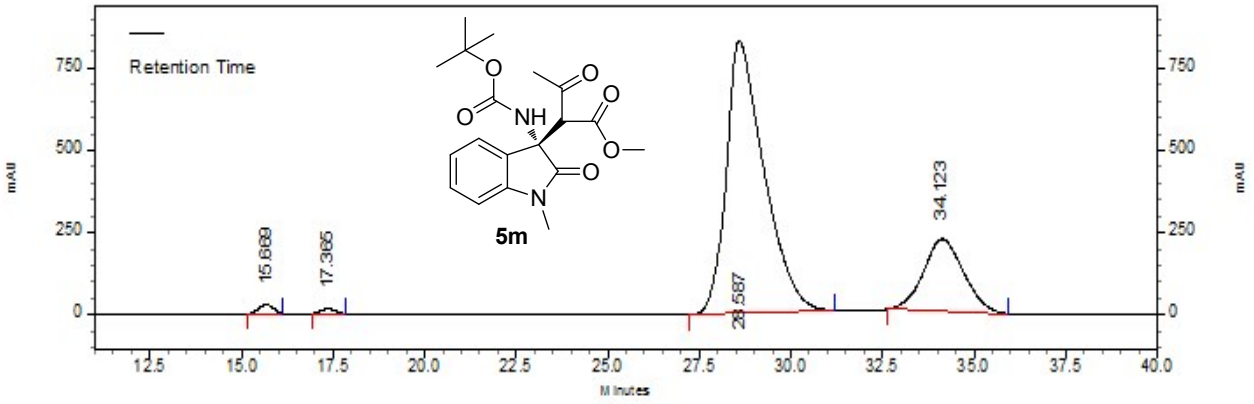
Pk #	Retention Time	Area	Area Percent	Height
1	9.248	149981	1.94	8758
2	10.261	7590272	98.06	373191
Totals		7740253	100.00	381949



3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	15.413	40246586	27.35	1288799
2	17.045	33271425	22.61	901611
3	28.917	39999609	27.18	585388
4	34.016	33661066	22.87	424125

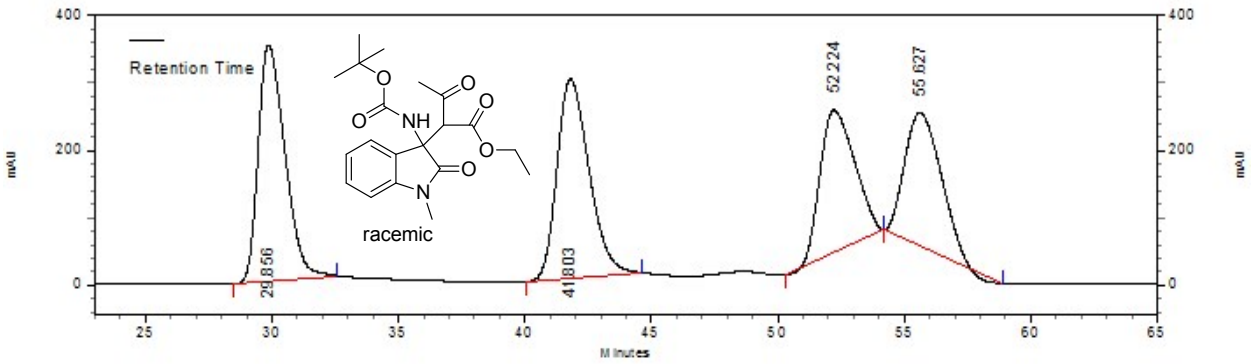
Totals				
		147178686	100.00	3199923



3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	15.669	785960	1.02	28078
2	17.365	432759	0.56	14728
3	28.587	59405700	76.83	829082
4	34.123	16693814	21.59	219006

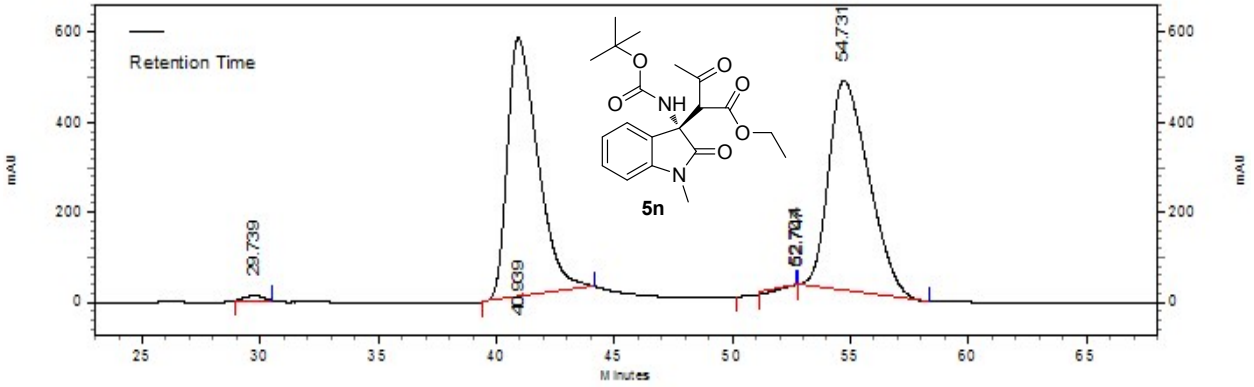
Totals				
		77318233	100.00	1090894



3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	29.856	25657395	28.07	350072
2	41.803	26037267	28.48	295363
3	52.224	20044595	21.93	211133
4	55.627	19681801	21.53	197626

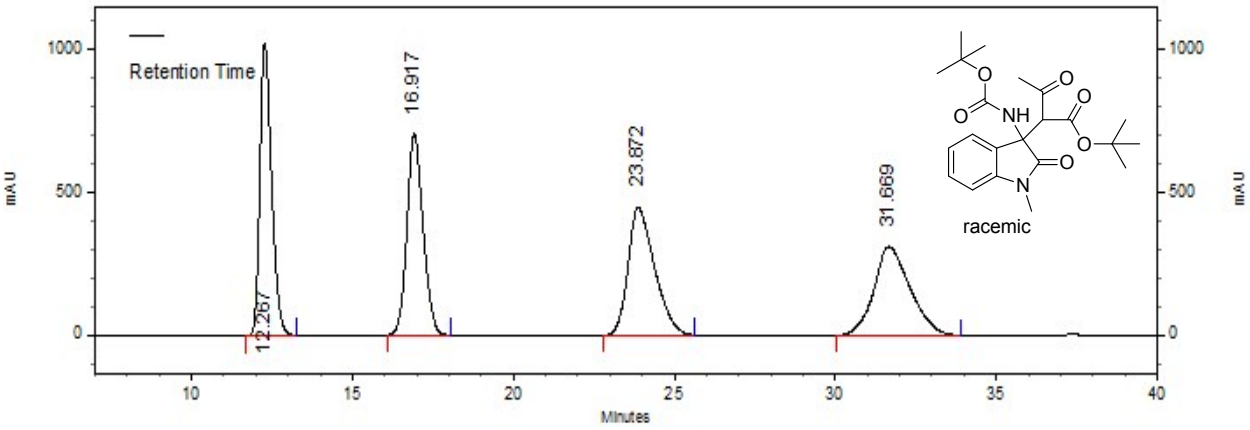
Totals				
		91421058	100.00	1054194



3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	29.739	616603	0.57	11162
2	40.939	51725824	47.78	576477
3	52.747	442206	0.41	17
4	54.731	55211778	51.00	466881

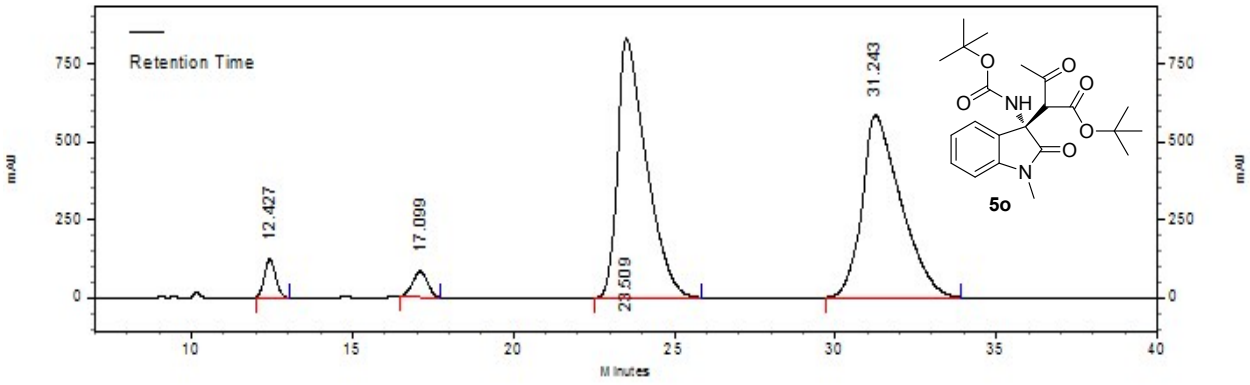
Totals				
		108248384	100.00	1054599



3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	12.267	25853632	25.73	1022331
2	16.917	24566640	24.45	703002
3	23.872	25677157	25.55	447132
4	31.669	24385402	24.27	310364

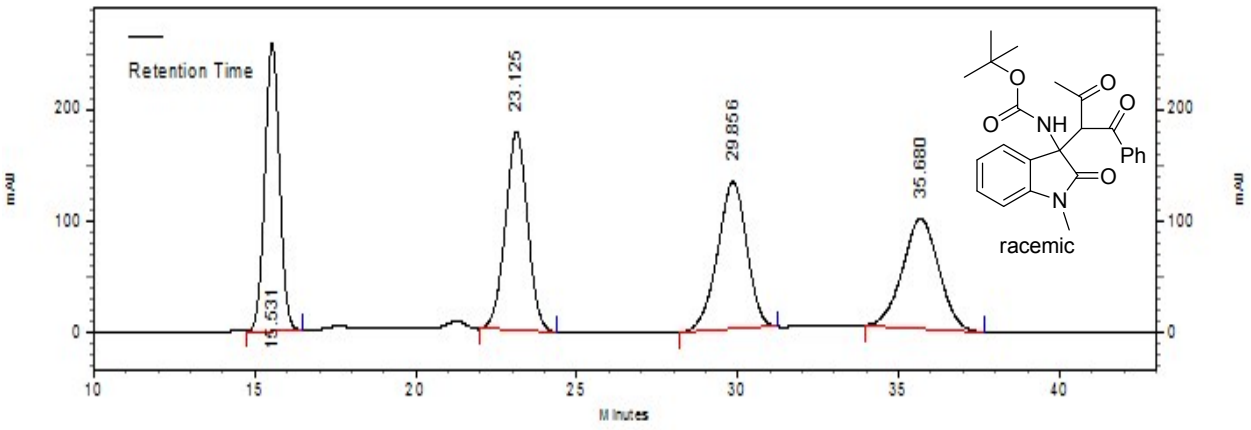
Totals				
		100482831	100.00	2482829



3: 254 nm, 8 nm

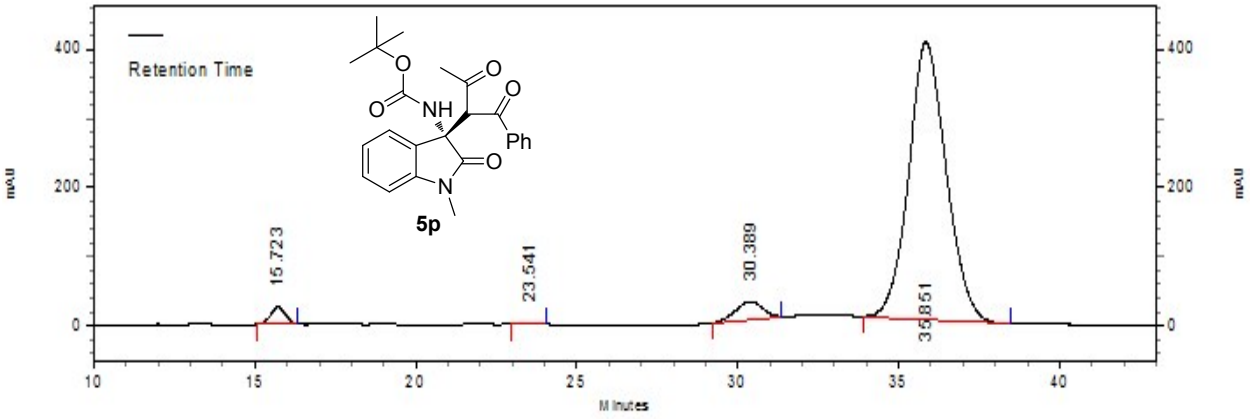
Pk #	Retention Time	Area	Area Percent	Height
1	12.427	3005386	2.83	123580
2	17.099	2687326	2.53	82760
3	23.509	51413117	48.47	830215
4	31.243	48974680	46.17	582900

Totals				
		106080509	100.00	1619455



3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	15.531	8139306	24.79	257482
2	23.125	8574362	26.12	178329
3	29.856	8402038	25.59	132360
4	35.680	7716533	23.50	98585
Totals		32832239	100.00	666756



3: 254 nm, 8 nm

Pk #	Retention Time	Area	Area Percent	Height
1	15.723	762394	2.18	24591
2	23.541	80017	0.23	2225
3	30.389	1514722	4.32	25880
4	35.851	32674347	93.27	402534
Totals		35031480	100.00	455230

5) **X-ray structure report for product 5g**

Experimental

Data Collection

A colorless chunk crystal of $C_{19}H_{23}BrN_2O_5$ having approximate dimensions of 0.470 x 0.390 x 0.320 mm was mounted on a glass fiber. All measurements were made on a Rigaku SCX mini diffractometer using graphite monochromated Mo-Kα radiation.

The crystal-to-detector distance was 52.00 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 8.9423(8) Å
b = 10.714(1) Å β = 94.596(3)^o
c = 10.4490(9) Å
V = 997.8(2) Å^3

For Z = 2 and F.W. = 439.31, the calculated density is 1.462 g/cm^3. Based on the reflection conditions of:

0k0: k = 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$P2_1$ (#4)

The data were collected at a temperature of 20 $\pm$ 1^{o}C to a maximum 2θ value of 55.0^o. A total of 540 oscillation images were collected. A sweep of data was done using ω oscillations from -120.0 to 60.0^o in 1.0^o steps. The exposure rate was 8.0 [sec./^{o}]. The detector swing angle was -30.80^o. A second sweep was performed using ω oscillations from -120.0 to 60.0^o in 1.0^o steps. The exposure rate was 8.0 [sec./^{o}]. The detector swing angle was -30.80^o. Another sweep was performed using ω oscillations from -120.0 to 60.0^o in 1.0^o steps. The exposure rate was 8.0 [sec./^{o}]. The detector swing angle was -30.80^o. The crystal-to-detector distance was 52.00 mm. Readout was performed in the 0.146 mm pixel mode.

Data Reduction

Of the 10269 reflections that were collected, 4547 were unique (R_{int} = 0.0684); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku).

The linear absorption coefficient, μ, for Mo-Kα radiation is 20.973 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.251 to 0.511. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 4547 observed reflections and 248 variable parameters and converged (largest parameter shift was 0.02 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0559$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1614$$

The standard deviation of an observation of unit weight[4] was 0.95. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.55 and -0.54 $e^-/Å^3$, respectively. The absolute structure was deduced based on Flack parameter, 0.036(10), using 2140 Friedel pairs.[5]

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Δf' and Δf'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10] crystallographic software package except for refinement, which was performed using SHELXL-97[11].

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(3) Least Squares function minimized: (SHELXL97)

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Flack, H. D. (1983), Acta Cryst. A39, 876-881.

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 4.0: Crystal Structure Analysis Package, Rigaku Corporation (2000-2010). Tokyo 196-8666, Japan.

(11) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{19}H_{23}BrN_2O_5$
Formula Weight	439.31
Crystal Color, Habit	colorless, chunk
Crystal Dimensions	0.470 X 0.390 X 0.320 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 8.9423(8) Å b = 10.714(1) Å c = 10.4490(9) Å β = 94.596(3) ^{o} V = 997.8(2) $Å^3$
Space Group	$P2_1$ (#4)
Z value	2
D_{calc}	1.462 g/cm^3
F_{000}	452.00
μ(MoKα)	20.973 cm^{-1}

B. Intensity Measurements

Diffractometer	SCX mini
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 30mA
Temperature	20.0^{o}C
Detector Aperture	75 mm (diameter)
Data Images	540 exposures
ω oscillation Range	-120.0 - 60.0^{o}
Exposure Rate	8.0 sec./^{o}
Detector Swing Angle	-30.80^{o}
ω oscillation Range	-120.0 - 60.0^{o}
Exposure Rate	8.0 sec./^{o}
Detector Swing Angle	-30.80^{o}
ω oscillation Range	-120.0 - 60.0^{o}
Exposure Rate	8.0 sec./^{o}
Detector Swing Angle	-30.80^{o}
Detector Position	52.00 mm
Pixel Size	0.146 mm
$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 10269 Unique: 4547 (R_{int} = 0.0684) Friedel pairs: 2140

Corrections	Lorentz-polarization Absorption (trans. factors: 0.251 - 0.511)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(Fo^2) + (0.1000 \cdot P)^2 + 0.0000 \cdot P]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	4547
No. Variables	248
Reflection/Parameter Ratio	18.33
Residuals: R1 ($I>2.00\sigma(I)$)	0.0559
Residuals: R (All reflections)	0.0683
Residuals: wR2 (All reflections)	0.1614
Goodness of Fit Indicator	0.952
Flack Parameter (Friedel pairs = 2140)	0.036(10)
Max Shift/Error in Final Cycle	0.016
Maximum peak in Final Diff. Map	0.55 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.54 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Br1	0.26330(6)	-1.20023(3)	0.68976(5)	5.59(2)
O1	0.3960(3)	-0.7981(4)	1.0328(4)	4.83(7)
O2	0.2760(6)	-0.4809(4)	0.7496(5)	6.7(1)
O3	0.3646(4)	-0.6795(3)	0.7415(3)	3.95(6)
O4	-0.1196(4)	-0.6230(4)	0.9239(5)	5.40(8)
O5	0.1167(6)	-0.6139(5)	1.1374(5)	6.4(1)
N1	0.3168(4)	-0.9436(3)	0.8810(4)	3.17(6)
N2	0.1881(5)	-0.6335(4)	0.8694(5)	3.98(8)
C1	0.3096(5)	-0.8331(4)	0.9454(4)	3.41(7)
C2	0.1661(5)	-0.7652(4)	0.8921(4)	3.01(7)
C3	0.1184(4)	-0.8420(4)	0.7758(4)	3.08(6)
C4	0.0084(5)	-0.8201(5)	0.6801(4)	3.99(8)
C5	-0.0173(6)	-0.9065(6)	0.5829(5)	5.1(1)
C6	0.0637(7)	-1.0163(6)	0.5869(5)	4.9(1)
C7	0.1738(5)	-1.0398(4)	0.6843(4)	3.91(8)
C8	0.2049(4)	-0.9498(4)	0.7789(4)	2.87(6)
C9	0.2780(5)	-0.5884(4)	0.7833(5)	3.91(8)
C10	0.4563(5)	-0.6613(5)	0.6311(5)	4.01(8)
C11	0.5778(7)	-0.5689(7)	0.6634(6)	5.9(2)
C12	0.3560(8)	-0.6221(9)	0.5143(6)	7.2(2)
C13	0.5196(8)	-0.7905(6)	0.6137(7)	6.0(2)
C14	0.0510(4)	-0.7842(4)	0.9961(4)	3.18(7)
C15	-0.1027(5)	-0.7312(5)	0.9543(4)	3.70(8)
C16	-0.2317(5)	-0.8193(5)	0.9553(5)	4.33(9)
C17	0.1037(5)	-0.7245(5)	1.1265(5)	4.14(9)
C18	0.1309(8)	-0.8138(7)	1.2366(6)	6.1(2)
C19	0.4410(5)	-1.0292(5)	0.9128(6)	4.61(9)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H2	0.1409	-0.5810	0.9137	4.77
H4	-0.0486	-0.7475	0.6804	4.79
H5A	-0.0887	-0.8909	0.5152	6.12
H11A	0.5342	-0.4899	0.6828	7.12
H11B	0.6375	-0.5595	0.5917	7.12
H11C	0.6400	-0.5976	0.7367	7.12
H12A	0.4147	-0.6148	0.4417	8.58
H12B	0.3106	-0.5431	0.5306	8.58
H12C	0.2791	-0.6837	0.4967	8.58
H13A	0.5777	-0.7910	0.5404	7.23
H13B	0.4389	-0.8493	0.6009	7.23
H13C	0.5825	-0.8131	0.6889	7.23
H14	0.0398	-0.8741	1.0096	3.82
H16A	-0.2853	-0.8206	0.8720	5.20
H16B	-0.2977	-0.7924	1.0180	5.20
H16C	-0.1952	-0.9015	0.9767	5.20
H18A	0.0624	-0.7961	1.3004	7.37
H18B	0.2321	-0.8047	1.2736	7.37
H18C	0.1156	-0.8978	1.2062	7.37
H19A	0.5050	-1.0316	0.8433	5.53
H19B	0.4024	-1.1112	0.9267	5.53
H19C	0.4975	-1.0010	0.9894	5.53
H6	0.057(8)	-1.09(1)	0.521(8)	9(2)

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br1	0.0950(4)	0.0357(3)	0.0848(4)	0.0040(3)	0.0262(3)	-0.0168(3)
O1	0.042(2)	0.066(3)	0.074(2)	-0.007(2)	-0.006(2)	-0.022(2)
O2	0.118(4)	0.027(2)	0.117(4)	0.003(2)	0.056(3)	0.012(2)
O3	0.058(2)	0.030(2)	0.065(2)	-0.001(2)	0.026(2)	0.001(2)
O4	0.057(2)	0.046(2)	0.104(3)	0.015(2)	0.018(2)	0.019(2)
O5	0.104(3)	0.057(3)	0.086(3)	-0.011(3)	0.035(3)	-0.032(3)
N1	0.034(2)	0.030(2)	0.058(2)	0.003(2)	0.012(2)	-0.002(2)
N2	0.056(2)	0.028(2)	0.070(3)	-0.001(2)	0.027(2)	-0.003(2)
C1	0.040(2)	0.032(2)	0.059(3)	-0.006(2)	0.013(2)	-0.005(2)
C2	0.039(2)	0.026(2)	0.052(3)	-0.001(2)	0.015(2)	-0.001(2)
C3	0.041(2)	0.030(2)	0.047(2)	-0.002(2)	0.010(2)	0.002(2)
C4	0.054(3)	0.044(3)	0.055(3)	0.002(2)	0.005(2)	0.007(2)
C5	0.071(3)	0.072(4)	0.050(3)	-0.009(3)	-0.004(3)	-0.000(3)
C6	0.083(4)	0.058(3)	0.046(3)	-0.012(3)	0.006(3)	-0.010(2)
C7	0.062(3)	0.037(3)	0.052(3)	-0.003(2)	0.021(2)	-0.006(2)
C8	0.040(2)	0.026(2)	0.045(2)	-0.005(2)	0.013(2)	-0.002(2)
C9	0.060(3)	0.028(2)	0.062(3)	-0.003(2)	0.016(2)	-0.002(2)
C10	0.054(3)	0.047(3)	0.053(3)	-0.005(2)	0.013(2)	0.006(2)
C11	0.065(3)	0.079(5)	0.081(4)	-0.020(3)	0.004(3)	0.016(4)
C12	0.077(4)	0.134(7)	0.059(3)	0.003(5)	-0.003(3)	0.004(4)
C13	0.090(4)	0.063(4)	0.082(4)	0.009(4)	0.043(3)	-0.000(3)
C14	0.042(2)	0.030(2)	0.050(2)	0.002(2)	0.016(2)	-0.001(2)
C15	0.042(2)	0.043(3)	0.057(3)	0.005(2)	0.012(2)	-0.002(2)
C16	0.044(2)	0.048(3)	0.073(3)	-0.001(2)	0.006(2)	0.005(3)
C17	0.052(3)	0.050(4)	0.057(3)	-0.002(2)	0.015(2)	-0.014(2)
C18	0.089(4)	0.088(5)	0.054(3)	0.006(4)	-0.006(3)	-0.008(3)
C19	0.048(3)	0.044(3)	0.084(4)	0.010(2)	0.005(2)	0.002(3)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Br1	C7	1.895(5)	O1	C1	1.208(5)
O2	C9	1.205(6)	O3	C9	1.341(6)
O3	C10	1.481(6)	O4	C15	1.209(6)
O5	C17	1.195(7)	N1	C1	1.365(6)
N1	C8	1.404(5)	N1	C19	1.459(6)
N2	C2	1.446(6)	N2	C9	1.344(7)
C1	C2	1.541(6)	C2	C3	1.501(6)
C2	C14	1.568(6)	C3	C4	1.365(6)
C3	C8	1.389(6)	C4	C5	1.380(7)
C5	C6	1.380(9)	C6	C7	1.381(7)
C7	C8	1.393(6)	C10	C11	1.489(8)
C10	C12	1.515(8)	C10	C13	1.512(8)
C14	C15	1.519(6)	C14	C17	1.544(6)
C15	C16	1.491(7)	C17	C18	1.501(8)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
N2	H2	0.860	C4	H4	0.930
C5	H5A	0.930	C6	H6	1.07(10)
C11	H11A	0.960	C11	H11B	0.960
C11	H11C	0.960	C12	H12A	0.960
C12	H12B	0.960	C12	H12C	0.960
C13	H13A	0.960	C13	H13B	0.960
C13	H13C	0.960	C14	H14	0.980
C16	H16A	0.960	C16	H16B	0.960
C16	H16C	0.960	C18	H18A	0.960
C18	H18B	0.960	C18	H18C	0.960
C19	H19A	0.960	C19	H19B	0.960
C19	H19C	0.960			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C9	O3	C10	121.9(4)	C1	N1	C8	110.9(3)
C1	N1	C19	119.8(4)	C8	N1	C19	128.8(4)
C2	N2	C9	123.8(4)	O1	C1	N1	126.1(4)
O1	C1	C2	126.2(4)	N1	C1	C2	107.6(3)
N2	C2	C1	113.5(4)	N2	C2	C3	115.9(4)
N2	C2	C14	110.2(4)	C1	C2	C3	102.0(3)
C1	C2	C14	105.2(4)	C3	C2	C14	109.3(3)
C2	C3	C4	129.8(4)	C2	C3	C8	108.4(4)
C4	C3	C8	121.8(4)	C3	C4	C5	119.4(5)
C4	C5	C6	119.6(5)	C5	C6	C7	121.2(5)
Br1	C7	C6	117.5(4)	Br1	C7	C8	123.1(3)
C6	C7	C8	119.1(5)	N1	C8	C3	109.9(4)
N1	C8	C7	131.4(4)	C3	C8	C7	118.7(4)
O2	C9	O3	126.6(5)	O2	C9	N2	122.9(5)
O3	C9	N2	110.4(4)	O3	C10	C11	110.5(4)
O3	C10	C12	109.5(4)	O3	C10	C13	102.1(4)
C11	C10	C12	111.9(5)	C11	C10	C13	111.3(5)
C12	C10	C13	111.1(5)	C2	C14	C15	112.4(4)
C2	C14	C17	112.8(3)	C15	C14	C17	107.7(4)
O4	C15	C14	121.6(4)	O4	C15	C16	121.9(4)
C14	C15	C16	116.4(4)	O5	C17	C14	121.0(5)
O5	C17	C18	123.4(5)	C14	C17	C18	115.6(5)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C2	N2	H2	118.1	C9	N2	H2	118.1
C3	C4	H4	120.3	C5	C4	H4	120.3
C4	C5	H5A	120.2	C6	C5	H5A	120.2
C5	C6	H6	129(5)	C7	C6	H6	109(5)
C10	C11	H11A	109.5	C10	C11	H11B	109.5
C10	C11	H11C	109.5	H11A	C11	H11B	109.5
H11A	C11	H11C	109.5	H11B	C11	H11C	109.5
C10	C12	H12A	109.5	C10	C12	H12B	109.5
C10	C12	H12C	109.5	H12A	C12	H12B	109.5
H12A	C12	H12C	109.5	H12B	C12	H12C	109.5
C10	C13	H13A	109.5	C10	C13	H13B	109.5
C10	C13	H13C	109.5	H13A	C13	H13B	109.5
H13A	C13	H13C	109.5	H13B	C13	H13C	109.5
C2	C14	H14	107.9	C15	C14	H14	107.9
C17	C14	H14	107.9	C15	C16	H16A	109.5
C15	C16	H16B	109.5	C15	C16	H16C	109.5
H16A	C16	H16B	109.5	H16A	C16	H16C	109.5
H16B	C16	H16C	109.5	C17	C18	H18A	109.5
C17	C18	H18B	109.5	C17	C18	H18C	109.5
H18A	C18	H18B	109.5	H18A	C18	H18C	109.5
H18B	C18	H18C	109.5	N1	C19	H19A	109.5
N1	C19	H19B	109.5	N1	C19	H19C	109.5
H19A	C19	H19B	109.5	H19A	C19	H19C	109.5
H19B	C19	H19C	109.5				

Table 8. Torsion Angles(O)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C9	O3	C10	C11	66.8(5)	C9	O3	C10	C12	-56.9(5)
C9	O3	C10	C13	-174.8(3)	C10	O3	C9	O2	-9.2(7)
C10	O3	C9	N2	169.9(3)	C1	N1	C8	C3	-1.2(5)
C1	N1	C8	C7	176.3(4)	C8	N1	C1	O1	-175.7(4)
C8	N1	C1	C2	7.9(4)	C19	N1	C1	O1	-2.7(7)
C19	N1	C1	C2	-179.1(4)	C19	N1	C8	C3	-173.4(4)
C19	N1	C8	C7	4.1(7)	C2	N2	C9	O2	166.9(4)
C2	N2	C9	O3	-12.2(6)	C9	N2	C2	C1	63.8(6)
C9	N2	C2	C3	-53.8(6)	C9	N2	C2	C14	-178.5(4)
O1	C1	C2	N2	47.3(6)	O1	C1	C2	C3	172.6(4)
O1	C1	C2	C14	-73.3(5)	N1	C1	C2	N2	-136.3(4)
N1	C1	C2	C3	-10.9(4)	N1	C1	C2	C14	103.1(4)
N2	C2	C3	C4	-47.5(6)	N2	C2	C3	C8	134.1(4)
N2	C2	C14	C15	61.7(4)	N2	C2	C14	C17	-60.3(4)
C1	C2	C3	C4	-171.3(4)	C1	C2	C3	C8	10.2(4)
C1	C2	C14	C15	-175.5(3)	C1	C2	C14	C17	62.5(4)
C3	C2	C14	C15	-66.7(4)	C3	C2	C14	C17	171.3(3)
C14	C2	C3	C4	77.7(5)	C14	C2	C3	C8	-100.8(4)
C2	C3	C4	C5	-178.7(4)	C2	C3	C8	N1	-6.3(5)
C2	C3	C8	C7	175.9(3)	C4	C3	C8	N1	175.1(4)
C4	C3	C8	C7	-2.7(6)	C8	C3	C4	C5	-0.5(6)
C3	C4	C5	C6	2.9(7)	C4	C5	C6	C7	-2.2(8)
C5	C6	C7	Br1	173.7(5)	C5	C6	C7	C8	-1.0(8)
Br1	C7	C8	N1	11.7(7)	Br1	C7	C8	C3	-171.0(3)
C6	C7	C8	N1	-173.9(4)	C6	C7	C8	C3	3.4(6)
C2	C14	C15	O4	-56.7(5)	C2	C14	C15	C16	124.4(4)
C2	C14	C17	O5	65.6(5)	C2	C14	C17	C18	-116.5(4)
C15	C14	C17	O5	-59.1(5)	C15	C14	C17	C18	118.8(4)
C17	C14	C15	O4	68.2(5)	C17	C14	C15	C16	-110.7(4)

Table 9. Possible hydrogen bonds

Donor	H	Acceptor	D...A	D-H	H...A	D-H...A	
N2	H2	O4	2.856(6)	0.86	2.38	115.09	intramol.
N2	H2	O5	2.928(7)	0.86	2.39	121.08	intramol.

Table 10. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
Br1	N1	3.410(4)	Br1	C19	3.275(6)
O1	O3	3.290(5)	O1	O5	3.430(6)
O1	N2	2.996(6)	O1	C3	3.540(5)
O1	C8	3.446(5)	O1	C9	3.538(6)
O1	C14	3.082(5)	O1	C17	2.971(6)
O1	C18	3.316(7)	O1	C19	2.818(7)
O2	C2	3.563(6)	O2	C10	2.862(7)
O2	C11	3.062(8)	O2	C12	3.022(9)
O3	N1	3.227(5)	O3	C1	2.767(6)
O3	C2	2.630(5)	O3	C3	2.852(5)
O3	C4	3.535(6)	O3	C8	3.266(5)
O4	O5	2.949(6)	O4	N2	2.856(6)
O4	C2	3.015(6)	O4	C3	3.599(6)
O4	C4	3.567(7)	O4	C17	2.996(6)
O5	N2	2.928(7)	O5	C2	3.094(7)
O5	C15	2.913(6)	N1	N2	3.514(5)
N1	C4	3.582(6)	N1	C14	3.236(5)
N2	C4	3.159(6)	N2	C8	3.524(6)
N2	C15	3.003(6)	N2	C17	3.010(7)
C1	C9	3.122(6)	C1	C17	2.981(6)
C1	C18	3.555(8)	C3	C6	2.733(7)
C3	C9	3.067(6)	C3	C15	3.064(6)
C4	C7	2.778(7)	C4	C9	3.566(7)
C4	C14	3.315(6)	C4	C15	3.250(7)
C5	C8	2.775(6)	C7	C19	3.242(7)
C8	C14	3.270(6)	C9	C11	3.056(8)
C9	C12	2.971(8)	C15	C18	3.586(7)
C16	C17	3.516(6)			

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Br1	H19A	3.155	Br1	H19B	2.845
Br1	H6	2.70(8)	O1	H2	3.422
O1	H14	3.278	O1	H18B	3.013
O1	H18C	3.382	O1	H19A	3.381
O1	H19B	3.535	O1	H19C	2.413
O2	H2	2.425	O2	H11A	2.467
O2	H11C	3.500	O2	H12B	2.427
O2	H12C	3.424	O3	H2	2.988
O3	H11A	2.637	O3	H11B	3.266
O3	H11C	2.618	O3	H12A	3.274
O3	H12B	2.655	O3	H12C	2.611
O3	H13A	3.181	O3	H13B	2.463
O3	H13C	2.514	O4	H2	2.383
O4	H4	2.985	O4	H14	3.140
O4	H16A	2.616	O4	H16B	2.655
O4	H16C	3.119	O5	H2	2.390
O5	H14	3.143	O5	H18A	2.661
O5	H18B	2.652	O5	H18C	3.125
N1	H13B	3.360	N1	H13C	3.520
N1	H14	3.005	N2	H4	3.034
N2	H14	3.295	C1	H2	3.098
C1	H14	2.593	C1	H18B	3.566
C1	H18C	3.417	C1	H19A	3.001
C1	H19B	3.103	C1	H19C	2.480
C2	H4	2.816	C3	H2	3.144
C3	H5A	3.211	C3	H13B	3.522
C3	H14	2.618	C4	H12C	3.523
C4	H14	3.480	C4	H16A	3.430
C4	H6	3.41(10)	C6	H4	3.227
C7	H5A	3.242	C7	H13B	3.297
C7	H19A	3.279	C7	H19B	3.217
C8	H4	3.244	C8	H13B	3.102
C8	H14	3.036	C8	H19A	2.852
C8	H19B	2.839	C8	H19C	3.326
C8	H6	3.28(9)	C9	H4	3.477
C9	H11A	2.802	C9	H11C	3.313
C9	H12B	2.723	C9	H12C	3.165

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C11	H12A	2.682	C11	H12B	2.679
C11	H12C	3.307	C11	H13A	2.705
C11	H13B	3.297	C11	H13C	2.630
C12	H11A	2.683	C12	H11B	2.667
C12	H11C	3.312	C12	H13A	2.682
C12	H13B	2.681	C12	H13C	3.321
C13	H11A	3.301	C13	H11B	2.706
C13	H11C	2.619	C13	H12A	2.718
C13	H12B	3.318	C13	H12C	2.648
C13	H19A	3.535	C14	H2	2.497
C14	H4	3.370	C14	H16A	3.202
C14	H16B	3.146	C14	H16C	2.529
C14	H18A	3.176	C14	H18B	3.213
C14	H18C	2.536	C15	H2	2.768
C15	H4	2.946	C16	H4	3.504
C16	H14	2.518	C17	H2	2.745
C17	H16C	3.538	C18	H14	2.529
C19	H13C	3.593	H2	H4	3.366
H2	H14	3.438	H4	H5A	2.317
H4	H16A	3.127	H5A	H6	2.527
H11A	H12A	2.974	H11A	H12B	2.519
H11A	H12C	3.548	H11A	H13A	3.588
H11A	H13C	3.490	H11B	H12A	2.505
H11B	H12B	2.947	H11B	H12C	3.538
H11B	H13A	2.584	H11B	H13B	3.582
H11B	H13C	2.955	H11C	H12A	3.549
H11C	H12B	3.555	H11C	H13A	2.938
H11C	H13B	3.480	H11C	H13C	2.409
H12A	H13A	2.552	H12A	H13B	3.012
H12A	H13C	3.580	H12B	H13A	3.568
H12B	H13B	3.533	H12C	H13A	2.910
H12C	H13B	2.476	H12C	H13C	3.528
H13B	H19A	3.215	H13C	H19A	2.957
H14	H16A	3.190	H14	H16B	3.150
H14	H16C	2.122	H14	H18A	3.143
H14	H18B	3.219	H14	H18C	2.126
H16C	H18C	3.522			

Table 12. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
Br1	O2[1]	3.071(4)	O1	C16[2]	3.496(5)
O1	C19[3]	3.258(7)	O2	Br1[4]	3.071(4)
O2	C16[5]	3.585(8)	O4	N1[5]	3.397(6)
O5	C6[5]	3.569(7)	O5	C7[5]	3.410(7)
O5	C8[5]	3.540(6)	O5	C16[5]	3.482(7)
N1	O4[6]	3.397(6)	C6	O5[6]	3.569(7)
C7	O5[6]	3.410(7)	C8	O5[6]	3.540(6)
C16	O1[7]	3.496(5)	C16	O2[6]	3.585(8)
C16	O5[6]	3.482(7)	C19	O1[8]	3.258(7)

Symmetry Operators:

(1) X,Y-1,Z
(2) X+1,Y,Z
(3) -X+1,Y+1/2,-Z+2
(4) X,Y+1,Z
(5) -X,Y+1/2,-Z+2
(6) -X,Y+1/2-1,-Z+2
(7) X-1,Y,Z
(8) -X+1,Y+1/2-1,-Z+2

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Br1	H5A[1]	3.265	Br1	H11B[2]	3.482
Br1	H12A[2]	3.413	Br1	H13A[2]	3.049
Br1	H16B[3]	3.201	Br1	H18A[3]	3.098
O1	H16A[4]	3.430	O1	H16B[4]	2.756
O1	H19A[5]	3.230	O1	H19B[5]	2.705
O1	H19C[5]	3.336	O2	H5A[6]	3.263
O2	H16B[7]	3.152	O2	H16C[7]	3.123
O2	H19C[5]	3.271	O3	H19C[5]	3.540
O4	H11C[8]	2.802	O4	H14[7]	2.833
O4	H18C[7]	2.771	O4	H19B[7]	3.079
O5	H14[7]	3.252	O5	H16A[7]	3.490
O5	H16C[7]	2.688	O5	H19A[5]	3.486
N2	H16C[7]	2.958	N2	H19C[5]	3.381
C1	H16B[4]	3.559	C4	H6[6]	3.24(10)
C5	H12B[1]	3.151	C5	H18A[9]	3.310
C5	H6[6]	3.54(11)	C6	H11B[2]	3.411
C6	H12B[1]	3.483	C6	H12C[1]	3.598
C6	H18A[3]	3.443	C7	H11B[2]	3.462
C7	H18A[3]	3.476	C9	H16C[7]	3.338
C9	H19C[5]	3.128	C11	H18B[5]	3.340
C11	H18C[5]	3.484	C12	H5A[6]	3.439
C12	H18B[9]	3.307	C13	H12A[2]	3.578
C13	H12B[2]	3.503	C13	H16A[4]	3.110
C15	H11C[8]	3.417	C15	H19B[7]	3.303
C16	H2[3]	3.197	C16	H11C[8]	3.429
C16	H13C[8]	3.126	C16	H19A[8]	3.412
C16	H19B[7]	3.020	C16	H19C[8]	3.149
C18	H11A[10]	3.582	C18	H12C[11]	3.242
C19	H16A[4]	3.366	C19	H16B[4]	3.562
C19	H16B[3]	3.204	C19	H16C[4]	3.543
H2	C16[7]	3.197	H2	H14[7]	2.895
H2	H16B[7]	3.446	H2	H16C[7]	2.271
H2	H18C[7]	3.198	H2	H19C[5]	3.419
H4	H11B[8]	3.518	H4	H11C[8]	3.308
H4	H13A[8]	3.569	H4	H13C[8]	3.381
H4	H6[6]	2.676	H5A	Br1[6]	3.265
H5A	O2[1]	3.263	H5A	C12[1]	3.439

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H5A	H12B[1]	2.583	H5A	H12C[1]	3.567
H5A	H13A[8]	3.199	H5A	H18A[9]	2.894
H5A	H6[6]	3.228	H11A	C18[5]	3.582
H11A	H13A[12]	3.256	H11A	H13B[12]	3.351
H11A	H18B[5]	2.891	H11A	H18C[5]	3.398
H11A	H19C[5]	3.461	H11B	Br1[12]	3.482
H11B	C6[12]	3.411	H11B	C7[12]	3.462
H11B	H4[4]	3.518	H11B	H13B[12]	3.060
H11B	H18B[5]	3.245	H11B	H18C[5]	3.405
H11B	H6[12]	3.082	H11C	O4[4]	2.802
H11C	C15[4]	3.417	H11C	C16[4]	3.429
H11C	H4[4]	3.308	H11C	H16A[4]	2.829
H11C	H18B[5]	3.345	H11C	H18C[5]	3.083
H11C	H19B[5]	3.570	H11C	H19C[5]	3.367
H12A	Br1[12]	3.413	H12A	C13[12]	3.578
H12A	H13A[12]	3.474	H12A	H13B[12]	3.177
H12A	H13C[12]	3.509	H12A	H18B[9]	3.072
H12A	H19A[12]	3.244	H12B	C5[6]	3.151
H12B	C6[6]	3.483	H12B	C13[12]	3.503
H12B	H5A[6]	2.583	H12B	H13A[12]	2.993
H12B	H13B[12]	3.422	H12B	H13C[12]	3.550
H12B	H6[6]	3.333	H12C	C6[6]	3.598
H12C	C18[9]	3.242	H12C	H5A[6]	3.567
H12C	H18A[9]	2.962	H12C	H18B[9]	2.671
H12C	H6[6]	3.151	H13A	Br1[12]	3.049
H13A	H4[4]	3.569	H13A	H5A[4]	3.199
H13A	H11A[2]	3.256	H13A	H12A[2]	3.474
H13A	H12B[2]	2.993	H13A	H16A[4]	3.595
H13B	H11A[2]	3.351	H13B	H11B[2]	3.060
H13B	H12A[2]	3.177	H13B	H12B[2]	3.422
H13C	C16[4]	3.126	H13C	H4[4]	3.381
H13C	H12A[2]	3.509	H13C	H12B[2]	3.550
H13C	H16A[4]	2.168	H13C	H16B[4]	3.525
H13C	H16C[4]	3.596	H14	O4[3]	2.833
H14	O5[3]	3.252	H14	H2[3]	2.895
H16A	O1[8]	3.430	H16A	O5[3]	3.490
H16A	C13[8]	3.110	H16A	C19[8]	3.366

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H16A	H11C[8]	2.829	H16A	H13A[8]	3.595
H16A	H13C[8]	2.168	H16A	H19A[8]	2.937
H16A	H19B[7]	3.302	H16A	H19C[8]	3.065
H16B	Br1[7]	3.201	H16B	O1[8]	2.756
H16B	O2[3]	3.152	H16B	C1[8]	3.559
H16B	C19[8]	3.562	H16B	C19[7]	3.204
H16B	H2[3]	3.446	H16B	H13C[8]	3.525
H16B	H19A[8]	3.534	H16B	H19B[7]	2.250
H16B	H19C[8]	2.889	H16B	H19C[7]	3.595
H16C	O2[3]	3.123	H16C	O5[3]	2.688
H16C	N2[3]	2.958	H16C	C9[3]	3.338
H16C	C19[8]	3.543	H16C	H2[3]	2.271
H16C	H13C[8]	3.596	H16C	H19A[8]	3.236
H16C	H19C[8]	2.960	H18A	Br1[7]	3.098
H18A	C5[11]	3.310	H18A	C6[7]	3.443
H18A	C7[7]	3.476	H18A	H5A[11]	2.894
H18A	H12C[11]	2.962	H18A	H6[7]	3.108
H18B	C11[10]	3.340	H18B	C12[11]	3.307
H18B	H11A[10]	2.891	H18B	H11B[10]	3.245
H18B	H11C[10]	3.345	H18B	H12A[11]	3.072
H18B	H12C[11]	2.671	H18C	O4[3]	2.771
H18C	C11[10]	3.484	H18C	H2[3]	3.198
H18C	H11A[10]	3.398	H18C	H11B[10]	3.405
H18C	H11C[10]	3.083	H19A	O1[10]	3.230
H19A	O5[10]	3.486	H19A	C16[4]	3.412
H19A	H12A[2]	3.244	H19A	H16A[4]	2.937
H19A	H16B[4]	3.534	H19A	H16C[4]	3.236
H19B	O1[10]	2.705	H19B	O4[3]	3.079
H19B	C15[3]	3.303	H19B	C16[3]	3.020
H19B	H11C[10]	3.570	H19B	H16A[3]	3.302
H19B	H16B[3]	2.250	H19C	O1[10]	3.336
H19C	O2[10]	3.271	H19C	O3[10]	3.540
H19C	N2[10]	3.381	H19C	C9[10]	3.128
H19C	C16[4]	3.149	H19C	H2[10]	3.419
H19C	H11A[10]	3.461	H19C	H11C[10]	3.367
H19C	H16A[4]	3.065	H19C	H16B[4]	2.889
H19C	H16B[3]	3.595	H19C	H16C[4]	2.960

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H6	C4[1]	3.24(10)	H6	C5[1]	3.54(11)
H6	H4[1]	2.676	H6	H5A[1]	3.228
H6	H11B[2]	3.082	H6	H12B[1]	3.333
H6	H12C[1]	3.151	H6	H18A[3]	3.108

Symmetry Operators:

(1) -X,Y+1/2-1,-Z+1
(2) -X+1,Y+1/2-1,-Z+1
(3) -X,Y+1/2-1,-Z+2
(4) X+1,Y,Z
(5) -X+1,Y+1/2,-Z+2
(6) -X,Y+1/2,-Z+1
(7) -X,Y+1/2,-Z+2
(8) X-1,Y,Z
(9) X,Y,Z-1
(10) -X+1,Y+1/2-1,-Z+2
(11) X,Y,Z+1
(12) -X+1,Y+1/2,-Z+1